

**GLOBALSTONE**  
c o n g r e s s 2 0 2 3

**7th EDITION**

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**MOSTEIRO DA BATALHA**  
BATALHA, PORTUGAL

**PROCEEDINGS**

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18TH - 23RD OF JUNE 2023

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**ASSIMAGRA**  
MINERAL RESOURCES OF PORTUGAL



**CLUSTER  
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**VII GLOBAL STONE CONGRESS 2023**  
**JUNE 18 – 23 | BATALHA – PORTUGAL**

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# **GLOBAL STONE CONGRESS 2023 | BATALHA, JUNE 18 – 23**

<https://globalstone2023.stonebyportugal.com/>

**NEW CHALLENGES ON DIMENSION STONES, FROM PORTUGAL TO THE WORLD**

**Responsibility for the information and views set out in this  
publication lies entirely with the authors**

Luís Lopes, Marta Peres, Célia Marques (Organizers)

Edition: Departamento de Geociências da Escola de Ciências e  
Tecnologia da Universidade de Évora

Cover: Inês Ribeiro, Luís Lopes

Back cover: Luís Lopes

Graphic design and pagination: Luís Lopes

Publication date: June 2023

Support Type: eBook

I.S.B.N.: 978-972-778-327-4

**How to cite publications in this proceeding's eBook (example):**

N. Careddu, L. Pia, O. Pandolfi, N. Santoro, S. DüNDAR. 2023. Study for the Implementation of an Integrated Monitoring System in Marble Quarries. Proceedings of the VII Global Stone Congress, Batalha, Portugal, 18-23 June 2023. Luís Lopes, Marta Peres, Célia Marques (Eds.). Departamento de Geociências da Escola de Ciências e Tecnologia da Universidade de Évora, Portugal. pp. 30 – 35. ISBN: 978-972-778-327-4



# GLOBAL STONE CONGRESS 2023 | BATALHA, JUNE 18 - 23

## PROGRAM

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- 17:30 Gathering in Mosteiro da Batalha and departure to Porto de Mós  
18:00 - 20:00 Reception Event & Sunset Drinks at Porto de Mós Castle  
20:30 Return to Batalha

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20:45 - 21:30	Return to Batalha	

**Friday, June 23th**

**Touristic visit**

09:00 - 09:30 Batalha - Fátima

09:30 - 10:45 Fátima Sanctuary Guided Visit (Building Stones)

10:45 - 11:00 Fátima - Serra de Aire Dinosaur Footprints Natural Monument

11:00 - 12:00 Serra de Aire Dinosaur Footprints Natural Monument Guided Visit

12:00 - 13:00 Serra de Aire Dinosaur Footprints Natural Monument - Nazaré

**13:00 - 14:45 Nazaré (Free Lunch)**

14:45 - 15:00 Nazaré - Alcobaça

15:00 - 17:00 Alcobaça Monastery Guided Visit

17:00 - 17:30 Alcobaça - Batalha

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## VII GLOBAL STONE CONGRESS 2023 | BATALHA, PORTUGAL 18 – 23 JUNE

### CLOSING SESSION

Batalha, June 22<sup>nd</sup>, 2023

It's time to say goodbye or see you soon... hope to see you at the next GSC, somewhere in the world.

The Global Congress 2023, the only international congress of Stone, has brought together over the last 5 days, companies, researchers, and all those who work in innovation, in Portugal, in the Center Zone, in Batalha.

It was 5 days of true sharing and knowledge. These were days when together we were able to discover what Natural Stone involves in Portugal, Quarries, Factories, Monuments, History, Culture and Art.

They were days of true networking, but above all where net friendship was created (I don't know if the term exists, but you get the idea).

To everyone who participated, who got involved, who sponsored and to the entire operational team, my thanks. Everyone was fantastic!!!

We celebrated Natural Stone and knowledge and I believe that we all evolved a little more.

It remains my/our commitment for the Global Stone Congress to continue traveling around the World promoting Natural Stone, sharing, knowledge and innovation.

See you soon...

Marta Peres  
Executive Director of Mineral Resources Cluster  
Global Stone International Committee Member

ASSIMAGRA – Portuguese Mineral Resources Industry Association – would like to thank all the participants, sponsors, speakers and organizers who made this 7th edition of the Global Stone Congress in the Central Region of Portugal a successful event. Also, a special thanks to the Presidents of the Municipalities of Batalha and Porto de Mós, and to the directors of the Monastery of Batalha and Feira Stone Ibérica for hosting the activities of this congress.

It was with great pride that we hosted this congress in the central region of Portugal, where the extractive and natural stone processing industry is one of the most important economies present.

There were 5 days of intense work and, certainly, an opportunity to get to know the natural stone industry in this region, but also to promote collaboration between different professionals and entities from different geographies around the world, to share knowledge, trends and technological advancements and an opportunity to grow together.

Thanks again and we look forward to seeing you all again on the next global stone.

Célia Marques  
Executive Vice President of ASSIMAGRA  
Portuguese Association of the Mineral Resources Industry

First, let me start by thanking you dear colleagues, dear participants, dear friends.

Without you we would not be here for sure!

You gave us what is more precious in life, your time to share, thank you!

Once a dear friend told me that landscape is nothing if there are no people to watch it... you are our landscape because, as you felt these days, our focus is on the people, besides the stone that gather all together, of course!

Even in the companies we visited you notice it. People came before and after all processes...

Saying that, I would like to address my recognition to all that made this congress happen.

First, to our small but precious team: André, Catarina, Célia, Inês, Nelson, Susana, Sílvia, and of course Marta!

This group really worked as a team; everyone knew exactly what they had to do... just so you to know our meetings took no more than a few minutes, just to delivery tasks. It is a real pleasure to count on you, thank you!

Without our sponsors the Global Stone Congress would not have been possible!

I must highlight the first line sponsors:

Joaquim Ruivo from the Batalha Monastery; José Frazão from ExpoSalão and Rogério Henriques to Stonelbérica; Jorge Vala from the Municipality of Porto de Mós; Carlos Rabadão from the Leiria Polytechnic Institute; Ana Pagará from the Alcobaça Monastery; Raúl Castro from the Municipality of Batalha, and, Isabel Damasceno (Congress President), from the Commission for Coordination and Regional Development of the Center.

A special word is due to our friends and Diamond sponsors:

José Júlio from Julipedra; Samuel Delgado from Solancis; Filipe Miguel from Marfilpe, and, Eliseu Frazão from Fravizel.

Last but not least, I also want to thank our Platinum sponsors and all the Entities that supported us from the beginning without questioning. We deeply acknowledge your support.

I am very biased to say but I think after a full week that passed in a glimpse, we all have learned a lot of new things and we are now closer related. That was our purpose from the beginning!

Until we meet again, thank you very much.

Luís Lopes  
Professor at the University of Évora  
Global Stone International Committee Member  
President of the Portuguese Association of Geologists (APG)

# **KEYNOTE SPEAKERS**

## Siegfried Siegesmund

Department of Structural Geology and Geodynamics of the Göttingen Center for Geosciences at the University of Göttingen (Germany)



**Prof. Dr. Siegfried Siegesmund** works in the Department of Structural Geology and Geodynamics of the Göttingen Center for Geosciences at the University of Göttingen (Germany). His main research focuses on regional and structural geology as well as applied geology. Aspects of his work on natural stones ranges from the exploration and evaluation of natural stone deposits to the optimized extraction of dimensional stones by using the new 3D-BlockExpert approach. Furthermore, his geomaterials research consists of the use of natural stones in architecture, building conservation and the preservation of historical monuments. Prof. Siegesmund has been involved in more than 400

publications (excluding conference papers), the co-author of several geological maps, the editor of 26 special volumes in national and international journals as well as scientific and popular science books. Among the best-known international publication is the well-received textbook "Stone in Architecture", which he published in the 5th edition with Prof. Dr. Rolf Snethlage at Springer-Verlag. So far, he has supervised around 150 Diploma, Bachelor and Master theses and 38 Dissertations. He is a member of the editorial board of the journal "Environmental Earth Sciences", where he is responsible for the subject area "Environmental Mineralogy and Petrography". Moreover, he is also internationally active as a publicly appointed and sworn-in expert in the field of "natural stones."

### Marble Decay – Phenomena, Processes and Diagnosis

Siegfried Siegesmund (ssieges@gwdg.de)

University of Göttingen, Geoscience Center Göttingen, 37077 Göttingen

Although at first glance it may seem that marble, due to its simple mineralogical composition, is a rock whose behaviour should be easy to understand, it is precisely marble that has numerous peculiarities due to its extraordinary physical properties especially of the calcite crystals. Marble as ornamental and dimensional stone as well in their natural environments show complex weathering phenomena. The magnitude of disintegration depends on the rock composition, rock fabric as well as on the climatic condition. The physical weathering of marbles due to thermal treatment is often discussed as the initial state of deterioration. Durability problems of marble also include the spectacular deterioration feature known as the so-called bowing behaviour. Microstructure-based finite-element analysis was successfully applied to simulate an actual degradation phenomenon of marble structures, i.e., microcracking. Both microcrack initiation and crack propagation were characterized, as were their dependence on lattice preferred orientation (LPO), grain shape preferred orientation (SPO), grain size, marble composition (calcite and dolomite) and grain-boundary fracture toughness.

Ultrasonic wave velocity measurements are a powerful tool for the damage assessment of marble. However, water saturation has an important influence on the magnitude and directional dependence of ultrasonic velocities. The effect of changing climate and, in consequence, different weathering actions can help to calculate or forecast risk numbers based on the Vp data in combination with the proposed decay index especially for marbles. For the protection of historically valuable, externally exposed marble objects, winter shelters were used as a reasonable measure for protecting marble sculptures, i.e., to reduce the effects of temperature and moisture fluctuation.

## Gurmeet Kaur



Dr. Gurmeet Kaur is the current chair of the Heritage Stone Subcommittee. She is working as an Associate Professor at the Department of Geology, Panjab University, Chandigarh, India. She is a co-leader of the IGCP Project HERITAGE STONES RECOGNITION: A STEP FORWARD (HerSTONES). She has been the lead guest editor for two special volumes in the journal *Geoheritage and Episodes*. She has convened many sessions on heritage stones at EGU. Dr. Gurmeet has more than 40 publications in international and national journals, and she has authored a book. She is serving as an associate editor for *Episodes*. She is also the editor of the monthly IUGS e-Bulletin. Dr. Gurmeet has delivered keynote lectures and invited talks at seminars and conferences.

### **IUGS Heritage Stones and UNESCO stone-built World Heritage Sites**

Gurmeet Kaur

Department of Geology, Panjab University, Chandigarh, India

Email: gurmeet28374@gmail.com

The International Commission on Geoheritage (ICG) is a commission of the International Union of Geological Sciences (IUGS) with the goal of finding and honouring exceptional geological heritage sites, stones, and collections. The Heritage Stone Subcommittee (HSS), a Subcommittee of the ICG, designates notable stones with cultural significance. Significant stones and their attributes should be included as an important criterion for the nomination of stone-built UNESCO Cultural World Heritage Sites since stones have played a vital role as recorders of our cultural progress.

## **José Delgado Rodrigues**



Geologist, Conservation Scientist

Consultant in Stone Conservation, Lisbon, Portugal

José Delgado Rodrigues received his degree in Geology in Coimbra University, Portugal, and the “Especialista” degree (PhD equivalent) in the National Laboratory of Civil Engineering (Lisbon, Portugal) where he also became Principal Research Officer, Head of the Geotechnique Department, and President of the Scientific Council. He has been working on the field of Conservation Science in which he has researched and taught in the last 40 years.

He participated in some relevant conservation interventions v.g. the Tower of Belém, Jerónimos Monastery, in Lisbon, Santa Cruz Church and Porta Especiosa in Coimbra, Cathedrals of Évora and Oporto, the Côa Valley rock art site, and the Segovia Aqueduct.

He taught graduate courses in the New University of Lisbon and in the post-graduate course in Évora University. He acted as invited professor in short courses and as invited lecturer in several universities and institutes around the world. He has supervised more than a dozen of PhD and MSc theses, and was the LNEC leading scientist in more than a dozen of international research projects. He published over 200 papers in congresses and international journals. He is currently reviewing papers for several international journals. He was Research Scholar at the Getty Conservation Institute to deepen knowledge in deterioration and conservation of granitic materials and to structure a publication in this same subject. He is Fellow of ISRM-International Society of Rock Mechanics and Rock Engineering. ORCID: 000 0002 5180 6130.

### **Stone in Architecture. Learning from Cultural Heritage Assets**

José Delgado Rodrigues

Geologist, Principal Res. Officer LNEC (Ret.), j.delgado.rodrigues@gmail.com

Anticipating the life cycle of a stone object and predicting its performance in time is a concern for stone designers and users in general. A commonly followed path to reach this aim is the (more or less extensive) laboratory characterisation of stone samples, which may include exposure tests and artificial ageing tests. In this presentation I will try to illustrate how the understanding of past behaviours, mainly in Cultural Heritage assets, may help to extract information to better decide in the present for the new uses of stone materials.

The move of learning from the past to apply in the present to anticipate the future requires the support of a theory, or at least a reasoning model to avoid, or to overcome, the accidental or anecdotal cases; Theories and models are not exempt of traps and proper actions to validate the conclusions must be executed. Cultural Heritage assets are a privileged group to carry out such validation exercises, since stones are there exposed for much longer than the usually required life cycle duration of modern constructions and have been subject to all kinds of external exposure conditions.

## Isabel Almeida



Isabel Duarte de Almeida is an Assistant Professor with 25 years of experience in higher education, and she is the coordinator of Sustainable Development Research Lines. She teaches in ISCTE-IUL, Portugal, and at the Lusitana University and participates in academic activities abroad: Institute for Management (IFM), University of Cambridge; University of Palermo (UniPa), Sicily, Italy; Technische Hochschule Mittelhessen (THM) in Germany. Dr Isabel Almeida graduated from the University of Lisbon (UL/FC, Portugal) with a 5-year degree in Biology. She has a postgraduate degree in Sociology, a second PhD in Sociology and Sustainability Management from the New University of Lisbon (UNL /FCSH, Portugal), and a postgraduate degree in Circular Economy from Berkeley University. Isabel has worked as a researcher at the Gulbenkian Institute of Science for 12 years. She has published indexed papers in Q1, and Q2 Scientific SCOPUS indexed publications, published many papers in peer-reviewed conference proceedings and is a reviewer for AoM and EurOMA conferences. She is also a reviewer for Springer's "Environment, Development and Sustainability" and the "Journal of Rural and Community Development". Her current research interests include Sustainable Development, Circular Economy, Corporate and Social Responsibility, Corporate Sustainability, and Green Supply Chain. Ongoing research projects:

Project I: "INOVMINERAL 4.0 -Advanced Technologies and Software for Mineral Resources". The project's overall objective is to reorient innovative industrial models for the extractive industry by developing advanced technologies, new products and software for the entire value chain: upstream processes, valorisation of mineral resources and market. Partners CEI (Coord.); FRAVIZEL; FRONTWAVE; STREAMVALUE CONSULTING LDA; MARFILPE; SOLANCIS; INSTITUTO POLITÉCNICO DE LEIRIA; FACULDADE DE CIENCIAS DA UNIVERSIDADE DE LISBOA; UNIVERSIDADE DO PORTO, ACPMR; INSTITUTO SUPERIOR TÉCNICO; SAVANNAH, among others.

Project II: "The value of transdisciplinary projects: a sustainable path in academic research". It is a transdisciplinary study on the future of teaching in the field of management, analysing the global, complex and diverse reality that determines professional and academic activity. The aim is to (i) identify problems in professional practice resulting from gaps in academic training, (ii) discuss proposals for sustainable solutions in the paradigm of transdisciplinarity, and (iii) overcome the boundaries of disciplinary domains inherent in the teaching and professional practice of managers.

Project III: "Business students' perceptions of ethics and sustainability in business". This research strand addresses business students' perceptions of business ethics and sustainability management by studying ethical dilemma cases.

## **The European Green Deal and the Dimension Stone Industry**

Isabel Almeida  
ISCTE-IUL, Portugal

The dimension stone sector, which encompasses the extraction, processing, and use of natural stone for construction purposes, plays a significant role in the building sector and is in line with the Green Deal objectives.

The Green Deal is a comprehensive framework aimed at promoting sustainable development and combatting climate change. The initiative aims to achieve carbon neutrality, energy efficiency and the use of renewable resources across a wide range of industries.

Several advantages are associated with the use of dimension stone in the building sector and are consistent with the goals of the Green Deal. Firstly, natural stone is a durable and long-lasting material, which makes it an attractive option for building construction. A building constructed with dimension stone is durable and requires fewer resources for maintenance and replacement than a building constructed with other materials. Further, dimension stone is a natural, abundant resource that can be sourced locally, thus reducing the carbon footprint associated with transportation. As a result of using locally available stone, less long-distance transportation is required, which results in lower greenhouse gas emissions.

As part of the Green Deal, emphasis is placed on building energy efficiency as well as promoting the use of renewable energy sources and reducing energy consumption. Dimension stone has excellent thermal properties that can enhance insulation and reduce energy consumption for heating and cooling. As a result, by integrating dimension stone into building designs, energy efficiency can be improved, resulting in a reduction in the use of fossil fuels to heat and cool buildings. Also, the dimension stone sector can also play a significant role in raising the principles of circular economy that are outlined in the Green Deal. The waste generated during the extraction and processing of dimension stone can be recycled and re-purposed for a variety of applications, thereby minimising waste, and conserving natural resources.

In summary, dimension stone and the Green Deal have a “symbiotic” relationship. Dimension stone is in fact compatible with the Green Deal's goals of sustainability, energy efficiency, and carbon neutrality. By leveraging the benefits of dimension stone, a greener and more sustainable future can be achieved.



## Guta Moura Guedes



Guta was born on the 23<sup>rd</sup> of July 1965, Torres Vedras, Portugal.

Daughter of Ruy de Moura Guedes e Maria Margarida de Moura Guedes, Guta has two sons, Rui Maria (1991) and Manuel (1995) and one granddaughter, Alice (2021).

She is a curator and a strategic designer. She co-founded the cultural non-profit association Experimenta, in Lisbon in 1998, of which has been Chairwoman since 2000. From 1999 until 2017 she dedicated herself to the Lisbon biennale, experimentadesign, as co-author and Director.

Actively engaged in the international design community as a curator, creative director, strategist and critical thinker since the early 90's, GMG has integrated several high-profile international committees and juries. She runs and curates exhibition projects in the area of design and architecture, working also as a consultant, as a teacher and as a speaker at debates and conferences in the area of design and strategic development, in Portugal and abroad.

She was Director of the Fundação Casa da Música and was named Administrator of the Fundação Centro Cultural de Belém, the most important cultural institution in Portugal, by the Portuguese Government. She studied Piano, Biology, Hotel Management and Design and was distinguished by the French Government with the "Ordre de Chevalier des Arts et des Lettres" for her work in the design and culture field around the world. She is considered since 2018 one of the 25 most influential women in Portugal.

Guta develops her professional career with the main objective of reinforcing the importance of culture as a pillar of a sustainable and egalitarian society, as an indispensable factor for human development.

## **Primeira Pedra**

Guta Moura Guedes  
Experimenta Design

During the 6 years of the First Stone program, the national natural stone industry joined for one of the biggest initiatives ever to promote the Portuguese natural stone sector. A program that brought together the industrial and the cultural sector - architecture, design, and the visual arts - bringing together 36 authors from 15 countries and 28 national natural stone companies, a pride for the national industry and for the image of Portugal in the world.

By inviting 36 of the most fascinating contemporary authors from the fields of design, architecture and the visual arts, the central dimension of this programme called for a strong emphasis on creativity and the ability to innovate and create different proposals. The results bring a new added value to society, exploring this sustainable material in an innovative way.

The programme produced 41 original concepts and a total of 77 pieces, all with different scales and functions, from a very delicate pair of rings to a children's slide, including three permanent installations – Mint Street, designed for London's Borough of Southwark, A Bench For a Tower, in New York and Ruisseau for the Vitra Campus' gardens in Weil am Rhein. They were all produced by 28 Portuguese quarrying and processing companies, partners of the programme.

Coordinated by ASSIMAGRA, conceived, and developed with experimentadesign, First Stone innovated an entire production and processing system, through an approach centered on creativity and culture.

The First Stone programme was divided into five main projects with distinctive curatorial premises: Resistance, Still Motion, Common Sense, Expanded and Fragile Mode Fragile. All of them presented specific approaches, objectives and consequently, briefings.

The first, Resistance, was specially aimed at the territory of Architecture, focusing on the theme of resistance in relation to the material itself and also to architecture as a form of resistance to banality; Common Sense integrated exclusively product and equipment designers, with a greater focus on the idea of simplicity and the use of stone in everyday domestic life; Still Motion challenged communication designers, very distant from a material like stone, about the concepts of beauty and meaning; Expanded directed its attention to the visual arts, exploring notions of space, scale, time and place; and lastly, Fragile Mode Fragile gathered architects and designers around the theme of human and environmental fragility.

As in all curatorial processes, the invited creators, confronted with the briefings, with the Portuguese stone and the numerous ways to work with it, defined and developed their own explorative paths. The results, the 77 pieces produced, showcase the freedom and the diversity of interpretations, which are part of the power of cultural territory.

The last exhibition of the First Stone programme was at Museu Nacional dos Coches, Lisboa, Portugal.

With the High Sponsorship of His Excellency the President of the Portuguese Republic Marcelo Rebelo de Sousa, this project was co-financed by the European Union through the Regional Development European Fund, within the scope of the Support System for Collective Actions (Internationalization), framed in PORTUGAL 2020.

## Hervé Beaudouin



Hervé Beaudouin, Born in Nancy in 1949.

Studied at Nancy School of Architecture, graduating in 1975.

Created the NIORT (Deux Sèvres) agency in 1976.

Following various encounters, with American architect Bruce GOFF, but also with mason Manuel Paiva, he became interested in materials and the principles of recovery and reuse.

Curious exploration of rural territories, and learning about local traditions and know-how, led to creative interpretations and transpositions.

In the early years, he developed a simple architecture based on geometric rigor and symmetry.

Small means: a lot of little...

A keen photographer inspired by Raoul Hausmann's pictures of Ibiza, he built up a collection of medium-format photographs of the country.

The succession of projects constitutes a series of exploratory experiments, enabling him to improve the implementation techniques that may be appropriate in economically constrained contexts.

Praise for the raw and the "ordinary", for textures, tactile walls, thickness and anchoring to the ground.

He advocates the stubborn use of local resources and the most suitable materials for simple techniques that can be practiced, without sophisticated tools, by the fabric of regional businesses.

His experience and predilection for materials and the minerality of stone have brought him into close contact with Portuguese architects such as Joao Luis Carrilho da Graça (Théâtre Auditorium de Poitiers - Conservatoire de Musique et de danse de La Rochelle) and Eduardo Souto de Moura, with whom he has worked on several projects, including one in Bordeaux (84 housing units).

In 1997, he was appointed Architecte Conseil de l'Etat, and was successively posted in Creuse, Essonne and Hauts de Seine.

In 2009, he was elected Vice-President of the Académie d'Architecture, Place des Vosges, Paris.

In 2016, one of the Agency's projects, the Maison des Habitants de la Gibauderie in Poitiers, was exhibited at the Venice Biennale.

The Agency's work has been the subject of numerous publications in French and foreign journals and covers a wide range of fields: municipal facilities, auditoriums, theaters, media libraries, housing, educational buildings, university buildings, museums, offices, business premises and more.

## Pierre Bidaud



Pierre Bidaud has been a stonemason for 30 years. After his Tour de France, he left for England in 1998, working mainly in restoration. He developed an interest in design and contemporary architecture and in 2005 began to work on new stone projects, as well as collaborations with designers for furniture.

A meeting with a structural engineer in 2009 reinforced his belief in his material as a supporting element in contemporary buildings. It is this that fuelled his motivation to embark upon research, and develop a new method of mineral construction, combining stone with steel and high-performance resins, and creating stone on steroids.

Pierre believes that the survival of the stonemasonry profession depends on re-establishing communications between professional men and women, engineers and architects. It is most important that each sector understands and questions the capacities, limits and desires of each other, to build more honestly and solidly.

For him, stone materials have an even more relevant place in modern construction, their carbon footprint is much lower than that of concrete, and the implementation of stone manufacture is now easier and faster.

For 10 years he has worked at The Stonemasonry Company with a growing team of stone enthusiasts, designing and developing stone staircases and structures using techniques such as post tensioning, and discreet steel reinforcement, to create works that appear to defy gravity whilst still being loadbearing.

For Pierre, the staircase is an object that requires a wide range of knowledge, it must be aesthetically appealing, harmonious in its set up, functional, and the ascent must be an experience of pleasure. Understanding the ergonomics of movement for each characteristic, and of course the structural complexities of stairs, requires solidity and integrity. We can thus speak of a mineral ascension.

Pierre believes, even more now, the stonemason, with their knowledge and materials, needs to again become a protagonist, a leader, and not a simple actor in modern construction. It is important that the stonemason finds its place as designer, maker and developer of stone solutions.

In 2022, along with Steve Webb of Webb Yates Engineering, Pierre and The Stonemasonry Company won The CDUK Award for Architecture at The Royal Academy of Arts. Intended to promote the use of both stone and timber as natural, low embodied carbon materials, the 'Equanimity' stone beam was designed to challenge perceptions of stone and timber as largely decorative and to demonstrate structural integrity. 'Equanimity' was deemed the work to best demonstrate the capacity of architecture to signal hope, illustrating the structural potential of materials with minimal embodied carbon.

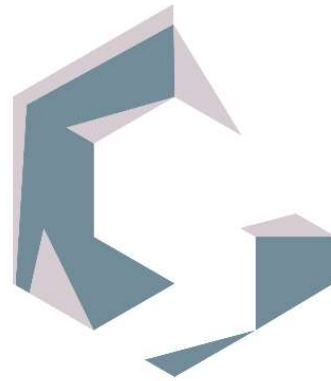
Pierre is also a strong advocate for the pivoting of the stone industry towards the manufacturing of pre-assembled stone elements with mechanical connections, to speed up, and improve installation time of stone structure systems. Coupled with a cross laminated timber (CLT) structure and an engineered timber frame, a new generation of carbon neutral hybrid construction should be proposed to architects, engineers, and clients.

Stone should again be considered as a commodity and not a luxury.

# **Global Stone Congress 2023 Presentations**

## **DISCLOSURE STATEMENT**

Responsibility for the information and views set out in this publication lies entirely with the authors



**GLOBALSTONE**  
c o n g r e s s 2 0 2 3

**Monday, June 19<sup>th</sup>**  
**PRESENTATIONS**

# STUDY FOR THE IMPLEMENTATION OF AN INTEGRATED MONITORING SYSTEM IN MARBLE QUARRIES

N. Careddu<sup>1\*</sup>, L. Pia<sup>1,2</sup>, O. Pandolfi<sup>2</sup>, N. Santoro<sup>2</sup>, S. Dündar<sup>2</sup>

(1) Department of Civil, Environmental Engineering and Architecture – Università degli Studi di Cagliari, Italy, [\\*ncareddu@unica.it](mailto:ncareddu@unica.it)

(2) Orlando Pandolfi & Partners Engineering, Carrara, Italy

**Summary:** *The paper describes the project for the implementation of an integrated monitoring system to solve, in an innovative and long-lasting way, a crucial geotechnical problem: the stability of the faces in marble quarries. The study was carried out at the “Cima Campanili” quarry, which is one of the main marble quarries in the Carrara producing area (Italy). Two different design methods are compared: a conventional method and an innovative one. Since the collected data come from different technologies and they refer to inhomogeneous situations, they must necessarily be processed with a specially validated method. For this purpose, the digital platform with cloud storage is the highest degree of innovation, easy and user-friendly for any type of user. This new method allows to carry out analysis over large areas, with high speed and ease of implementation. Finally, some economic considerations about the two methods have been quickly discussed.*

**Key words:** *marble quarrying, rock-mass stability, twin quarry, monitoring*

## 1. Introduction

Marble quarries are a huge challenge for mining engineers and quarrymen, who have to deal with a complex works in order to quarry high quality materials while ensuring the sustainable development of the rock-mass and guaranteeing safe conditions for workers and equipment.

One of the main issues in Dimension Stone quarries are the problem of stability and monitoring of the faces.

In order to introduce a new method for monitoring the rock-mass stability, a study was carried out at the “Cima Campanili” quarry, which is one of the most important marble quarries in the Carrara producing area, and it is currently exploited by open-cast method.

The innovative criterion is to integrate the traditional method with technologically advanced method. The first method gives a risk reduction assessment based on statistical geo-structural and morphological analyses. The advanced method are based on technologies as remote sensing, lidar and fiber-optic sensors. Therefore, the new criterion consists of the design of an integrated monitoring system followed by input of data into an innovative digital platform.

The data, which are collected in real time by the platform, will be processed and the obtained results will be available almost-real-time by a visual displaying that could be easily accessible to all. Therefore, this information will be at the fingertips of an unlimited number of users, who can access it at any time and from any device with a wireless connection. In this way, it will

be possible to safely carry out the quarry from a sustainable development perspective, providing decision-makers with greater awareness thanks to the various and easily accessible information.

This study aims to implement a pilot project designed by the authors, while validating the method and results.

## 2. Context

Carrara marble is well known worldwide. It is a reference in the natural stone world market, although it has lost the lead in export volumes in recent years. The deposit, one of the largest in the world, has only been exploited to a small extent, and there are currently about 115 active quarries in the territories of Massa-Carrara.

Both the aesthetic and mechanical characteristics of this marble depend on its carbonate origin and their subsequent metamorphosis: the vast area occupied by the deposit resulted in very different conditions of formation, tectonics, and impurity development providing different commercial types of the stone.

The “Cima Campanili” quarry has been surveyed in this study; it is located in the Municipality of Carrara inside the marble producing area of Colonnata along the eastern slopes of Mount Campanile.

Quarrying method is based on large descending benches; diamond-wire technology is applied for separating the bench in slices which are then tipped on a bed of rubble set in place for attenuating the impact.

### 3. General considerations

The quarry exploitation cycle (Fig. 1) is a standard workflow that begins with the quarrying plan (analysis of stone deposit and design) and it ends with land rehabilitation, through quarry's works that goes hand in hand with periodic checking and improvement.

This graphical representation refers to the new operative methods, mainly linked to the introduction of digitisation and new management tools.

The traditional method, still dominant today, is represented by the same cycle without the incremental improvements in the individual steps, making it too strict after only a few months of quarrying.

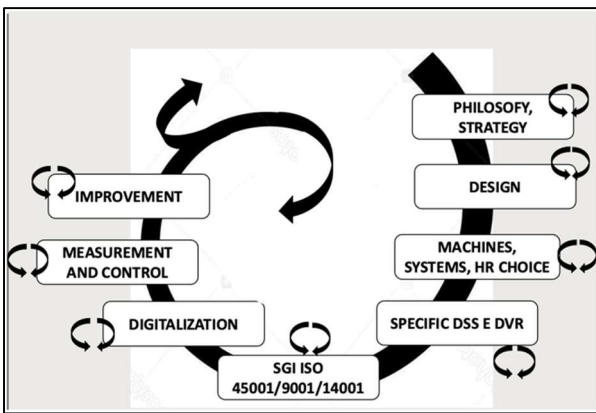


Fig. 1. Quarry exploitation cycle (by Pandolfi).

In fact, with the traditional approach, it often happens that project variants are needed due to the complexity of the stone deposits and the forecast, which may not match exactly to the real situation. For this reason, each phase of the complete cycle contains a separate cycle within it, which allows it to be optimized according to the new knowledge gained from the rock-mass. In addition, needs may also change, for example due to a fluctuation in market value.

Continuous improvement allows the implementation of techniques that maximize production, safety and environmental protection; in fact, the dimension stone quarry is a complex system that encompasses the areas shown in Fig. 2.

Quarry management (Fig. 3) is carried out through inputs (energy and resources) and it produces outputs. The latter include both desirable ones (commercial blocks, but also semi-irregular ones) and undesirable ones such as scraps and debris.

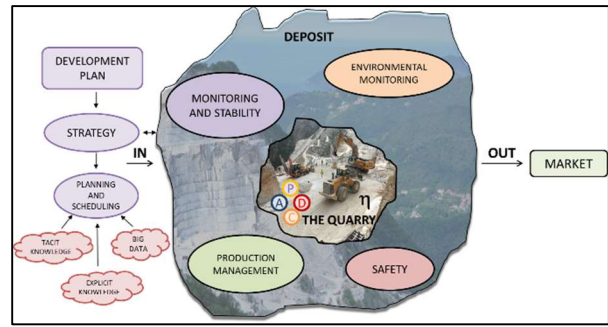


Fig. 2. The complex system quarry.

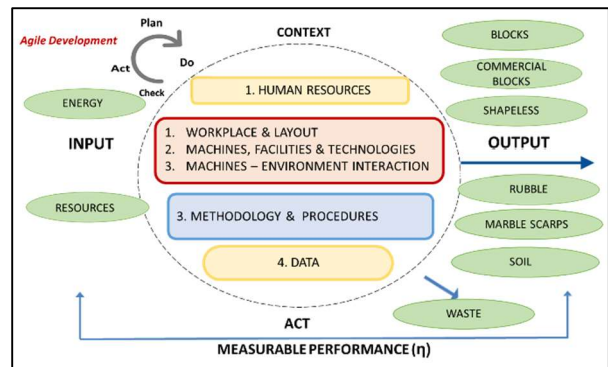


Fig. 3. Diagram of the complex system quarry with its input and output.

The exploitation of the quarry is based on improving the performance of the production processes - which were already shown in Fig. 1 -, so as to obtain the best yield, which can be of a different nature depending on the process under consideration (Fig. 4).

The indicator that is normally used is called the *Key Production Index* (KPI), which assumes different units of measurement depending on the type of process, the goal and the "point of view".

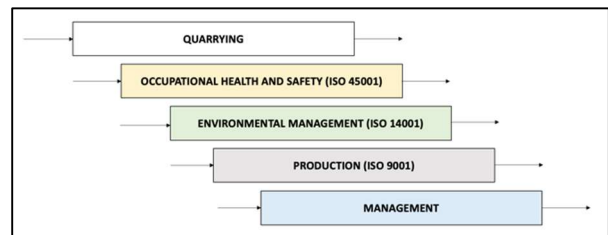


Fig. 4. Diagram of the complex system quarry with its input and output.

Monitoring involves the collection of data over time, such as measurements and analysis obtained by using instrumentation and adopting techniques which are adequate for the purpose. Measurements can be both quantitative and qualitative and they include:

- Mountain performance
- Economic performance
- Health and safety performance
- Environmental performance



Depending on quarrying method, the amount of volume and the organization of the quarry, both the appropriate monitoring analyses and periods between measurements are chosen.

Therefore, the design of the system follows the PCDA logic, with the repetition of the cyclic phases shown in Fig. 5.

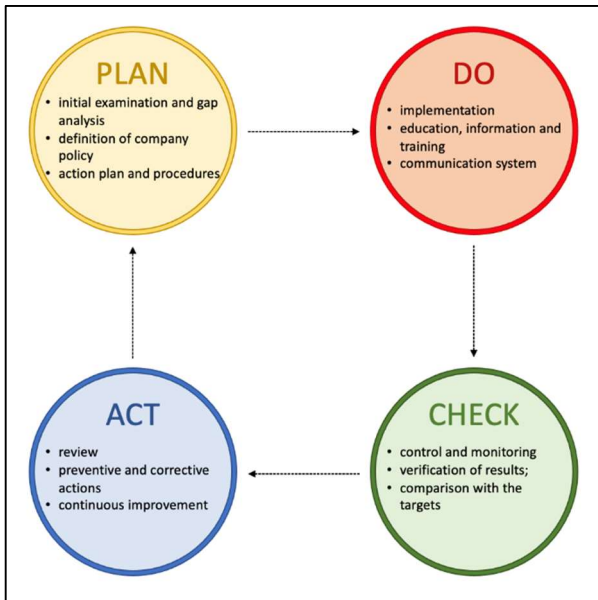


Fig. 5. PDCA logic diagram.

#### 4. Conventional method

The first design method is based on an analysis of the critical points to be solved and the study of the solution. In practice, the stability of the rock slope (or quarry face) is studied by means of discontinuity analysis using the widely established classical statistical methods.

The next step is to propose some risk reduction measures; for example, if we considered the placement of rockfall barriers as a design solution, we would use rockfall analysis software and then we will design the intervention.

The stability analysis of the rock slope starts with a geostructural survey of the rock mass in the accessible areas of the quarry and continues with the processing of the surveyed data using stereographic representations and Barton's strength criterion. As is well known, the stability analysis of rock masses takes into consideration two main aspects: the intrinsic characteristics of the rock and the characteristics of the discontinuities.

In this study, the aim was to understand the behaviour of the blocks that could detach from the rock slope.

Figures 6 and 7 show some moments of the discontinuity investigations *in situ* and the roughness measurement.

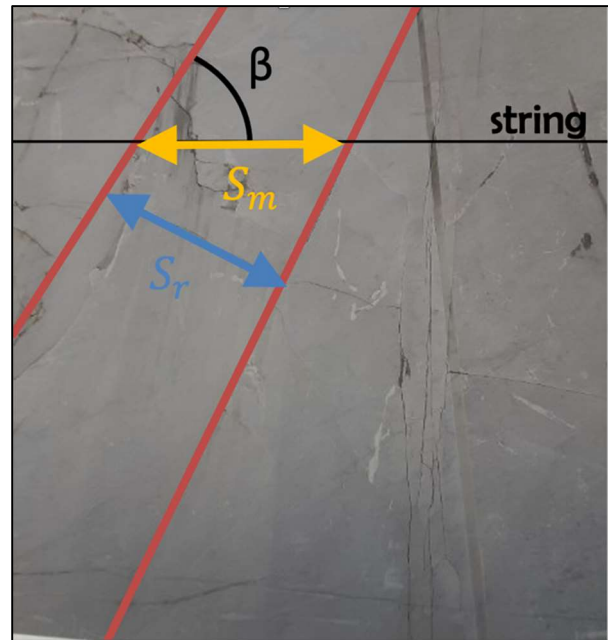


Fig. 6. Measurement of spacing and slope of discontinuities.



Fig. 7. Roughness measurement using the Barton comb.

Measurements (158 in total) on the discontinuity planes have been statistically processed; five main families of discontinuities, corresponding to nine pole groupings, have been identified (see Tab. 1 and Fig. 8).

Table 1. The five main discontinuity families.

SET	SUB-SET	DIPDIR/DIP
K1	K1.1	76/118
	K1.2	70/278
K2	K2.1	82/322
	K2.2	79/123
K3	K3.1	80/089
	K3.2	75/265
K4	K4	53/250
K5	K5.1	54/197
	K5.2	53/155

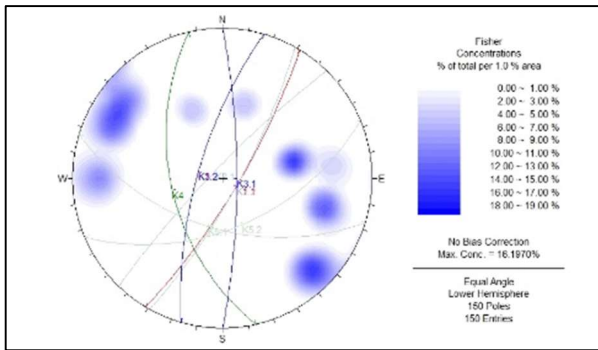


Fig. 8. Stereographic representation of the discontinuities detected in the study.

### 5 New method: integrated monitoring system

In section 3, it was mentioned that a quarry must be exploited basing on the analysis and development of operational processes, e.g., the up-line cutting is surely critical because the machine - rock-mass interaction holds strategic involvements for quarrying safety, production and cost management.

To improve all elements of the production processes and to keep track of the progress of work, it is therefore necessary to measure each process and verify each its change over time.

Therefore, measurable and SMART (Specific, Measurable, Accepted, Realistic, Timely) KPIs must be identified and chosen in consultation between the company and its consultants.

The proposed innovation is therefore the creation of a digital twin of the quarry; it must be flexible and scalable according to the context, and can be built in six simple steps:

- *creation*: setting up a large number of sensors to monitor the physical process, acquiring the key data of the process and its context;
- *network transmission*: allowing the digital twin to exist and contributing to the continuous interchange that occurs in real time between the real world and the digital model. Refinement of the raw data and elimination of unnecessary data is included therein;
- *aggregation*: the data obtained must be collected physically or in the cloud before being processed and analyzed;
- *analysis*: it allows visualisation and focussing using appropriate software: this produces graphical or other representations that help the user better understand the meaning;
- *assessment*: differences between the real and the digital can be highlighted, so as to identify areas where

further investigation may be necessary or should be discarded;

- *action*: having identified areas that need intervention, action can be taken to improve the actual similarity between the real world and the digital model.

In most of the Apuan quarries, there are 4.0 machines equipped with IoT (Internet of Things), which send data to be fed into digital systems. Digitalization is based on the massive collection of data, i.e. a time-varying, high-frequency data amount.

In this case study, the goal was achieved with the 3!PETRA digital platform, which simplifies the management of the quarry by means of the Twin Marble Quarry™ (Fig. 9).

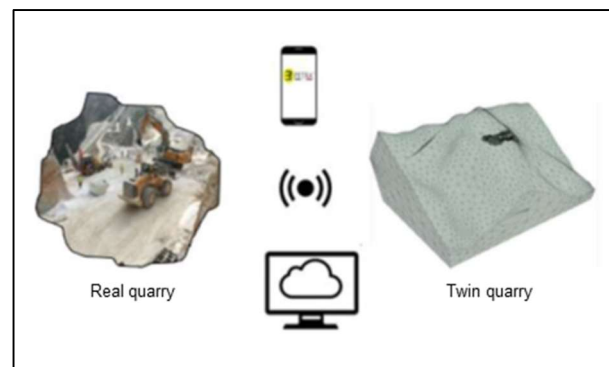


Fig. 9. The Twin Marble Quarry™ (by Pandolfi).

The survey began with an in-depth examination of remote sensing technologies and the assessment of their strengths and weaknesses.

In particular, great importance was given to the accuracy of the measurement, which must guarantee a resolution comparable to that of standard geotechnical measuring instruments.

However, examination of the quarry-3D-models has shown that the level currently achieved is not comparable with that resulting from the use of traditional geotechnical equipment.

An innovative technique, not yet applied in the field of rock and stone mechanics, was therefore adopted. It is based on an original workflow, to be applied on rock slopes studies in stone quarries, by using artificial intelligence algorithms and data management in the 3!PETRA platform.

### 6. Results and discussion

The combined use of surveying techniques such as laser, drones and special filming machines enabled the construction of an extremely accurate 3D model.

The validation of the workflow required a detailed analysis of the *in situ* direct measurements, which was carried out in successive phases:

1. laser-photogrammetric survey;
2. implementation in the 3IPETRA platform;
3. visual sensor testing.

The first phase allowed the construction of a 3D model of the quarry using the most innovative laser-photogrammetric survey techniques.

The quarry covers an area of approximately 200,000 m<sup>2</sup> and it is characterized by both natural and artificial rock slopes that reach more than 150 m from ground level. This results in the obvious inaccessibility of a large part of the site. The general survey was subsequently deepened in the test areas of the subsequent phases.

Unmanned Aircraft Systems (UAS) are best suited to survey vast and inaccessible areas. Therefore, two drones were used at the same time, taking thousands of photos of the site and allowing it to be totally surveyed.

The subsequent survey campaign was carried out with the additional aid of a laser scanner to better study the area identified for testing (approximately 200 m<sup>2</sup>). The point clouds obtained in the construction of the 3D model were then processed to obtain the textured mesh.

In the second phase of the work, the 3D reconstruction was imported into the 3IPETRA platform. The system (shown in Fig. 10) allows the visualization and interaction with both point clouds and meshes.

Currently, the 3D model, uploaded onto the platform, is connected to a database; this makes possible the integration of data from different sensors in real time.

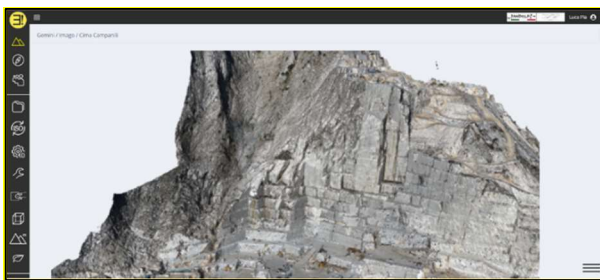


Fig. 10. 3D model imported into the 3IPETRA platform.

The third phase involved the testing of a visual sensor that will be used in the Integrated Monitoring System. The purpose of the test was to verify the accuracy achievable with such a sensor.

In the test area - prior to carrying out the survey for the reconstruction of the 3D model - an easily accessible

bench was chosen. A target, i.e. an artificial and clearly recognizable element, measuring 60 cm x 60 cm was attached on the bench vertical wall (Fig. 11).



Fig. 11. The target attached on lower part of the bench.

A calibration slide with different micro-meter scales from 0.1 mm to 0.01 mm was placed inside the target.

The workflow was then used. The high number of surveyed data were processed using image reconstruction algorithms based on A.I. This resulted in an orthophoto of the target with a resolution of approximately 0.01 mm (Figure 12).

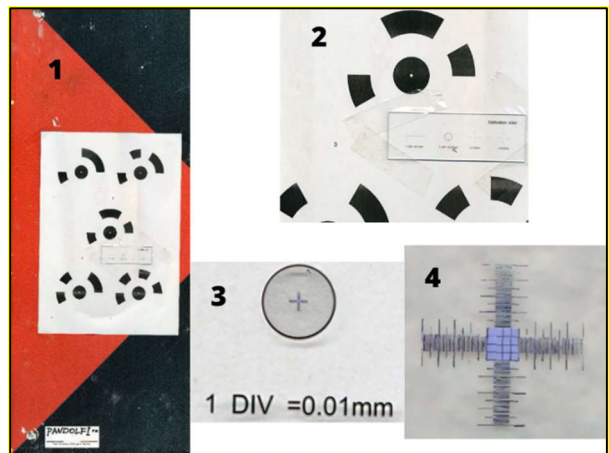


Fig. 12. High resolution orthophoto of the target.

Considering the accuracy achieved by traditional geotechnical sensors, such as crack meters (0.01 mm), the result obtained is in agreement with the aim of the study. This demonstrates the feasibility of integrating the data obtained with traditional and innovative methodologies.

## 7. Conclusions

Although the study is still in progress, it has proven to have excellent potential.

By combining conventional techniques with innovative ones, it is possible to achieve optimal results from every point of view, while managing to compensate for the various criticalities of the instruments when taken individually.

- calibration of the monitoring instruments which will be chosen, both conventional and remote sensing;
- installation of the monitoring instruments;
- installation and setting of the monitoring unit and data log for real-time data acquisition;
- implementation of the monitoring data in the platform.

To complete the structure of the Integrated Monitoring System, the following steps will be taken in the coming months

**Acknowledgments:**

The authors would like to thank Successori Adolfo Corsi Carrara S.r.l. for their willingness to perform the measurements at the Cima Campanili quarry.

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# THE IMPORTANCE OF STRUCTURAL AND SEDİMENTOLOGICAL STUDIES IN A MARBLE QUARRY; AN EXAMPLE FROM BİLECİK MARBLE QUARRIES (TURKEY)

Atiye Tugrul<sup>1\*</sup>, Murat Yılmaz<sup>1</sup>, Serdar Akgündüz<sup>1</sup>, Göksel Dursun<sup>1</sup>, Erdi Avci<sup>1</sup>, Hakan Elçi<sup>2</sup>

(1) İstanbul University-Cerrahpaşa, Faculty of Engineering, Geological Engineering Department, Büyükçekmece, İstanbul, Turkey  
[\\*tugrul@iuc.edu.tr](mailto:tugrul@iuc.edu.tr)

(2) [Dokuz Eylül Üniversitesi Torbalı Meslek Yüksekokulu, Geoteknik Programı, Torbalı, İzmir](#)

## Abstract

There are very rich natural stone resources in Turkey, and high quality products have been produced for the world market with modern production methods from these resources in recent years. Turkey's natural stone export revenue in 2022 has exceeded 2 billion dollars. This study was carried out to emphasize the effects of tectonic-controlled structures (faults, cracks, folds...) and sedimentological features on the quarry productivity in the marble quarries in Bilecik, and to determine the geological factors that affect the development of the patterns observed in the marble quarries, and to explain the effects of these patterns on the marble quarrying. Within this scope, stratigraphic, sedimentological, structural and engineering geology studies were conducted at different stratigraphic levels in the quarries producing marble in the region. The marble patterns observed in the quarries in this region were determined as Bilecik Beige (Bilecik Beige-oolithic, Bilecik Beige-reefal, Bilecik Beige-fossiliferous, Bilecik Beige-nonfossiliferous) and Rosalia (Rosalia breccia, Rosalian stylolitic). The geological parameters controlling the formation of these patterns and the quarry productivity are (1) the stratigraphic level at which the marble quarry is excavated, (2) the lithological and sedimentological characteristics of the rocks, (3) the characteristics of the faults (types, dip directions, dips) passing through or near the quarry, (4) fracture-crack density in the region, (5) secondary mineral developments and the abundance of these mineral veins.

**Key words:** Marble pattern, Faults, Sedimentological features, Bilecik, Turkey

## 1. Introduction

The mineral wealth of a country depends on the geological structure and geological evolution of that country. Because of its geological evolution, Turkey is located both in the Alpine-Himalayan mountain formation system and has been affected by immature tectonic activities. For this reason, it has rich resources in terms of marble, limestone, travertine, granite and basalt in various colours and patterns formed in a wide geological time. When the carbonate rocks in our country are evaluated in terms of quality, colour, pattern and reserve, they have the characteristics and potential to compete with the carbonate rocks around the world.

The block efficiency is low because larger blocks cannot be obtained while cutting blocks in limestone quarries operating in Turkey. The most important parameters affecting the quarry productivity are the facies change occurring during the formation process of the limestone and the deformation of the limestone quarries by tectonism due to the geological conditions of the country.

The study area is located in the province of Bilecik on the Sakarya Continent of the Western Pontides (Figure 1a). The Western Pontides region includes the Istranca Massif, the Thrace Basin, the Istanbul-Zonguldak Union, the Biga-Armutlu-Ovacık Zone, and the Sakarya Zone (Yılmaz et al., 1997; Okay and Tüysüz, 1999; Yiğitbaş et al., 1999; Elmas, 2012). The basement of the Sakarya Zone consists of metamorphic rock assemblages such as high-grade schists, gneisses, amphibolites and metaophiolites (Yılmaz et al., 1981). This metamorphic basement (Uludağ Group and Karakaya Complex) is unconformably overlain by a transgressive Jurassic-Cretaceous sequence (Figure 1a). The province of Bilecik, where the study area is located, is one of the leading marble production areas in the west of the Sakarya Zone. The marble quarries in the northern areas of Bilecik were excavated within The Middle-Upper Jurassic Bilecik Limestone, Cretaceous aged Vezirhan Formation and/or at the contacts of these two units (Figure 1b). Block stone of different colours and patterns are produced from 14 marble quarries (Quarries: K, V, N, T, E, G, S, W, H, O, U, F) in this region, controlled by some geological factors.

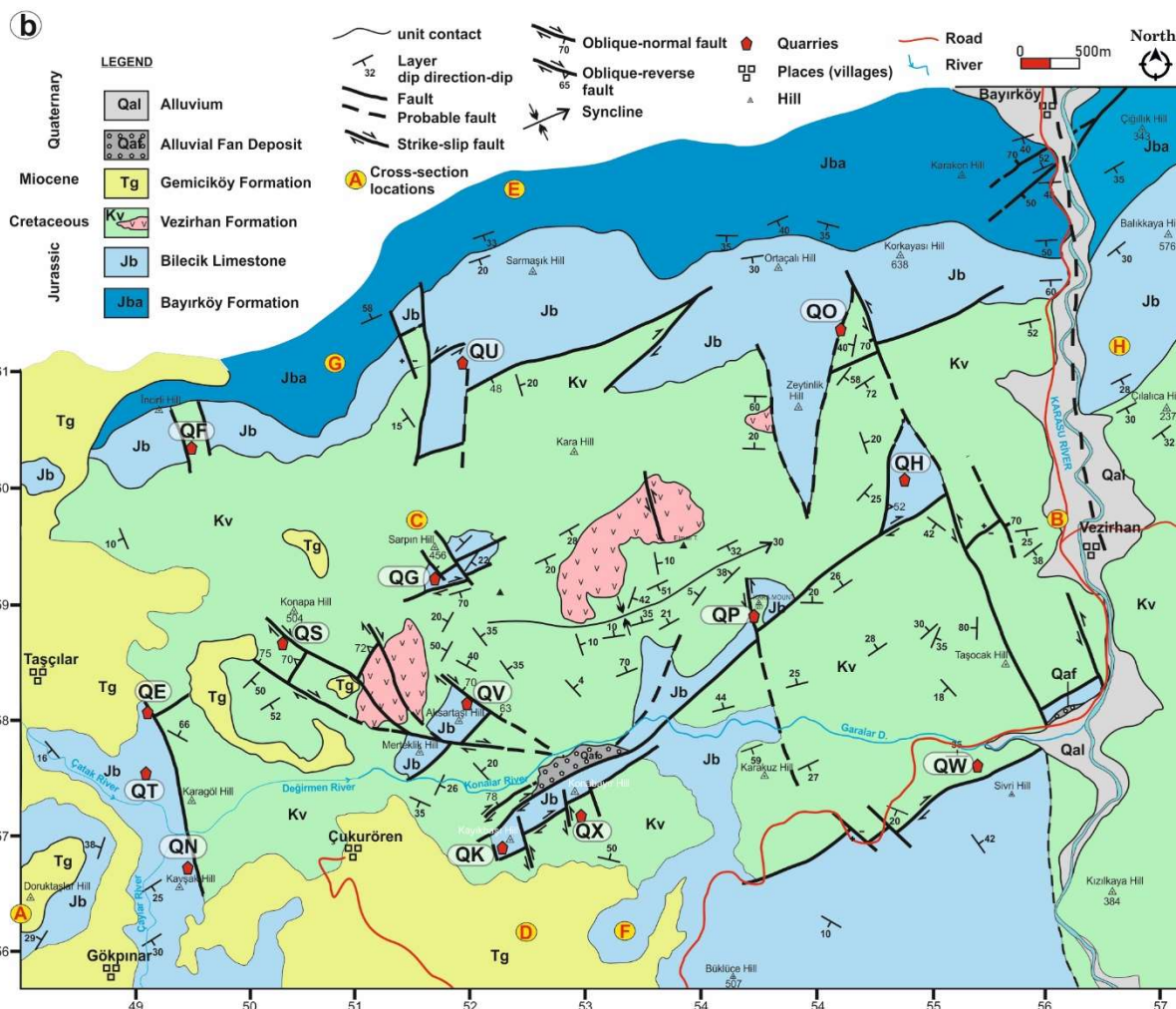
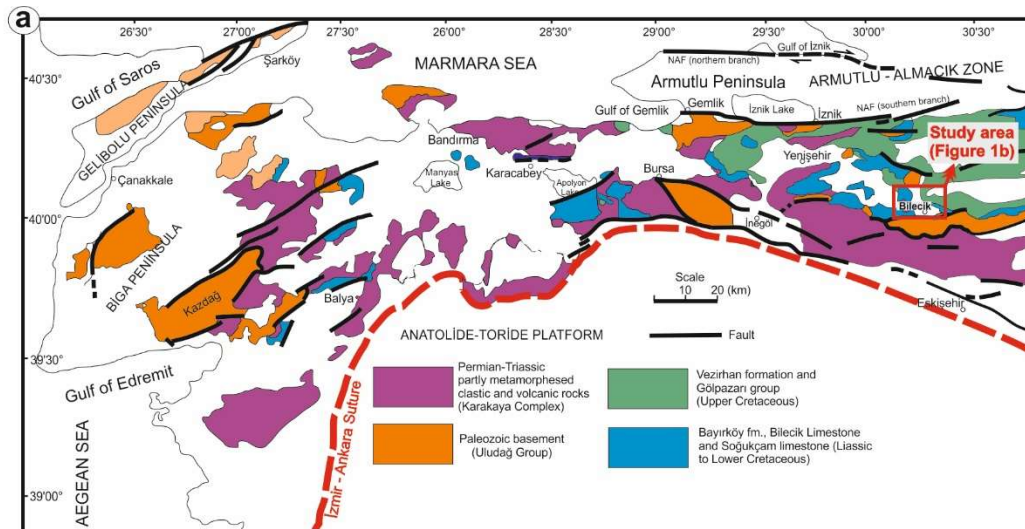


Figure 1. a. Simplified geological map of Sakarya Continent and major tectonic units of western Turkey separated by the sutures (modified from Yılmaz et al., 1997). b. Geological map of the study area (northern Bilecik, Turkey).

Studies on identifying the geological parameters controlling the marble patterns and block efficiency from quarries in Turkey are quite limited. It is crucial to determine the marble pattern and block efficiency that will be extracted from a quarry in advance for marble mining. This study aims to highlight the effects of

structures that have developed under tectonic control (faults, fractures, folds, etc.) on the selection of marble quarry locations and quarry productivity, to identify the geological factors affecting the development of patterns observed in marble quarries, and to explain the effects of these patterns on marble quarrying.

## 2. Geology of the study area

In the vicinity of the study area, the basement of the Sakarya zone is formed by the Söğüt metamorphics, which are composed of high-grade schists, gneisses, amphibolites, and migmatites of the Lower Paleozoic age (Çoğulu et al. 1965; Delaloye & Bingöl 2000; Okay et al. 1996, 2002; 2006; Topuz et al. 2007). This metamorphic basement is unconformably overlain by a transgressive Jurassic-Cretaceous sequence, which is observed extensively within the study area. The Bayırköy Formation, which is composed of Lower Jurassic aged conglomerate, cross-bedded sandstone, and debris flow deposits, represents the base of this transgressive sequence. This unit stratigraphically includes carbonate lenses and limestone intercalations upward. The Middle-Upper Jurassic aged Bilecik Limestone conformably overlies the clastic sediments of the Bayırköy Formation. The stratigraphically lower portions of the Bilecik Limestone consist of oolitic

limestone levels developed in environments where wave activity was effective in shallow marine settings. Stratigraphically, this limestone, which are rich in fossils and have a reefal character, are characterized by micritic limestone with low fossil content in the upper levels. The Vezirhan Formations, which have a deep-sea character, consisting of Cretaceous-aged chert limestone, red radiolarite, and pelagic limestone, conformably overlay these units. The Vezirhan Formation starts with semi-pelagic limestone that is white or cream-coloured, porcelain-like in appearance, nodular or banded with clay interlayers at the base. Above this level, pinkish-brown pelagic limestone, chert, and radiolarites are present. Greenish volcanic and volcanoclastic layers are also found as intercalations within this sequence. These units are unconformably overlain by the Miocene aged Gemiciköy Formation, consisting of coarse-grained clastics such as conglomerate and sandstone (Figure 1-2).

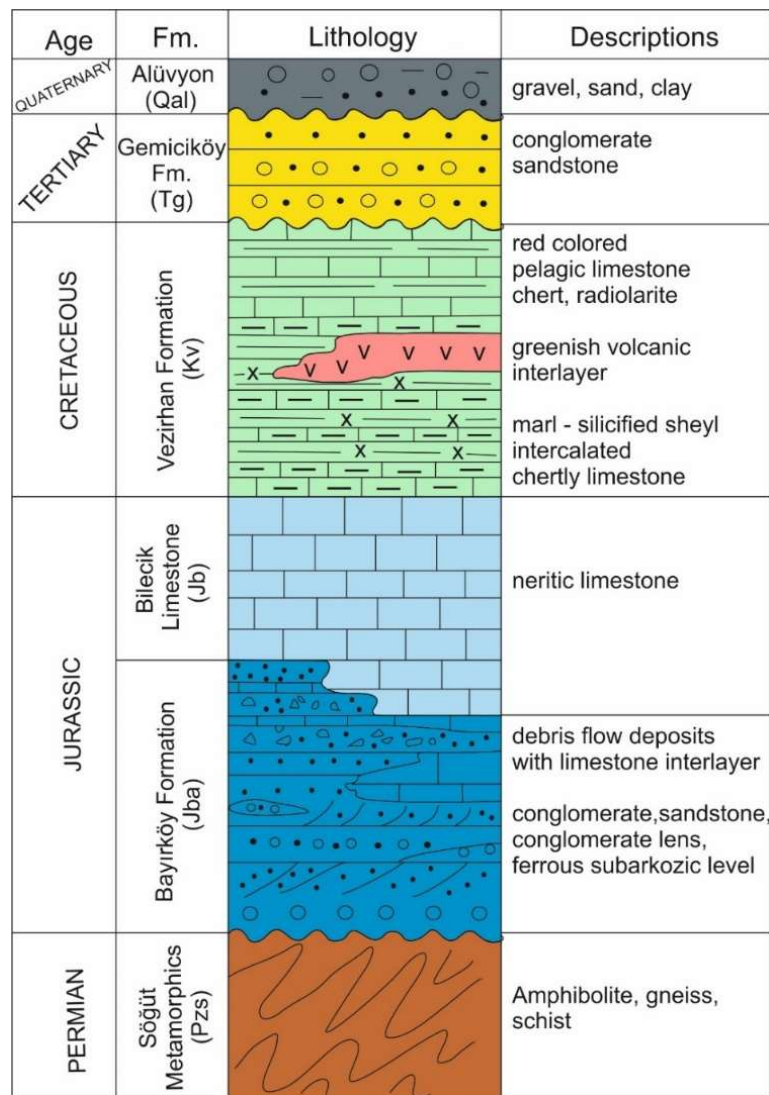


Figure 2. Generalized stratigraphic section of the study area (Northern Bilecik, Turkey).

### 3. Geological Parameters Affecting The Marble Texture

#### 3.1. Lithological and sedimentological features

The QV, QT, and QU quarries are located in areas where the shallow marine character of Bilecik Limestone's lower levels are outcropped stratigraphically. Therefore, the Bilecik Beige-Fossil and Bilecik Beige-Oolitic patterns with a low abundance of fossils and/or an oolitic patterns are extracted from these quarries (Figure 3a-b). The QK, QG, and QH quarries are excavated in the Middle-Upper Jurassic-aged Bilecik Limestone's stratigraphically middle levels. These levels consist of limestone with abundant fossil reef-like characteristics. From these levels, the Bilecik Beige-Reef pattern is extracted (Figure 3c). In the quarries QP and QO, excavated at the contacts between Bilecik Limestone and Vezirhan Formations, the Bilecik Beige-

Reef pattern belonging to the middle levels of Bilecik Limestone is observed.

Additionally, unlike the other quarries, blocks are extracted from lithologies belonging to the Vezirhan formation in the QP and QO quarries. In the QP and QO quarries, there is the Rosalia pattern extracted from the burgundy coloured pelagic limestone (Figure 3e). Furthermore, the greenish-beige coloured clay limestone with shale intercalations of the Vezirhan Formation also crop out at shallow depths in the QO quarry. These lithologies are less durable and more prone to plastic deformation compared to other rock types in the quarry. Therefore, zig-zag folds originating from tectonism have developed in the rocks at these levels of the Vezirhan Formation. The Zig-Zag Pattern, which arises from the relationship between rock type and tectonism, can be observed in the blocks extracted from these levels (Figure 3i).



Figure 3. Marble patterns from the quarries in the study area.



### 3.2. Faults

The study area is located in a tectonically active region influenced by the North Anatolian Fault Zone (NAFZ) and the Eskişehir Fault Zone (EFZ) in terms of its geological location. The faults passing through the marble quarries in this region and their vicinity are given in the geological cross-sections in Figure 4. When the geological map of the study area (Figure 1b) and its cross-sections (Figure 4) are examined, it can be seen that the marble quarries in this region are generally aligned along these fault zones. The faults generally follow the contacts between the Middle-Upper Jurassic Bilecik Limestone and the Cretaceous-aged Vezirhan Formation. The blocks (footwall and hanging wall blocks) on both sides of the fault plane in the areas where the faults pass have undergone brittle deformation to form fault breccia. Brecciated patterns

developed under tectonic control are observed in the blocks extracted from the marble quarry along these zones (Figure 3f). These brecciated patterns, observed in pinkish-red colours, are commercially called Rosalia breccia.

The quarries where the Rosalia breccia pattern is extracted are QK, QP, QO, and QU, and to a lesser extent, the QG and HDF quarries (Figure 1b). Apart from these brecciated zones, faults also cause the formation of various fracture systems in rocks, and the formation of these fracture systems leads to the formation of veins by various minerals settling in these fractures. In fact, the main factor controlling these triggering elements is faults. The strike, dip, and characters of the faults also play an important role in the marble patterns extracted from the quarries.

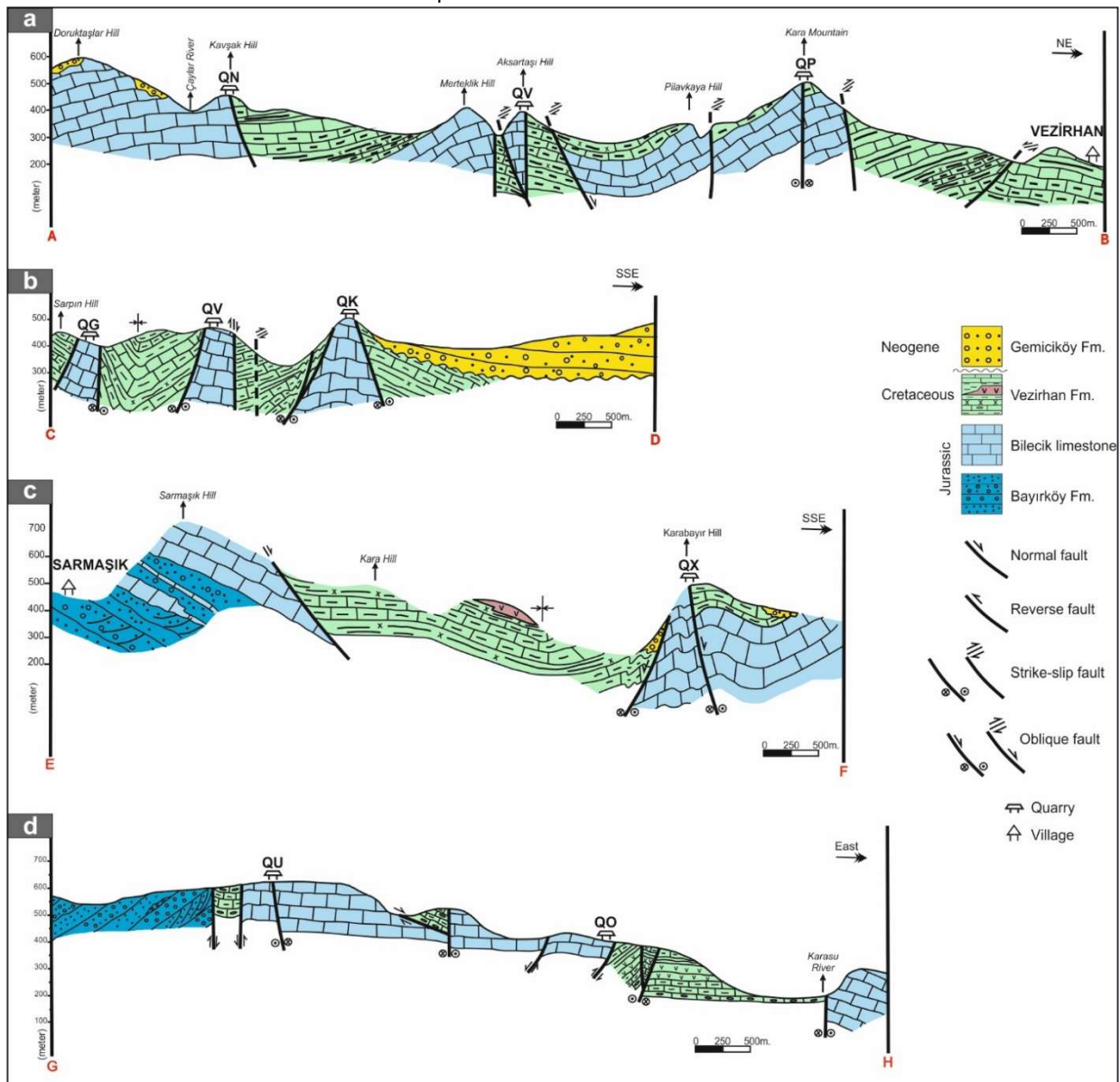


Figure 4. Geological cross-sections along the quarry sites and routes passing through the vicinity of the study area (see Figure 1b for section routes).

As part of this study, QK, QV, QN, and QH quarries were studied in detail. Within the scope of the detailed study, the detailed geological and structural geological characteristics of the four quarries were mapped, and pattern changes on a quarry basis were determined.

The QK quarry is surrounded by faults on all four sides (Figure 5). Along the right-lateral strike-slip fault zone

passing north of the quarry, a wide alluvial fan deposit has developed on the northern slope of Konabayır Hill. In addition, the left-lateral strike-slip fault zones observed in the western and eastern parts of the quarry have caused the displacement and fragmentation of levels belonging to the Bilecik Limestone extracted from the quarry.

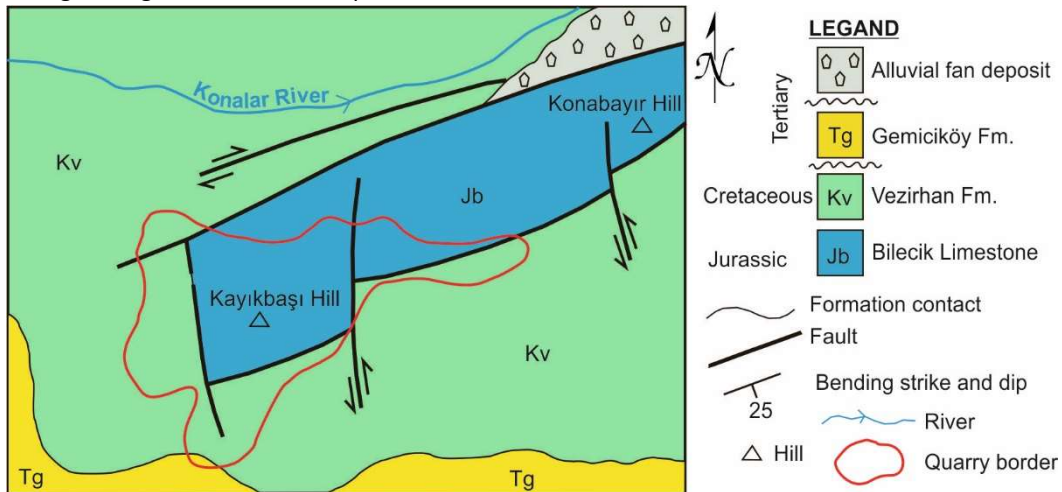


Figure 5. Geological map of the QK Quarry and its vicinity.

There is a vertical slip on the southern part of the QV Quarry and a right-oblique normal fault dipping east on the eastern part (Figure 6). As the fault, that borders the QV Quarry from the east dips outside of the quarry, rather than inside, when going deeper into the quarry, blocks with less effect from the fault, which are more

clear-in pattern and have better strength, are reached (Figure 9a). The dip direction of faults is towards the outside the quarry ensures that the blocks extracted from the quarry are less affected by the fault. Therefore, no tectonic breccia pattern is observed in the Vezir Quarry, or it is observed locally.

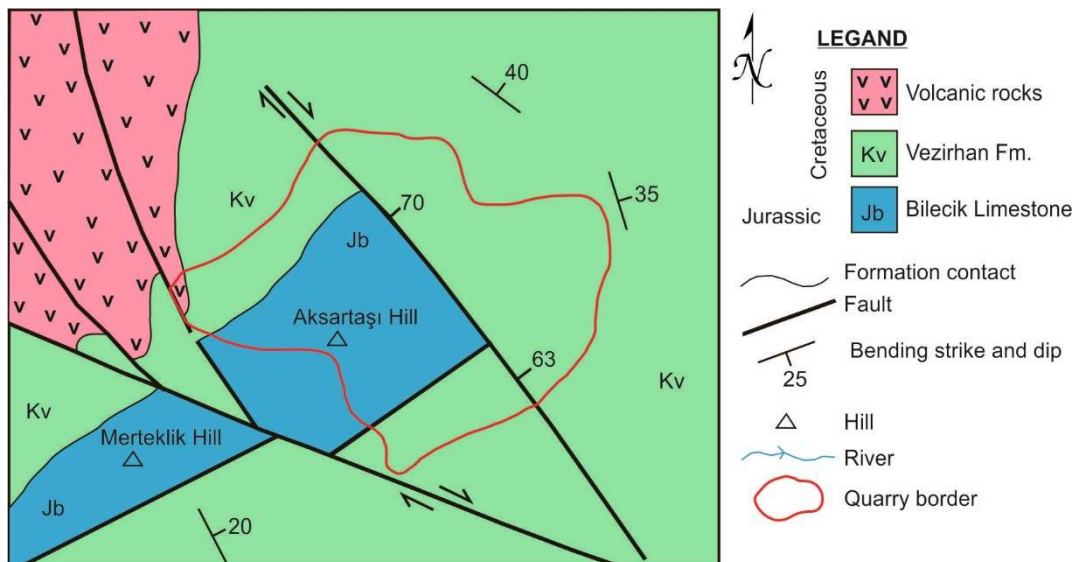


Figure 6. Geological map of the QV Quarry and its vicinity.

There is a fault that extends towards the QN quarry to the east of the QT quarry and is located at Kavşak Hill in the south (Figure 1b and Figure 7). Since this fault is located away from the quarry and dips to the east (away from the quarry), it does not significantly affect the patterns in the QT and QN quarries. Additionally,

there is a secondary fault connected to this main fault located just north of the QT Marble quarry. This fault is also a normal fault dipping to the north (away from the quarry) and does not significantly affect the patterns in the QT quarry. There is also a fault that extends along the N20W strike in the east of the QN quarry and dips

towards NE (Figure 9c). This fault, similar to the fault in the QT quarry, dips both towards the edge of the quarry and outside the quarry, so it moves further away from

the quarry as depth increases. This does not have a significant negative impact on the Bilecik Beige pattern and block efficiency in the quarry.

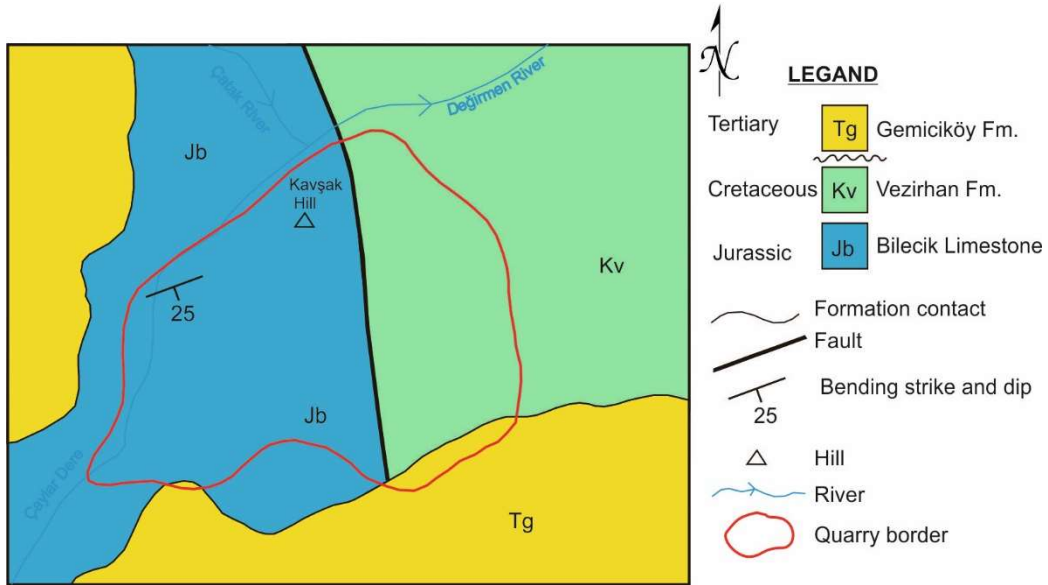


Figure 7. Geological map of the QN quarry and its vicinity.

The Bilecik Limestone, which is the rock type extracted from the QH quarry, is surrounded by faults on all sides at this location (Figure 8). The fault passing through the quarry and forms the boundary between the Bilecik Limestone and the Vezirhan Formation is a reverse fault with approximately N-S strike and a 52-degree dip to the south (Figure 9d). The Bilecik Limestone in the hanging wall block of the fault are pushed towards the red-brown pelagic limestone of the Vezirhan

Formation. This reverse fault movement has caused significant deformation and fracturing in the rocks in both blocks. Additionally, since this fault dips into the quarry, the fault zone is approached as depth increases in the quarry (Figure 9d). This negatively affects the block efficiency in the QH quarry. The blocks extracted from the HDF quarry show a Rosalia (breccia) pattern due to the tectonic faults in the quarry (Figure 3f).

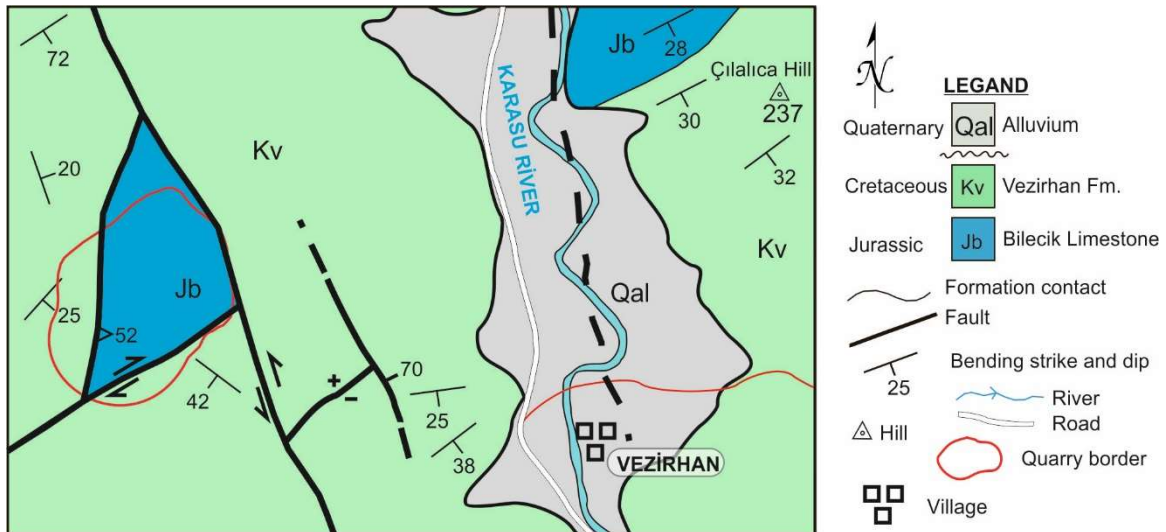
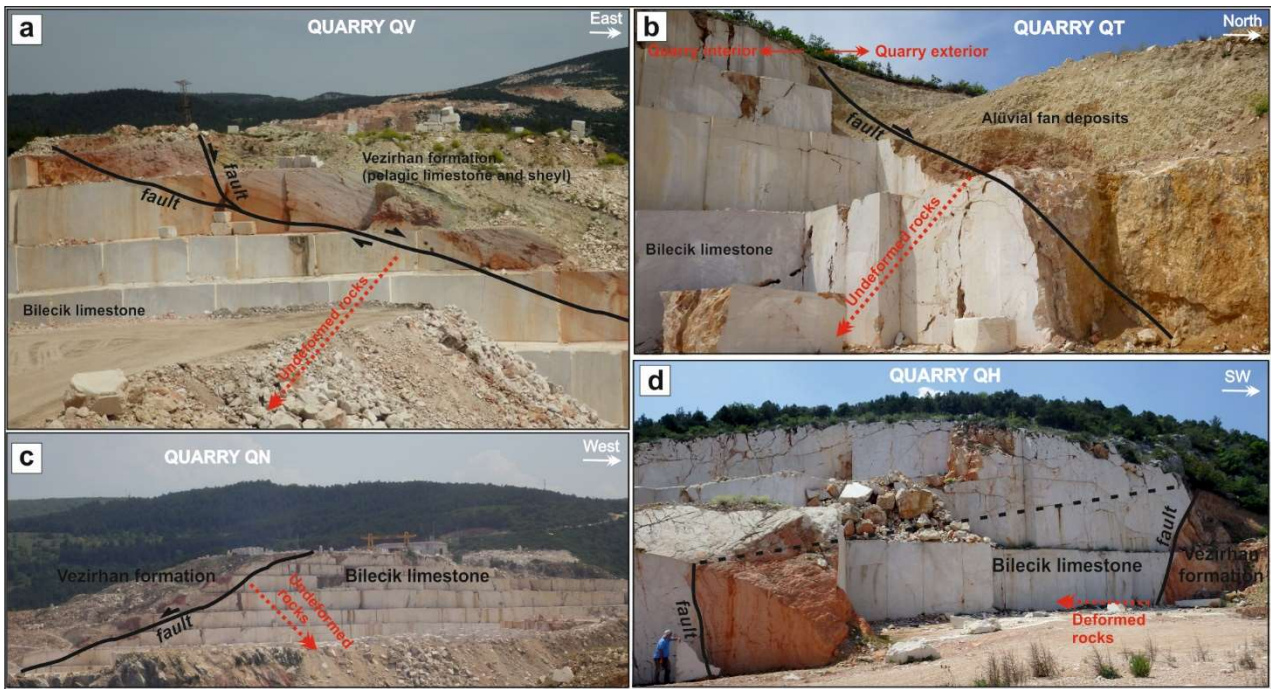


Figure 8. Geological map of the QH quarry and its vicinity.



**Figure 9.** The extents of the faults passing around the QV quarry (a), QT quarry (b), QN quarry (c), and QH quarry (d), and their effects on the rocks within the quarries.

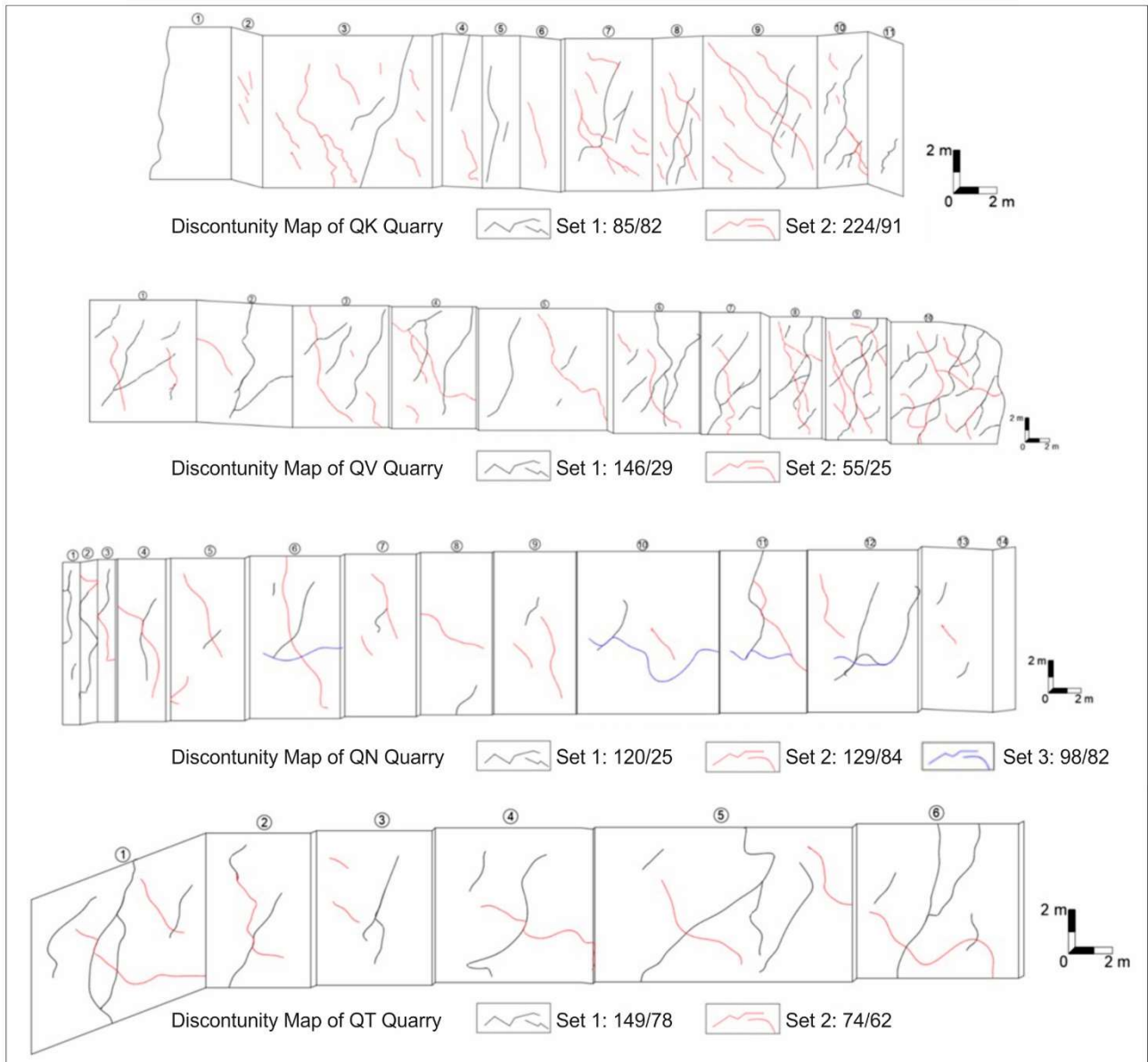
### 3.3. Discontinuity

The fracture-crack systems observed in the marble quarries in the study area are all of tectonic origin. These include tension cracks (1) developed by tension, shear cracks (2) formed parallel to joint planes by shear movement, stylolite (3) developed in rocks parallel to the principal stress direction, and sigmoidal cracks (4) formed in brittle-ductile shear zones. Tension cracks and sigmoidal cracks are typically later filled with aragonite minerals, forming aragonite vein patterns. These tectonic fractures are present in each quarry in the region, but are more commonly observed in QP, QK, QE, QF, QU, and QO quarries, which are located on or near fault zones. Stylolitic fracture systems are typically observed in the pelagic limestone levels of the red-bordeaux coloured Vezirhan formation or in the breccia levels of the tectonic contacts between the Bilecik Limestone and Vezirhan Formation. This stylolitic fracture pattern is called the Rosalia-stylolitic pattern (Figure 3e) and is mostly extracted from QP, QO, and QK quarries.

The production of block stone in quarries is a long process that requires detailed engineering geology

studies (such as fieldwork, discontinuity properties, lineament analysis, colour-pattern tracking, etc.) to estimate the potential production of block stone, determine the block sizes to be produced, manage the quarry according to the geological conditions and advance the production.

There are many parameters that affect the efficiency and production of block stone quarries. Among these parameters, those of geological origin directly affect efficiency and production. In this study, QK, QV, QN, and QT quarries were studied in detail. Within the scope of the detailed study, engineering properties of discontinuities were determined using the scanline method in accordance with the principles stated in ISRM (2007), and quarry discontinuity maps were prepared based on production benches for the relevant four quarries (Figure 10). The dominant orientation of the discontinuities in the quarry was determined using Dips 6.0 software (Rockscience, 2012) (Figure 11), and the spacing and aperture of the discontinuities were also determined. In addition, the block size index, volumetric joint count, and block quality designation that affect the block efficiency in the quarry were determined according to the discontinuities (Table 1).



**Figure 10.** Discontinuity maps prepared in the relevant quarries, (a) QK, (b) QV, (c) QN, (d) QT.

**Table 1.** Properties of discontinuities in QK, QV, QN and QT quarries.

Quarry name	Number of discontinuity	Discontinuity orientation	Discontinuity spacing	Discontinuity aperture	Block size index (I <sub>b</sub> )	Volumetric joint count (joint/m <sup>3</sup> )	Block quality designation (BKG,%)
QK	95	Set1:85/82 Set2:224/91	2,90 m	17 mm	4,84	0,81	%80
QV	169	Set1:146/29 Set2:55/25	10 m	23 mm	2,50	0,91	%78
QN	96	Set1:120/25 Set1:129/84 Set2:148/85 Set3:98/82	14,9 m	29 mm	3,49	0,65	%83

QT	61	Set1:149/78 Set2:74/62	12,65 m	36 mm	3,73	0,53	%88
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In light of these data, the block size that can be obtained from QK, QV, and QT quarries is controlled by 2 sets of discontinuities, while in QV quarry it is controlled by 3 sets of discontinuities. Discontinuities in QK quarry are defined as "very wide-spaced discontinuities", and those in QV, QN, and QT quarries are defined as "intensely wide-spaced discontinuities" (ISRM, 1981). Based on the data of volumetric joint count, all quarries examined are defined as sparsely jointed rock mass (Palmstorm, 1982 and 1996). The block size index is greater than 3 for all quarries except QV quarry, where it is 2.50. The Block Quality Index (Elçi and Türk, 2014) is greater than 50% for all quarries examined (Table 1).

### 3.4. Aragonite veins

As a result of tension and sigmoidal fractures in the quarries being worked on, aragonite minerals have filled in to form honey-coloured aragonite veins. The thicknesses of these veins range from a few millimeters to 15-20 centimeters, sometimes appearing as continuous bands while in other places they are discontinuous, fragmented by small faults. Especially the thin aragonite veins do not pose a problem in block production, but they do disrupt the homogeneity of the marble pattern. Aragonite veins can be observed within Bilecik Beige and Rosalia patterns (Figure 3b-c). However, in the QP quarry excavated on the strike-slip fault zones, the Aragonite pattern consisting of only aragonite or the brecciated-aragonite pattern with a very high aragonite ratio is extracted (Figure 3g-h). In fact, the thicknesses of aragonite veins can reach several meters, and the extracted blocks can be entirely composed of aragonite.

### 4. Conclusion

- Quarries for marble production in the northern areas of Bilecik were excavated within the Middle-Upper Jurassic Bilecik Limestone of the Jurassic-Cretaceous aged transgressive succession, Cretaceous Vezirhan Formation and/or at the contacts of these two units.
- The size of the blocks produced from these quarries is generally controlled by two, and rarely by a third set of discontinuities. The discontinuities in the quarries are "intensely wide-spaced" and "very wide-spaced". This indicates the low jointed nature of the limestone mass and allows for the production of economically valuable blocks from block quarries excavated within these limestone formations.

- The marble patterns in this region are identified as Bilecik Beige Pattern (Bilecik Beige-oolithic, Bilecik Beige-reefal, Bilecik Beige-fossiliferous), Rosalia Pattern (Rosalia-brecciated, Rosalia-styrolitic), Aragonite Pattern, Brecciated Aragonite Pattern, and Zig-Zag Pattern.
- The Bilecik Beige-oolithic pattern is stratigraphically extracted from the basal levels of the Bilecik Limestone, the Bilecik Beige-reefal pattern is extracted from the middle levels of the Bilecik Limestone with a shallow marine coral reef character, the Bilecik Beige-fossiliferous pattern is also extracted from the shallow marine middle levels, and the Bilecik Beige-nonfossiliferous pattern is extracted from the uppermost levels that represent a deeper marine environment.
- The Rosalia pattern is divided into two types: Rosalia-brecciated and Rosalia-styrolitic. The Rosalia-brecciated pattern is a brecciated pattern extracted from the fault zones where the Bilecik Limestone and Vezirhan Formation are in contact. The Rosalia-styrolitic pattern is extracted from the pelagic limestone of the Vezirhan Formation that represent a deep-sea environment with a reddish-brown colour.
- In addition to the Rosalia-brecciated pattern, Aragonite and Brecciated Aragonite patterns, which develop with the effect of secondary aragonite minerals emplaced in the fracture-cracks, are extracted from the fault zones.
- The geological parameters that affect marble production, block efficiency, and marble pattern in the study area (Bilecik North Quarries - Western Turkey) are (1) the lithological and sedimentological properties of the sequence, (2) faults (type, strike, dip, and density), (3) cracks-fractures, and (4) aragonite-filled veins.

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# Colour changing in light-sensitive minerals exposed to UVA<sub>340 nm</sub> rays

F. Sitzia\*<sup>1,2</sup>, C. Lisci<sup>1,2</sup>, P. Moita<sup>1,2</sup>, S. Bottura-Scardina<sup>1</sup>

(3) HERCULES Laboratory, Institute for Advanced Studies and Research, University of Évora, Largo Marquês de Marialva 8, 7000-809 Évora (Portugal),

(4) Geosciences Department, School of Sciences and Technology, University of Évora, Rua Romão Ramalho 59, 7000-671, Évora (Portugal)

\* corresponding author: fsitzia@uevora.pt

## Abstract

Some minerals suffer from colour variations when exposed to incident sunlight. The minerals which possess this property are defined as “photosensitive”, and their colour changes especially under UV radiation. This work targeted this phenomenon and selected 27 among the most widespread and widely occurring mineral phases for. Initially, the chosen minerals have been characterised chemically (SEM-EDS) to detect common impurities often responsible for the chromatic alteration; subsequently, these minerals were exposed to UVA<sub>340 nm</sub> rays at 0.35 W/m<sup>2</sup>. The chromatic shifts were measured in the CIELAB space immediately after the artificial ageing to identify the changes with respect to the initial colour. The results show that only 11% of the minerals displayed slight chromatic changes, i.e. imperceptible to the naked eye, while 89% of the chosen minerals manifested visible color change with relative perceptual difference  $\Delta E$  ranging from 3 to 32.

**Key words:** artificial ageing, light-sensitive minerals, QUV, ultraviolet, colourspace

## 1 Introduction

Ultraviolet (UV) is a form of electromagnetic radiation with wavelength  $\lambda$  ranging from 10 nm (corresponding to ca. 30 PHz frequency) to 400 nm (750 THz). Shorter than visible light but longer than X-rays, UV radiation (10 - 400 nm) can be further classified into the sub-categories UVA, UVB, UVC by wavelength, as specified in the ISO-21348 standard.

UV radiation is an important spectral component of daylight. Sunlight in vacuum consists of about 50% infrared (IR), 40% visible light (Vis) and 10% UV in terms of energy, equivalent to a total intensity of about 1400 W/m<sup>2</sup> (Kopp and Lean 2011). To reach sea-level, the solar radiation must pass through multiple atmospheric layers, thus seeing the relative amounts of these energetic components changing from the values in vacuum. The layers of gas enveloping the Earth can, indeed, block around 77% of the solar UV at its zenith. In these latter conditions, sunlight consists of approximately 44% of Vis light ( $390 < \lambda < 700$  nm), 3% IR ( $700 \text{ nm} < \lambda < 1 \text{ mm}$ ) and UV ( $280 < \lambda < 400$  nm) (Bennson 1984). As to the relative amount of each sub-component, above 95% of the terrestrial UV radiation is UVA (the least energetic component), whereas UVB represent only 5% and almost no UVC (the most energetic end of this electromagnetic range).

The effects of UV-exposure on a certain substance depend on its nature. In organic materials such as polymers, UV-induced photodegradation caused by

radiation in the 300 - 400 nm range affects their aesthetic properties with subsequent discoloration, yellowing and surface whitening (Cogulet et al. 2016), (Rajagopalan and Khanna 2014), (Rosu et al. 2009). In inorganic materials, UV exposure can similarly cause alterations of their chemical structure, in spite of a higher resistance to photodegradation than organic compounds. The possible outcome is that such highly-resistant materials like stones can experience reversible or irreversible colour changes under exposure to sunlight, particularly to UV rays. This effect is called *photosensitivity*.

Previous research has targeted mineral photosensitivity, reporting a direct correlation between UV irradiance over a given period of time, rock discoloration and gloss decrease (Careddu and Marras 2013), (Navarro et al. 2019), (Sitzia et al., 2021a), (Sitzia et al., 2021b).

Before these works, further literature documented further instances of light-induced changes in mineral specimens. A notable case in point is proustite (Ag<sub>3</sub>AsS<sub>3</sub>), whose crystals showed chromatic alterations ever since first extracted and brought to the surface (Virgil Lueth 2018). Aside from this minerals, intentional Vis-induced colour changes of a specific group of silver halide minerals (AgBr, AgI, AgCl) represented the basis of film photography (Bringley et al. 2005). Accidental changes were reported for Hackmanite (Na<sub>8</sub>Al<sub>6</sub>Si<sub>6</sub>O<sub>24</sub>(Cl,S)<sub>2</sub>) from Bancroft, Ontario, which exhibits a slow-bleaching blue coloration upon exposure to 1850 Å (185 nm) ultraviolet. This additional shade alters the original red-



purple coloration of this mineral (Kirk 1955), altering its final appearance. Finally, corderoite, ( $\text{Hg}_3\text{S}_2\text{Cl}_2$ ) from Miocenic sediments in Nevada and regular Hackmanite are reportedly photosensitive (Foord et al. 1985).

Some colour changes may be reversible upon removal of the light source or through irradiation (Nassau 2013) (Horak. J.M. 1994) or do not alter the physical or chemical properties of the specimen, as with some quartz ( $\text{SiO}_2$ ) varieties (Rossman 2019). For those minerals that manifest light-sensitivity, this behaviour forms three categories (Howie 1992), (Nassau 2013):

- I) (possibly reversible) light-induced colour changes without any other physical or chemical alteration—e.g. the faded colour of blue celestite ( $\text{SrSO}_4$ ) may return to its original colour if stored in the dark.
- II) (irreversible) light-induced decompositions producing significant bulk physical or chemical changes (LD). Good instances of this second class include red  $\alpha$ -HgS (trigonal, cinnabar), where light causes a phase transition of its black polymorph  $\beta$ -HgS (cubic, metacinnabar) at room temperature (Ballirano, Botticelli, & Maras, 2013). Similarly, red-orange coloured realgar ( $\text{As}_2\text{S}_3$ ) converts permanently to yellow pararealgar ( $\text{As}_4\text{S}_4$ ) at low temperature (Douglass, Shing, & Wang, 1992).
- III) (irreversible) light-accelerated surface reactions with air, moisture, and/or pollutants (LA). Instances for this last class are vivianite ( $\text{Fe}^{2+}3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ ) darkening with possible crystal decohesion upon light exposure.

Light may cause chemical rearrangement of the mineral structure by converting one phase into another (realgar to pararealgar, (Brunton et al. 1985)) or releasing volatile species even at room temperature, such as metallic mercury (Howie 1979).

Colour variations upon light exposure become crucial when minerals are considered for industrial and economical purposes. This phenomenon is critical for building material, although changes upon sunlight exposure is a slow process that becomes visible only in the longer run. Discoloration was reported in ornamental stones: in fact, exposure to UV  $_{340\text{ nm}}$  light proved to display considerable effects in the optic properties of a wide variety of Portuguese limestones (Lisci et al. 2022).

Given the above considerations, this study intends to provide a systematic description for a set of common phases present in sedimentary, magmatic and metamorphic rocks. A group of 27 minerals was subjected to artificially induced alteration through UV irradiation in controlled conditions, and the ensuing colour changes were assessed after the process. The provenance of the chosen minerals is mostly continental Portugal and secondarily Italy, Spain, Morocco, Brazil, China and Afghanistan.

Before exposure to UV, the minerals have been analysed in order to obtain information about their elemental and crystallographic composition (SEM-EDS and  $\mu$ -XRD).

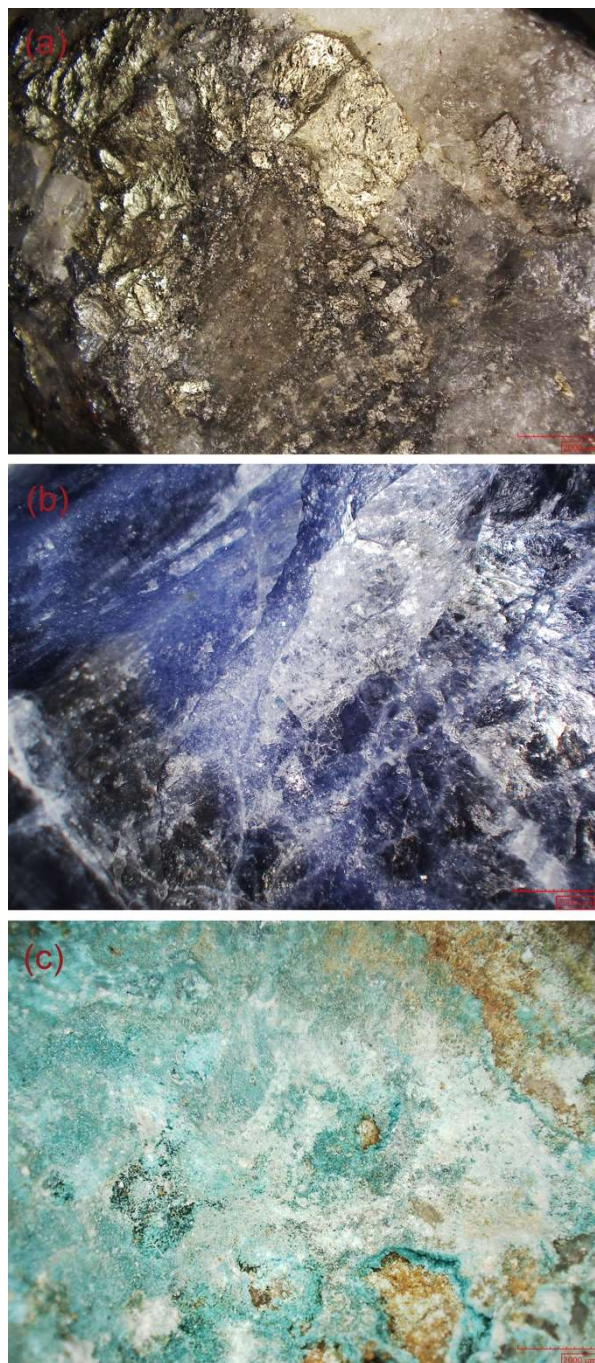


Fig. 1. Example of selected minerals: (a) arsenopyrite, (b) sodalite, (c) malachite.

## 2 Materials and methods

Table 1 provides an essential description of the provenance, classification and crystallographic properties of the minerals selected for the study. Those samples have been collected from private collections, and the criteria for their selection were frequency of occurrence and susceptibility to colour shifts, based on

bibliographic research. Before and after UV exposure, the crystallographic and elemental profile of the samples was determined with  $\mu$ -XRD and SEM-EDS respectively (Table 2). The colour of the chosen samples was measured before and after exposure with a routine involving HSI and digital processing; UV changes were induced by performing photoaging tests with an UV aging test chamber.

### **2.1 $\mu$ -XRD analyses**

A Bruker AXS D8 Discover XRD with a Cu K $\alpha$  source, operating at 40 kV and 40 mA and a Lynxeye 1-dimensional detector was used to obtain detailed information about the crystallographic structure and chemical composition of the chosen samples. Scans were performed from 3 to 75° 2 $\theta$ , with 0.05° 2 $\theta$  step and 1 s/step measuring time by point. Diffract-Eva software from Bruker with PDF-2 mineralogical database (International Centre for Diffraction Data-ICDD) was utilized to interpret the scans. During the  $\mu$ -XRD experiments, a Goebel mirror and 1-mm collimator were employed according to previous methods (Sitzia et al. 2021a), (Sitzia et al., 2021).

The  $\mu$ -XRD characterization on the crystals could be difficult due to the scarcity of reflections. It often happens that the diffractogram only presents a peak that the interpretation software is not able to associate with the correct mineral. Often, the diffractogram is completely flat. In some cases, as in the beryl, 3 diffractograms were performed by moving the crystal to different positions and the final diffractogram was the sum of the 3. In other cases the diffraction software immediately identified the mineral.

### **2.2 SEM-EDS analyses**

To understand the elemental composition, minerals were analysed by variable pressure HITACHI S3700N SEM coupled with a Quantax EDS microanalysis system. The Quantax system was equipped with a Bruker AXS XFlash Silicon Drift Detector (129 eV FWHM spectral resolution at Mn K $\alpha$ ).

Standardless PB/ZAF quantitative elemental analysis was performed using the Bruker ESPRIT software. The operating conditions for EDS analysis were as follows: backscattering mode (BSEM), 20 kV accelerating voltage, 10 mm working distance, 120  $\mu$ A emission current, and 40 Pa pressure in the chamber. The detection limits for major elements (> Na) were in the order of 0.1 Wt. %. The instrumental statistical uncertainty was 1 $\sigma$ .

### **2.3 Colorimetric analyses**

To detect the colour, Vis-NIR HSI hypercubes were acquired with a Specim IQ<sup>®</sup> hyperspectral camera by Specim, Spectral Imaging LTD. (Oulu, Finland). The camera is equipped with a Si-CMOS sensor serving as a

photographic camera with resolution of 512 x 512 pixel (5 Mpixel) and a spectral detector (400-1000 nm spectral range). The radiation is allowed into the camera through two distinct objectives, one for the spectral analysis and the other for the photographic recording. The hypercubes were acquired by placing the reference for white contextually to every acquisition, without spectral binning (204 channel/pixel), with spatial resolution in the 0.1-0.3 mm/pixel range and integration time in the 96-116 ms range. Spectral extraction and mapping were performed as single-pixel extraction of the irradiance values with the ENVI<sup>®</sup> 5.3 software by L3Harris Geospatial (Broomfield, CO, USA).

Before the UV exposure test (moment 0), the colour of the minerals was measured on 5 representative areas by using a routine based on HSI and MATLAB programming. The adopted procedure relies on the initial acquisition of irradiance values for the white standard, a black reference material and the areas to inspect with the HSI camera. Those values were directly fed to a custom-made function written in MATLAB<sup>®</sup> environment (R2020b by MathWorks). This function was written to perform automated data averaging, conversion to apparent reflectance (dark-subtracted) first, and to the CIELAB colour space as recommended in the ASTM E308-22 standard, the adopted chromaticity coordinates x, y and z specific for the 10° Supplementary Standard Observer, 5 nm spectral step and D65 input Standard Illuminant. The final conversion to the perceptual space (CIELAB colour space, L\*, a\*, b\*) adopted the X<sub>n</sub>, Y<sub>n</sub> and Z<sub>n</sub> values of the Standard Illuminant D65 (output illuminant) and CIE XYZ-to-CIELAB colour conversion formulas.

The process was computed with a custom-made, automated procedure by using an iterative MATLAB code. For each mineral, 5 representative areas were chosen and sampled digitally with HSI before exposure (moment 0).

After 2 days of UV exposition performed by the QUV spray chamber (Q-LAB) mineral colours were measured. Following the exposure, the colour coordinates were measured on the same spots as moment 0 and by adopting the same HSI-MATLAB routine as before.

### **2.4 UV photoaging tests**

The QUV-spray Q-LAB climatic chamber is equipped with 8 UV lamps with a maximum life of 5000 hours work. These lamps provide the best possible simulation of sunlight in the critical short wavelength region from 365 nm down to the solar cut-off of 295 nm. Its peak emission is at 340 nm.

Lamps intensity expressed in W/m<sup>2</sup> can be regulated by the operator in the range 0.35 <  $\lambda$  < 1.55 W/m<sup>2</sup> in order to equalize, accelerate (up to 2 times) or even set an irradiance (I) lower than the natural one. For this

purpose it is necessary to specify the order of magnitude of the UV<sub>340 nm</sub> radiation in the natural environment.

According to Q-LAB, an irradiance of 0.35 W/m<sup>2</sup><sub>340 nm</sub> is equivalent to noon summer sunlight and 1.36 W/m<sup>2</sup><sub>340 nm</sub> represents 2X solar maximum. By using this irradiation, the test is accelerated. The irradiation of 0.35 W/m<sup>2</sup><sub>340 nm</sub> is the so-called *average optimum* and is equivalent to March/September middle-latitude sunlight for “average” or low UV exposure. For our test the chamber was set by performing a new cycle “Sitzia standard” consisting of 48 h of UV exposure at 0.35 W/m<sup>2</sup><sub>340 nm</sub> with 45°C temperature. This cycle exposes the minerals to a non-accelerated test with a temperature low enough not to create mutations in the chemical structure of the same.

As above mentioned, the test last for 48 hours where minerals were exposed to the following irradiance energy: because 1 W/m<sup>2</sup><sub>340 nm</sub> corresponds to 1 J/m<sup>2</sup> · s, 0.35 W/m<sup>2</sup><sub>340 nm</sub> = 0.35 J/m<sup>2</sup> · s. In consideration of the above conversion factor, the irradiation of a 48-hour photoaging test would amount 0.06 MJ/m<sup>2</sup>.

In fact, (0.35 J/m<sup>2</sup> · s) · (1.728 · 10<sup>5</sup> s) = 60480 J/m<sup>2</sup>, or 0.06 MJ/m<sup>2</sup>.

### 3 Results and discussion

The chromatic effects of the UV aging tests are shown in Figs. 2 and 3, while the complete chart of discoloration is given in Fig. 4. This latter figure reports for each mineral phase two squares filled with a solid colour, one corresponding to the original appearance and the other to the colour following the UV-photoaging tests. The changes occurred in the study are also given in numerical form at the end of each row. This value is expressed as the distance metric ΔE of perceptual colour difference, calculated with the formula:

$$\Delta E = ((L^*_{\text{post}} - L^*_{\text{pre}})^2 + (a^*_{\text{post}} - a^*_{\text{pre}})^2 + (b^*_{\text{post}} - b^*_{\text{pre}})^2)^{0.5}.$$

When ΔE < 2.3, the colour separation is not detectable; otherwise (ΔE > 2.3), the human eye becomes capable of perceiving those differences. In this case, the colour separation is classified as JND (just noticeable difference) (Witzel et al. 1973). Using this value as a reference of perceptual colour separation, the numerical data in Fig. 4 are evaluated. At a close look, it becomes apparent that only 3 samples of 27 (11% of the total) had ΔE < 2.3. In these samples (gypsum, halite, quartz) the colour changes are, indeed, so minimal that they could not be observed with the naked eye. As for the remaining 24 mineral samples (89%), the colorimetric variations were so strong that they became perceptually noticeable. Within this group, ΔE ranges from 3 to 32.

Excluding the samples with subperceptual colour changes, the data allow drawing three clusters of minerals. The first includes minerals displaying relatively low colour distance (2.3 < ΔE < 6), and it includes

orthoclase, beryl, arsenopyrite, cinnabar, malachite, talc, barite and kyanite. A second cluster gathers minerals with average colour shift (6 < ΔE < 20) like calcite, topaz, galena, muscovite, fluorite, sodalite, aquamarine beryl and aragonite. The final group is of those minerals which manifest strong colour variation (ΔE > 20), namely the cluster of tourmaline, apatite, hematite, biotite, lazurite, optical calcite and grossular. Within this latter class, particularly notable are apatite (ΔE = 30), hematite (ΔE = 26.3), lazurite (ΔE = 27), optical calcite (ΔE = 32), and grossular (ΔE = 20.1).

Among these minerals with the greatest change, further considerations must be drawn. Two of them, i.e. lazurite and grossularia, display indeed a strong colour shift, yet their relative ratio between the a\* and b\* component does not seem to have been significantly affected. However strong is their ΔE, one stays predominantly blue coloured (lazurite), while the other remains red (grossularia). Different is the case of apatite, whose initial green colour had scaled down to a dull grey with the UV irradiation. Thus, this mineral loses completely its characteristic green component. Another outstanding case is that of optical calcite, those initial yellow shades to a light grey with only a vestigial, weak yellow component.

Also worthy of notice is the fact that the minerals with ΔE > 20 did not restore their initial original colour afterwards (i.e. in dark). In fact, galena (ΔE = 12.1) regained its original colour.

The causes of colour variation are quite complex and would require dedicated attention to elaborate hypotheses about their causes. However, this work attempts to advance some theories based on background research.

Colour arises from interaction of white light with the mineral surface, where it may be reflected, transmitted or absorbed. Giving a rough approximation of the ensemble of all radiative interactions, the portion that reached the eye is the one that is not absorbed, i.e., only the scattered component. The portion that undergoes absorption promotes electronic transitions of the valence shells of minerals, and the exact phenomena form a long list of radiative interactions.

Cinnabar is known for displaying colour changes with UV irradiation by forming black metacinnabar. This latter phase owes its dark colour to an hypsochromic shift of the bandgap edge by 1.2 eV (380 nm) of the initial 2.0 eV edge (610 nm) of α-HgS, with the result that the black mineral absorbs radiation in the UV range and becomes invisible to the eye (Dickson and Tunell 1959). In our case, the observed colour shift was rather weak (ΔE = 5.7), and no evidence of this structural rearrangement in the mineral structure was gathered.

In other minerals, the colour variations could be due to the oxidation of organic matter, iron (from Fe<sup>+2</sup> to Fe<sup>+3</sup>) and manganese (from Mn<sup>+2</sup> to Mn<sup>+3</sup>).

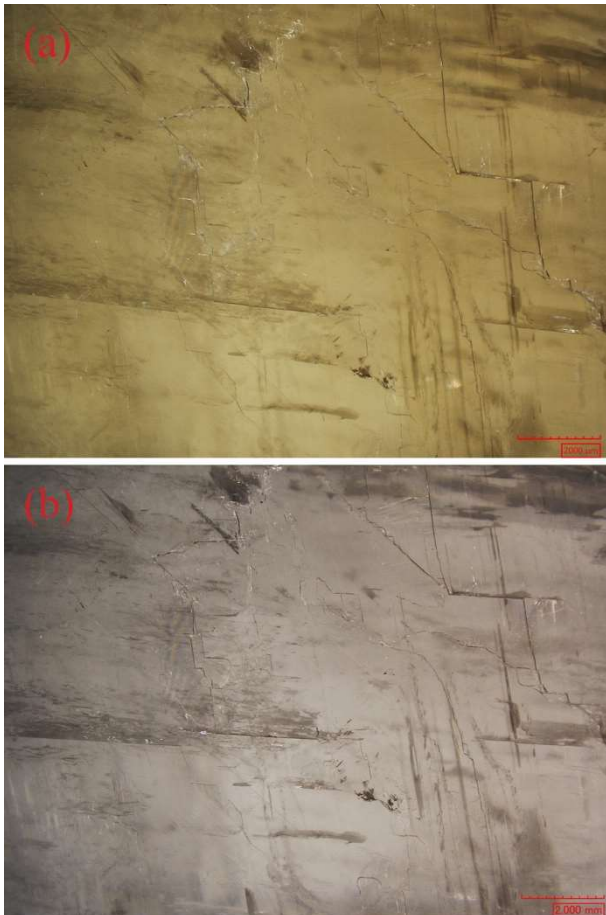


Fig. 2. Colour change of the optical calcite. (a) pre UV exposure, (b) post UV exposure

Other kinds of colour variation, especially those related to a decrease of the  $L^*$  coordinate, may arise from micro-fractures of the crystalline lattice, with subsequent morphological alterations. This has proved to be a crucial factor since different degrees of surface roughness proved to impact significantly the colour of minerals and stones (Sitzia et al. 2022), (Sitzia et al. 2023). An important study on light sensitive minerals was published in 1992 (Nassau 2013). In that paper, the author identified 35 light-induced color changes, 8 light-induced decompositions, and 78 light accelerated surface reactions. These changes he related to the 15 physical and chemical causes of colour (Rogers 1964).

One of the simplest cases of colour modification is the case of fluorite, whose colour arises from the occurrence of the so-called *hole colour centres* or *F-centres*. More specifically, coloration in this class of minerals is the result of an electron taking the place of a vacancy of defective halide ions (F) that had been previously removed. The absence of the ion leaves unpaired electrons in the crystalline lattice that are free to interact with the incoming radiation, allowing the striking light to promote those unrestricted electrons. When irradiated by natural sources, these electrons may get displaced, perturbate the electronic cloud producing

the original colour and alter the original colour. A possible outcome of the striking of external energy in thermic or radiative form on this kind of minerals is that their F-center may be removed. As a result, their colour may fade or disappear (Nassau 2013). This could be the case for the fluorite in our study, that displayed indeed  $\Delta E = 7.9$ .

Another interesting case with considerable colour shift ( $\Delta E = 27$ ) is lazurite. The colour of this mineral arises from charge transfers in gaseous polysulfide ions trapped within the aluminosilicate cage, especially  $S_3^{2-}$ . Degraded lazurite occurs when aluminium is removed from the aluminosilicate cage, causing the network to open and release the sulfide-based chromophore, thus discoloration (Cato, Borca, Huthwelker, & Ferreira, 2016). This value tends to increase, leaving the mineral dull and tendentially white greyish. As a matter of fact, the lapis lazuli gemstone showed a positive change ( $+\Delta L^*$ ) in the perceptual brightness.

Two minerals that displayed a substantial colour change are apatite and optical calcite. Both minerals are already known to be sensitive to light. Calcite itself is known to become colourless following exposure to UV radiation, although some American varieties can turn pink. Colour change of some calcites seems to be reversible if left in complete darkness for a few days; however, this was not the case for the calcite in this study.

The light sensitivity of calcite arises from the presence of impurities or structural defects within the crystal lattice. Calcite crystals can contain trace amounts of transition metal ions trapped within their crystalline lattice that can act as chromophores like manganese ( $Mn^{+2}$ ) or copper ( $Cu^{+2}$ ). Chromophores are responsible for absorbing light and causing the calcite crystal to undergo a change in its electronic structure, resulting in a change in colour. Calcite crystals may also contain such structural defects as vacancies or dislocations, which can trap and absorb radiative energy (Howie 1992). This can result in the excitation of electrons within the crystal lattice and subsequent changes in the crystalline optical properties, such as their colour or opacity.

Calcite crystals can exhibit light sensitivity due to the presence of organic compounds, such as humic acids or other carbon-containing molecules, which can interact with light and cause the Calcite to darken or become opaque.

In the apatite sample, when light interacts with crystals, it can cause the atoms or ions within the crystal lattice to shift or rearrange, leading to changes in a crystal's properties. Additionally, apatite crystals contain trace amounts of impurities, such as transition metals, sodium or which can also contribute to their light sensitivity. In our apatite a percentage of 0.36 % Na has been found (Table 2). These impurities can absorb light energy and undergo photochemical reactions, leading to changes in the crystal's properties. The exact mechanisms behind the light sensitivity of apatite are complex and can vary

depending on the specific type of apatite and the environmental conditions. However, it is generally believed that the light sensitivity of Apatite is related to the crystal's ability to absorb and interact with light energy, causing changes in its structure, composition, or properties (Nassau 2013). It is worth noting that not all apatite minerals are light sensitive to the same degree, and the extent of their light sensitivity can vary depending on factors such as crystal size, impurity content, and the wavelength and intensity of the incident light. The light sensitivity of Apatite can have various practical applications, such as in photoluminescent materials, optical devices, and geological dating techniques (Howie 1992).

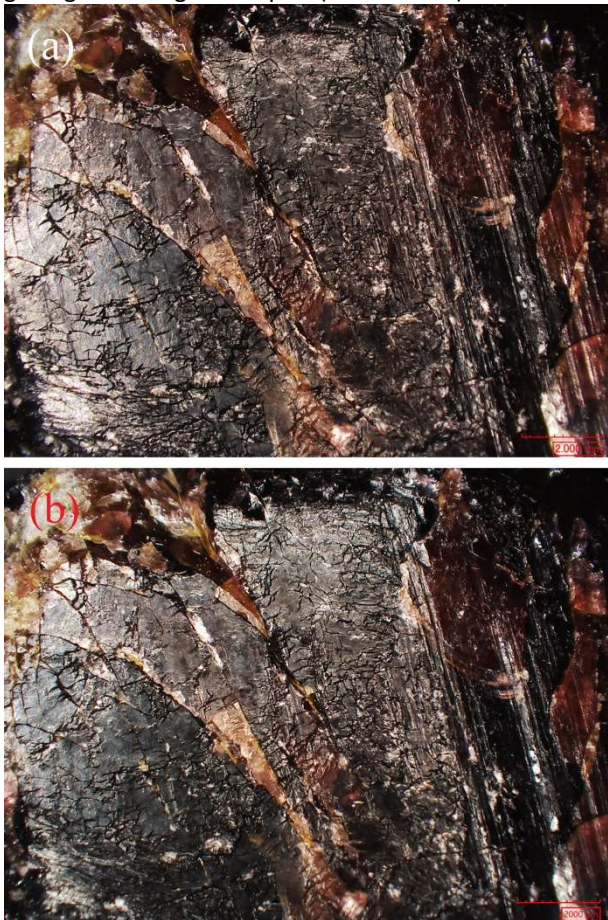


Fig. 3. Colour change of grossular. (a) pre UV exposure, (b) post UV exposure

#### 4 Conclusions

Color change following exposure to ultraviolet is a fairly common problem affecting a large number of minerals. Already in our experiment, 89% of the minerals subjected to aging showed more or less intense changes. In stones, the total chromatic alteration is due the sum of the colour change of the minerals constituting the paragenesis. This can lead to a real building pathology defined as "chromatic alteration" according to the ICOMOS glossary. The situation can often be the cause of disputes between customer and stone supplier. In the last few cases, the authors have witnessed whitening problems of dark stones rich in organic matter (e.g. quartzites, dark marbles) laid outdoors. Solar radiation oxidizes organic matter with a colour change which consists in a decrease of L\*parameter.

The interactions crystal-sunlight are well monitored and known in museum environmental, where expansive collection of minerals can be present. In this case, the materials are not directly exposed to sunlight but to artificial ones (Atalaya 2020).

If it is necessary to display the collection, the light lamps could be not be superior to 50 lux (corresponding to 50m cd), (Horak. J.M. 1994). The same authors referred that by reducing the intensity of illuminance, we can only minimize the damage, although note that light remains cumulative.

Regarding the acceptable limits of annual light exposure, highly sensitive minerals should have a maximum annual light exposure of  $1 \cdot 10^4$  Lux/h per year (Homem 2006).

Table 1: Provenience, classification, and crystallographic properties of the selected minerals.

Mineral	Chemical formula	Nickel-Strunz classification	Provenience	Crystal system
Tourmaline	$\text{NaMg}_3(\text{Al}, \text{Mg})_6\text{B}_3\text{Si}_6\text{O}_{27}(\text{OH})$	Cyclosilicate (8/E.19X)	Portugal	Trigonal
Orthoclase	$\text{KAlSi}_3\text{O}_8$	Tectosilicate (09.FA.30)	Portugal	Monoclinic
Diopside	$\text{MgCaSi}_2\text{O}_6$	Inosilicate (09.DA.15)	Portugal	Monoclinic
Gypsum	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Sulphate (07.CD.40)	Portugal	Monoclinic
Arsenopyrite	$(\text{FeAs})\text{S}$	Sulphide (02.EB.20)	Portugal	Monoclinic
Beryl	$\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$	Cyclosilicate (9.CJ.05)	Portugal	Hexagonal
Calcite	$\text{CaCO}_3$	Carbonate (5.AB.05)	Italy	Trigonal
Cinnabar	$\text{HgS}$	Sulphide (2.CD.15a)	Spain	Trigonal
Apatite	$\text{Ca}_5(\text{PO}_4)_3(\text{F}, \text{Cl}, \text{OH})$	Phosphate (08.BN.05)	Portugal	Hexagonal
Malachite	$\text{Cu}_2\text{CO}_3(\text{OH})_2$	Carbonate (5.BA.10)	Spain	Monoclinic

Talc	Mg <sub>3</sub> Si <sub>4</sub> O <sub>10</sub> (OH) <sub>2</sub>	Phyllosilicate (9.EC.05)	Italy	Monoclinic
Topaz	Al <sub>2</sub> SiO <sub>4</sub> (F,OH) <sub>2</sub>	Nesosilicate (9.AF.35)	Portugal	Orthorhombic
Hematite	Fe <sub>2</sub> O <sub>3</sub>	Oxide (4.CB.05)	Portugal	Trigonal
Galena	PbS	Sulphide (2.CD.10)	Italy	Cubic
Biotite	K(Mg, Fe) <sub>3</sub> AlSi <sub>3</sub> O <sub>10</sub> (F,OH) <sub>2</sub>	Phyllosilicate (9.EC.20 09)	Portugal	Monoclinic
Muscovite	KAl <sub>2</sub> (AlSi <sub>3</sub> O <sub>10</sub> )(F,OH) <sub>2</sub>	Phyllosilicate (9.EC.15)	Portugal	Monoclinic
Barite	BaSO <sub>4</sub>	Sulphate (7.AD.35)	Morocco	Orthorhombic
Fluorite	CaF <sub>2</sub>	Fluoride (3.AB.25)	Italy	Cubic
Quartz	SiO <sub>2</sub>	Tectosilicate (4.DA.05)	Morocco	Trigonal
Lazurite	Na <sub>3</sub> Ca(Al <sub>3</sub> Si <sub>3</sub> O <sub>12</sub> )S	Tectosilicate (9.FB.10)	Afghanistan	Cubic
Sodalite	Na <sub>8</sub> (Al <sub>6</sub> Si <sub>6</sub> O <sub>24</sub> )Cl <sub>2</sub>	Tectosilicate (09.FB.10)	Brazil	Cubic
Aquamarine beryl	Be <sub>3</sub> Al <sub>2</sub> Si <sub>6</sub> O <sub>18</sub>	Cyclosilicate (9.CJ.05)	Brazil	Hexagonal
Aragonite	CaCO <sub>3</sub>	Carbonate (5.AB.15)	Morocco	Orthorhombic
Optical calcite	CaCO <sub>3</sub>	Carbonate (5.AB.05)	China	Trigonal
Kyanite	Al <sub>2</sub> SiO <sub>5</sub>	Nesosilicate (9.AF.15)	Italy	Triclinic
Grossular	Ca <sub>3</sub> Al <sub>2</sub> (SiO <sub>4</sub> ) <sub>3</sub>	Nesosilicate (9.AD.25)	Italy	Cubic
Halite	NaCl	Chloride (3.AA.20)	Portugal	Cubic

Table II: Chemical composition of the selected minerals (wt. norm. percentage).

Mineral	Chemical composition
Tourmaline	0.49 % Na, 1.67 % Mg, 0.46 % Ca, 1.82 % Ti, 37.60 % Fe, 27.46 % Al, 30.49 % Si
Orthoclase	0.92 % Na, 11.71 % K, 0.13 % Ca, 18.55 % Al, 51.46 % Si, 46.91 % O
Diopside	0.16 % Na, 8.86 % Mg, 20.12 % Ca, 0.26 % Cr, 2.94 % Fe, 0.27 % Al, 22.18 % Si, 45.22 %
Gypsum	23.64 % Ca, 59.75 % O, 16,61 % S
Arsenopyrite	35.05 % Fe, 47.73 % As, 17.22 % S
Beryl	0.77 % Na, 0.09 % Ca, 8.49 % Al, 25.66 % Si, 65 % O
Calcite	4.79 % Ca, 34.14 % C, 61.07 % O
Cinnabar	0.67 % Na, 0.63 % K, 0.35 % Mg, 1.24 % Ca, 74.51 % Hg, 1.56 % Al, 3.99 % Si, 15.23 % S
Apatite	0.36 % Na, 24.98 % P, 7.77 % F, 66.66 % Ca, 0.23 % Cl
Malachite	0.13 % Mg, 0.98 % Ca, 2.49 % Fe, 44.74 % Cu, 0.90 % Al, 11.87% C, 0.61 % Si, 37.01 % O
Talc	0.14 % K, 15.20 % Mg, 0.90 % Fe, 0.25 % Al, 23.33 % Si, 59.88 % O
Topaz	4.98 % Al, 18.79 % C, 2.19 % Si, 41.80 % O, 32.24 % F
Hematite	1.85 % Rb, 54.74 % Fe, 0.13 % Ni, 0.38 % C, 42.89 % O
Galena	81.38 % Pb, 6.74 % O, 11.88 % S
Biotite	0.66 % Na, 8.13 % K, 0.11 % Mg, 1.91 % Fe, 20.21 % Al, 19.62 % Si, 49.01 % O, 0.34 % F
Muscovite	0.80 % Na, 9.43 % K, 1.67 % Fe, 20.66 % Al, 20.19 % Si, 47.25 % O
Barite	2.56 % Rb, 0.88 % Mg, 1.72 % Ca, 47.74 % Ba, 37 % O, 10.11 % S
Fluorite	41.93 % Ca, 9.05 % C, 49.02 % F
Quartz	0.78 % Na, 15.21 % Si, 84.01 % O
Lazurite	0.58 % Na, 0.69 % K, 1.47 % Mg, 27.96 % Ca, 0.84 % Fe, 1.21 % Al, 2.59 % Si, 63.55 % O, 1.11 % S
Sodalite	17.87 % Na, 13.87 % Al, 14.44 % Si, 46.85 % O, 6.97 % Cl
Aquamarine beryl	4.31 % Al, 13.62 % Si, 73.40 % O
Aragonite	30.14 % Ca, 0.58 % Sr, 11.81 % C, 57.48 % O
Optical calcite	12.39 % Ca, 22.76 % C, 64.85 % O

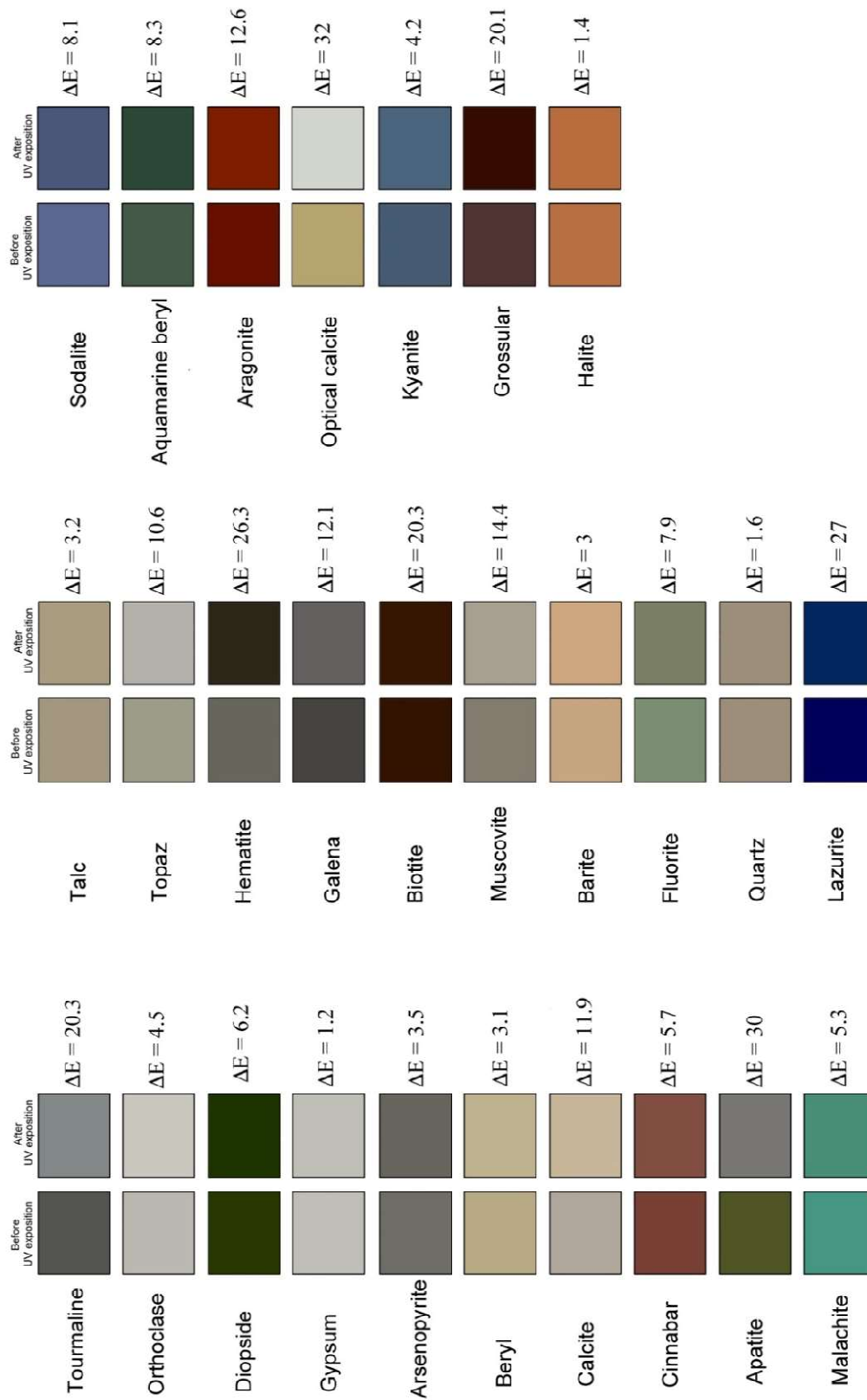


Fig. 4. Colour variation chart of the selected minerals before and after 48 h UV exposure.

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# ORNAMENTAL STONES VARIETY IN NAMIBE, ANGOLA: METAGREYWACKES AND MIGMATITES, GRANITES AND MARBLES

J.V. Lisboa<sup>1\*</sup>, L. Duarte<sup>2</sup>, J.F. Máximo<sup>2</sup>, A. Oliveira<sup>1</sup>, J.F. Suarez<sup>3</sup>, J.F. Rodrigues<sup>1</sup>, J.M. Plastov<sup>4</sup>, J.L. Lobón<sup>3</sup>

(5) Laboratório Nacional de Energia e Geologia (LNEG, Portugal), [\\*vitor.lisboa@lneg.pt](mailto:vitor.lisboa@lneg.pt)

(6) UTE-PLANAGEO,

(7) Instituto Geológico y Minero de España (IGME, Spain),

(8) Instituto Nacional de Geologia (IGEO, Angola)

**Summary:** *In the province of Namibe, the potentiality in ornamental stones is diversified and high. In addition to marble, new rocks from this region have found their way onto the market: metagrauwakes and migmatites, and light-toned granites, predominantly grayish with two micas, and biotitic. The potential resources are very high, but mostly not evaluated. The main constraints to the exploitation are related to the high intensity of fracturing, the occurrence of enclaves in the migmatites, and textural/chromatic heterogeneities in the marbles, besides its occurrence in very steep reliefs. The lack of roads and access to the resources is a major difficulty. The analyzes and technological tests carried out to characterize the samples did not show significant variations in the physical-mechanical behavior at a regional level in any of the studied lithologies, these being in general high quality ornamental stones for indoor or outdoor use, namely for pavement, cladding and lining.*

**Key words:** *gneiss-type stones, granite, marble, stone properties, ornamental potential*

## Introduction

The variety of rocks with ornamental potential in southwest Angola is very high, but still little known, despite the marketing efforts carried out by extractive sector companies operating in the region. In fact, besides the worldwide known black granites from Huíla province, other stones from this vast region are establishing themselves in the ornamental stones' world market. Examples of such stones are the metagreywackes and migmatites, a multitude of potential granitic types, and also marbles, besides other rocks. Regarding the ornamental aptitude, except for the stones being explored, the characteristics of all these potential ornamental stones are practically unknown.

This paper's goal is to make a preliminary presentation of the geological, physical-mechanical characteristics, and potential of some of these lithotypes, from the ornamental stone viewpoint: metagreywacke and migmatite, marble and leucogranite.

The present study follows on from work recently carried out for the Chart of Industrial Rocks and Minerals of Angola (CARMINA), which is a subproject of the National Geology Plan (PLANAGEO) for the Geological Survey of Angola (IGEO).

## Geological setting

During PLANAGEO's geological mapping work and aiming at structuring the Unified Legend of the Geological Chart of Angola, in addition to the new geological units purposed, others were redefined, encompassing the most recent knowledge. This new nomenclature is adopted in the present work.

### Metagreywackes and migmatites

Metagraywackes and migmatites are dark rocks that show a relatively similar color and general textural appearance. The metagreywackes are part of the Namibe Group ( $\approx 1.86 - 1.81$  Ga) represented only in the province of Namibe and extending its southernmost outcrop to the Cunene river (Silva et al., 2021a). This is an essentially siliciclastic sedimentary protolith unit, whose dominant lithologies are metagreywacke rocks with intercalations of chloritic phyllites. In these siliciclastic lithologies of low metamorphic grade, thin beds of microconglomerates interspersed in the metagreywackes are observed. These are often feldspathic and, to a lesser extent, quartzwackes. Some of these rocks, when purer, can approach to quartzites, although true quartzites are not common.

Migmatites occur in the study area in several limited zones and are part of the Epupa-Namibe Metamorphic Complex ( $\approx 1.83-1.77$  Ga) (Silva et al., 2021a). These

rocks range from paraderived metatexites to diatexites with metasediment restites. They are considered paraderived protoliths, given the abundance of metasedimentary rock relics not completely digested by anathetic melting. The field observations, together with the radiometric ages obtained so far in the PLANAGEO work, allows to consider the hypothesis that this migmatitic set derives from the melting of the Namibe Group (Silva et al., 2021b).

### Marbles

In the metagreywackes and phyllites of the Namibe Group, intercalations of very fine-grained black amphibolites (thickness can vary from a few meters to a few tens of meters) and marbles occur. These are predominantly calcitic and dolomitic marbles, in some cases granatiferous, locally quite thick, reaching several hundred meters and, one kilometer thick in the Macota mountain range (Silva et al., 2021b). These carbonated lithologies are easily visible in the landscape, since they form imposing residual reliefs. To the NE, marbles from the Namibe Group were also mapped, and integrated into a unit called “Calcitic and dolomitic marbles” (Gutiérrez-Medina, 2021), which outcrops in Serra da Lua and in small isolated massifs nearby.

### Granites

The granites with ornamental potential in SW Angola include varied lithotypes integrating geological units of Paleoproterozoic age, many of them already defined in the existing bibliography. The granites discussed herein, are examples of this ornamental potential: the leucocratic granites and leucogranites associated with the Epupa-Namibe Metamorphic Complex (Silva et al., 2021b) in the Macota region, and, to the west, in the Caraculo area, granites from the Intermediate to Acid Magmatism Suite (G3 Suite), also associated with the Epupa-Namibe event (Escuder-Viruet, 2021). In this suite which is made up of granites, two main lithofacies were defined: granites and granodiorites with biotite, and porphyroid granites and granodiorites with biotite. G3 Suite corresponds to the “Granitos de Caraculo” by Carvalho (1982, 1983) and by Carvalho et al. (2000), or the “Late to post-tectonic granite of the Serra de Gandarengos type” by Pereira et al. (2013) The G3 suite consists of granitic massifs that present a weak or microscopically unidentifiable deformation, and were implanted in epizonal conditions, developing halos of contact metamorphism in the host materials when there is a marked contrast of metamorphism T.

### Methodology

During the implementation of the CARMINA subproject, diverse outcropping lithologies in predefined areas of the Huila and Namibe provinces were geologically surveyed and sampled to assess its ornamental potential. The preliminary interpretation of collected data, followed the basic methodological procedures for the realization of the CARMINA sheets, which includes field and laboratory work.

The field analysis to identify potential areas for exploration of ornamental stone, first comprised the mapping distinction and characterization of occurring lithotypes, fracturing survey and sampling in existing quarries, active and inactive, and significant outcrops. The characterization of lithotypes was primarily made according textural, mineralogical, and alteration criteria, and petrographic analysis in laboratory. Lithological description encompassed the stone’s macroscopic, microscopic-petrographical study and identification of flaws susceptible to decrease the ornamental potential (such as, xenoliths, filonean intrusions, grain gradations and crystal size heterogeneity, color heterogeneity particularly in marbles, sulphides in granitoids).

The petrographic analysis and tests to determine the mechanical properties of the selected rocks were performed in the laboratory. These tests comprised water absorption, apparent density, open porosity, compressive strenght and bending strength.

The physical-mechanical tests considered most relevant for the technological characterization of ornamental rock were carried out at the Geoscientific Laboratory of IGEO, in Lubango, the testing procedures following the European Standards (EN). The tests included the determination of apparent density, open porosity, water absorption at atmospheric pressure, compressive and flexural strength in selected samples of the considered lithologies both in quarries and outcrops of the study area.

### Results and discussion

#### Metagraywacke and migmatite

The metagreywackes of the subunit “Chlorite phyllites, metagreywackes and rare quartzites” of the Namibe group (Lisboa and Duarte, 2021) and the migmatites of the Epupa-Namibe Metamorphic Complex are currently very ssought after by the extractive industry in the province of Namibe.

This lithology occupies a vast area, but only the massive rock bodies have the potential to be exploited. The lithofacies with ornamental potential can be differentiated from the geological unit, being more

compact, and less fissile and clayey (Fig. 1a). It is a dark gray to black metasedimentary rock, quite heterogeneously fine grained, with numerous quartz and carbonate exudation veins (Fig. 1b); the foliation is intense, with strike trending from NE to NW, generally sub-vertical or with a high dip. The observed mineralogical paragenesis is essentially quartz, feldspars, sometimes with amphiboles and sulphides. Thus, despite this subunit being very deformed with intense foliation, areas where it is possible to obtain quality blocks (these always require treatment with resins) exist and can be found.



Fig. 1. Metagreywacke quarry in the Virei municipality. (a). Note the thin superficial alteration layer, and stone without significant fracturing.; (b). large block of metagreywacke, DFG quarry.

In migmatites, the migmatization process originates a very heterogeneous rock, which is generally characterized by having two or more petrographically distinct parts. The part neofomed during the migmatization process (neosome), typically divides into a portion characterized by having pale quartz-feldspathic or feldspathic colors of medium to coarse grain (leucosome), and portions of dark color (dark gray to black), which are enriched in ferromagnesian minerals (melanosome). The melanosome represents the residual (restitic) fraction, which is usually finer in

granularity. The migmatites are gray in color, with whitish quartz-feldspathic dilatational (leucosomatic) structures/channels, which develop flow folds, sometimes ptigmatic, characteristic of migmatitic domains. Fracturing, although generally intense, in several observed outcrops allows, as it is spaced, exploration of blocks.

Metagreywackes and migmatites occur in domes of variable dimensions, which emerge from the surface in rounded shapes and often, although not outcropping, are found very close to the surface at economic exploration depths, so that prospecting methods, namely seismic or georadar, can provide good results. These domes on the surface, characteristically present a very high foliation, appearing (particularly concerning the metagreywackes), that the rock is intensely fractured and altered; this aspect is superficial, as below the alteration layer, which is very thin, the rock generally appears unaltered and compact (Fig. 1a). In migmatites, in addition to high fracturing, the frequent presence of enclaves is a conditioning factor to be considered during the prospecting and research phase. In sites with ornamental stone potential, spacing between fractures allows the extraction of large blocks (Figure 1b), but the high foliation at the surface does not allow the evaluation of spacing between joint sets.

A feature valued by the extractive industry in metagreywackes and migmatites, is the yellowish tones in the veins, which are due to the oxidation of sulphides and can be found close to the surface, because in depth, this oxidation disappears. However, the presence of these same sulfides (pyrite) or Fe oxides disseminated in these rocks, in fine grains or aggregates, also constitutes a flaw, since their presence, occasionally very frequent, causes brownish stains due to oxidation, on freshly cut blocks. Also, the greater the contrast between the leucosome/melanosome pair, and the respective rhythmic succession of the different parts that make up the migmatite, the greater the value of the blocks produced. Also, the existence of flow structures that produce convoluted (ptigmatic) folding and attribute “visual effects” to the rock, is a factor that value the blocks, as long as they do not increase their degree of fracturing.

There are some quarries exploiting these lithotypes close to the Namibe-Virei road and, therefore, with access to the port of Namibe.

#### Petrographic analysis

The microscopic study reveals a metamorphic rock nature, granoblastic-banded texture, inequigranular, with the following mineralogical composition: quartz (90-95%) and biotite (5%), which is replaced by chlorite, as main minerals, and accessory opaque minerals (<1%).

Quartz crystals form a banded aggregate with grains <200 microns. Among the quartz grains appear biotite crystals with greenish colors (chloritized) forming bands parallel to each other (biotite < 100 microns). Fractures occur perpendicular to the compositional bands, filled with quartz and biotite (Fig. 2). The deformation is manifested by the degree of crushing of the quartz and biotite crystals.

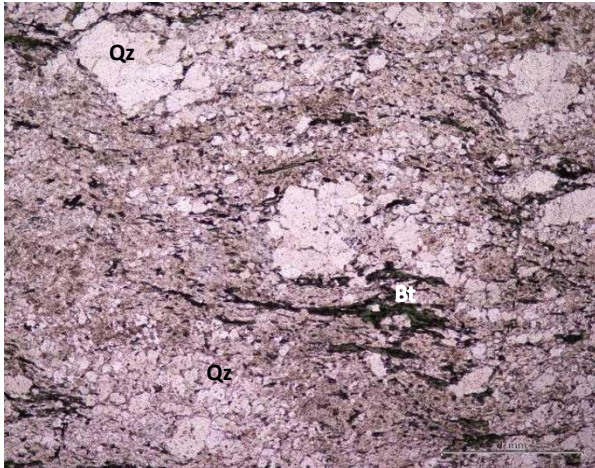


Fig. 2. Microphotography under the petrographic microscope of metagreywacke sample (// nicols) from an outcrop in the Virei area. Scale bar: (bottom right) 1 mm. Symbology: Qz – Quartz, Bt – Biotite/Chlorite

#### Physical-mechanical properties

The metagreywackes and migmatites tested have very similar technological properties on average, with values that differ little, indicating a slightly more favorable behavior of the greywackes (Table I). However, given the reduced number of samples tested, this behavior cannot be conclusive.

Density is compatible with gneiss-type rocks, with all samples showing values higher than the minimum established by ASTM and CEN/TR 17024 standards (Molina, 2017).

Regarding open porosity and water absorption, the stones present low and homogeneous values, which are significantly below the maximum values accepted by the specifications for both properties (Molina, 2017); only one sample slightly exceeds the minimum values.

Compressive strength is mostly high (only one sample of greywacke shows a minimum outlier) and compatible with the density and compaction of these stones. Flexural strength is also high, far exceeding the specified minimum (Molina, 2017).

The few outliers verified in some properties, namely those of mechanical resistance, are related to the banded texture (different mineralogy) of these stones.

Both lithologies reveal a technological behavior that can be considered similar and that allows their outdoors and indoors use as ornamental stone, after adequate treatment to avoid cracking and oxidation when sulfides or Fe oxides are disseminated in the stone.

Table I. Results of technological tests carried out on: A. metagreywacke samples (n=6); B. Migmatite samples (n=3)

#### A. Metagreywacke

Test	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2678	37	2740	2640
Open porosity (%)	0,6	0,1	0,7	0,5
Water absorption (%)	0,3	0,1	0,3	0,2
Comp. strenght (MPa)	138	56	236	82
Flex. strenght (Mpa)	27,0	12,0	47,4	19,0

#### B. Migmatite

Test	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2630	40	2670	2590
Open porosity (%)	0,9	0,4	1,4	0,6
Water absorption (%)	0,4	0,1	0,5	0,3
Comp. strenght (MPa)	172	43	220	136
Flex. strenght (Mpa)	17,3	5,5	22,8	11,8

#### Marble

Unlike the metagreywackes and migmatites that only recently triggered the interest of the extractive industry, for a long time the marbles of the Namibe Group have been sought after as ornamental stone. These rocks always outcrop in very steep slope reliefs (Fig. 3) reaching vast areas such as in the Macota, Lua, and Cuanhangue-Coatchila mountains, besides other sites. At the time of the field work, there were active quarries in the Lua and Cuanhangue-Coatchila mountains, and a quarry in intermittent activity in the Macota mountain.

The marbles show a predominant white color, with frequent gray intercalations, but also layers of varied colors, from blue to green, and pink. The granularity varies from fine to coarse, predominantly medium to coarse, with well-developed crystals. The foliation they present is mostly very high, with predominantly NW-SE orientation in the Macota region and high dips.

The layers of calcitic and dolomitic marbles thickness range from centimeter to decimeters up to 3 m thick and over, with intercalations of calc-silicate schist, hornfels, quartzite, amphibolite and gneiss. These intercalated beds produce an increase in hardness that makes extraction and subsequent processing difficult. High fracturing, combined with poor chromatic and textural uniformity (e.g. granularity) of layers, creates another

constraint to exploration, making it difficult to plan and obtain a stable and homogeneous production. The presence of quartz inlays, of different sizes and shapes, is frequent in the marbles of Serra da Lua and Macota, which, when the rock is white, look like gray spots. These occurrences, in general, do not limit the extractive potential of the rock.

Although fracturing is generally high, in certain sites it does not prevent the extraction of blocks with commercial dimensions, and homogeneous areas in terms of grain and color, also occur.



Fig. 3. Front of an old marble quarry on a steep slope, SE of Macota mountain. White marble with sparse light gray intercalations.

There is no mining information available for the marble occurring areas, but in a quarry in Serra da Lua, marbles evidencing in general, high fracturing, provided an average yield of 5%, and 80% in the best sites, however to reach these, others with 0% yield had to be removed.

#### Petrographic analysis

The marble thin sections observed under a petrographic microscope are mostly made up of calcite and rare dolomite (Fig. 4), although pyroxene is also observed (not in the sample shown). It has a granoblastic texture with a heteroblastic trend, with medium grain (1-5 mm) and fine grain (<1 mm) being these grains mostly recrystallized calcite and rare dolomite. The larger grains mostly have sutured edges due to recrystallization, sometimes straight to curved. The fine-grained generation has mostly curved to straight edges. Ductile deformation is evident in some calcite crystals with undulating extinction and formation of subgrains; also visible fractures, mainly intragranular with calcite recrystallization in the filling, and intergranular fracturing, sometimes open. This fracturing can decrease the mechanical strength of the rock. It is therefore a marble that was subject to recrystallization and mainly plastic deformation.

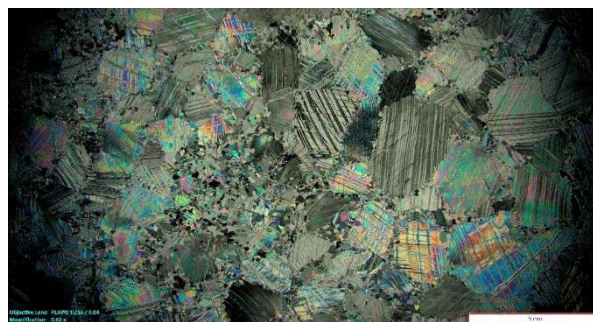


Fig. 4. Microphotograph under the petrographic microscope (NX) of a marble sample from Macota mountain. Obj: PLAPO 1.25x/0.04, Ampl. 0.62x. Scale: 5mm white bar.

#### Physical-mechanical properties

The sampled marbles did not reveal significant differences in physical-mechanical behavior in the different areas tested, showing regionally, a behavior considered homogeneous. Regarding density, the values exceed the minimum required, although for more demanding specifications, the values obtained can sometimes be considered slightly low for ornamental marble (Table II). The open porosity always presents very low values and the water absorption is also low, although it slightly exceeds the minimum recommended (0.2%) by the ASTM standard in 2 samples (Molina, 2017).

The compressive strength and flexural strength values show some dispersion (Table II). Regarding compressive strength, the values can be considered mostly medium, compatible with the values for quality ornamental marble (Moura, 2006). Flexural strength values, in general, significantly exceed the minimum indicated in the ASTM C503 standard (Molina, 2017). Particularly in the area of Monte Cuanhangue-Coatchila (Bibala municipality), where ornamental marble is exploited, the values of the physical-mechanical properties suggest a more consistent quality of the rock.

Table II. Results of technological tests carried out on marble samples (n=16)

Test	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2681	41	2760	2610
Open porosity (%)	0,5	0,5	2,1	0,2
Water absorption (%)	0,2	0,2	0,9	0,1
Comp. strenght (MPa)	89	54	238	36
Flex. strenght (Mpa)	13,3	6,6	22,1	4,0

#### Granite

Until now, the leucocratic granites occurring in the municipality of Virei (Cahinde-Virei road surrounding

area and south) have not interested the extractive industry for ornamental purposes. On the contrary, granites with similar characteristics, occurring in the Caraculo and Naobva areas, municipality of Bibala, in the region surrounding the road to Namibe, were at the time of the field work, in the initial phase of exploration in two quarries.

In the municipality of Virei, the leucocratic granite outcrops of the Epupa-Namibe Metamorphic Complex are hundreds of meters in size, and although they usually present pronounced fracturing, several domains were recognized, locally with spacing between fractures greater than 5m. The granites color is whitish, cream to light gray (which differentiates them from the gray lithotypes), fine to medium grained, although coarse grained facies occasionally occur. Macroscopically, they present a homogeneous textural appearance, equigranular and isotropic, with rare biotitic-amphibolic enclaves (few millimeters to centimeter extent, rarely decimeters), widely spaced. They correspond to “two micas” leucogranites with mineralogy consisting of feldspar, quartz, biotite and muscovite. The presence of Fe oxides was observed at several points. In general, the rock is unaltered, with virtually no alteration layer.

In the municipality of Bibala, the lithotype described as Granites and granodiorites with biotite (not porphyroid) has affinity with the above described leucocratic granites. These granites, also outcropping in domes, are being sought after for ornamental rock, due to the possibility of obtaining blocks of commercial size (Fig. 5). They are medium grained, less often fine-grained, with few scattered feldspar phenocrysts (0.5 to 1.5 cm length). They present a dominant light gray color, and may have greenish tones due to chloritization, and yellowish to brownish tones due to alteration of the biotite. This facies differs from the porphyroid facies due to its lighter color, lower biotite content and greater abundance of quartz.

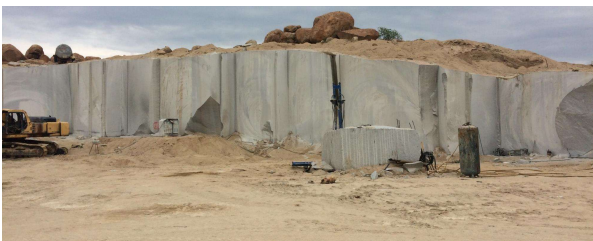


Fig. 5. Quarry in Bibala municipality (Caraculo area) exploiting a light gray, medium-grained granite (Granites and granodiorites with biotite lithotype) in a dome with widely spaced joints.

Spacing between fractures is large, in some cases higher than 15 m. Significant flaws in the stone, which can limit its exploitation and commercialization, were not

observed in the studied area, giving good perspectives for the extractive industry settling.

#### Petrographic analysis

The microscopic analysis of granite thin sections (Granites and granodiorites with biotite lithotype) shows a rock with a holocrystalline texture, inequigranular, medium-grained, made up of an allotriomorphic quartz crystals mosaic (0.1 to 5mm) showing deformation structures (associated with sutured edges and undulating extinction), potassium feldspar with characteristic microcline twinning, and hypidiomorphic plagioclase crystals (< 5mm), completely altered to sericite. The biotite crystals are partially chloritized (Fig. 6). Mineralogical composition: quartz (45%), plagioclase (35%), microcline (10%) and biotite (10%), as main minerals, accessory opaque minerals (1-2%), and as secondary minerals, chlorite and sericite. No major deformation is observed.



Fig. 6. Microphotography under the petrographic microscope of a granite (+ nicols) in the Caraculo area. Obj: U MPLFL 5x/0.15, Ampl. 5x. Scale: 1 mm (bottom right). Symbology: Qz – quartz, Pl – plagioclase, Mc – microcline

#### Physical-mechanical properties

The tested granites (Table III) have medium to high density, characteristic of this lithology, being the values obtained higher than the minimum value recommended for quality granite (>2560 kg/m<sup>3</sup>), according to the specifications in the ASTM and CEN/TR 17024 standards (Molina, 2017). The open porosity values are also within the values admitted by the standards; the same for most of the water absorption values, although these tend to be slightly high. The compressive strength is high and so is the bending strength, reflecting the high quartz component in these granites; however, a limited number of bending strength tests were carried out, so

the results relating to this property are not very conclusive.

There are no significant differences between the physical-mechanical behavior of the granites sampled in the municipalities of Virei and Bibala.

Table III. Results of technological tests carried out on: A. Leucocratic granite samples (n=3); B. Granite samples from Caraculo area (Granites and granodiorites with biotite unit) (n=6)

A. Leucocratic granite samples (Virei municipality area)				
Test	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2650	40	2690	2610
Open porosity (%)	0,8	0,5	1,3	0,5
Water absorption (%)	0,3	0,2	0,5	0,2
Comp. strenght (MPa)	221	31	244	186
Flex. strenght (Mpa)	20,6	9,5	31,4	14
B. Granite with biotite samples (Caraculo area, Bibala municipality)				
Test	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2593	46	2650	2530
Open porosity (%)	1,0	0,4	1,6	0,7
Water absorption (%)	0,4	0,1	0,6	0,3
Comp. strenght (MPa)	207	38	245	156
Flex. strenght (Mpa)	18,6	0,6	19,0	18,1

## Conclusions

The exploration of ornamental marbles in the province of Namibe has been practiced for a long time, with interruptions, but only more recently has the exploration of other rocks such as granites and, above all, metagreywackes and migmatites started to attract the attention of the ornamental stone market.

The metagreywackes and migmatites, are rocks that despite their different geological nature, exhibit a relatively similar appearance at the chromatic and textural level, which in commercial terms are included in the granites. These rocks are currently sought after by the ornamental stone market, due to their originality and technological characteristics, and due to the outcrops' location near the road that gives access to the port of Namibe, facilitating the possibility of export. There are several active quarries, mostly close to the Virei-Namibe road. Both lithologies outcrop in large areas in Namibe, especially the metagreywackes, but their ornamental potential is limited to compact bodies, less fissile and clayey; migmatites, are mainly conditioned to the non-occurrence of excessive enclaves, which is a frequent flaw; both lithologies have

the potential strongly conditioned to the occurrence of slightly fractured bodies, as intense fracturing is frequent. The bodies with the greatest potential generally occur in domes. The analysis and physical-mechanical tests carried out revealed that both rocks have essentially quartzose mineralogical composition, and very similar technological properties, slightly better in metagreywackes.

The calcitic and dolomitic marbles, display varied colors although mostly white, with frequent intercalations of different shades, predominantly gray. granularity is medium in most outcrops. Foliation is generally very high and frequent intercalations of other metasedimentary and igneous lithologies occur. The frequent intense fracturing, associated to the outcrop's steep slopes, and the heterogeneity of the stone expressed in varied intercalations, variations in color and granularity, are the constraints to the exploitation of marble. The physical-mechanical characterization revealed homogeneous technological behaviour of the samples among the studied areas.

The granites mentioned in this work, occurring in the municipalities of Virei and Bibala, correspond to light gray facies, with a predominance of whitish to cream colors in the municipality of Virei, and light gray, sometimes with pink, greenish and yellowish tones in the municipality of Bibala. The granularity is predominantly medium in both areas and mineralogically they differ mainly because the leucogranite outcropping in the municipality of Virei is a two-micas granite and the lithotype Granites and granodiorites with biotite outcropping in the Bibala region is biotitic with accessory muscovite. The two lithotypes outcrop in domes and even though fracturing is often high, it locally allows the extraction of large blocks for ornamental stone, especially in biotitic granites where the spacing between fractures is often high. Heterogeneities and flaws in these stones are scarce.

In terms of technological properties, there are no significant differences between two-micas granites and biotitic granites. Some high compressive and bending strength values reflect the high quartz mineralogical content.

The results of the analyses and tests reveal that all the studied lithologies are resources for quality ornamental stone, which can be used indoors and outdoors, namely for application in pavement, cladding and lining. The main constraints on the ornamental potential of these stones are mainly correlated with the possibility of textural heterogeneity and the intense fracturing.

A common factor to all mentioned lithologies, of which the metagreywackes and migmatites stand out for its

greater originality, is their enormous outcropping areas, enclosing a potential that is still virtually unknown, but which is very high. Particularly, the marbles field survey was constrained, not only by the outcropping pattern, but by the lack of roads that gave access to the outcrops.

The lack of roads is perhaps the biggest constraint to the development of the ornamental stone industry in this rather unpopulated region, since the potential is high.

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# ORNAMENTAL STONES WITHIN THE INSPIRE DIRECTIVE INFRASTRUCTURE: THE NEED FOR CHANGE

J. M. F. Carvalho<sup>1\*</sup>, J. V. Lisboa<sup>1</sup>, A. Pereira<sup>1</sup>

(1) LNEG - Laboratório Nacional de Energia e Geologia, IP, \*[jorge.carvalho@lneg.pt](mailto:jorge.carvalho@lneg.pt)

**Summary:** *The INSPIRE data model for mineral occurrences is still based on concepts and vocabularies that do not entirely respond to current nomenclature and categorization of ornamental stones, despite harmonization efforts made by several projects funded by the European Commission. Proposals for change the INSPIRE structure and mineral resources vocabularies concerning ornamental stones are herein presented. Regarding the INSPIRE commodity type code-list, we propose to replace the label dimension stone by ornamental stone, change its definition, and reformulate seven types of ornamental stones with new labels and definitions. Ornamental Stone is proposed as the mineral deposit type label and a new definition is presented; about the end use potential type value, the distinction between construction aggregates and ornamental stones is made; two new code-lists are proposed, one for the stone's colour and another for the stone's commercial names.*

**Key words:** *Ornamental Stones, Databases, INSPIRE Directive, Mineral Resources*

## INTRODUCTION

The INSPIRE Directive (<https://eur-lex.europa.eu/eli/dir/2007/2/2019-06-26>) aims the establishment of an infrastructure for spatial information in Europe that will enable the sharing of environmental spatial information among public sector organizations, facilitating public access to spatial information across Europe and assisting in policymaking across boundaries. It entered into force in May 2007. In practice, the availability of spatial data by public sector organizations must be INSPIRE compliant. This is the case of national geological surveys when disclosing geological maps, mineral resources databases, etc.

Several projects funded by the European Commission intended for the collection and structuring of Pan-European data on mineral resources have been contributing to these harmonization efforts in accordance with the INSPIRE Directive. Minerals4EU (<https://cordis.europa.eu/project/id/608921>), was one of the first European projects dedicated to this matter, but others followed, such as the MICA project (<https://cordis.europa.eu/project/id/689648>) and, more recently, the MINTELL project integrated in the GeoEra funded programme (<https://geoera.eu/projects/mintell4eu7/>). Also integrating the GeoEra programme, the Eurolithos project (<https://geoera.eu/projects/eurolithos1/>) was targeted to the inventory and INSPIRE compliant harmonization of European ornamental stones data.

The work carried out in the Eurolithos project clearly showed the existence of errors and incongruities in the INSPIRE nomenclature and categorization of ornamental stones. Therefore, from the point of view of non-IT specialists, this work presents a proposal to change the INSPIRE structure and vocabularies of mineral resources concerning ornamental stones.

## WHAT ARE ORNAMENTAL STONES?

First, there is a question of name: dimension stones, building stones, natural stones, decorative stones, and ornamental stones. To which mineral raw material do these names refer? Moreover, what do some authors mean when they combine some of these names, such as “building and ornamental stones” (Barale et al., 2020), “dimension and ornamental stones” (Scrivano & Gaggero, 2020), “natural stone for ornamental use” (Careddu et al., 2020), “natural and building stones” (Tokmak & Dal, 2020), and “building and dimension stones” (Bloise et al., 2021)?

As far as we understand, all these names refer to the same mineral raw material: naturally occurring rocks sufficiently consolidated to enable them to be cut or shaped into blocks or slabs for structural and decorative purposes in the construction of buildings and other structures.

This concept includes magmatic, sedimentary, and metamorphic rocks processed into various shapes, from the small cubes used for sidewalks and thin slate

slabs for roofing, to the large slabs for cladding and flooring, statuary, tombstones, etc.

The decorative purpose is at the heart of this definition, which is why we prefer the more comprehensive name Ornamental Stones, even if they are named Dimension Stones by ASTM International, and Natural Stones by CEN (the European Committee for Standardization), when considered from a commercial point of view as a construction material that must be governed by standards.

If in ancient times these stones were essentially used for their structuring capacity, from the first houses and fortifications made of bare stone to palaces and other more recent monuments, nowadays they have been replaced by iron and bricks. Today, these stones are mainly used for their ornamental capacity and status of having something unique, made of a natural product almost not processed, the structuring capabilities being only used occasionally or secondarily.

#### HOW ARE ORNAMENTAL STONES ADDRESSED BY INSPIRE?

Ornamental stones are a mineral raw material and for that reason are treated in INSPIRE under the Mineral Resources theme. According to the INSPIRE Thematic Working Group Mineral Resources (2013), the core data model for Mineral Resources is based on the EarthResourceML (<http://earthresourceml.org/>), which is a data model from the IUGS Commission for the Management and Application of Geoscience Information describing the geological features of mineral occurrences, mines and mining activity. EarthResourceML vocabularies are the ones adopted by INSPIRE Mineral Resources theme, which is in the process of adapting to changes arising from the Minerals4EU project (available at: <https://inspire.ec.europa.eu/file/1618/download?token=KIMfj4uo>), as well as from the MINTELL project (available at <https://geusgitlab.geus.dk/m4eu/2020-m4eu>).

Regarding ornamental stones, code-lists and vocabularies from EarthResourceML obey to the concepts born in United States of America almost one hundred years ago with Bowles & Coons (1933). They used the term dimension stone as a *general term covering stones sold in the form of blocks which are cut to definite shapes and often to specified sizes*, to distinguish them from those used as aggregates in building construction. Later, Currier (1960) and Barton (1968) improved their concepts, defining also eight types of dimension stones (basalt, granite, greenstone, limestone, marble, miscellaneous, sandstone, and slate) and supporting the design of the ASTM C 119 -

Standard Terminology Relating to Dimension Stone. With minor changes, they last until today in EarthResourceML, INSPIRE, Minerals4EU, and MINTELL.

Within the INSPIRE/Minerals4EU/MINTELL code-lists and vocabulaires, which from now on we will refer solely as INSPIRE code-lists, the ones more relevant to ornamental stones are part of the Mineral Resources data model, namely:

- Commodity type, where there is a code for Dimension Stone, which is defined from Wikipedia as a *natural stone or rock that has been selected and fabricated (i.e., trimmed, cut, drilled, ground, or other) to specific sizes or shapes*. The Commodity Type code-list also includes codes and definitions for the eight types of dimension stones (basalt, granite, etc.).
- Deposit Group type, where the more appropriate code for dimension stones is Bulk Rock Material.
- Deposit Type with a specific code for dimension stone
- Enduse Potential type includes three relevant codes:
  - Building and Dimension Stones, which are defined as *Natural Stone used in a block, flag or slate form for construction or decorative purposes, or artifacts, e.g., millstones*.
  - The former is a subsidiary of the Construction code: *Material used in the construction industry. Includes aggregate, dimension & ornamental stones (granite, gabbro, travertine, etc.), gypsum, anhydrite, cement limestone, limestone for lime, marble, sand and gravel*.
  - A third last code is a subsidiary of the Non-Metallic Minerals code-list: it is the Building Raw Material defined as *mineral occurrences including Aggregate; Dimension & ornamental stones (granite, gabbro, travertine, etc.); Gypsum, anhydrite; Cement limestone; Limestone for lime; Marble*.

Clearly there are inconsistencies, as is the case of the same rock name being used to identify different commodities or the use of the composite name "Ornamental and Dimensional Stones". Moreover, the definitions used for the eight different types of dimension stones also have some problems:

- They are too long and full of small not-relevant particularities. For instance, the definition for granites is: *Commercial granites include all feldspathic crystalline rocks of mainly interlocking texture and with individual mineral grains that are visible to the naked eye. This category includes such*

rock types as anorthosite, gneiss, granite, granodiorite, monzonite, syenite, and all other intermediate igneous and coarse-grained metamorphic rock types. The primary colors of commercial granites are white, gray, pink, and red; green and brown are secondary colors. Although black granites are also included in this category and range in color from dark gray to black, they are not true granites mineralogically but rather mafic rocks, such as diabase, diorites, gabbro, and similar rocks.

- Are not consistent with one another. Comparing, for instance, the above granite definition and the slate definition (*Commercial slate is a microgranular metamorphic rock formed by the recrystallization of clay sediments, such as claystone, shale, or siltstone. Characterized by excellent parallel cleavage, slates may be easily split into relatively thin slabs*), nothing is said about the color of slates, and nothing as well is said about the origin of granite-type stones.
- Make use of very specific terms hardly known by geologists and other professionals. As examples, the definition of basalt includes the term “traprock” and the definition of sandstone includes “flagstone”.
- Inconsistently, integrate definitions for other types of rocks. As an example, the limestone definition (*commercial limestones are rocks of sedimentary origin that primarily are composed of calcium carbonate with or without magnesium. Included in this category are limestone, dolomite, dolomitic limestone, and travertine, which is a calcitic rock that is precipitated from hot springs*) includes a definition for travertine, but not for dolomite or dolomitic limestone.

## PROPOSALS FOR CHANGING INSPIRE

### The commodity type values

Besides the inconsistencies presented in the previous chapter, a major question must be raised: if there are several European standards dealing with ornamental stones and calling them Natural Stones, why are they named as dimension stones in the more recent INSPIRE Mineral Resources theme?

We do not have an answer for it, therefore our logical reasoning would be to propose a change of the INSPIRE commodity name to Natural Stone. However, bearing in mind that we are not dealing with commercial product standards, but with the categorization of mineral resources for the purposes of standardization and interoperability of spatial data,

we prefer to use the name Ornamental Stones, for the reasons we wrote earlier.

Thus, we propose that the label dimension stone be replaced by ornamental stone in the commodity type code-list of INSPIRE. We also propose to change its definition to: naturally occurring rock sufficiently consolidated to enable it to be cut or shaped into blocks or slabs for structural and decorative purposes in the construction of buildings and other structures.

Still in the commodity type code-list, we propose that INSPIRE should only consider seven different kinds of ornamental stones with the following labels and definitions:

**Basalt:** Commercial basalt includes extrusive igneous rocks that are too fine grained to be included in the granite type of stones (e.g., andesite, basalt, and dacite).

**Granite:** Commercial granites include all feldspathic crystalline rocks of mainly interlocking texture and with individual mineral grains that are visible to the naked eye (e.g., anorthosite, gabbro, gneiss, granite, monzonite, and syenite).

**Limestone:** Commercial limestones are rocks of sedimentary origin that primarily are composed of calcium carbonate with or without magnesium (e.g., limestone, marly limestone, dolomitic limestone, dolostone, and travertine).

**Marble:** Marble is a metamorphic rock containing more than 50% of carbonate minerals, resulting from metamorphic recrystallization of limestones or dolostones.

**Miscellaneous Ornamental Stones:** Commercial stone types that do not easily fall into the other categories, such as amphibolite, chert, serpentinite, and steatite.

**Sandstone:** Commercial sandstone is a lithified sediment chiefly comprising quartz or quartz and feldspar clasts cemented by calcite, clay, iron oxides, or silica (e.g., sandstone, quartzite, greywacke, and breccia).

**Slate:** Commercial slate is a microgranular metamorphic rock that may be easily split into relatively thin slabs.

### The Mineral Deposit Type value

The mineral deposit type values indicate the style of mineral occurrence or deposit. We propose that the mineral deposit type label should be Ornamental Stone with the following definition: Ornamental stone deposits are igneous, sedimentary, or metamorphic rocks that are sufficiently consolidated to enable them to be cut or shaped into blocks or slabs.

### The End Use Potential type value

About the end use potential, various interdependent changes are proposed.

The label Construction should have its definition changed to: Material used in the construction industry. Includes building stones, gypsum, anhydrite, limestone and marble for cement or lime, sand, and gravel.

Under the Construction umbrella, a new code label building stones should replace aggregates and building and dimension stone labels. Its definition should be: Building stones are consolidated or unconsolidated rocks directly used in building construction with minor or no processing or beneficiation.

Finally, under building stones, two new codes are proposed:

- Construction aggregates (definition as in INSPIRE)
- Ornamental stones. Definition: stones used for structural and decorative purposes as cladding, paving or roofing materials in the construction of buildings, other structures, and artifacts.

### The need for new code-lists

The colour of ornamental stones is one of its main distinctive characteristics, if not the most important. Therefore, any queryable mineral resources database that includes ornamental stones should have them characterized by their colour. However, INSPIRE does not have a code-list of colours.

Several colour standards could be applied to describe ornamental stones. However, a fast web review of existing ornamental stones directories (e.g. <https://www.naturalstone-online.com/> or <https://www.natural-stone-database.com/>) shows that the usual colours are not too many. From work carried out during the Eurolithos project, it was concluded that simple colour names should be used rather than the fancy names that are sometimes used for the composite names of stones (Carvalho & Heldal, 2020), and the list of colours of Table I was adopted. For these reasons, we believe that this list of 20 colour names should also be adopted by INSPIRE.

Table I - List of colour names to be considered in Mineral Resources and Geology themes of INSPIRE.

Ornamental Stone colour values	
BEIGE	GREY-GREEN
BLACK	LIGHT GREY
BLACK-GREEN	LIGHT PINK
BLUE	MULTI-COLOURED
BLUE-GREY	PINK

BROWN	RED
DARK GREY	REDDISH BROWN
GREEN	WHITE
GREY	WHITE-BLACK
GREY-BROWN	YELLOW

Another important issue when referring to ornamental stones is the trade name by which they are known and marketed (e.g., Moca Creme, Azul Bahia, Carrara Bianco). In the business world of ornamental stones, these commercial names are often more relevant indicators of the stone quality than available results of their technical properties. For this reason and as for colours, a queryable database of ornamental stones must include the respective commercial names. The European Standard EN12440 - Natural Stone Denomination Criteria integrates a list of most European commercial names of ornamental stone names that is regularly updated. It should be considered in INSPIRE's Mineral Resources theme.

### CONCLUSÕES

For more than ten years, the European Commission has been financing projects wholly or partially dedicated to the harmonization of spatial data on mineral resources in accordance with the INSPIRE Directive. Despite this, regarding ornamental stones, the INSPIRE data model for mineral resources is still based on concepts and vocabularies born in the United States of America nearly 100 years ago, not considering the type of mineral deposits from which this raw material comes, nor the specificities and reasons underlying its current use and application. That is why we have proposed some changes to the INSPIRE mineral resources data model.

Probably the most impactful proposal is changing the name of the commodity. Instead of *dimension stone*, we propose *ornamental stone*. We know that the name Dimension Stone has been used for decades, mainly in Anglo-Saxon speaking countries. On the other hand, we also know that Natural Stone is the most used name in the industrial and commercial circles and, above all, we are aware of the use of Natural Stone in the European standards. However, because this mineral raw material is mainly used today for its decorative role, we prefer the more comprehensive name Ornamental Stone.

Thus, the commodity ornamental stone should be defined as naturally occurring rocks sufficiently consolidated to enable them to be cut or shaped into blocks or slabs for structural and decorative purposes in the construction of buildings and other structures. In addition, Ornamental Stone is the parent group of the different types of ornamental stones for which we

also proposed changes. The last proposed modifications to existing INSPIRE code-lists refer to the end use potential, where we make the distinction between construction aggregates and ornamental stones.

Finally, two new code-lists are proposed, one for the colour of the stones and another for their commercial names, which are two main features when characterizing ornamental stones.

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## ORNAMENTAL STONES FROM THE CUNENE ANORTHOSITE COMPLEX, ANGOLA: THE “NEGRO” LITHOTYPE ANORTHOSITE AND THE RED GRANITES

J.V. Lisboa<sup>1\*</sup>, J.C. Sousa<sup>2</sup>, J.F. Máximo<sup>2</sup>, A. Oliveira<sup>1</sup>, J.F. Suarez<sup>3</sup>, B. Barros<sup>2</sup>, J.F. Rodrigues<sup>1</sup>, J.M. Plastov<sup>4</sup>, J.L. Lobón<sup>3</sup>

- (9) Laboratório Nacional de Energia e Geologia (LNEG, Portugal), [\\*vitor.lisboa@lneg.pt](mailto:vitor.lisboa@lneg.pt)  
(10) UTE-PLANAGEO,  
(11) Instituto Geológico y Minero de España (IGME, Spain),  
(12) Instituto Nacional de Geologia (IGEO, Angola)

**Summary:** *The anorthosites (“black granites”) and associated red granites occurring in selected areas of the Cunene Anorthosite Complex (SW Angola) were assessed regarding its ornamental potential. The results of the field survey in an area of the Huíla province, analysis and tests carried out on collected samples reveal that anorthosites are high quality ornamental stones for indoor or outdoor use, namely for pavement, cladding and lining. The exploited anorthosites occur in varied lithotypes, in terms of texture, mineralogy and color, which is generally the most differentiating factor. One main trade lithotype, the “Negro Angola” has its central extraction area in the Tchiquatite quarrying site, due to the finer granularity and greater homogeneity of the stone when compared to other sites in the region. The red granites were studied in two areas of the complex revealing ornamental quality but the potential is low due to alteration, intense fracturing, and frequent flaws.*

**Key words:** *black granite, red granite, ornamental potential, physical-mechanical properties*

### Introduction

The most worldwide well-known ornamental stones in Angola are the anorthosites commercially referred to as “black granites” of which the most recognized is the “Negro de Angola”. The characteristics that favor the anorthosite exploitation as ornamental stones, are related to decorative criteria where the intense black stands out, although they occur with other chromatic tones and with varied iridescences.

These rocks occur in southwestern Angola, in one of the largest anorthositic complexes in the world the Cunene Anorthosite Complex (CAC). Several bodies of acidic rocks are associated with the CAC, mostly red granites, locally with significant ornamental potential and target for exploration.

Although the “black granites” have been known and exploited for over 50 years, they have been scarcely studied, especially from an ornamental point of view and given their huge area of occurrence, the potential, although admittedly high, is little known.

The anorthosites exploited as ornamental stone in the CAC occur in varied lithotypes, in terms of texture,

mineralogy and color, which is generally the most differentiating factor. Black, brownish, greenish olivine-rich lithotypes occur, with different granularities and mineralogical proportions. Two main commercial lithotypes can be considered, the “Negro Angola” (“Negro”) and the “Marron”.

The anorthositic rocks of the CAC have great potential for use as Ornamental Stones, as demonstrated by the number of existing quarries in vast areas of the massif, which, however, represent a fraction of the total area. The investigation carried out allowed an extension of the areas under exploitation to other potential quarrying areas, whose validation will require a detailed survey. These areas are in the central zone of the massif, approximately between the localities of Dongue and Chibemba (Huíla province) being intersected by a granitic intrusion (mostly red granite and granite porphyry), which generates a NE-SE belt that intersects the CAC and separates the anorthosites in two compositional regions.

The present study intends to preliminary assess the ornamental stone potential of the anorthosites occurring in the Dongue area (Chibia municipality), mainly associated with the “Negro” lithotype, north of

the NE-SW trending red granites belt (RGB) which are also assessed, based on the characterization of selected samples. This paper follows on from work recently carried out for the Chart of Industrial Rocks and Minerals of Angola (CARMINA), which is a subproject of the National Geology Plan (PLANAGEO) for the Geological Survey of Angola (IGEO).

### Geological setting

The Mesoproterozoic Cunene Anorthosite Complex (1.50 – 1.34 Ga) is an extensive igneous basic massif composed predominantly of anorthositic, leucotroctolitic and troctolitic rocks, which also includes smaller volumes of gabbros and norites (e.g., Carvalho and Alves, 1990; Silva, 1992; Sleijko et al., 2002). The CAC extends from the Quipungo region (Angola) to the north of Namibia (Zebra Mountains), covering an outcropping area of about 15,000 km<sup>2</sup>, which could be substantially larger (Oliveira, Sousa, 2021). In Angolan territory, where about 75 to 80% of the massif outcrops, the CAC develops with a longitudinal direction close to N-S along about 300 km, and transverse extension that can vary between 25 to 50 km (Pereira et al., 2013).

This huge anorthositic body has been classified as a Massif-Type Anorthosite according to Ashwal's (1993) classification. The information gathered refers, in the majority, the CAC as an igneous assemblage of the Proterozoic Massif-Type Anorthosite type (e.g., Ashwal, 1993; Morais et al., 1998; Sleijko et al., 2002; Mayer et al., 2004; Gleißner et al., 2010). Other opinions consider the CAC as a layered intrusion like the Bushveld massif (e.g., Stone and Brown, 1958; Silva, 1990; 1992; Maier et al., 2008).

The mineralogical constitution of the CAC rocks varies around terms with variable amounts of plagioclase, olivine, ortho and clinopyroxenes. The anorthosites (*s.l.*) generally present plagioclases measuring between 1 and 5 cm, containing iron oxide lamellae in a pseudo-ophyctic matrix of orthopyroxene and olivine.

Regarding the anorthosite composition, the following can be seen:

- predominant troctolitic (olivine-rich) composition north of the RGB; anorthosites are essentially medium-grained and dark in color, homogeneous, without or with few iridescences, minor mafic content and heterogeneities (the “Negro Angola” belongs to this major lithotype);

- preferably more gabbroic (pyroxene-rich) composition south of the RGB; anorthosites are medium to coarse grain, grayish to brownish or purplish in color, with very frequent textural and mineralogical heterogeneities, higher frequency of iridescences provided by labradorite.

The felsic rocks associated with the CAC are referred to as the Red Granite Suite (Milani et al. 2022) and include granitoids, syenite, mangerite, and charnockite. The most frequent lithologies correspond to red granites, which occur in the north (Matala region), in the RGB and between this belt and the river Cunene, frequently reaching large areas. According to geochronological evidence, the suite emplacement has a coeval to younger relationship with the anorthosites (Milani et al. 2022).

Geomorphologically, the CAC in the studied area constitutes, for the most part, a generally flattened zone with several outcrops. These are in general, of small size, surrounded by extensive zones with an important weathering mantle. However, there are also numerous inselberg type reliefs (Fig. 1), and it is on these that exploration for ornamental stone extraction is currently focused.

### Methodology

During the implementation of the CARMINA subproject, rocks were surveyed and sampled in the CAC area to assess its ornamental potential. The preliminary interpretation of collected data concerning the anorthosites and granites occurring in the CAC central area followed the basic methodological procedures for the realization of the CARMINA sheets.

The methodology applied includes field and laboratory work, in accordance with the one adopted for carrying out the CARMINA sheets, first comprising aerial photograph interpretation to recognize granite outcrops, altered zones, fracturing patterns, distinct zones and fracturing intensity.

The field analysis to identify potential areas for exploration of ornamental stone, first comprised the mapping distinction of occurring anorthositic lithotypes, fracturing survey and sampling in quarries and significant outcrops. The distinction of anorthositic lithotypes was primarily made according to textural, mineralogical, geomorphological, and alteration criteria, and petrographic analysis in laboratory. Lithological description encompassed the anorthosite's macroscopic, microscopic-petrographical study and

identification of flaws susceptible to decrease the ornamental anorthosite's potential (such as, enclaves, abundant pyroxene, layering and filonean intrusions, grain gradations and crystal size heterogeneity, rare spots of sulphides).

The petrographic analysis and tests to determine the mechanical properties of the selected stones were performed in the laboratory. These tests comprised water absorption, apparent density, open porosity, mechanical resistance to compression and bending strength.

## Results and discussion

### Anorthosite

The areas of occurrence of inselbergs north of the RGB are generally associated with higher ornamental potential anorthosite and the "Negro" lithotype. In this anorthosite, the occurrence of a relatively high content in pyroxenes is frequent, possibly associated with olivine; the average composition of the plagioclases is also invariably poorer in sodium.

Generically, these rocks are essentially made up of dark plagioclase, coarse-grained, in the order of 1-2 cm, rarely  $\geq 3$  cm, displaying accessory small crystals of mafic minerals, such as olivine, or black pyroxene with dark green hues, the latter generally appearing in crystals of around 5 mm. Rarely, small crystals or masses of titanomagnetite and ilmenite are observed, as well as small sheets of biotite. Occasionally, coarser granulometry zones occur, in which the crystals, whether of plagioclase, pyroxene, or magnetite, can reach decimetric dimensions, occurring singly, or else forming zones of variable geometry, but always well delimited, of mafic cumulates; zones of magmatic layering may exist, with this mineral association. Distinctive aspects of the "Negro" lithotype, in addition to fine granularity and dark color, is greater textural homogeneity compared to other lithotypes, and the absence or rare iridescence. The fracturing intensity is usually lower when compared to other lithotypes.

The so-called Tchiqatite quarrying site is the main extraction area for this lithotype, due to the finer granularity and greater homogeneity of the rock when compared to other sites in the region (Fig. 1).



Fig. 1. Anorthosite quarry (Friconde) with inselberg relief in background in the Tchiqatite quarrying site.

The fracturing survey of the anorthosite occurrences in the Tchiqatite region was strongly limited both by its outcropping pattern (mostly in rounded boulders and blocks), and by the cover and ongoing extraction work in quarries. The survey on the quarry fronts made it possible to identify a generalized trend towards decreased fracturing in depth, reflected both in the sub-horizontal and sheet fractures. Given the small number of simultaneous strike and dip determinations at each location, preventing a fracturing statistic in individual stations, all the collected data were compiled, allowing to verify the occurrence of two dominant NE-SW fracture directions, and another fracture direction, NNW-SSE to NW-SE. In the NE-SW direction, the fractures dip range between subvertical and  $70^\circ$  NW and SE; there is a more frequent set  $N50-60^\circ E$ ;  $70^\circ SE$ . In the NNW-SSE direction, the slopes are mostly subvertical and to a lesser extent with inclinations to the W; according to the NW-SE most of the fractures are also subvertical with some leaning  $70$  to  $80^\circ$  to the E (Fig. 2)

The spacing between fractures verified in the outcrops and quarries is quite variable. According to García (1995), wide (2.5-6.25m) to moderate (1-2.5m) spacing predominate. In most outcrops it is common to find very wide (6.25-15.62m) to extremely wide ( $>15.62$ m) spacing between more fractured areas with clusters, which cause an apparent greater fracturing.

To the south of the Tchiqatite region towards the RGB, there tends to be a relatively significant dispersion of fracture directions, although with a predominance of NE-SW fractures. The spacing between fractures tends to be smaller, with moderate spacing (1-2.5m) predominating.



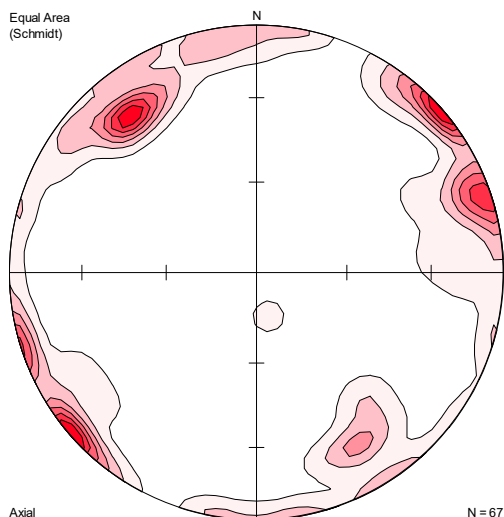


Fig. 2. Density contour diagram (fracture poles) in the Tchiqatite region. N=67

### Petrographic analysis

The thin sections of anorthosite samples (Fig. 3), observed under the petrographic microscope, are holocrystalline, phaneritic, medium to coarse grained, composed mainly of plagioclase, which is twinned according to the albite law, occasionally denoting some deformation as well as undulating extinction, being still characterized by pericline twinning.

Intergrown with plagioclase, olivine occurs (sometimes in smaller inclusions in plagioclase) in association with relatively sparse orthopyroxene and clinopyroxene (marked by exsolutions of opaques along their cleavages) in reaction rims (corona). The presence of opaque minerals in exsolutions is verified along the irregular fractures of the olivine and on its edges. There is also rare biotite, of fine granulometry and associated with opaque minerals in the olivine border. Plagioclase is also characterized by micro-fracturing of varying intensity with or without filling, by fine material that may correspond to clayey minerals and micro inclusions of opaque mineral (Fe oxides), in “dust” and needles.

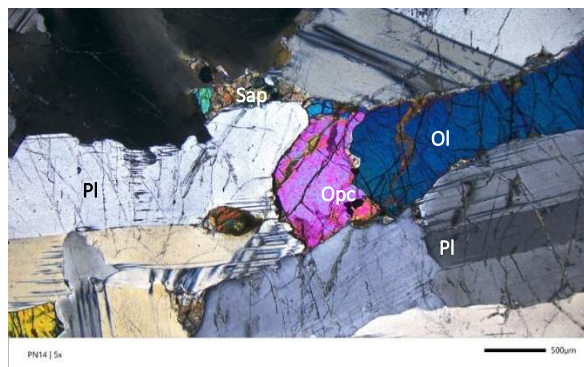


Fig. 3. Microphotography under the petrographic microscope of an anorthosite sample (+ nicols). Obj: U MPLFL 5x/0.15, Ampl. 5x. Scale: 500µm black bar. Symbology: Ol – olivine, Pl – plagioclase, Opc – opaque, Sap – saponite (bowlingite)

### Physical-mechanical properties

The physical-mechanical tests considered most relevant for the technological characterization of ornamental stone were carried out at the Geoscientific Laboratory of IGEO, in Lubango. They included the determination of apparent density, open porosity, water absorption at atmospheric pressure, compressive and flexural strength in samples of selected anorthosites both in quarries and outcrops of the study area (Table I).

Table I. Results of technological tests carried out on anorthosite samples (n=22)

	Average	St Dev.	Max	Min
App. density (kg/m <sup>3</sup> )	2775	55	2960	2690
Open porosity (%)	0,2	0,1	0,4	0,1
Water absorption (%)	0,1	0,0	0,2	0,1
Comp. strenght (MPa)	165	22	202	105
Flex. strenght (Mpa)	18,4	3,9	25	11,5

The obtained values reveal a high density (slightly higher in the Tchiqatite region) reflecting the good quality of the anorthosite, and in line with the data declared by the quarrying companies. All tested samples have values well above the minimum values required for this type of stone, greater than 2600 kg/m<sup>3</sup>, according to ASTM and CEN / TR 17024 standards (Molina, 2017)

Regarding open porosity and water absorption, the stones present very low (Moura, 2006; Molina, 2017) and homogeneous values.

The medium to high values values obtained in the compressive strength tests are in line with those expected, given the density and compaction of the anorthosites. These values are lower than those presented by the quarrying companies, certainly due to the location of part of the sampling points, in superficial areas of the massif, where alteration processes can lower those values. The data obtained in the compressive strength tests indicate medium-high quality for the anorthosites.

Considering the possible uses, based on the outcrop characteristics and the obtained results, flexural strength is the parameter of greatest interest. Based on the North American ASTM standards, the results obtained show values well above the minimum required, set at 8.3 MPa (Molina, 2017). According to Moura (2006) proposal, most samples can be classified as medium-high quality in terms of this parameter.

Regarding resistance to compression and flexion, there is a tendency for slight loss of quality in anorthosites from north (Tchiquatite region) to south (RGB area). This may be influenced by a coarsening of grain and pyroxene enrichment.

In conclusion, the results of the analyzes and tests reveal that these anorthosites constitute ornamental stones of high quality for interior or exterior use, namely for application in pavement, cladding and lining. The main constraints on the ornamental potential of these stones are mainly correlated with the possibility of textural heterogeneity and the characteristics of fracturing.

It was observed during the field work, the abandonment of many extraction sites, often with dismantling fronts <10m height. The justification, excluding non-geological aspects intrinsic to the exploration companies, seems to be associated with fracturing, which is more intense close to the surface, and with several common flaws in the anorthosites: closed joints, thin veins with whitish zeolite/clay minerals filling, "stained" appearance of the stone, which is due to variations in the mafic minerals content, occurrence of mafic cumulates (megacrystals of pyroxenes and magnetite), and magmatic layering (Fig. 4). It is also common practice to widen quarries on the surface rather than deepen quarries. The lack of knowledge about the characteristics of the stone in depth may contribute to this fact, since the existence of a drilling plan is not frequent, with some companies preferring not to invest in deeper exploitation.



Fig. 4. Anorthosite slab from Metarochas quarry showing magmatic layering with pyroxene cumulate crystals.

Within the scope of the PLANAGEO project for the Geological Institute of Angola (IGEO), in which these studies were carried out, potential areas for the ornamental anorthosite occurrence were demarcated. In the area that this study reports to, north of RGB, the total of those potential areas sums 165 km<sup>2</sup>, which will correspond to very high ornamental stone resources and, despite the existence of a very significant extractive activity in this area, the great part remains unexplored.

#### Red granite

The red granites outcropping areas addressed in this work, despite corresponding to a vast area summing up over 200km<sup>2</sup>, only correspond to a small fraction of the total outcropping area of the Suite of Red Granites. They refer to the RGB area, NE of Chiange (Huila), and the occurrences of the region of Otchindjau, Cahama (Cunene). In these areas currently there is no quarrying activity.

Throughout the entire surveyed area, there are also granodiorites, granitic porphyries and mylonites, almost always with the presence of potassium feldspar, giving rise to red to pink lithologies, with different textures and granulometries. The occurring granitic lithotypes with red tones, are predominantly biotitic, medium to coarse granularity ( $\leq 1\text{cm}$ ), with a porphyritic tendency, often with grain orientation. These lithologies making up the set known as red granites are usually found in shallow outcrops, or forming small elevations with generally small blocks, denoting high levels of fracturing and deformation. At the surface, alteration is highest in the Cahama region.

## Petrographic analysis

The analyzed sample (Fig. 5) is holocrystalline, phaneritic, with xenomorphic to hypidiomorphic texture, porphyritic, with fine to coarse granulometry (with the presence of alkaline feldspar and quartz phenocrysts). Alkaline feldspar is found sub-euhedral to anhedral and is relatively abundant, characterized by perites (plagioclase exsolutions) and Carlsbad twinning, less frequent a potassium term characterized by checkerboard twinning (microcline). In alkaline feldspar phenocrysts, a poikilitic texture is sometimes observed, with inclusions of other minerals. The sample is also composed of anhedral to sub-euhedral quartz, showing an undulating extinction, and more dispersed, sub-euhedral biotite, preferably with tabular shapes, greenish brown in color, chloritized (focusing on the edges of the crystals) (Fig. 5). It is common to form aggregates with chlorite and an opaque mineral, bordered by chlorite. The crystals in the sample, especially those of alkali feldspar, are affected by a reddish-brown dust of iron oxides. These also appear to occur in numerous fluid inclusions in quartz. Some intragranular fractures are observed, especially in quartz phenocrysts, which are always filled with chlorite and/or iron oxides. Plastic deformation and recrystallization of the quartz are evident, these phenomena contributing to the stone's physical and mechanical properties, referred to below.

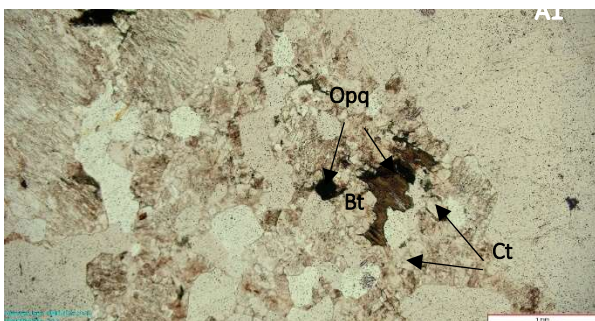
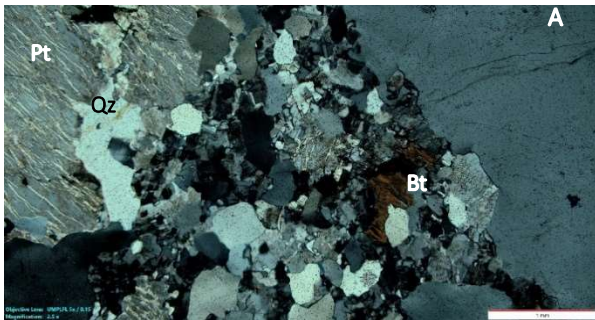


Fig. 5. Microphotographs under the petrographic microscope of Sample 1 of red granite. Obj: UMPLFL 5x/0.15, Ampl. 2.5x. Scale: 1mm white bar. A) NX, A1) N//. Symbology: Qz – quartz, Bt – biotite, Opq – opaque, Ct – chlorite, Pt – perites.

The more frequent mineralogical composition of these granites is as follows: quartz (40%), plagioclase (25%) and potassium feldspars (20%) as the dominant minerals; biotite (7%), opaques (5%) and pyroxene (<10%) as accessory minerals; sericite and chlorite as secondary minerals.

## Physical-mechanical properties

The granites tested have medium to high density (Table II), characteristic of this lithology and higher than the minimum value recommended for this type of stone, greater than 2560 kg/m<sup>3</sup>, according to the specifications in the ASTM and CEN / TR 17024 standards (Molina, 2017).

The open porosity and water absorption show low values (Table II) revealing good quality (Moura, 2006; Molina, 2017) and, therefore, with values below the maximum recommended for the best quality products, either for the open porosity (< 1%) and for water absorption ( $\leq$  0.4%) (Moura, 2006).

Table II. Results of technological tests carried out on 2 red granite samples (n=22)

Test	Sample 1	Sample 2
Apparent. density (kg/m <sup>3</sup> )	2610	2720
Open porosity (%)	0.6	0.3
Water absorption (%)	0.3	0.2
Compressive strenght (MPa)	245	118
Flexural strenght (Mpa)	20.2	15

Compressive strength demonstrates a good capacity to respond to requests, in line with quality ornamental granites.

Based on the North American ASTM standards, which establish recommended values for flexural strength depending on the type of stone, the result obtained (Table II) is considerably higher than the minimum required (8.3 MPa, Molina, 2017) and the value for ornamental granite of best quality (> 10 MPa, Moura, 2006), for this parameter.

In conclusion, the results of analyzes and tests carried out show that both sampled granites have ornamental quality and can be used in all types of applications,

whether indoors or outdoors. The granite sampled in RGB area (Sample 1) has a higher hardness resulting from the higher quartz content, revealed in very high compressive and flexural strength. The possibility of excessive heterogeneity, mainly textural, and the characteristics of fracturing must be considered as constraints to the ornamental potential of these stones.

Although the lack of positive reliefs in these lithologies, their alteration and the intense fracturing observed, limit their use in obtaining Ornamental Stone, mainly regarding the production of large blocks and plates, the important cartographic expression that presents this unit together with its textural variations, allow admitting the existence of localized areas where the necessary requirements for a possible exploitation are fulfilled.

### Conclusions

Ornamental stone anorthosites in the studied area have a very high potential. Only the “Negro” lithotype occurs, with relatively homogeneous characteristics, without significant flaws, differing from other sites south of the RGB, where the occurrences of this lithotype are much more dispersed and where layering and lithotype variations are more frequent. The frequency of zones with spaced fracturing, also allows the extraction of large blocks in many outcrops, mostly in inselberg reliefs, although there are quarries in flattened domes.

At the mineralogical and petrographic level, plagioclase is associated with smaller and variable amounts of

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olivine and pyroxene with opaque mineral exsolutions. Plagioclase is characterized by micro-inclusions of opaque mineral (Fe oxides) occurring both in crystallographically aligned needles, and very fine particles (“dust”) in the crystals, often showing micro-fracturing with and without filling, which may be reflected in the strength of the stone. From a physical-mechanical viewpoint, the anorthosites are dense stones, with low porosity and water absorption and, in general, high resistance to bending, which fully satisfy the minimum values and those recommended by the standards. They thus constitute high quality natural stone for interior or exterior use, namely for application in floors and wall cladding. The main constraints to ornamental potential are the possibility of occurrence of heterogeneities, mainly textural, and fracturing.

Red granites are chromatically attractive, and their physical-mechanical properties are quite suitable for use as ornamental stone, both in interiors and exteriors. The lack of outcrops showing positive reliefs, the granite’s alteration, and fracturing are however, strong constraints to obtain ornamental stone, particularly concerning the production of large blocks and slabs. The ornamental potential in the studied areas is low, but considering the vast area that the red granites occupy together with their textural variations, the existence of localized areas where the necessary requirements for a possible exploitation can be fulfilled is a strong possibility.

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## EXOTIC STONES: A MILESTONE IN NATURAL STONE SECTOR

M.H.B.O. Frascá<sup>1\*</sup>, N.F. Castro<sup>2</sup>

(13) MHB Geological Services, [\\*mheloisa2@yahoo.com.br](mailto:mheloisa2@yahoo.com.br)

(14) Centre for Mineral Technology, CETEM/MCTI.

**Summary:** *The natural stones, commonly called exotic, comprise a special group characterized by unusual patterns and relatively rare geological occurrence. They are currently the flagship with the highest added value of Brazilian natural stone exports and are used domestically and internationally for the internal and external cladding, flooring and decoration of luxury buildings or residences. Its high price, in addition to the intrinsic exclusivity of almost all slabs, stems from the high costs of locating the deposits, mining exclusively with diamond wires, transport, processing, including several steps of reinforcement and resining, and finally, polishing. The development and use of all these state-of-the-art technologies, exposed in this article, make it possible to point to exotic rocks as a milestone in natural stones' production.*

**Key words:** *exotic stones, Brazil, pegmatites, quartzites, processing technologies*

### Introduction

Exotic, according to Merriam-Webster dictionary (2023), means either "strikingly, excitingly, or mysteriously different or unusual" or "introduced from another country: not native to the place where found". Exotic stones in Brazil seem to cover both definitions since they constitute a set of rare rocky materials and are currently the flagship of exports. Technically, they can be defined as rocks that usually have a differentiated aesthetic appearance and a relatively rare geological occurrence, whether of an igneous, metamorphic, or sedimentary nature.

The term started to be used by the natural stone sector around the 90s of the 20<sup>th</sup> century when it began the production and marketing of some very heterogeneous rocks in several aspects (granulometric, mineralogical, textural, and structural), namely the pegmatitic rocks, which are characterized by very large feldspar mega crystals. Frascá (2007) also called these "decorative stones" due to their typical low mechanical strength and limited production, and thus commonly used as interior cladding.

Gradually, other distinct rock types were introduced into the market thanks to the geological singularities of the Brazilian territory and technological developments that facilitated their extraction and processing. The "exotic" quartzites are the best example of top-ranked stones nowadays.

Although the innovations in the natural stone industry are extended throughout the sector, regarding exotic stones, pegmatites could be considered as "Phase 1" and quartzites as "Phase 2", in technological terms. Therefore, the first can be related to the new resin-bonding technologies in the 1990s and early 2000s, and the second to the additional advances in equipment and inputs for cutting and polishing, specifically new diamond tools and resins, together with CNC (computer numerical control) technologies.

The lack of sharing the provenances and technological properties, which seem to be predominantly linked to marketing strategies, is a relevant characteristic of these rocks. So, the primary objective of this paper is to gather the available information to contribute to a better knowledge of these stony materials.

### Brazilian and international market

Montani (2021) points Brazil as one of the main world stone producers, regarding chromatic diversification or the exclusive nature of a good proportion of the offered products, which seems to refer to exotic stones.

In 2020, Montani (2021) positioned Brazil as the fourth leading quarrying producer (circa 5% of world production), the fourth leading exporter of stone in quantity (circa 2,000 tons and 4% of global shares) as well as of processed stones (5,7%). China, India, and Turkey precede Brazil in almost all positions.

The export data provided by Centrorochas (2023) shows that a large part of the stone material currently exported refers to quartzite slabs, exotic silicate rocks, and massive quartzite blocks.

In terms of buyers, the USA is the major importer, predominantly of processed stone. In 2022, the USA, China, Italy, and Mexico, in that order, represented the leading destinations for Brazilian exports, with the USA responsible for 58% of revenues in this period (ABIROCHAS 2022). The same publication mentioned that the stones sold under the code 68.02.99.90 (massive quartzite slabs and exotic silicate stones) had an average price of US\$ 2,300/t, representing US\$ 356.6 million and 154,800 t of the material exported.

Regarding stone consumers, after China (33.1 %), India (8.2 %) and the USA (5.7 %), Brazil is an expressive consumer, using circa 3.3 % (56.4 mill m<sup>2</sup>) of the world stone produced, according to Montani (2021).

#### **Main rock types and technological properties**

As a result of the great geodiversity and the improvement of the geological knowledge of the Brazilian territory, together with the search for detailed information on the likely places of occurrence of the rare materials, it was possible to discover and diversify the range of options for Brazilian natural stones.

Figure 1 (a, b, c) shows some rough blocks to illustrate the huge diversity of patterns and geological nature exhibited by exotic stones. They may be pegmatitic, quartzitic and schistose rocks that, in addition to each type's characteristic mineralogy, quartz and feldspars, quartz and quartz and micaceous minerals, respectively, additionally contain a significant variety of subordinate and accessory minerals, making their petrographic classification very difficult in routine characterization studies.

Likewise, the structural, textural and granulometric heterogeneity also difficult an accurate determination of their technological properties.

Besides, although their properties may meet the requirements for application in flooring or wall covering, in addition to other decorative options, due to the very coarse grain size and the high degree of cracking or fracturing of some lithotypes, their mechanical strength and porosity/water absorption may be relatively lower or higher, respectively, than typical granites or quartzites.

Thus, studying an pegmatite exotic stone called Karnaval (from Borborema, in the northeast region of the state of Paraíba, and geologically associated with the Seridó Fold System), Pazeto et al. (2017) indicated two main petrographic factors contributing to its brittleness and lower flexural strength: (a) the extremely coarse grain size, configuring a high degree of discontinuities and poor mineral interlocking, and (b) the surfaces of weakness related to feldspar mega-crystals cleavage plans.

On the other side, wear abrasion and chemical attack resistance are high for most quartzites due to their high quartz content.

Zagôto et al. (2022) proposed a natural stones quality index based on the technological test results of 285 natural stones. The quartzite group (61 samples), in its natural state, showed the highest mechanical strength dispersion, which was attributed to the microcracking density.

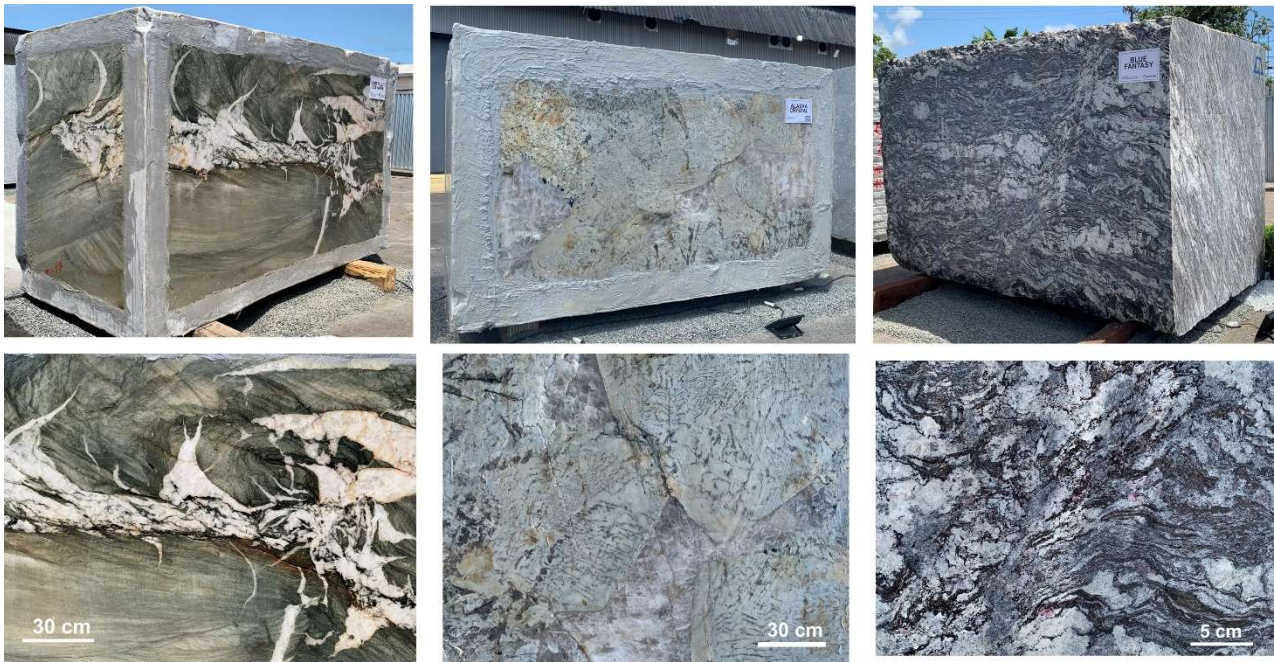


Fig. 1. Examples of exotic stones: quartzitic rock (a), pegmatitic rock (b), and schistose rock (c).

Furthermore, characterization results of several quartzites from Ceará (Santos et al., 2017) showed that some may have low mechanical strength, but also low water absorption. In this case, Nunes (2018) found that some quartzites, even with a high density of microcracks, have most of them not connected, thus inhibiting the percolation of water. Consequently, reinforcing the blocks and slabs is of utmost importance.

### Occurrences

Singularity and differentiated geological aspects, such as structure, grain size and colour are the main requirements for a rock to be "exotic". Thus, they are mostly stone materials of restricted occurrence, with smaller reserves than traditional deposits of granites and marbles.

Another remarkable feature is the insufficient knowledge of the exact locations of exploitation, mainly aiming to avoid competition and maintain exclusivity.

Pegmatitic rocks and quartzites are the main stone types commercialized as exotic stones, followed by schists and some very rare igneous rocks (such as metamorphosed pillow lavas).

The leading producers of exotic stones are Minas Gerais, Paraíba, Rio Grande do Norte, Ceará and Bahia (ANM, 2023). Quartzite slabs and exotic silicate rocks from the states of Ceará and Espírito Santo, as well as massive

quartzite blocks from the state from Bahia and Minas Gerais, are the main exported items, according to Centrorochas (2023).

However, it is important to mention that the state of Espírito Santo has, by far, the largest processing park and is the largest Brazilian exporter of natural stone. On top of this, companies from that state also quarry and process natural stones in other states. This explains its presence in the statistics and not necessarily due to its significant occurrences of exotic stones.

Data available at the National Mining Agency (ANM, 2023) show more than 1,000 companies with licensed areas, explicitly registered to produce dimensioned quartzite, alone or with other substances. Half of the areas are for dimensioned quartzite alone. Though this does not mean they are effectively exploited, it clearly shows the market interest.

The mentioned exotic silicate stones, which include pegmatites and differentiated granites, are those whose provenance is most challenging to determine since they are late phases of granitic intrusions that can occur in the most distinct orogenic belts scattered through the Brazilian territory (Figure 2).

Thus, it is more difficult to identify the licensed areas for pegmatite natural stone as it is usually registered as granite.

Within the tectonic complexity of the Brazilian territory, there are several regions with a high concentration of pegmatites, called pegmatitic provinces, generally



named according to their geographic distribution. Dimension pegmatitic stones have been quarried in the Eastern and Northern provinces since the beginning of the 21<sup>st</sup> century (Beurlen, 2009; Pedrosa-Soares et al., 2009) from barren exposures of fresh rock, sometimes together with associated aplites and granites.

Several quarrying operations are carried out in the pegmatitic occurrences reported by Teixeira (2012) in the Seridó Belt, on the border of the states of Paraíba and Rio Grande do Norte, as well as in the northeast of the state of Minas Gerais, and some in other states of the country (i.e., Ceará and Goiás).

In the state of Minas Gerais, most of the countless pegmatites are Late Neoproterozoic intrusions of granitic residual melts (Pedrosa-Soares et al., 2009) of the Araçuaí Folded Belt, related to Gondwana amalgamation.

As for quartzites, they have been used as building stones, in Minas Gerais, since the Portuguese colonization (Costa, 2015). The quartzite flagstones from the region of São Tomé das Letras, named 'Pedra São Tomé' and 'Pedra Mineira', have been exploited for several decades and domestically used and exported as rough slabs or setts for external paving. Geologically, they belong to the Andrelândia (Neoproterozoic) and Canastra (Mesoproterozoic) Groups, both in the Tocantins orogenic system, which lies between the Amazonian and São Francisco cratons (Hasui 2012). Some predominantly white quartzites, such as the commercial 'Montblanc', from the Espinhaço Supergroup (Paleo/Mesoproterozoic), are currently

exploited in the north-eastern part of Minas Gerais, as well as in the south of the State of Bahia.

Also, regarding well-known quartzites, the "exotic" blue quartzites (Frască; Castro, 2022), also in the Espinhaço Supergroup, have been mined since the 1960s close to the city of Oliveira dos Brejinhos, state of Bahia.

Thus, due to the tradition and presence of mining companies in these areas, the exploitation of other types of quartzites, included here as exotics, and occurring in these geological and geographic contexts, is expected. For instance, quartz stones with green-coloured fuchsite crystals are exploited close to emerald occurrences in northeast Bahia.

Bahia is also known for the diversity of coloured and banded quartzites: 77 of the 118 commercial varieties presented in the Atlas of Dimension Stones of Bahia (Iza et al., 2022) are quartzites.

Another important site for quartzite production is the state of Ceará where some well-known whitish beige quartzites, such as 'Perla Santana', 'Perla Venata', 'Taj-Mahal', 'Matira' and others, are being mined are being mined in the northwest of the state, in the Médio Coreaú folded region, belonging to the Borborema Orogenic System, which comprises diversified Precambrian lithotypes of different ages. They are pure quartzites, sometimes micaceous and ferruginous and fine- to medium-grained (Nunes, 2018), of the Neoproterozoic metasedimentary sequence of the Martinópolis Group (Santos, 2004).

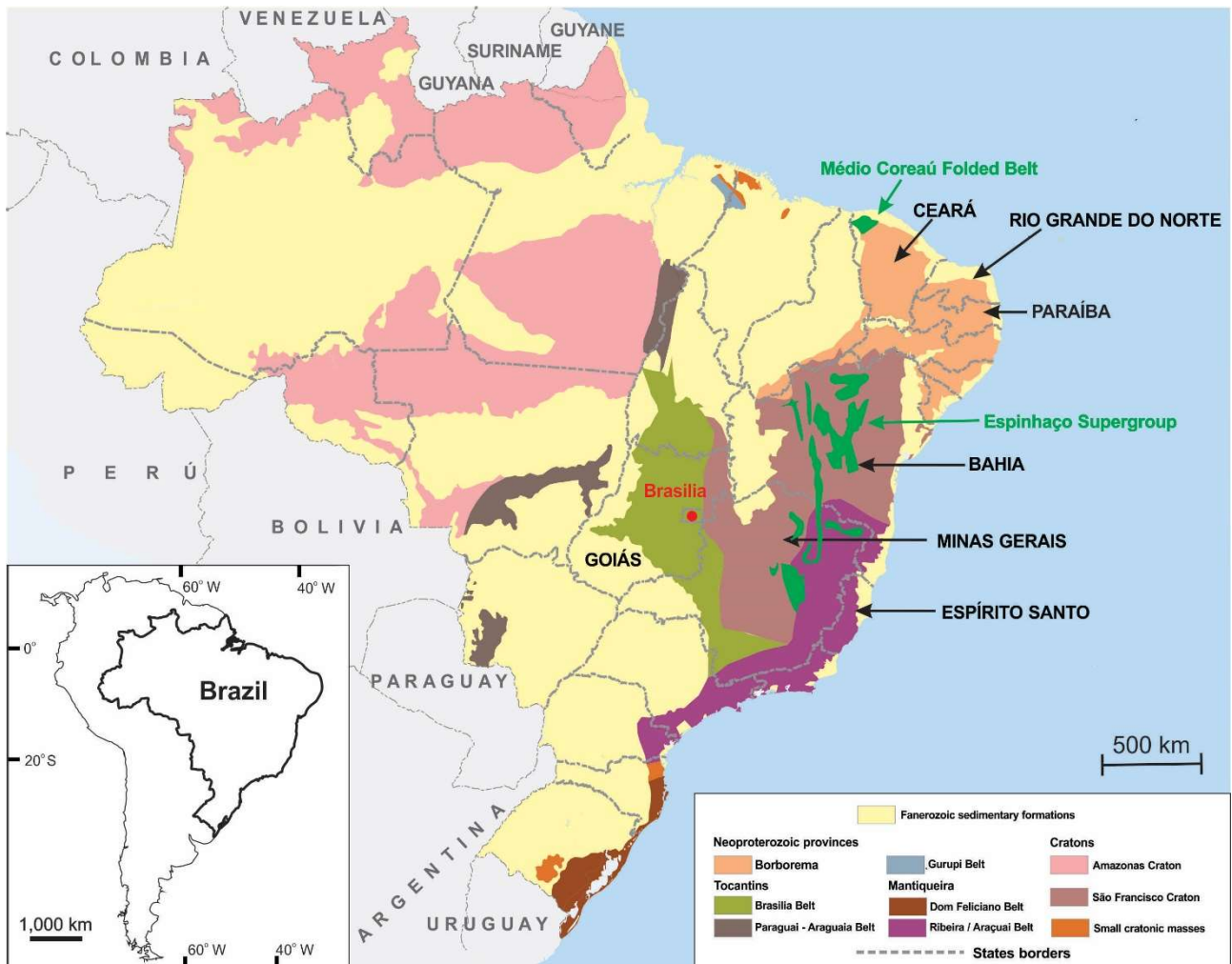


Fig. 2. Simplified map of Brazilian Geotectonic Provinces, with location of geological features and states mentioned in the text (Sources: Franz et al., 2014; Silva et al., 2014).

### Mining and processing technologies

The Brazilian stone industry has achieved a high technological level regarding the quarrying and processing of exotic stones during the last decades, partly due to the increasing production of exotic stones. European companies, most Italian, have been working together with stone producers to improve the cutting and resining of hard and heterogeneous stones, so Brazil is frequently considered a research laboratory for diamond wire and resin companies. Although the national industry has also developed and provided cutting equipment, tools, and chemical products, most of them are still imported.

Exotic stones are usually quarried in open pits with the medium-to-high benches method. Diamond wire is the only cutting technology. Primary cuts on benches of six to fifteen meters high and secondary cuts on the laid benches to produce the blocks are done by diamond

wire equipment, while drilling is used for auxiliary operations.

Most blocks are superficially reinforced on-site, or at least their corners (see Fig. 1 a, b), with a mixture of resin and mineral fillers over which a fiberglass mesh is fixed. More valuable stones can be reinforced twice in the quarry to allow their safe transportation, and sometimes, even the benches' wall is reinforced with the same system before laying them down to the quarry (Figure 3 a).

Production is slow, and costs are high as diamond wire, drilling rods and bits need more frequent replacement than in quarries of traditional silicate rocks, which are more homogeneous and can be quarried in high or ultra-high benches resulting in higher production rates. Moreover, especially regarding pegmatites, the more exotic patterns occur as several meters width veins inside the rock massif, requiring continuous and careful quarry planning and leading to a low recovery.

additionally, exotic materials, which have a high selling value, allow a profitable quarry production of small blocks for slabs or other products (i.e., furniture, artworks), beyond producing the market-demanded big blocks.

The quarried blocks go straightforward to exportation or to a processing facility to be cut into slabs and polished. The blocks can again be reinforced with resin, mineral filler and a fibreglass mesh and cut into slabs in multiwire saws (Figure 3 b, c). Before the multiwire cutting machines, a quartzite block took around ten days to be cut in a gang saw, while now, that task takes just several hours (Souza et al., 2012; Careddu; Cai, 2014).



Fig. 3. Exotic stones production: quartzite quarry showing benches and blocks reinforcement (a); pegmatite block being reinforced at the quarry (b); and a multiwire saw cutting a reinforced quartzite block (c).

The slabs polishing process involves the following sequence: the back of the slab reinforcing with fibreglass; restoring (filling of cavities with the same rocky material), when needed, and subsequent rubbing; slab resin impregnation; rough polishing; first resin coating; medium polishing; second resin coating; fine polishing; and inspection (Martins; Silveira, 2020). The slab drying and resin curing are done mainly by gas or electric ovens, but there are also microwave and UV systems. Almost all Brazilian companies have automatic polishing lines, and several have fully automatized production facilities, including robots and vacuum systems for resin application. (Figure 4).



Fig. 4. Exotic stones production: automatized processing plant (a); cavities for filling (b); robotized resining for fibreglass fastening (c); automatic resin coating (d).

Pazeto et al. (2020) conducted a study to determine the mechanical performance of the reinforcing process adopted by industry (netting) by testing specimens of Karnaval pegmatite reinforced with different thermoset resins and glass fibres. An important finding, in terms of costs, was that the post-curing process of the epoxy resin commonly applied in the industry proved to be more efficient in meeting a better mechanical performance than adding low-density fibreglass to the reinforcing procedure.

Resining has also experimented with a remarkable evolution in Brazil to achieve the best surface quality through less time-consuming application methodologies and cost savings (Martins; Silveira, 2020). Regarding the resins applied, some polyester-based systems are used for the blocks' reinforcement (Camargo et al., 2022), but most are epoxy resin-based systems. They require rigid health and safety measurements for their use, beyond being originated from a non-renewable source. Brazilian research has been studying natural resins to substitute them in the industry for several years, but epoxy resins are still the most commercialized (Dorigo et al., 2020; Silveira et al., 2020).

### Uses

Although the scarcity of information describing the final application of the several exotic stone types, the best indications are that interior decoration is their principal use, even in Brazil, mostly due to their high price and beauty.

Best examples are countertops, tables, bathrooms, swimming pool areas, gourmet kitchens and many others (Figure 5).



Fig. 5. Examples of uses of exotic stones: kitchen with 'Ornamental Granite' (a) and 'Perla Venata' (b); dining room with 'Quartz Blue' (c); bathroom with 'Taj Mahal' (d), and pool area with 'Yellow Bamboo' (e). Photos by Paulo Giafarov (a, c, e) and Liliane de Lucca (b, d).

## Conclusions

Exotic rocks are a particular group of natural stones characterized by special patterns and, in most cases, by their rare occurrence.

To the exotic stones high market price due to their aesthetic patterns, it must be added the high production costs for that, as it is used high tech cutting tools,

abrasives, and resins in a consumption much higher than for common natural stones.

Along with that are the mining operations, which generally involve the movement of large amounts of burden and rock massif to reach the desired product, often with several quarry fronts opened without success. Transportation costs are expensive because of the long distances to the processing or exporting facilities.

As for the final products' elaboration in Brazilian workshops, providing more technical information about the finished slabs properties would help improve the quality of natural stone applications and reduce costs.

The technical advances that currently allow the exploitation, processing, and use of rocky materials, unfeasible until the end of the last century, opened great market opportunities, especially concerning competitiveness against the ceramic industry.

The major bottleneck compared to ceramics, at least in Brazil, continues to be the proper dissemination of products' technical properties and consumer assistance.

**Acknowledgments:** The authors would like to express their acknowledge to Liliane de Lucca (Defruili Mármore) and Paulo Giafarov (DGG Stones) for the pictures.

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## How testing and quality assurance can make marble a durable option for exterior cladding

Björn Schouenborg<sup>1</sup>, Bent Grell<sup>2</sup>, David West<sup>3</sup> and Paola Blasi<sup>4</sup>

<sup>1</sup> RISE Research Institutes of Sweden, Built Environment, Box 857, SE-50115 Borås, Sweden

<sup>2</sup> Grell Consult, Borrebyvej 26, DK-2700 Brønshøj, Denmark

<sup>3</sup> Inhabit, 88 Phillip St., Sydney, NSW, 2000, Australia

<sup>4</sup> Arup, 8 Fitzroy Street London W1T 4BJ, United Kingdom

**Key words:** *Marble, Cladding, Quality assurance, Standardisation, Case study*

### Abstract:

Many prominent 20th Century buildings around the world were clad with marble. Some of them have experienced severe problems with rapidly deteriorating marble panels and accompanying safety issues, e.g. La Grande Arch de la Défense in Paris, Finlandia Hall in Helsinki, Australia's National Library in Canberra, and Amoco Oil Building in Chicago. The stories of these buildings have attracted great attention around the world, and perhaps unfairly, created a perception that marble is unsuitable for use as exterior cladding.

From the late 1990s, the use of marble as exterior cladding material reduced significantly, and it was even forbidden in some countries, regardless of reference projects. In response to this problem, the European research project TEAM was initiated. That comprehensive project on marble resulted in test methods for discrimination between marble suitable and unsuitable for outdoor use. The most important test is the 2013 European Standard EN16306.

One of the remaining obstacles for using marble, which this standard identifies as suitable for use, is ensuring traceability of its characteristics all the way from the quarry, through block production to the final panels installed on the building. With the rise of green building rating systems and CE Marking in Europe, there has been increased interest in documentation of the full life-cycle of building materials, and systems for ensuring traceability.

This presentation explores the potential for traceable systems to facilitate an integrated quality assurance during marble production. Such an approach would provide confidence that marble panels installed on a building are consistent with the samples originally tested and specified. The presentation will outline the detailed elements for quality control, from geological mapping of the quarry, through tracking of block extraction, to production of panels, and installation of the cladding. It will also provide information about some recent examples of marble façades.

# SMARTSTONE - BUILDING THE STONE OF THE FUTURE

I. Rondão<sup>1\*</sup>, V. Francisco<sup>1</sup>

(1) Technological Centre for Ceramic and Glass (CTCV), Coimbra, Portugal, \*[ines.rondao@ctcv.pt](mailto:ines.rondao@ctcv.pt)

**Summary:** *The Natural Stone sector currently faces challenges due to the constant emergence of new alternative products, often cheaper and with new responses to consumer needs. Complementarily, the rapid technological progress, the global trends towards sustainability, and the competitiveness of processes and products, in the national and international markets, constitute a framework of high demand for these products and companies to remain competitive. The work presented here aims to contribute to new and Innovative Natural Stone Products, differentiated and with added value. A perspective of what could become the products of Natural Stone in the medium- / long-term.*

**Keywords:** *Natural Stone, innovation, multifunctionality, added value*

## Introduction

In 2019, a total of 58.2 million tons of products derived from natural stone were produced in Portugal, corresponding to a production value of 809 million euros, according to data from the General Direction of Energy and Geology (DGEG). This amount is divided by different production sectors of mines and quarries (Figure 1) from the following sectors: metallic minerals, construction minerals, industrial minerals and ornamental.<sup>1</sup>

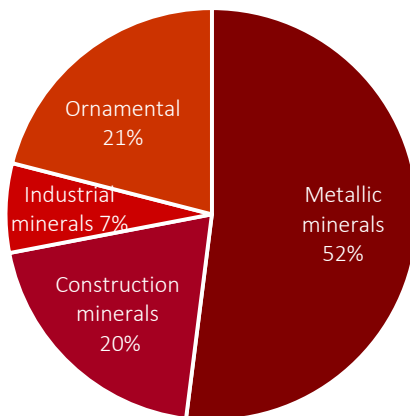


Figure 1- Weight of the different sectors of Portuguese mines and quarries in the total value produced in Portugal, in 2019 (Source: DGEG). Definitive data in 2019. Data referring to Portugal continental.<sup>1</sup>

The production and exploitation of Stone, historically, is essentially concentrated in two regions of the globe. In Europe, where Italy, Spain and Portugal stand out, and in Asia, where the two giants China and India prevail. These two regions are responsible for about 80% of the world's marble and granite production. However, countries like Turkey, Iran, and Brazil have been gaining market share.<sup>2</sup>

In Portugal, we can find deposits of the main types of ornamental stones: marble, granite, limestone and slate, scattered throughout the country. The sector that explores this type of stone is mostly made up of family-

owned companies, Micro, Small and Medium Enterprises (SMEs), according to data from INE. The activity of these industries results essentially in two types of saleable stone products, raw stone and processed stone.<sup>1</sup>

In the last years, there has been a growth in the volume of exports, largely due to the effort that has been made by the industries and associations in the sector to diversify markets, but also related with the increase in demand and improvement in the international market conjuncture.<sup>3</sup>

Simultaneous to the growth of the Stone Sector in Portugal, in the last years, there has also been an accentuated development of national and international competition, to which is added the rise of new competing products and constant technological advances. It is in this ecosystem that the Natural Stone Sector is challenged to gain sustainability and competitiveness.

The challenges that are currently imposed on the Natural Stone Sector include the constant appearance of new alternative products, rapid technological advances, and the global trend towards greater sustainability and competitiveness of processes and products in the national and international markets.

## Innovation in the Natural Stone

Innovation comes then as an essential tool to consolidate the degree of diffusion in the domestic and international markets. Innovation can be at the level of processes, with investment in new exploration technologies, or at the level of products, adding value through proposals of innovative design and/or giving them functionalities and disruptive applications that increase the range of uses, beyond the traditional ones.



The great technological advances in the Stone Sector in Portugal occurred with the insertion of diamond-cutting tools and the adoption of modern management practices in the early 1980s. Until then, the exploration of natural stone was made in small industries, in a typical artisan-family structure.

With the emergence of new tools, a mass production was possible to be started. However, in the late 80s, the precision requirements of these tools were too low, or even non-existent, to meet the tight tolerances demanded by the construction industry. Therefore, the first precision-cutting machines emerged, and those were already programmable to some degree. This advance allowed the development of integrated mass production lines.

At the beginning of the '90s, Computerized Control Systems and CNC (Computerized Numerical Control) technologies arrived. With this comes the Stone Machining Centers, which use innovative technology for plate processing, such as laser and water jet.

In the new millennium, an effort has been made by the companies to present diversified and differentiated products, with many custom-made works. The consumer is now more informed and demanding, the product requirements start to request technological advances. In this way technology will have to become more flexible and efficient in terms of sustainability and competitiveness, allowing to do better with less cost, without forgetting environmental and occupational safety issues.<sup>4</sup>

The industries of the sector have invested in putting on the market innovative products and disruptive approaches, namely by associating architects and other artists to reinterpret the Stone products. Examples of this are the works resulting from the "Primeira Pedra", a

project promoted by ASSIMAGRA. Some examples of the works produced are presented in Figure 2. Those are good examples of the incorporation of design in products, generally seen as traditional.

In addition, to the new design, technological development can also encompass new functionalities and even multifunctionalities. In this sense, it is necessary a greater articulation of companies with the scientific and technological system in order to develop technical skills, appropriated to the development of those new products.

### Multifunctional Stone Products

In the case of stone products, multifunctionality means that they have more functions than the usual ones: decorative, structural, cladding and flooring. To achieve multifunctionality, it is necessary to consider the intrinsic properties of stone, namely the fact that it is a material of natural origin that is not subject to processing, and where usually only some kind of surface finishes are applied. These are materials with high density, although variable, depending on the type of stone and genesis conditions, and with an equally variable chemical composition.

Therefore, multifunctionality cannot be achieved by modifying the overall composition or changing the production process, as it is done in other types of materials, once these parameters are determined by the type of stone. The path to multifunctionality is then through modification of the surface properties or by the association of other devices, which confer other characteristics (Table I).



Figure 2- Examples of works resulting from the "Primeira Pedra" project promoted by ASSIMAGRA.

Table I-Multifunctionality of Natural Stone, by modifying the surface characteristics or by incorporation of devices.

MULTIFUNCTIONALITY IN NATURAL STONE	
By surface modification	By incorporating devices
Impermeability	Integrated control systems
Anti-slip	Photovoltaic
Antifungal	Thermal
Self-cleaning	Backlighting
Anti-graffiti	Permeability
Photoluminescent	Sensors support
Relief tactile	

These are just a few examples of functions that can be added to Natural Stone, some of which already exist in the market, others exist for other classes of products such as ceramics, and can easily be adapted for the Natural Stone.

### Multifunctionality through surface modification

In general, surface treatments aim to protect and preserve natural stone products. In some of the functions mentioned in Table I, there are already solutions on the market, however, developments are needed in terms of adhesion, mechanical and physical stability, transparency and maintenance of surface color and brightness.

In many cases, the durability of these treatments is short and they are applied as a corrective and not preventive measure and may damage the stone surface. The developments in this area have been towards the creation of surface treatments with greater durability, of a non-organic matrix that can be applied in a preventive way, requiring a minimum of maintenance actions.

The stone, although a dense material, presents some porosity, which varies depending on its type. Many surface treatments are based on the elimination of open porosity, preventing the accumulation of dirt and debris in these places and facilitating the removal of dirt from the surface.

In addition to functional requirements, surface treatments must meet some fundamental criteria, such as good adhesion, mechanical and physical stability, transparency, colorless and not changing the shine of the surface.

Water is one of the agents responsible for the degradation of stone materials, which leads to the fact that one of the primary conservation treatments for these materials is protection against water penetration on the stone surface. The simplest form of this treatment is the application of a polymeric resin that covers the entire surface of the stone, creating a

homogeneous layer that covers the pores and roughness, preventing the water from coming in. Other properties can be associated with this coating, through the selection of resins with different characteristics, such as the anti-slip effect, or through the combination with other materials, such as titanium dioxide nanoparticles (TiO<sub>2</sub> NPs) adding hydrophobic and self-cleaning properties. In fact, these waterproofing films often serve as a substrate for other multifunctionalities of Stone products.<sup>5</sup>

The growth of microorganisms is one of the most common processes in the degradation of stone materials. Among these organisms, fungi have the greatest impact, causing changes in colour, composition and even morphology. The aim of antifungal treatment is essentially to inhibit the growth of these species by targeting their preferred growth sites. Nanoparticles, especially zinc oxide (ZnO NPs) and magnesium oxide (MgO NPs), are examples of solutions already studied for this purpose. Due to their small size, nanoparticles are a good solution to act on pores and cracks with small dimensions. There are also some studies with TiO<sub>2</sub> NPs, however, these need the incidence of light to activate the photocatalytic effect that causes the degradation of contaminants, and its application is limited to outdoor spaces. However, these self-cleaning coatings research is also going into finding alternatives or modifications to TiO<sub>2</sub> NPs for indoor spaces or spaces with little incident sunlight.<sup>6</sup>



Figure 3- Operation to remove graffiti from Stone buildings using low-pressure water and detergent or solvent.

The application of graffiti on cultural heritage or on unauthorized buildings can be considered an act of vandalism, which can result in damages to this heritage that are difficult to repair. To prevent the paints used in graffiti from adhering to the stone surface, compounds that give it anti-adhesive properties are used. Graffiti applied to surfaces with this type of treatment is easily removed with low-pressure water and detergent or solvent (Figure 3). There are products on the market that are based on aqueous solutions of fluoroalkylsiloxane, such as "Protectosil Antigraffiti" commercialized by Degussa, or organically modified

silica (ormosil). These products change the surface energy and hinder the adhesion of the paints, although another critical factor is roughness. Therefore, this aspect must be considered when selecting the type of stone to be used in places where it may be subject to vandalism.<sup>7,8</sup>

Another multifunctionality achieved by surface modification is the creation of photoluminescent surfaces. Those surfaces can work as a complementary lighting system to the installed one. The use of photoluminescence makes it possible to have lighting without installing any additional infrastructure to the flooring itself and with zero energy consumption, since photoluminescent materials retain sunlight, reflecting it at night and during low light hours. There are numerous works on the photoluminescence of materials and in the case of stone materials, this property can be achieved by applying a photoluminescent film on the stone surface.

#### **Multifunctionality by incorporating other devices**

The integration of devices in stone products, to turn them multifunctional, finds preferential application in Habitat products, meeting the requirements of an intelligent house, although there may be other applications outside this scope.

There is a wide variety of devices that can be applied to stone products. Some examples are sensors for movement, temperature, humidity, luminosity, noise, or even pollution. Besides adding value to the stone product, the implementation of these devices opens the door to new design proposals.

This way, it is possible to increase the degree of customization of the product, highly valued by today's consumer. Also, some of these solutions can result in energy savings, for example, dimming lighting systems. Motion sensors, often used in alarm systems, can be discreetly incorporated into the stone making them more difficult to detect.

A current trend is the integration of technology into physical objects and infrastructures. An example is the replacement of common switches by solutions integrated into the Stone wall covering, which involves not only the study of the function but also the methodology of application on-site, the interface with the user and the system for subsequent maintenance.

This functionality can be ensured through a capacitive sensor placed on the back of the stone. The integration of electronic components in stone would require compatibility with conventional construction procedures and a reliable application system is essential

to ensure that the products can be easily disassembled in case the system needs maintenance.<sup>9</sup>

Stone materials are applied in many outdoor spaces, where they are directly exposed to sunlight. The application of solar panels, not as an external device, but as an integral part of these surfaces, allowing them to maintain their aesthetic appearance, is the concept behind the concept of a photovoltaic stone.

The Tesla company is developing photovoltaic roofs, in which ceramic pieces are also photovoltaic panels (Figure 4). However, in the stone sector, few developments have been made in this field, largely because the current photovoltaic cells de-characterize the surface, as they do not allow the preservation of its aesthetic aspect, which is one of the added values of the Stone product.<sup>9</sup> The developments for this application, to be viable, should be in the sense of enabling better incorporation of these panels in the stone materials making their presence less noticeable, preferably without loss of electrical efficiency.



*Figure 4- Roof with photovoltaic panels integrate the surface of the roof tiles (Source: Electrical Trade Magazine).*

The radiant floor in interior spaces is not a novelty, the innovation is the application of radiant systems that allow heat parts used in urban furniture (benches) and in floors for the waiting areas of citizens in winter, such as public transport stops. The installation of these systems brings comfort to citizens and increases safety in the use of public areas, despite the climate. An example of this installation is installed in the city of Castellón (Spain) at the public transportation stop in front of the train station. Another recommended use is as thermal sidewalks in areas with a risk of ice formation. The intention is to heat the surface but not so much that the surrounding space can be heated since it is an open space and this heat will be lost to the atmosphere.<sup>9</sup>

Another multifunctionality is the backlighting of Stone surfaces, which can be a signalization alternative in public spaces such as subway stations, functioning as a

safety distance delimiter, for emergency signalling, among others. Backlighting requires the installation of a light source and its electrical circuit on the back of the stone must contain a hole or slot, for example with the design of emergency exit signs, which allows the light to exit. In the case of floor applications, it is essential to fill this opening with a transparent and impermeable material that prevents water and other debris from entering the lighting system.<sup>9</sup>

One problem in large urban centres is rainwater management, since most surfaces (streets and buildings) are impermeable, leading to difficulties in the drainage of these waters when precipitation reaches very high values. Given that many of these surfaces are covered with stone materials, it is imperative to re-evaluate their application and ways to avoid this problem. The solutions may involve creating systems or configurations that turns the stone more permeable, allowing water to infiltrate into the ground beneath it, or in more advanced systems, which in addition to incorporating permeable sidewalks, make it possible to reuse the water that passes through them, minimizing the consumption of potable water for applications such as watering urban gardens.<sup>9</sup>

## Conclusions

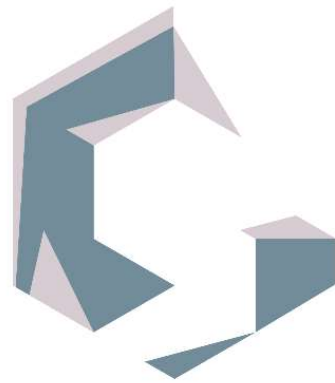
The continuous monitoring of market trends and technological advances is a crucial aspect currently faced by the Natural Stone sector. The integration of cutting-edge technologies to get innovative products, in terms of design and multifunctionality, adding value to them, is a strategic move to keep Natural Stone competitive in a market where alternative materials, with disruptive approaches for the same application, are appearing every day.

In this work, a survey of some possible multifunctionalities that can be conferred to Natural Stone was developed. Some of these already exist on the market but require further development before they can be used on a large scale. Others have been applied to other types of materials such as ceramics but can easily be transposed to natural stone. Nonetheless, there are many other multifunctionalities that will be interesting to integrate. In this field, the imagination and demands of the consumer will be the limit, buoyed by the technological advances of the moment.

**Acknowledgements:** The authors acknowledge the support of “Stone 4.0 Age” Project, promoted by ASSIMAGRA - Portuguese Association of the Mineral Resource Industry, with the support of COMPETE2020 – FEDER.

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**GLOBALSTONE**  
c o n g r e s s 2 0 2 3

**Tuesday, June 20<sup>th</sup>**  
**PRESENTATIONS**

# THE GEOGRAPHICAL INDICATION AS A SUCCESS INDICATOR IN THE SOCIOECONOMIC DEVELOPMENT INCENTIVE OF THE LOCAL PRODUCTIVE ARRANGEMENT IN THE ORNAMENTAL STONES SECTOR IN THE NORTHWEST REGION OF ESPIRITO SANTO

A.R.F. Custodio<sup>1\*</sup>, Q.C. Gomes<sup>2</sup>, D.O. Caverzan<sup>3</sup>, A.R. de Paula<sup>4</sup>

- (1) Angelo Roberto Fiorio Custodio, angelo.custodio@ifes.edu.br - NEPRO/IFES - Campus Barra de São Francisco\*
- (2) Quéren Coutinho Gomes – Scientific Initiation Scholarship Holder NEPRO/IFES/FAPES
- (3) Daniele Oliveira Caverzan – Scientific Initiation Scholarship Holder NEPRO/IFES/FAPES
- (4) Alexandre Ribeiro de Paula – Scientific Initiation Scholarship Holder NEPRO/IFES/FAPES

**Summary:** The research was carried out with the objective of verifying the real meaning of the Geographical Indication - GI Granites Northwest of Espírito Santo on the socioeconomic development of the Local Productive Arrangement - LPA of the Ornamental Stones sector in the northwest of Espírito Santo. For that, it was conceptualized what is a GI, before Brazilian and international law, how the ornamental stone sector was organized in Brazil, but especially in Espírito Santo, with emphasis on the northwest. Then, the Human Development Indexes of the northwest region of the state and its main cities were analyzed, so that, finally, the correlation between the elements presented could be traced. The research concludes that yes, the GI has the symbolic value of representing the entire history of investments in the LPA of Ornamental Stones, as long as it is embraced by the entire chain, a great difficulty to be overcome by those involved.

**Key words:** *Geographical Indication, Ornamental Stones, Development, Northwest, Espírito Santo.*

## 1 – INTRODUCTION

This article was developed in the context of the granting of the GI - Geographical Indication to Granites extracted from the northwest region of the state of Espírito Santo, located in the southeast region of Brazil, by the INPI – National Institute of Intellectual Property, in 2023, through the work of ANPO – Northwest Association of Ornamental Stone Producers in partnership with the Brazilian Micro and Small Business Support Service - Sebrae-ES and was motivated by the search for understanding the real meaning of this event. Therefore, the following problem was established: **What is the real meaning of granting the Geographical Indication to Granites in the Northwest of Espírito Santo on the socioeconomic development of the LPA - Local Productive Arrangement of the Ornamental Stones sector in the northwest of Espírito Santo?**

The justification behind this work is to analyze the trajectory of public and private investment in the sector of Ornamental Stones in Espírito Santo and draw a parallel with the history of the Human Development Indexes - HDI of the Northwest of Espírito Santo, since this region carries with it a history of having the lowest indexes in the state, at the same time that it concentrates a wide range of investments in the sector of extraction and processing of Ornamental Stones, being responsible for a large part of the exports of this product at national level and having great expressiveness in the GDP - Gross Domestic Product of Espírito Santo. Therefore, understanding this contrasting environment becomes a topic of paramount

importance for the academy, as well as representing a huge step in the geological, economic and social heritage of the people responsible for, in fact, making this sector what it is. This can be represented, symbolically, in the IG seal of origin granted to the sector.

In order to achieve the general objective of verifying the real meaning of the GI Granites Northwest of Espírito Santo on the socioeconomic development of the LPA in the Ornamental Stones sector in the northwest of Espírito Santo, it was necessary to overcome four specific objectives related to the central aspects of the problem. They are: Conceptualizing what Geographical Indications are; To analyze the socioeconomic development of the LPA of Ornamental Stones in the northwest of Espírito Santo; Analyze the history of Human Development Indexes in the northwest region of the state of Espírito Santo and, finally; Demonstrate the real meaning of a Geographical Indication concession on local development around a specific sector.

In order to carry out such a demonstration, it was necessary to collect historical data on the development of the entire northwest region, focusing on the main cities in the region, such as Colatina, Nova Venécia, Barra de São Francisco and Ecoporanga, among others, as well as on the ornamental stone sector in their late emergence in the region and to trace a correlation between these two elements in search of understanding about how these factors are related and their real meaning.

The main hypothesis of this article goes back to the assertion that investment in the ornamental stone sector had the role of reversing the historical trend of the HDI in the northwest region of Espírito Santo, while the secondary hypothesis states that the granting of a geographical indication seals the success of an investment policy in a specific sector.

It should be noted in advance that Espírito Santo is notoriously recognized for the production of marble and granite, however this research will be limited to the extraction of granites only, since the marble production areas are located in the southern region of the state, where highlights the city of Cachoeiro de Itapemirim. This limitation exists because the Geographical Indication is linked only to the production of granites extracted in the northwest region. However, it is not possible to analyze the history of the sector, without mentioning the extraction of marble in the south of Espírito Santo, where the sector, in fact, began in the state.

## 2 – GEOGRAPHICAL INDICATIONS

The legal framework for Geographical Indications in Brazil takes place on May 14, 1996, the day on which the Law nº 9.279 was enacted with the aim of regulating the rights and obligations relating to intellectual property in the Brazilian territory and deals, in its title IV, specifically with geographical indications. Its article 176 defines what a GI is: “constitutes a Geographical Indication the Indication of Origin or the Designation of Origin”, therefore presenting the two possible types of GI, hereinafter presented only as IO and DO (Law 9.279, 2023).

The IO's e DO's are specified in articles 177 and 178 of the same law, they are:

Art. 177. Indication of Origin - IO is considered to be the geographic name of a country, city, region or locality in its territory, which has become known as a center of extraction, production or manufacture of a certain product or provision of a certain service.

Art. 178. Denomination of Origin - DO is considered the geographic name of a country, city, region or locality in its territory, which designates a product or service whose qualities or characteristics are due exclusively or essentially to the geographic environment, including natural and human factors.(Law 9.279, 2023).

In the law, it is possible to perceive that a DO is a more complex point than an IP, since two crucial factors are considered beyond the mere quality of the product, natural and human, which must be closely related in a causal link. The law 9,279 also requires that, in the case

of a DO, the product or service undergoes a process of meeting quality requirements and also establishes the INPI as the body that will establish the conditions for registration of geographical indications in Brazil (Law 9.279, 2023).

In a quick concept, Gurgel (2005), places the Geographical Indication as a “territorial mark”, in addition to citing that historically the concept of a GI was already present in the 4th century BC, in Ancient Greece, with the trade of Corinthian wines , Icarus, Rhodes and also in the Roman Empire itself with Carrara Marble and Falerne wines. At this point, it can be seen that an ornamental stone is at the origin of the GI concept and carries with it all the weight of surviving for centuries recording the history of those who used it.

When dealing with French Wines and making a parallel with Brazilian Wines, both with their respective distinct Geographical Indications, the author Niederle (2011) does so by highlighting the commitment to quality as a central element of what may or may not receive the seal of approval of a GI. Something also advocated by Mafra (2008) who focuses on building a market, bringing to light the need for the product, in addition to its intrinsic qualities, to also be a product of value, which generates desire, which meets a demand and which moves the economy.

In this economic aspect, one cannot talk about development without bringing up concerns with the sustainability issue and at this point the GI's have a strategic and political role of great interest, as can be seen in the work of Velloso (2008) who brings us a direct correlation between Geographical Indication and sustainable territorial development. It is obvious that, in this case, the focus was not on the environmental aspect, but on the sustainable development of society as a whole, addressing qualitative aspects of the place, culture, history, tradition and environmental conditions that would be “imbued” in the final project, in this case the wines of the “Goethe” variety, produced based on “local know-how”, another characteristic element of a GI and more prominent in the DO processes, which emphasize the human aspect, which is active, when relating it with the natural elements, which are passive.

Another important point that Vellozo (2008) points out is that the GI's were not designed to develop a territory, since the product itself already existed and would continue to be produced with or without the recognition promoted by the GI and that the main issue , for the longevity of a GI, lies in the adherence of those who compose it, placing an extra weight on the human factor involved in granting and maintaining this seal. Therefore, it can be said that recognition can be given, but the “sustainability” factor, in this case, is linked to

the maintenance of the GI by the public entities that make up society and the market.

This point of view is shared by Maiorki and Dallabrida (2015) whose study points out that the granting of a GI can indeed be a possible vector for the development of territories and regions, but that this does not occur autonomously, but rather with the contribution of civil society and sectors of the economy that are part of the object of the Geographical Indication. That is, far beyond the natural aspect, the preponderant factor for the success of a GI is not in the product itself, in what nature gives us, but in what society is capable of doing with what we have been given. Hence the passive aspect of the natural factor and the active aspect of the human factor, present in the Causal Link required in a DO process. In this way, the political-social factor emerges as an essential element in understanding the real value of a GI over the mere product it represents.

The relationship between Geographical Indications and Sustainability is present in several other studies, such as Neiva et al. (2011) who see in the GI an opportunity to add value while preserving the Curraleiro cattle, raised by a Kalunga community, a quilombola remaining located in the state of Goiás; Dallabrida (2012) and the production of mate herb from native herbs in Brazil; Prado et al. (2022) who defends the social process in the construction of a GI for sustainable territorial development, as well as Gargur (2008) who openly defends the Geographical Indication as a tool for social inclusion.

The vast majority of products that are presented to the market with the seal of a GI are, generally, agricultural products, such as wines (Niederle, 2011), meats (Brandão et al., 2012), mate herb (Dallabrida, 2012), Palm oil (Conceição et al., 2021) and so many others. However, there are industrialized products with greater added value that also received this seal, among them we can mention footwear from Franca, São Paulo (Gurgel, 2005), footwear and leather goods produced in the Jacuípe-Bahia basin (Das Neves et al., 2019) or the furniture hub of Marco, in Ceará (Da Silva Florência et al., 2021). Even with such diversity, this is a relatively recent research topic and it is necessary to highlight the great potential yet to be explored by a GI, and to better understand its implications and limitations (Reis, 2016), but which can be resolved with due attention to the territorial governance, as shown by De Lima Fante and Dallabrida (2016). The human, political and social factor, once again, is essential.

With a view to the political aspect that governs the regulation of Brazilian GIs, these are internationally recognized, as they are in accordance with Decree No. 635, of August 21, 1992, which “Promulgates the Paris Convention for the Protection of Industrial Property,

revised in Stockholm on July 14, 1967”, where the Federative Republic of Brazil undertakes to fully follow what was established in this convention regarding intellectual property (Decree 635.1992). Add to this the agreement on aspects of Trade-Related Intellectual Property Rights (Decree 1,355, 1994) and the Brazilian market is aligned with international demands for free trade and defense of the origin of products traded between the signatory countries.

### **3 – THE ORNAMENTAL STONES SECTOR IN THE NORTHWEST OF ESPÍRITO SANTO**

Since ancient times, the most emblematic constructions made by human hand have used stones as a basic raw material, but it was with the Italians from the city of Carrara, in the region of Tuscany, whose capital is Florence, the cradle of Renaissance architecture, that the rock came to be treated as an ornamental element and even a work of art, and not just a structural element. Carrara marble has been famous since ancient Rome, when it was used to build the Pantheon. Many renaissance sculptures, such as David by Michelangelo were also carved from Carrara marble (Wikipedia: Carrara, 2023). Currently, Carrara Marble continues to be extracted and marketed worldwide as one of the most valuable ornamental stones in the world and its origin is protected by the same international laws that govern GI’s in Brazil.

Through the work of Frascá (2021) it is possible to cross the history of Brazilian dimension stones with the construction of Brasília. The construction of the new federal capital took place at an accelerated pace and after the shock of Oscar Niemeyer’s daring architectural lines, the second most notable element is the white marble coating. Still according to Frascá (2021) although many documents present the origin of the materials as being Rio de Janeiro, it is possible to verify that many of these materials were, in fact, from the city of Cachoeiro de Itapemirim, in the south of the state of Espírito Santo.

In 1957, the first block of marble from Espírito Santo was extracted, which, after being processed in Rio de Janeiro, was destined for an important scenario in Brazilian history, the construction of Brasília (A Tribuna, 2011, as quoted in Frascá, 2021).

Delighted with the marble from Cachoeiro de Itapemirim, Rio de Janeiro was, for a long time, the main buyer of our stones (...) from there, dozens of trucks would leave loaded with the “White Gold” of prosperity, already benefited, heading to Brasília (...) (Sabadini et al. 2018 as quoted in Frascá, 2021).

The same author also highlights the important maintenance work of these monumental constructions,



which are already over 60 years old, and the decision to use national materials greatly facilitates this work, “since the quarries of Cachoeiro remain active” (Frască, 2021).

In this way, the genesis of the exploitation of dimension stones in Espírito Santo was born together with the construction of Brasília in the late 1950s and entered the 1960s, when the sector was consolidated in the region of Cachoeiro de Itapemirim, with the exclusive extraction of marble. . This material, given its prestige for more than 50 years, has a Geographical Indication, restricted to the municipalities of Cachoeiro de Itapemirim and Vargem Alta.

It is important to highlight that at this time, little granite was used, as stated by Frasca (2021) in his article on the construction of Brasília, as used in “much smaller quantities”, because they were much denser and difficult to process.

The area that comprises the extraction of granites as Ornamental Stones occurs naturally in the northwest region of the State of Espírito Santo, having as a natural dividing line the coastal plains to the east, where there is no presence of the rocky pontoons characteristic of the northwest region of Espírito Santo and the course from Rio Doce, to the south. This watercourse also represents the southern limit of a large stone mass that comprises the entire region where these granites are naturally found. The predominant geological formation in this area is known as the Carlos Chagas Suite, which is the common origin of all granites in the region. In the Tectonic Map of the State of Espírito Santo, published by Vieira et. al. (2015) it is possible to notice this massif under the northwest region of ES, represented by the magenta color, predominant on the map.

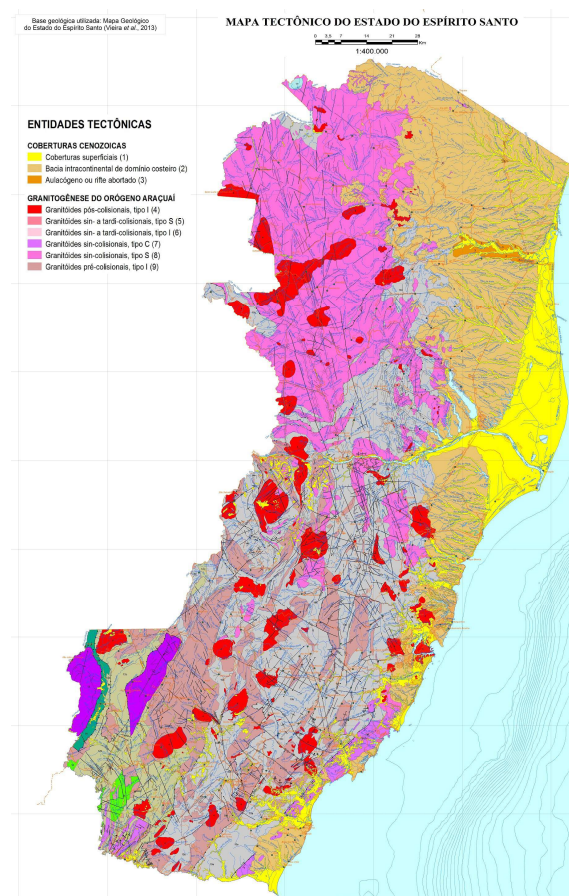


Fig. 1. Tectonic map of the state of Espírito Santo – Highlight for the concentration of granitoids in the northwest region, represented by the magenta color.

The sector of extraction and processing of Granites as ornamental stones only really started in the northwest region of Espírito Santo in the mid-1980s, with the advent of drilling and cutting tools using diamond materials, which raised the productivity of the sector to the level nowadays, where Espírito Santo stands out as the main supplier of ornamental stones for export. According to Findes (2020), Espírito Santo's participation in the export of ornamental stones went up from 79.9% in 2018 to 81.8% in 2019, evidencing the State's role in this economic segment, exporting its materials to 106 different countries and accounting alone for 10% of the GDP – Gross Domestic Product of Espírito Santo.

Exports from Espírito Santo registered an increase of 4.43% in revenue, equivalent to almost US\$ 828 million. Espírito Santo has the largest productive arrangement of stones in Brazil. There are around 1,600 companies in the State that generate more than 25,000 direct jobs and 100,000 indirect jobs and are present in 78 municipalities (Findes, 2020).

This is a thriving sector of great importance for the economic scenario of Espírito Santo and Brazil. According to Abirochas (2021) the main factors that are

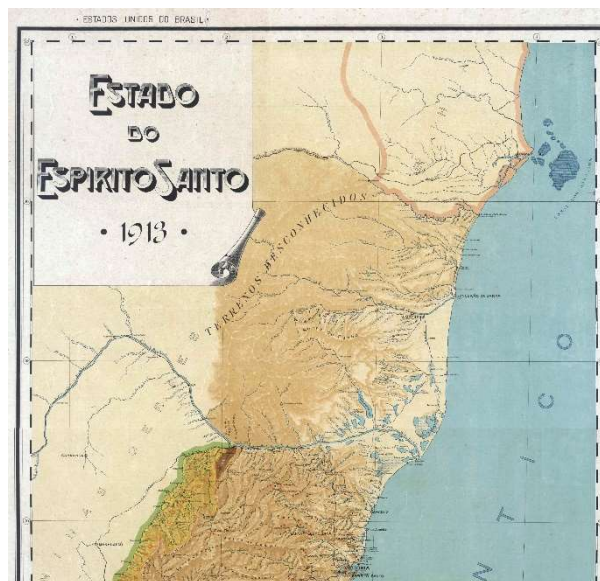
directly affecting this sector and will guide the future directions of the sector are: The consolidation of diamond wire technology, through multiwire looms and single wire machines, which increased the productivity of sawing polished and cutting sheets in the quarries and opens up margins for the sale of finished products; Changes in pace in the domestic and foreign markets, and variations in demand for sheet metal and finished products; The high cost and shortage of skilled labor and; The expansion of environmental and mining licensing requirements, and legal uncertainty.

These are specific technical factors, but they bring to light, once again, the importance of the human factor surrounding the development of this very important industry. The social value of the industry indirectly emerges in the report presented by Abirochas (2021) when it states that, given the policy that governs the sector, “mining activities migrate to areas of economic frontier, where the regulatory environment allows competitive survival”. This statement corroborates the hypothesis presented in this article, since the legislation and inspection itself force mining to seek to move away from the big centers, migrating to the interior, where the levels of development are lower and, that through the installation from these industries there is the possibility that, along with the mining industry, progress will also arrive.

#### **4 – ANALYSIS OF THE HUMAN DEVELOPMENT INDEX OF THE NORTHWEST REGION OF ESPIRITO SANTO**

Following the reasoning of this article, as well as the data already presented, it is important to analyze the region in which the process of extraction and processing of granite as an ornamental stone is most concentrated in the State of Espírito Santo, the Northwest region. According to ANPO (2023) there are 21 mining municipalities in the northwest region, organized in the Consortium of Mining Municipalities of the Northwest of Espírito Santo – COMMINES, comprising the entire region limited to the south by the River Doce and to the east by the coastal plains.

The history of the development of the state of Espírito Santo was strongly affected by the discovery of gold in the territory of Minas Gerais, since the former Captaincy of Espírito Santo was reduced to a small coastal strip, forbidden to develop for more than 200 years to protect the mines with the chain of mountains that naturally, until today, divide the two states. To the north of the Doce River, in the entire northwestern region of Espírito Santo, the Botocudos Indians lived, who were anthropophagous (Garbe, 2014) and that kept away any foreigner who ventured through those parts. An official map of Espírito Santo, dated 1913, shows this entire region as “uncharted terrain” (Mello, 1912).



*Fig. 2. Official map of Espírito Santo, Northwest region as “uncharted terrain” (Mello, 1912).*

In other words, development arrived late in the northwest of Espírito Santo. According to Araújo (2019), the region only began to be populated in 1927, when a group of farmers from Minas Gerais, Colatina and São Mateus, looking for vacant lands, settled at the confluence of the São Francisco and Itáunas rivers. However, in 1937, a territorial dispute between Minas Gerais and Espírito Santo, gave rise to an armed conflict that was only resolved in 1963 and the place became known as the Contestado Region.

The consequences of this troubled history delayed the socioeconomic development of the area, which is reflected in the Human Development Indexes of the Region.

For Lorena and Leite (2011), when analyzing the Municipal Human Development Index – IDHM of the state of Espírito Santo, referring to the year 2000, it was possible to verify that the worst indices are concentrated in the northwest, which is basically an agricultural region, which has been suffering from the effects of the desertification process, already advanced in some areas, which contributes to the reduction of production and consequently to the low development of the municipalities and the low income of the region.

When analyzing the public data from the IBGE - Brazilian Institute of Geography and Statistics on the HDI data of some cities in the northwest region, it is possible to verify that the values start considerably low in 1991 and suffer a significant increase over time, mainly in the indices in the city of Barra de São Francisco, which increased by 70.75% between 1991 and 2010 (IBGE, 2023).

CIDADE	IDH	ANO
Colatina	0,546	1991
	0,657	2000
	0,746	2010
Nova Venécia	0,459	1991
	0,627	2000
	0,712	2010
Barra de São Francisco	0,4	1991
	0,565	2000
	0,683	2010
São Domingos do Norte	0,445	1991
	0,562	2000
	0,682	2010
Ecoporanga	0,397	1991
	0,542	2000
	0,662	2010

Tabela. 1. Série histórica – IDH/Município (IBGE, 2023)

The second highest variation was in the city of Ecoporanga, which varied 66.75% between 1991 and 2010, followed by Nova Venécia, with 55.12%, São Domingos do Norte with 53.26% and with the lowest variation Colatina, with 36, 63%. The choice to analyze these municipalities was due to the importance of the city in the mining sector and by reference to the city of Colatina, which, due to its location, on the banks of the Rio Doce, is the largest city in the region and also the closest to the capital. In contrast, the city of Ecoporanga is the furthest away and was not the one that presented the greatest variation, that is, the highest index of improvement in the HDI, but the city of Barra de São Francisco, considered the nerve center of the ornamental stone trade today.

## 5 – CORRELATION BETWEEN HDI AND GI IN THE ORNAMENTAL STONE SECTOR

Considering what has been exposed so far in this study, it appears that there are signs of a positive correlation between the development of the Dimension Stone mining sector and the Development indexes of the municipalities in the northwest region.

This is a region with elements of delay in its historical development, from the “uncharted terrain” in 1913, to an armed conflict over territory, which was only pacified in 1963, and years after the inauguration of Brasília and the consolidation of Cachoeiro de Itapemirim as a marble production hub in the south of the state.

According to ANPO (2023), the first company regulated in the extraction of dimension stones in the northwest region only took place in 1987. Therefore, it can be said that its installation should not have a significant effect on the HDI measured in 1991, and it is then possible to consider this marks as a reliable parameter for a

reference of a municipality without any type of relevant industrialization.

It is therefore verified that the investment in the extraction of granites in the region accompanies the data collected on the HDI and the consequent formation of a network of suppliers and service providers that will give rise to the complex of the Local Productive Arrangement of Ornamental Stones, which in the following decades will be the target of actions and public policies that will make the sector develop and consolidate.

It is important to highlight that the “notion of a production chain comes from a political choice” (Gurgel, 2005). This happened, in fact, with the marble and granite sector, with the maturity of the sector and its adaptation to the market, with the migration or expansion of several companies from the Cachoeiro region to the northwest region, seeking to include granite in their product portfolio.

This influx of companies, capital and investments also forced the labor market to adapt by attracting and retaining increasingly qualified professionals to occupy the positions demanded by the sector, as well as the indirect services that local commerce now offers to adapt to the demands of the new residents. This movement was noted by civil and public entities that began to include the sector in a set of actions aimed at the regulation and development of the industry, such as the state and municipal governments and autarchies such as the Federal Institute of Espírito Santo - IFES, Findes and Sebrae -ES.

Macedo (2011) in his study on the inclusion of mining activity in the Municipal Master Plans - MMP, focusing on the dimension stone extraction industry in the northwest of the state of Espírito Santo, concludes that the planning of mining activity and its insertion in the MMP of small municipalities demographic, where mining is an important factor in generating employment and income, is a fundamental tool for territorial organization and sustainable local development. The author also states that public policy actions by the federal government, through the Ministry of Mines and Energy, combined with actions by local public authorities and the productive sector, show the possibility of tracing paths to improve living conditions, transforming a in a process of building an effectively sustainable development model for the region.

Given this direct relationship between the public sector, private investment and socioeconomic development in the region, verified in the data presented, it can be said that the consolidation of this policy will in fact be endorsed by the representation of the seal that registers the value of the origin of the granites extracted from the

Northwest Region of Espírito Santo in the face of all global trade.

## CONCLUSION

This article was made with the objective of verifying the real meaning of the GI Granitos Noroeste do Espírito Santo on the socioeconomic development of the LPA of the Ornamental Stones sector in the northwest of Espírito Santo and based on the data presented, it can be stated, with certainty, that this seal represents the apex of a set of efforts and investments to consolidate a productive sector that had a direct positive effect on the Human Development Indexes in the municipalities that have mining as an important part of their management strategy.

It can be said that, yes, historically the entire Northwest region was in a situation of backwardness and low level of attention by the public power, as well as in attracting investments to the region. Even with the arrival of the coffee industry in the late 1920s, these levels did not improve significantly until the 1990s, when the granite dimension stone extraction sector was definitively established in the region and more markedly after the 2000s with the arrival of cutting technologies based on diamond tools.

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There is enough data to state that investment in an LPA of Ornamental Stones has the power to reverse the rural exodus, bringing work, employment and development to the interior and, consequently, improving the lives of those who live in these regions. This is a viable and interesting strategy for the public authorities in other Brazilian states where mining is also present and which also have characteristics for the creation of new dimension stone GIs.

Finally, it is possible to state that the adoption of the Geographical Indication, based on a Denomination of Origin process, concentrates the real human value on the riches that nature offers to society and that it has the potential to take all these elements as a complement to the products already marketed, giving them another layer of differentiation for a market as competitive as the current one.

It is a fact that this process of adopting a GI still has a series of difficulties to be overcome, among them, in the first place, the cultural factor, where the success of this initiative will only be achieved with the adoption of all those who make up this chain. It will take a huge effort to convince entrepreneurs and commercial sectors to adopt and incorporate this value into their products. A GI can lose all its value if there is no commitment from the parties.

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# PORTUGUESE LIMESTONES AND CULTURAL HERITAGE: APPLICATIONS AND PATTERNS OF DEGRADATION THROUGH TIME

A.G. Costa

Labtecrochas-CPMTC – Federal University of Minas Gerais (UFMG), Belo Horizonte, Brazil, ag.costa@uol.com.br

**Summary:** *Of the Portuguese limestones applied in cultural heritage buildings, but also in contemporary ones, there are types extracted in the vicinity of Lisbon, which historically were exported to countries, such as Brazil. Extracted in old quarries, located mainly in the region involving the sites of Pêro Martins, Negrais, Men Martins and Sintra, in Portugal, it is observed that they are almost completely deactivated and without production, having been, in part, replaced by other new ones. From this set of limestones, those types historically identified as Lioz and others stand out, such as the Negro de Mem Martins and the Azul de Sintra, the latter presenting characteristics of a marble. These limestones, with the longest and most continuous periods of application, both in Portugal and in Brazil, will be treated in the present work observing this chronology of applications and considering their most typical degradations over that time.*

**Key words:** *lioiz, limestone, types, application, degradation*

Outcropping in the region of Lisbon, these limestones were historically applied in buildings of this city and, certainly, it was from there that they were transported for applications in Brazil, especially during the period when the Brazilian territory belonged to Portugal, from colonial times until its status as a United Kingdom with Portugal and Algarves.

Extracted in old quarries (Fig. 1), in the District of Lisbon, Council of Sintra (Aires Barros, 2001) and

involving the sites of Pêro Martins, Negrais and Sintra, it can be said, from a petrogenetic point of view, that the different types of Lioz are considered to result from geological processes which date back to the Cretaceous (Casal Moura et al., 2007; Lopes, 2015) and according to these authors they were formed in a shallow sea environment, warm and clear waters, leading to the proliferation of carbonated skeleton of organisms and coral banks.



Fig. 1. Old and contemporary limestone quarries of the Lioz type, located in the District of Lisbon (Photo: Antônio Gilberto Costa).

From a petrographic point of view, it can be said that Lioz in general can be identified as microcrystalline and hard limestone, and characterized as very compact, with relatively low porosity. Calcitic in composition, though with some content of clay minerals, a part of

them is characterized by the presence of rudist fossils (Fig. 2a). In general, all these limestones are calciclastic and bioclastic rocks, and may contain styliolites (Fig. 2b) and be very sparitized (Casal Moura et al., 2007).



(a)



(b)

Fig. 2. Photographs of different types of Lioz limestones, presenting: (a) somatofossil contents of rudist bivalve shells in different cuts; and (b) styliolitic structures (Photo: Antônio Gilberto Costa).

Another striking feature of these limestones is the extensive chromatic variation they can present (Fig. 3), ranging from whitish tones to cream or beige (most frequent *Lioz*), sometimes pink with salmon pink venules, such as *Encarnadão de Lameiras*, or with thick elements and areas or pink lines, ranging to the red (*Encarnadão*). Some, such as the *Encarnadão Chainette*, bear a purplish hue with yellowish spots, whereas others are red with white or yellow spots, up until the golden-yellow limestone (*Amarelo Negrais*). In the case of the red variations, some present yellow-orange spots, such as the *Pedra Furada Encarnadão*. In this case, it is one which corresponds to a variation in terms of facies in relation to yellow, and whose extraction took place in the Montelavar Parish, in the Sintra Municipality and Lisbon District, in areas very close to those where the yellow type was extracted.

According Costa (2022), in Portugal and Brazil, examples of constructions with applications of different types of *Lioz* limestones (*Lioz*, *Chainette*, *Encarnadão*, *Amarelo de Negrais*) are abundant. Regarding the Portuguese case, the period of their constructions covers an extensive time interval, encompassing the 20th century, as in the cases of coatings in Lisbon subway stations and in the construction of the Belém Cultural Center in 1992, where varieties of *Lioz* were more frequently used – more specifically those identified as *Lioz Abancado*, also known as *Lioz* of Pero Pinheiro. In the Brazilian case, the oldest applications date back to the 18th century, despite also reaching the 20th with some

examples of internal coatings of yellowish, reddish and whitish *Lioz* varieties, as in the case of the Museum of Modern Art of Belo Horizonte, from 1943, and the 1947 Gustavo Capanema Palace, located in the city of Rio de Janeiro.

From the collection of stones applied in Cultural Heritage elements in Brazil and Portugal, materials of limestone composition stand out. Among them, there are the varieties of *Lioz*, described above and which correspond to the most applied in both countries (Silva, 2007; Costa, 2009).

From the set of important Portuguese monuments (Fig. 4a), some of the oldest, such as the Cathedral of Lisbon (built from the 12th century and partly with the use of clear *Lioz* limestones) present a construction history of many centuries, whereas others, such as the Jerónimos Monastery, the *Nossa Senhora de Loreto* Church, from 1518, and the Tower of Belém, from 1519, located in the region of Lisbon, had their constructions started and completed in the first years of the 16th century, and can even be considered contemporary with most part of the Brazilian history. The Jerónimos Monastery, for example, whose construction started around the year of 1501, has a predominance, among its limestones, of the whitish, fossiliferous or not, sometimes with stylotites *Lioz* types. In the building, there are combinations involving types such as *Encarnadão* and *Amarelo Negrais*, but also the *Azul de Sintra*, which is not a *Lioz* type, on floor coatings or as ornamental details. Constructed later, mainly throughout the 18th and 19th centuries, Portuguese

buildings such as the former *Convento de Mafra* Palace, built in the early 18th century and using limestones from the regions of Sintra and Pêro Pinheiro, such as the *Amarelo Negrais*, the *Encarnadão Chainette*, the Saint Florian Pink, the *Lióz Azulino*, but also the *Negro de Mem Martins*, which is also not a *Lioz* type; or the *Basilica da Estrela*, from 1790, which has a *façade* in

*Lioz*, panels in *Amarelo Negrais* and columns in *Encarnadão*; or the *Santo Domingos* Church, with limestone columns from 1834 and the *D. Maria II* National Theater, from 1846, can be considered contemporary in relation to Brazilian buildings, in which *Lioz* was also applied.



Fig. 3. Photographs of different types of *Lioz* limestones applied in buildings in Brazil and Portugal, presenting extensive chromatic variations (Photo: Antônio Gilberto Costa).

As examples of these contemporary Brazilian buildings (Fig. 4b), the following can be mentioned: the Jesuit Church of Salvador (Head Church from 1765), which has *façade* coverings in white *Lioz* and external floors in yellow, cream and red types; the *Conceição da Praia* Church, built in Salvador between 1739 and 1765 and featuring a frontispiece also in *Lioz* cream; the *São Bento* Monastery of Rio de Janeiro, built throughout the 18th century and with internal floors with beautiful compositions of different *Lioz* types; as well as the *Santa Isabel* Theater in Recife, of 1850 and with *Lioz* on the *façade* and its columns; and also the *Paço Real* of Rio de Janeiro. The *Paço* of Rio de Janeiro, which was the seat of government and residence of the Rio de Janeiro Captaincy governor between 1743 and 1763, and of the Viceroy of Brazil until March 8, 1808, has, in addition to structural blocks and applications of *Lioz* ornamentals, yellowish, reddish and grayish varieties, which were used to coat their floors.

Already exposed for long periods, and as a result of extrinsic factors such as humidity, climatic conditions and pollution, as well as intrinsic factors such as their own compositions, both in the aforementioned Portuguese and Brazilian buildings, there occur degradation degrees as a consequence of processes in these limestones, namely those of erosive character (Rodrigues, 2007). According to the nomenclature proposed by the International Council on Monuments and Sites (ICOMOS), there is an extensive loss of matrix and differential erosion, which indicates the reduction of surfaces controlled by the presence of fossil elements, carbonate resistance and the presence of secondary minerals (Vergès-Belmin *et al.*, 2008). Usually applied in floor and wall coatings, indoors or outdoors, they may present, in addition to the loss of their original surface, the loss of parts due to the presence of clays, for example, as in cases of applications both in floor covering and in walls subjected to conditions of greater humidity (Costa, 2021).





(a)



(b)

Fig. 4. Portuguese and Brazilian monuments that stand out for the use of Lioz-type limestone rocks. (a) from left to right: Mafra Palace in Mafra; Basílica da Estrela in Lisbon; and Jerónimos Monastery in Lisbon; (b) from left to right: Conceição da Praia Church of Salvador; São Bento Monastery of Rio de Janeiro; and Santa Isabel Theater in Recife (Photo: Antônio Gilberto Costa).

In this study, historical and contemporary applications, mainly involving different types of *Lioz* were considered, aiming to evaluate the influences of

processes over degradation degrees in these limestones, as in the cases, for example, of the Cathedral of Salvador and the Jerónimos Monastery, in Lisbon (Fig. 5).



Fig. 5. Photographs, on the left, of the external floor of the Cathedral of Salvador, state of Bahia, Brazil; and, on the right, of the internal floor of the Jerónimos Monastery, in Lisbon, Portugal. Limestone pieces presenting chromatic variation, the presence of fossils, loss of matrix and differential erosion (Photo: Antônio Gilberto Costa).

To illustrate problems involving internal applications, examples such as the internal floor and wall covering inside the *Basílica da Estrela*, in Lisbon (Fig. 6a), were

considered. In another application, in Brazil, the use and degradation of the *Amarelo Negrais* limestone was considered. Applied in the 1940s decade, on internal

floors of the Museum of Modern Art, in Belo Horizonte (Fig. 6b), the rock is also partly reddish or red, associated with yellow, and presents differential erosion and loss of matrix.

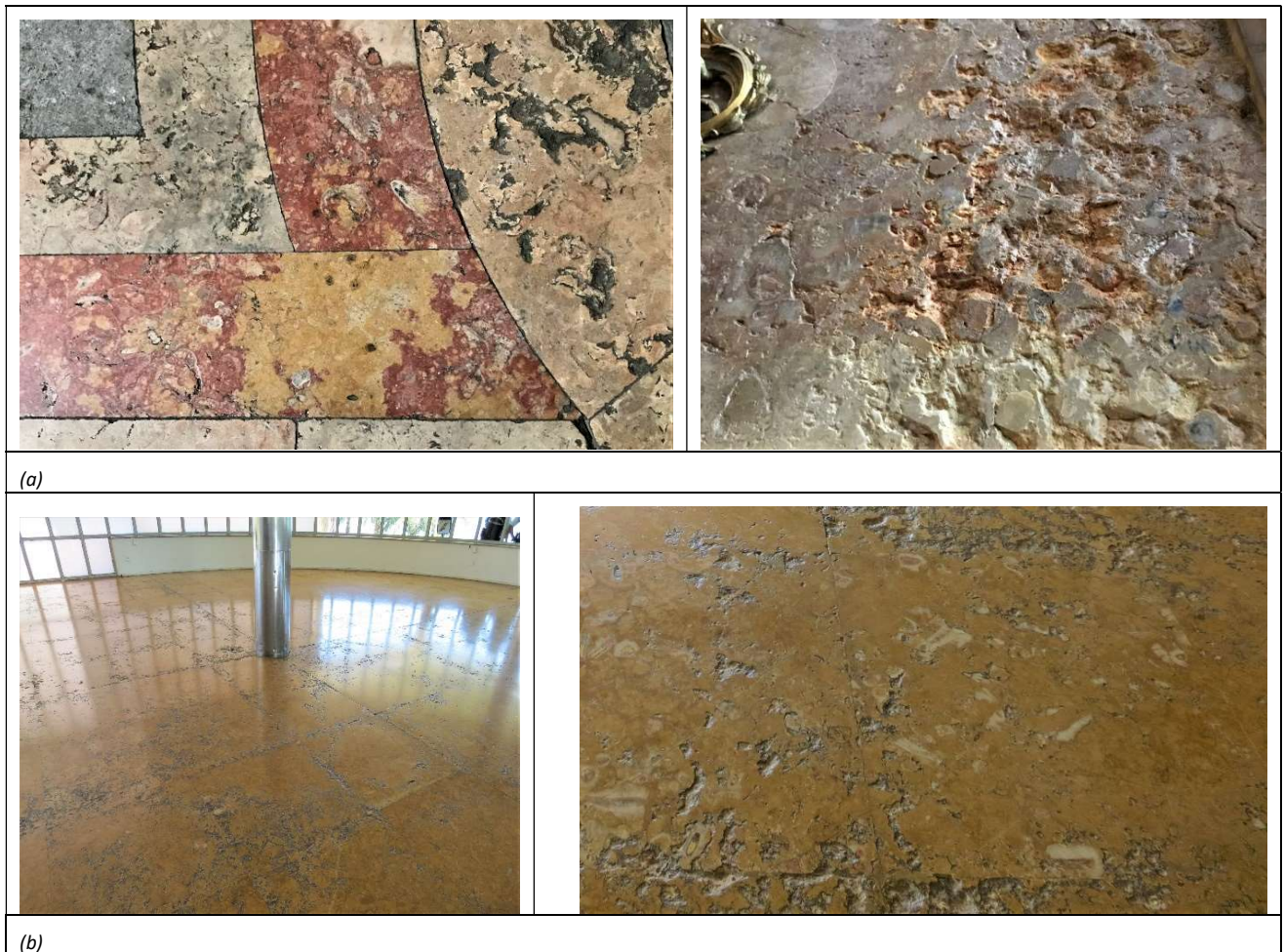


Fig. 6. Photographs (a) of internal floor and wall covering (right) applications of the Lioz inside the Basilica da Estrela, in Lisbon, Portugal, and (b) of internal floor of the Museum of Modern Art, in Belo Horizonte, Brazil. The rocks present chromatic variations, presence of fossils, differential erosion, and loss of matrix (Photo: Antônio Gilberto Costa).

#### Acknowledgments:

To LABTECROCHAS-CPMTC/UFMG for financial support for research activities and participation in Global Stone 2023 activities.

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# A study of the stone landscape technology in Suzhou's Canglang Pavilion represented by Taihu stone and Yellowstone

Jie Wu<sup>1,2\*</sup>, Yuege Ren<sup>1</sup>, António Candeias<sup>2</sup>, Xinyu Yang<sup>1</sup>, Haoran Wu<sup>1</sup>, Minmin Xu<sup>3</sup>

(15) School of Architecture, Soochow University, \*jwu@suda.edu.cn

(16) HERCULES Laboratory, Institute for Advanced Studies and Research, Évora University, Évora, Portugal

(17) Suzhou Key Laboratory of Novel Semiconductor-optoelectronics Materials and Devices, Soochow University

**Summary:** *The Canglang Pavilion is the oldest surviving garden in Jiangnan. It was built in the Song Dynasty, it has been restored and rebuilt many times, and there are still many details to appreciate about its long history and distinctive rock and mountain gardening techniques. This paper intends to study the design ideas and characteristics of the mountain and stone landscape of Canglang Pavilion through a combination of historical documentary analysis and field research sampling. The landscape art of Canglang Pavilion is analysed in terms of spatial patterns and the physical characteristics of the rock materials, detailing the regional adaptations and cultural representations of Taihu stone and Yellowstone in the garden. The layout, material characteristics and cultural features of the Taihu stone and Yellowstone in the Canglang Pavilion are discussed in the context of the eclectic garden layout and ingenious construction techniques of the Jiangnan gardens since the Song Dynasty.*

**Key words:** *the Canglang Pavilion, Taihu stone, Yellowstone, classical gardens, stacked stones*

## 1. Introduction

The stacking of hills and stones is one of the key gardening techniques in Chinese gardens. In gardens, hills and stones can form the main landscape of the garden; divide and organize the garden space; arrange courtyards, revetment, berms and retainers; and also set up naturalistic flower platforms. Mountains and rocks are combined with garden buildings, paths and plants to form a varied landscape to vigour natural interest and to integrate garden buildings into the landscape environment<sup>[1]</sup>. In Chinese gardens, no matter how big or small, there are almost always rocks in every garden.

In classical Chinese gardens, mountains and rocks were used to imitate natural landscapes, and the artificial way of creating landscapes with rocks went through several stages from the Han Dynasty to the Qing Dynasty. From the Song to the Ming dynasties, the technology of landscaping reached unprecedented heights. In order to build the Genye, Emperor Huizong of the Northern Song Dynasty set up the Yingfeng Bureau in Suzhou, collecting the flowers, trees and stones from Jiangsu and Zhejiang and transporting them to Kaifeng City in batches, with ten ships as a platform, known as Huashigang. The gardens of China, as represented by the gardens of Suzhou, have taken the technique of landscape gardening to the extreme, perfectly blending culture, art and natural beauty.

Stone-related theories form an important part of classical gardens. For example, *The Stone Book Of Yunlin*, written by Wan Du in the Song Dynasty, was the first monograph on stones in China. The 116 species of famous stones are discussed, and their origins are examined in detail, as well as their respective characteristics in terms of method of extraction, shape, colour, texture, sound when struck, hardness, lustre, crystal form, transparency, moisture absorption, and use<sup>[2]</sup>. *The Stone Book Of The Suyuan* by Youlin Lin of the Ming Dynasty provides a comprehensive account of the types of stone used in classical gardens and their characteristics<sup>[3]</sup>. *The Yuanye* by Cheng Ji of the Ming Dynasty contains a special chapter on the collection of hills and the selection of stones. The essence of the book is to map the land, mountain decoration and borrow scenery, and the record of Yellowstone appeared for the first time<sup>[4]</sup>. *The Superfluous Things* by Zhenheng Wen of the Ming Dynasty states that 'water and stones in gardens are the most indispensable'<sup>[5]</sup> and The Chapter on stones in the late Ming and early Qing dynasties in Yu Li's *The Leisurely Sentiments* describes the characteristics and layout of five types of stones such as stone walls, stone caves and small scattered stones<sup>[6]</sup>. In the modern day Dunzhen Liu 's *The Classical Gardens of Suzhou* describes the three parts of a stacked mountain, namely materials, general arrangement in detail and detailed techniques, giving the reader a more in-depth understanding of the creation of stone mountains, thus showing the importance of rocks in gardens<sup>[7]</sup>. In 1993,

Hongxun Yang published a chapter on the construction of hills and stacked rocks in his *Treatise On Gardens In Jiangnan*, detailing the creation of earthen and stone hills in gardens<sup>[8]</sup>. Mr Zhaozhen Meng's *A Brief Knowledge Of Rockery* discusses the stylistic types of classical rock-picking techniques<sup>[9]</sup>. Mr Xun Cao conducted an in-depth study of the life and techniques of some famous rock-pickers and Mr Jianzheng Sun's book *Rockery in Ancient Architecture* provides a more comprehensive study of rock-picking and mountain-moving techniques<sup>[10]</sup>. The design of classical Chinese gardens, whether royal gardens or private gardens or temple gardens, is inseparable from the use of stones and mountains, and this is also the case in Suzhou.

The Canglang Pavilion is the oldest garden in Suzhou, and most of the research on it has focused on the creation of a view, such as the theme of "turning reality into emptiness" and "Canglang", and has elaborated on its intention through the piling of hills and water, architectural layout and planting. The study of stone scenery, as an example, has been less well researched. The focus of this paper includes the development and evolution of the artistic characteristics of stone scenery in the Canglang Pavilion, and summarises the spatial layout and mood-building of Taihu and Yellowstone scenery in the gardens of Jiangnan.

## 2. Overview of the Canglang Pavilion

### 2.1 History of the construction of the Canglang Pavilion in Suzhou

The Canglang Pavilion is located on Canglang Pavilion Street, Sanyuanfang, in the southern part of the old city of Suzhou, and is the oldest of the surviving classical gardens in Suzhou. The construction history of the Canglang Pavilion is very long and can be divided into five stages: the Song Dynasty, the Yuan Dynasty, the Ming Dynasty, the Qing Dynasty and the post-Republic of China.

The Canglang Pavilion was built from the time Su Shunqin bought land and water stones in the Northern Song Dynasty, and has undergone the development of private gardens (Song Dynasty) - temple gardens (Yuan Dynasty) - government gardens (Ming and Qing Dynasty) - public gardens (after the Republic of China). Since the Qing Dynasty, the Canglang Pavilion has been rebuilt by successive local officials, and the style of "Chongfu Guangshui, not similar to the city" at the beginning of the founding of the People's Republic has not changed. With the idea of "Canglang", to pursue the virtue and spirit of Shunqing Su, the benchmark of famous men can be long shown to future generations, which is one of the models and representatives of many classical gardens in Suzhou and even China. In 2000, Canglang Pavilion was

inscribed on the UNESCO World Heritage List as an addition to the Suzhou Classical Gardens World Heritage Site.

### 2.2 The Art of Gardening at Canglang Pavilion, Suzhou

The Canglang Pavilion is a unique piece of gardening art, with a pool of green water surrounding the garden before you even enter the gate. The main feature of the Canglang Pavilion is the rocky landscape, and as you enter through the entrance you are greeted by a rocky hill on which the Canglang Stone Pavilion is built. Underneath the hill is a pond, and the hill is connected to the water by a double corridor. The Mingdao Hall in the south-east of the rockery is one of the main buildings in the garden, along with the Five Hundred Famous Sages Ancestral Hall, the Watch Hill Building and the Cui Linglong Pavilion (Fig. 1).

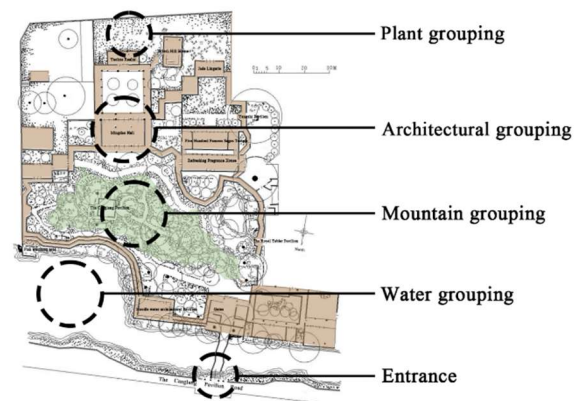


Fig. 1 General layout of the Canglang Pavilion

From the perspective of its artistic approach, one important feature of the spatial pattern is that the landscape comes first, followed by the architecture. Starting from the entrance and running north to south, the axis consists of the water group, the mountain group, the building group and the plant group. The water group includes the river and the revetment landscape along the waterfront, with Yellowstone being used in the stone scenery. The mountain group is dominated by Taihu stones, which are an important part of the garden. The architectural group is built around the mountain, with buildings such as Mingdao Hall, the Five Hundred Famous Sages Ancestral Hall, Qingxiang Hall, Cuihenglong and Guanshan Building, all facing the mountain and closely linked to the rocks. The plant group is the deepest group, creating a quiet, profound and tranquil atmosphere<sup>[11]</sup>.

## 3. Application of Taihu stone in the Canglang Pavilion

### 3.1 The artistic characteristics of Taihu stone

#### 3.1.1 Natural features

Taihu stone, also known as rockery stone and vault stone, is a limestone formed by slow erosion and can be

divided into two types: "water stone" and "dry stone". The "water stone" and the "dry stone" are taken from underwater in Taihu Lake and from the cliffs, respectively. The "water stone" is smoother and rounder than the "dry stone" [12].

Taihu stones form a unique shape due to the erosion of water and air, with self-generated holes and a rich variety of contours, with exquisite and transparent features and different forms [13].

The natural characteristics of Taihu stones include both beauty of form and colour. The traditional appreciation of stones is based on the criteria of "thinness, transparency, leakage and wrinkles" proposed by Fu Mi in the Song Dynasty, which reflects the beauty of the shape of Taihu stones. The term "thin" refers to the beauty of the form of a Taihu stone, with its slender lines in appearance. "Transparent" refers to the delicate texture of a Taihu stone, with its exquisite holes. "Leaky" refers to the naturally formed gaps in the stone, which are connected to each other and to the left and right, and are characterized by strong connectivity and irregularity. "Wrinkled" refers to the varied surface texture of the stone. This unique characteristic brought different visual experiences to the literati and formed different aesthetic concepts. Juji Bai of the Tang Dynasty described it in *Stones in Taihu lake* as follows: "Three mountains, five mountains, a hundred caves and a thousand ravines, all in one single strand." This shows that Taihu stones had an all-encompassing natural character in the eyes of the literati [14]. In addition to its shape, the colour of the stone was also appreciated by the literati, and was often a light and elegant greenish-grey, as well as white and greenish-black, which would demonstrate harmonious and natural with other colours in the garden.

In gardens, Taihu stones are treated by two techniques: 'placing stones' and 'collecting hills', for single stones and fragmented stones respectively. A good example of this is the Guan Yunfeng in the Liuyuan Garden in Suzhou, and a good example of the 'mountain' is the Four Seasons rockery in the Yangzhou Garden.

### 3.1.2 Cultural features

During the Qin and Han dynasties, the spread of the idea of the immortals inspired people to pursue the immortal world, and the immortal mountains depicted in the portraits were very similar to the Taihu Lake stones. From the Eastern Jin dynasty to the Tang dynasty, stone appreciation gradually flourished and people became interested in the peculiarly shaped Taihu stones. During the Northern Song Dynasty, Emperor Huizong organized the collection of the Taihu stones and other exotic stones from the south of the Yangtze River, and built the royal garden of Genyue, with an area at the entrance

where the Taihu stones were placed in groups. At the same time, Taihu stones began to appear in large numbers in paintings, such as Hanchen Su's *Autumn Garden with Infants* [15] and were used in gardens during this period, both in isolation and in groups. The rise of the style of mountain stacking and the painter's reproduction of landscape painting has a close link, Taihu Lake stone because of its varied form and more as the "stacked stone for the hole" of choice material, favoured by the gardeners. The famous garden builder Ji Cheng in the "Garden Ye" described in detail about the Taihu Lake stone stacked hole approach. In short, since the Yuan Dynasty, the stacking of stones for the mountain has enriched the expression of Taihu stone, and also reflects the development of garden stacking techniques and the flourishing of landscape painting.

## 3.2 The landscape art of Taihu stone in the Canglang Pavilion

### 3.2.1 Layout features of the Taihu Stone

The Canglang Pavilion is one of the oldest gardens in Suzhou, and the application of Taihu stones is characterized by two main levels: firstly, the extraction of natural landscape forms to create the landscape, and secondly, the flexibility of layout to suit the local context [16]. The Taihu stones are used as a symbol of natural mountains and are mainly used to stack stones to form mountains. Influenced by the traditional Geomantic learn, most garden rockeries are arranged on the north side of the main building. However, in the Canglang Pavilion, the panoramic landscape can be constructed according to local conditions. The main spatial layout is the central layout method, which is opposite to the hall and other buildings, and then matches with the water and follows the trend. Taihu Stone is mainly used for the western section of the real mountain forest and the Liuyu stone in the northeast of the Liuyu Lake (Fig. 2).

Most literati love the natural interest of Taihu stones, both 'like' and 'unlike', where the wonder of real mountains lies in their falseness and the beauty of fake mountains lies in their authenticity. The Taihu stones are exquisite in form and have the characteristics of being upright, smooth, transparent and versatile. The real mountain cave formed from this stack has a stone peak on top of which there are some ancient and famous trees such as Piaoshu, beech, camphor and cypress, which is quite clever.

### 3.2.2 The construction techniques of Taihu stones

The rock formations in the Canglang Pavilion are dominated by Taihu stones and Yellowstones in the selection of materials. Table I- Table III show the physical property indexes and microstructure characteristics of

Taihu stone and Yellowstone by sampling and testing in this study. The samples for SEM-EDS analysis were taken from the site (Fig. 3), and the test samples for mechanical indexes were taken from stone suppliers in Suzhou and its surrounding areas. The main component of Taihu stone is  $\text{CaCO}_3$ , and Mg, Al, Fe and S components are also found in Taihu stone in Canglang Pavilion. The measured compressive strength is about

55MPa, similar to marble and higher than ordinary concrete (C40), and the measured water absorption rate is about 0.64%, higher than that of Yellowstone in Canglang Pavilion. Therefore, It has enough compression strength for rocks piling or mountain moving and because of the fine texture and suitable for waterscape integration, mellow and ethereal.

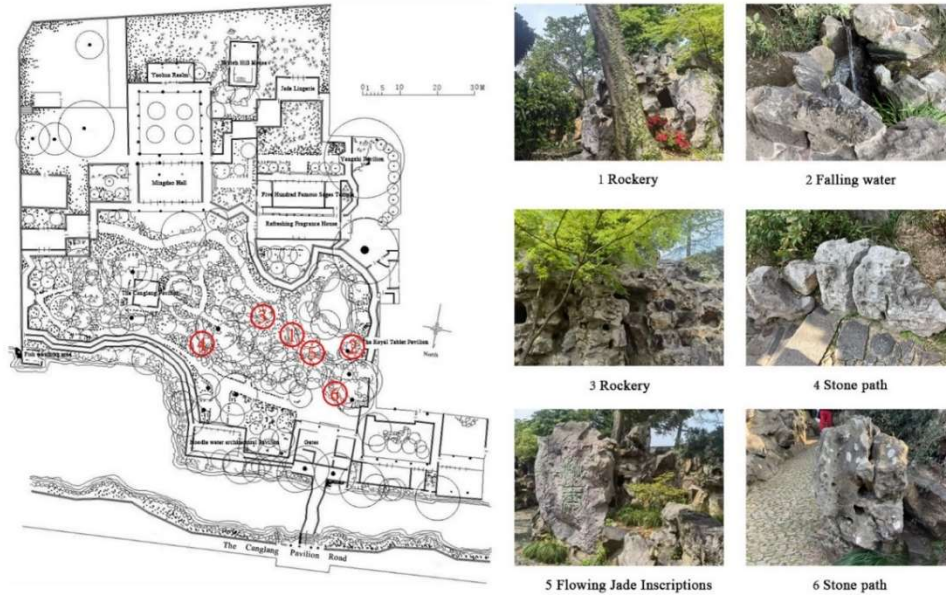

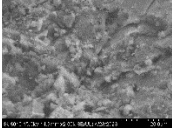


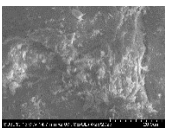



Fig. 2 Layout of Taihu Stone

Table I Comparison of Taihu stone and Yellowstone indicators

Species	Natural forms	Microstate	Sampling specimens	Compression strength (MPa)	Bulk density ( $\text{g}/\text{cm}^3$ )	Water absorption (%)
Taihu stone				55	2.54	0.64
Yellowstone				160	2.50	0.47

The rockery in Canglang Pavilion is an earthen stone hill, and the whole garden is laid out with the hill as the centrepiece, the mountain radiates a variety of surrounding garden elements, and at the same time dominating the whole garden. The mountain is oriented east-west, with its peaks, hills and trees, and it has the steadiness of a Northern Song landscape painting. Unlike other gardens, the Canglang Pavilion does not have a certain amount of space to dig pools of water or pile up mountains, but rather focuses on the footing of the 'real mountain and forest' and the configuration of the trees, using earth instead of stone, saving both

labour and material resources. More Taihu stone cliff paths at the foot of the mountain, rocks and soil, false mountain and real mountain into one, the most natural commission of the wonderful. The Taihu stones are mainly used in the western section of the real mountain forest, and in the pond area to the north of the real mountain forest, a completely different layout is adopted from the centralized arrangement of the mountain, i.e. the waterscape is integrated into the outside and inside of the garden. As the whole of the Canglang Pavilion is in the lake and a body of clear water passes around the garden, there is no large water

surface inside the garden, only a small pond in the northern part of the True Mountain Grove, called Liu Yu Tam. Looking down from the true mountain forest gives a sense of the hydrophobic endlessness at the foot of the mountain, and through this practice of small pools, the continuity of the river bank and the extension of the line of sight are shown, echoing the water tail under the bridge and suggesting the unending flow of water.

Table II Test results of Taihu stone composition

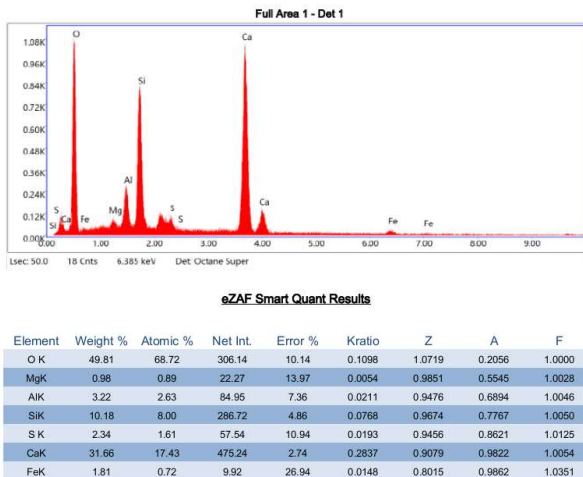
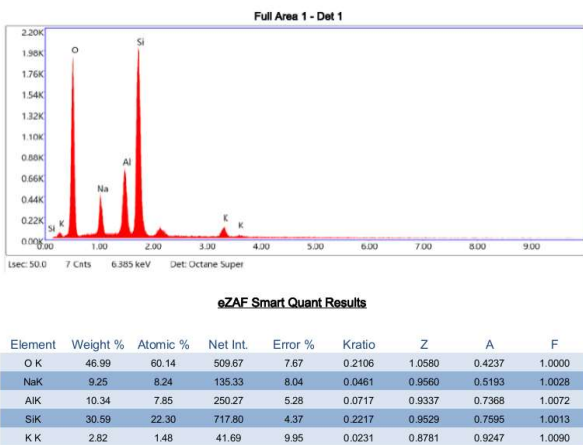


Table III Test results of Yellowstone composition



(a) Taihu stone (b) Yellowstone

Fig. 3 Photos of sampling sites

The use of Taihu stone in the Canglang Pavilion includes both borrowing and drawing in scenery. "The borrowed scenery is the most important part of the garden, and the Taihu stones can be used to create the scene

together with the "other stones". As Yellowstone is muddier in form and physical properties, while the physical properties of the Taihu stones themselves are more ethereal, so when collecting mountains, the Yellowstone should be more varied to show the ethereality, while the Taihu stones should not be too trivial to show the mellowness. The layout of Taihu stone and Yellowstone in the real mountain forest is different, Taihu stone is located in the western section of the rockery, more backlit and standing, although the light and shadow is not rich enough, but through the lake stone holes can bring clever changes. The Yellowstone is used in the eastern section of the rockery, mostly on the sunny side, adding light and shadow to the otherwise heavy rockery. The flowers and trees in the Canglang Pavilion are also the object of the Taihu Stone borrowing, and their configuration is dominated by trees, cypress and pine in deep colours with the colours of the lake stones, filling in the gaps between the rocks with flowers and trees to achieve the effect of hiding the awkwardness, while the flowers and trees can also be used as a background to enrich the layers of the landscape and enhance the shaping of space. The Taihu Stone uses water to achieve the purpose of increasing the level of space. The Taihu Stone at the junction of Liu Yu Lake and the rockery has a small iterative spring injected into it, with the water reflecting the Taihu Stone rockery, making the natural transition of the mountain colours into the water. The landscape elements in the garden, such as the garden road, rocks, flowers and trees, and water bodies all have the role of attracting scenery, and the cliff paths in the lake rock rockery have a stronger role in attracting scenery compared to the flat garden road, and the spatial feeling brought by the different paths will be different for visitors walking in them.

#### 4. Application of Yellowstone in the Canglang Pavilion

##### 4.1 The artistic characteristics of Yellowstone

###### 4.1.1 Natural characteristics

Yellowstone is an orange-yellow sandstone, often produced in places such as Huangshan Mountain in Changzhou, Jiangsu and Yao Feng Mountain in Suzhou, Jiangsu. In colour, Yellowstone is dark red, brown, slightly tawny and sometimes light red, brown and other colours. The stone is not only beautiful, but also very durable, and its colour does not fade after long periods of exposure to wind, sun and water erosion. Yellowstone has a variety of textures, often striped, laminated or blocky, with sharp angles and a folded profile. Quartz grains and other mineral grains are evident on the surface of the stone, and these form the distinctive texture and pattern of Yellowstone. Yellowstone has weathered over time in nature along calcite joints,



creating a structure of varying sizes, concave and convex in and out. In this feature, Yellowstone is often used as a material for stacking hills and peaks in gardens, forming fake hills that look like real hills.

The history of Yellowstone employed in garden construction is relatively late. Before the late Ming Dynasty, there was little records on Yellowstone. Until Chen Ji in 1631 completed the draft of the Garden Ye wrote "(Yellowstone) is produced everywhere, its quality is firm, not into the axe, its text is ancient and clumsy.....common people only know its naughty ram, but do not know its wonderful." This is the first time that Yellowstone has been recorded in a specialized garden work, where the record "is produced everywhere" indicates that Yellowstone was produced in many places and easy to obtain, while "the common people only know that it is stubborn, but do not know the wonders of it", indicates that Yellowstone was not yet widely used.

#### 4.1.2 Cultural characteristics

Yellowstone has a long history in classical Chinese garden construction, and although it was less used before the late Ming Dynasty, its earliest use dates back to the Middle Tang Dynasty. With its rich texture and colour, Yellowstone can form images of various natural scenes such as mountains, rivers, cliffs and caves. In classical gardens, through the use of these symbolic meanings of Yellowstone, the taste and cultural cultivation of the garden owner, as well as the celebration and pursuit of the beauty of nature, can be demonstrated.

Yellowstone is not only a material but also a symbol. Yellowstone symbolizes the spirit of nature and the realm of humanity, expressing the harmonious relationship between man and nature. Yellowstone is widely used in classical Chinese gardens to create humanistic landscapes, such as the elegant realm of the literati and garden architecture, reflecting its importance in the realm of humanity. The realm of nature refers to the value and significance of the natural landscape, which in traditional Chinese culture is not only a natural environment, but also a source of human life and soul. As a natural stone with natural form, texture and colour, Yellowstone is widely used in classical Chinese gardens to create natural scenarios such as landscapes, ponds and rockeries, reflecting its importance in the realm of nature.

### 4.2 The creation of Yellowstone in the Canglang Pavilion

#### 4.2.1 The layout of the Yellowstone

The Yellowstone in the Canglang Pavilion is mainly used in four places: the entrance revetment; the rocks by the Liuyu Lake in the garden; the rockery in the central

eastern section of the garden and the stone house under the Watch Hill (Fig. 4). In order to achieve a natural effect, the entrance and the revetment of Liuyu Lake use natural stone as a berm, imitating the natural spring pool and built, while the berm stone can also prevent rainwater erosion caused by soil loss, to protect the role of the waterfront. During the Northern Song Dynasty, Shunqin Su built the garden on the site of Sun Chengyou's pond, and the rockery was built as an earthen hill with stones at the time when Shunqin Su first built the garden. During the Kangxi period of the Qing Dynasty, Luo Song rebuilt the Canglang Pavilion and moved the pavilion at the water's edge to the top of the earthen hill, changing the rockery on the south bank of the water to the center of the garden and laying down the basic pattern of the garden thereafter, at which time the rockery was more earthy and less rocky, still dominated by earth. The eastern section of the rockery is a remnant of the Qianlong period, due to the fact that the terrain is flatter here with the view from a height, and although the rocks are numerous, it is generally more open.

Before the end of the Ming Dynasty, the material commonly used for rockeries in Jiangnan gardens was mainly Taihu stone. However, the demand for stone increased dramatically during the late Ming period when the gardens in Jiangnan flourished, leading to a shortage of Taihu stone resources and the danger of depletion. As a result, there was an urgent need for a new variety of stone to meet the needs of rockery techniques. As given in Table I, Yellowstone, with its hard texture and widespread production, became an alternative material for garden stonework. It was able to express the emerging genre of mountain stacking at the time and was in keeping with the aesthetic interests of the literati gardeners. As a result, Yellowstone became more and more widely used in gardens, and eventually became an important and indispensable material in garden landscaping, alongside Taihu stone.

#### 4.2.2 The construction techniques of Yellowstone

As can be seen from the measured results in Table I and Table III, the Yellowstone in Canglang Pavilion is mainly composed of  $\text{SiO}_2$ , and also contains a certain proportion of metal element Al, reflecting the presence of aluminosilicates. The measured compression strength is nearly three times that of Taihu stone, and the water absorption rate is only 0.47%. It is hard in texture, but the temperature difference in freezing and weathering erosion will cause disintegration along the joint surface. Therefore, Yellowstone is not suitable for the landscape of falling water, and its solid texture, rough texture and earth color, make it especially suitable for the use of stacking mountains and stones, and suitable for mountain scenery.-



Fig. 4 The Yellowstone layout



As can be seen from the Canglang Pavilion map, the landscape of the Canglang Pavilion had already been built with stacked hills and stones during the Qianlong period (Fig. 5), whereas it was previously recorded in the literature as a hill of earth and stone<sup>[11]</sup>. Based on the predominance of Yellowstone in the eastern section of the Canglang Pavilion, Dunzhen Liu believes that these hills may have been built at an earlier date, and it can be assumed that they are a large number of hills that survived from the Qianlong period. Yellowstone is hard, easy to mine, and is widely used in the construction of treacherous landscapes, while Suzhou Yao Feng Mountain is one of the main sources of Yellowstone. It can therefore be argued that the Yellowstone rockeries in Canglang Pavilion existed in the Qianlong period or even earlier.

The rockeries in the eastern part of the Canglang Pavilion adopt the theory of stacked hills, which advocates natural forms by mixing earth and stone, and seek to restore the natural landscape nature of Chinese gardens through the naturalistic technique of 'earth and stone', avoiding the traces of excessive artificiality. From the Song Dynasty onwards, one of the major trends in Chinese garden art was the specialization of gardening techniques. Since the Song Dynasty, the specialization of gardening techniques has become an important direction in the development of Chinese gardens. While this led to a greater sophistication in the skills of hill-stacking in later gardens, it also led to a lack of innovation in hill-stacking in Ming and Qing gardens. In the late Ming and early Qing dynasties, three famous gardeners, Cheng Ji, Nan Yuan Zhang and Yu Li, strongly advocated the use of a mixture of earth and stone to stack hills, opposing pure stone stacking, reflecting the creative concepts of the literati gardening of the time and resisting the vulgarization trend of artisanal

gardening. The colour of the Yellowstone and its ability to withstand long periods of exposure to the wind and the sun make it an antique, solid feature, in keeping with the "retro" aesthetic taste prevalent in the late Ming.

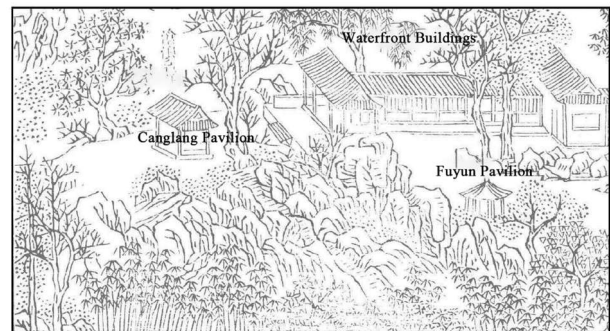


Fig. 5 The Canglang Pavilion and the rockery in The Canglang Pavilion

Photo credit: Gao Jin. A Catalogue of Famous Places on the Southern Tour. Gu Wu Xuan Press

## 5 Conclusion

In the late Ming dynasty, gardening flourished in the south of the Yangtze River, and Yellowstone was increasingly used in gardens where the supply of Taihu stone exceeded the demand, resulting in a situation where both Taihu stone and Yellowstone were used in gardens. Therefore, the use of Yellowstone and Taihu stone in gardens became more and more widespread and sophisticated, thus greatly enriching the types of stones in gardens. This paper explains the significance of the research on the art of stone scenery in the Canglang Pavilion, based on an analysis of the landscape architecture of classical gardens, and through a study of the Canglang Pavilion and an analysis and comparison of relevant historical documents, it compares the landscape art of Taihu stone and Yellowstone in the Canglang Pavilion in four aspects: layout, material characteristics, application features and construction

techniques. The main findings of the study are as follows:

(1) The mountain and water bodies in the Canglang Pavilion occupies about half of the park, and the dynamic and static settings of the mountain and water bodies set the overall tone of the garden. The water space, the mountain space and the architectural space in the overall garden space are basically kept intact, with only the compound corridor and the water body used as an enclosure, greatly enhancing the openness of the site, while also highlighting the natural appearance of the mountain and the water body themselves.

(2) Taihu stone vein hidden, the shape of the curious, the texture of the horizontal, interesting pits, highly freehand natural characteristics, material characteristics and cultural characteristics, so that it has become a combination of natural and artificial media. The application of Taihu stone in the Canglang Pavilion is

mainly mountain-moving, and it simulates the natural landscape close to each other. The round and delicate stone points are used to place equal emphasis on nature and skill through various landscaping techniques such as borrowing and guiding the scenery.

(3) Yellowstone texture is hard and thick, the mountain approach is made of stone layers, the shape edges and corners are obvious, the joint plane is straight, the transverse texture is evident, can reflect the natural vertical and horizontal rock joints of the mountain, with strong light and shadow effects. The application of Yellowstone in the Canglang Pavilion is mainly based on the revetment and the sunny side of the rockery. The stacked earth and stone mountains are closer to the state of the natural forest, with the natural interest of returning to the original nature.

**Acknowledgments:** This research was funded by National Key R&D Program of China (2021YFE0200100); 2021 Policy Directed Program of Jiangsu Province (BZ2021015).

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## **Trigaches Marbles (São Brissos, Beja, Portugal): petrographic and geochemical characterization of a historical dimension stone**

J. Rosa<sup>1</sup>, N. Moreira<sup>2\*</sup>, J. F. Santos<sup>3</sup>, S. Ribeiro<sup>3</sup>

<sup>1</sup> Departamento de Geociências, Universidade de Aveiro, 3810-193 Aveiro, Portugal, \*[nafm@uevora.pt](mailto:nafm@uevora.pt)

<sup>2</sup> Instituto de Investigação e Formação Avançada - Universidade de Évora, Instituto de Ciências da Terra (ICT) - Polo de Évora, Departamento de Geociências - Universidade de Évora, Colégio Luís António Verney, Rua Romão Ramalho, 59, 7000-671 Évora, Portugal

<sup>3</sup> GeoBioTec, Departamento de Geociências and Laboratório de Geologia Isotópica, Universidade de Aveiro, 3810-193 Aveiro, Portugal

### **Abstract**

The exploitation of carbonate crystalline rocks in the Ossa-Morena Zone (OMZ) dates back more than two thousand years. Several historical exploitation poles in OMZ were identified based on the identification of distinctive marble lithotypes in Roman consumption areas, and some of those historical exploitation places were, subsequently, identified in situ through geo-archaeological works (e.g. Almadén de la Plata, Estremoz Anticline). The distinctive macroscopic features of Trigaches Marbles also allowed to recognize their use on Roman architecture, but also in several buildings, namely in southern Portugal, along numerous historical periods until present days, which indicates a long lived history of exploitation of this dimension stone. The Trigaches Marbles are exploited in Trigaches - São Brissos parish (Beja, Alentejo), located at the SW border of the OMZ. The geological unit containing the Trigaches Marbles has been assigned to Cambrian (as most of the exploited marbles on OMZ), outcropping in a N-S elongated strip with approximately 1.5 km<sup>2</sup> of area. This unit contacts with Carboniferous magmatic bodies to the west and north, and with a predominantly metasedimentary unit, composed of schists and subordinate metavolcanic rocks, to the east.

At macroscopic scale, Trigaches Marbles can be light grey, dark grey or grey-scale banded marbles, showing a poorly developed subvertical to steeply dipping metamorphic banding, subparallel to the main unit trend. Marbles are mainly composed of calcite (generally above 97% of volume - just one sample has a proportion around 95%), exhibiting a well-developed non-oriented granoblastic texture, commonly with tendency towards polygonization of calcite grains. As minor mineral phases, quartz, opaque minerals, biotite or muscovite, and sporadically, scapolite and pyroxene were identified. Marbles are inequigranular, generally coarse to very coarse grained, although sometimes medium grained calcite is also present (grain size ranges from 0.5mm to 20-40 mm). Calcite crystals commonly present type 4 twins, indicating medium-high temperature recrystallization, in accordance with the grain size.

Major element geochemical data show great homogeneity, in accordance with mineral modal composition: CaO ranges between 52.2 and 54.8% whilst MgO varies from 0.4 to 1.0%, in accordance with the calcite nature of these marbles and the absence of dolomite; low concentrations of Al<sub>2</sub>O<sub>3</sub> [0.8 - 0.1%], K<sub>2</sub>O [0.0 - 0.2%] and Na<sub>2</sub>O [0.0 - 0.3%] agree with low phyllosilicate contents and the absence of feldspars s.l.; SiO<sub>2</sub> can reach 2% of whole rock being related mostly to the presence of quartz (can reach 3% of modal composition); LOI range between 42.5 and 43.9%, mainly indicative of the CO<sub>2</sub> content.

### **Acknowledgments**

Noel Moreira thanks the financial support provided by the ICT, under contract with FCT (Fundação para a Ciência e a Tecnologia) (REF: UIDB/04683/2020 e UIDP/04683/2020). Other authors receive the financial support provided by GeoBioTec, under contract with FCT (REF: UIDB/04683/2020).

# Investigation of the collapse of a four-span masonry arch bridge, a case study from Hungary

R. Varró<sup>1</sup>, P. Görög<sup>2</sup>,

(18) Richárd Varró, PhD student, Budapest University of Technology and Economics, Department of Engineering Geology and Geotechnics, [varro.richard@edu.bme.hu](mailto:varro.richard@edu.bme.hu)

(19) Péter Görög, associate professor, Budapest University of Technology and Economics, Department of Engineering Geology and Geotechnics, [gorog.peter@emk.bme.hu](mailto:gorog.peter@emk.bme.hu)

**Summary:** *With their unique combination of strength and beauty, stone arch bridges have been a hallmark of engineering and architectural design for centuries. Built from natural materials, such as stone, these structures have stood the test of time and played a crucial role in the development of transportation and commerce. However, with age came the need for proper maintenance and preservation to ensure their longevity and continued service to communities. This paper highlights the importance of maintenance and preservation by presenting a case study of the collapse of a four-span bridge in Hungary. The failure, a result of a combination of construction faults, environmental factors, and inadequate maintenance, sheds light on the need for proper infrastructure inspection and maintenance. The study provides load-bearing calculation of the bridge under different conditions and valuable insights for developing better maintenance practices. It serves as a cautionary tale for other communities to ensure the safety and stability of their critical infrastructure.*

**Key words:** *masonry arch bridge, failure, load-bearing capacity, maintenance*

## Introduction

Masonry arches have been utilized for over a millennium. In Hungary, there are over 1,500 stone masonry arches, constructed mainly during the 18th and 19th centuries. Unfortunately, these structures have received limited attention in Hungary, leading to severe deterioration (Gálos & Vársárhelyi 2006). Masonry arch bridges in Hungary were predominantly built during the 18th and 19th centuries using different materials based on the local geology. Stone was used in mountainous regions, while brick was the primary material in lowland areas. However, mixed masonry structures are also present. As traffic loads have significantly increased over the centuries, maintenance and investigation of these bridges have become crucial. Mapping the damages and determining load-bearing capacity are essential issues (Arias et al., 2007, Bögöly et al., 2015), but investigating these structures is challenging due to their complex behaviour. Bouzas et al (2023) did a reliability-based structural investigation of a five-span historical masonry arch bridge from Spain with on-site assessment and limit analysis. Additionally, determining input parameters for efficient numerical modelling is a significant challenge. The on-site assessment is essential to determine the necessary input parameters for numerical modelling. Nowadays, near the conventional measurements methods TLS and UAV measurements are also used to measure the geometrical parameters of masonry arch bridges (Kassotakis & Sarhosis 2021, Cavalagli et al., 2020). Though existing structures are difficult to study due to monument protection, knowledge of material parameters is vital for global investigations. Therefore,

the non-destructive methods have an important role in the testing methodology (Conda et al., 2017, Balla et al., 2019).

By presenting a case study of the collapse of a four-span bridge in Hungary, this paper underscores the significance of maintenance and preservation. To determine the causes of the collapse on-site investigations and limit-analysis technic had been used. The collapse, caused by a combination of construction defects, environmental factors, and insufficient upkeep, emphasizes the necessity for adequate infrastructure inspection and maintenance.

## Methodology

Due to the tight space and dense vegetation around the bridge, only conventional measurement techniques could be used during the initial assessment. However, during on-site measurements, the height of the arches was accurately measured. To obtain the true geometry of the structure, a new measuring method was developed. The arch height was measured every 10 cm along the length of the opening, allowing for a precise determination of the arc shape. This detailed information was crucial for any further analysis and repair of the structure.

Due to the bad condition of the bridge, it was possible to collect and test some of the sandstone blocks that had fallen from the pier. Ultrasonic pulse velocity measurements were taken on the samples, and uniaxial compressive strength values were estimated based on these results. The UCS value can be determined from the

ultrasonic pulse velocity, as outlined in Cargill and Shakoor's 1990 research.

For the numerical investigation of the bridge, the LimitState RING software was utilized. This program idealizes a masonry arch bridge structure as an assemblage of rigid blocks and employs computational limit analysis methods to analyze the ultimate limit state. The software determines the amount of live load that can be applied before structural collapse, taking into account various factors such as backfill, the effect of the neighboring span, connections between the blocks, and any failures in the structure (Gilbert & Melbourne 1994). The accurate geometry obtained from the field surveying was crucial for developing a correct numerical model that could aid in identifying any critical points that require immediate attention in terms of repair and maintenance.

### Description of the bridge under study

This elegant four-span bridge was a remarkable feat of engineering for its time and was located in town of Piliscsaba (Fig.1). The bridge was constructed around 1890, during the height of the Austro-Hungarian Empire's prosperity. Its graceful arches were formed in perfect half-circles using traditional brickwork, which gave the bridge a distinct and elegant appearance. Interestingly, two other very similar bridges were also built in the city during this period (Fig. 1).



Fig. 1. The location of the bridge in Hungary

The voussoirs of the arches were crafted using the multiring technique, which involved constructing the arch using three layers of brick, resulting in a thickness of 0.45 meters. This technique was popular in the 19<sup>th</sup> century and helped create robust and durable structures capable of withstanding the test of time. The bridge's piers were constructed using sandstone, which provide a stable foundation capable of supporting the weight of the structure. The use of brick for the rest of the bridge would have been a common choice during the time period in which the bridge was constructed, as brick was a readily available and cost-effective building material.

The combination of sandstone piers and brick construction created a unique and striking visual contrast, with the smooth and textured surfaces of the two materials complementing each other (Fig. 2).



Fig. 2. Bridge of Piliscsaba seen from the north

The bridge's spans are 3.3 meters wide. The height of the opening, at the middle 5.7 meters, was sufficient for the water to flow through underneath without any obstruction. The bridge's length of 25 meters and width of 4.9 meters made it a significant structure for the time period in which it was constructed, and it provided an essential link for the local community, enabling them to cross the water with greater ease and convenience. The surface course of the bridge appears to be incomplete, which means that there is no top layer to protect the underlying structure from external factors such as weathering, traffic, and other environmental conditions. This missing layer may have caused severe damage to the bridge over time, leading to structural issues that could compromise its safety and integrity (Fig 3). The result of the geometrical measurements of the bridge are on Fig. 4.



Fig. 3. The surface course of the bridge

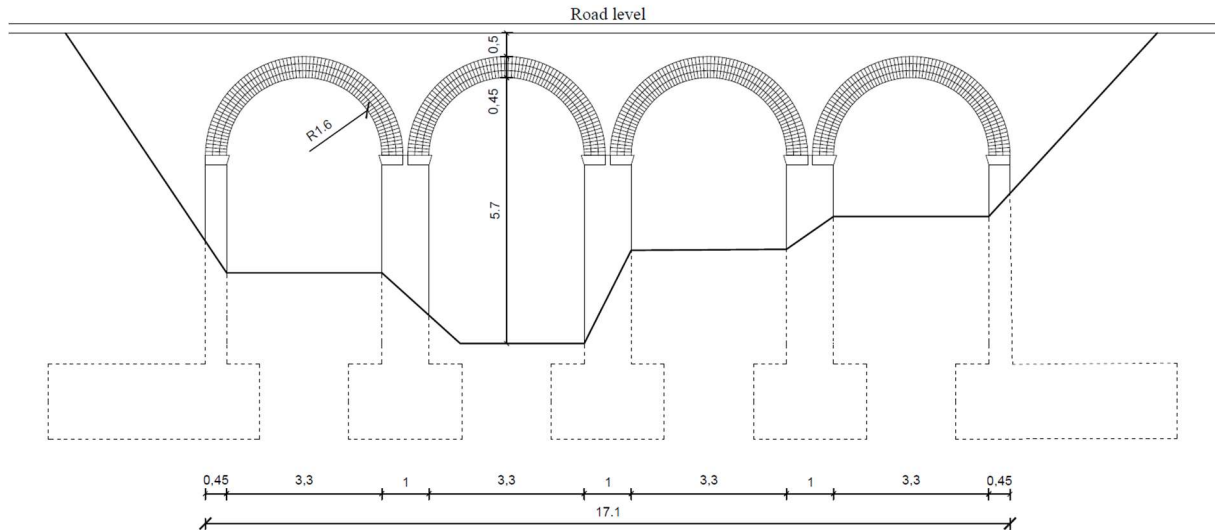


Fig. 4. Dimensions of the bridge of Piliscsaba

### Structural problems

Despite its impressive appearance when built, the bridge had suffered significant structural damage over time, primarily due to several factors. The harsh environmental conditions in the area, including exposure to wind, rain, and extreme temperatures, had taken their toll on the structure. Additionally, the bridge had been constructed with several geometric faults, which had further weakened its overall stability.

The neglect of critical repairs and reinforcements on the bridge over the years had taken a significant toll on its overall condition. The bridge had been left to deteriorate without any maintenance, which had allowed the surrounding vegetation to grow uncontrollably, covering and entangling the structure (Fig. 5). The thick roots of the plants had penetrated deep into the brickwork, causing cracks and destabilization. The overgrowth had also prevented proper drainage, causing water to accumulate and seep into the masonry, further weakening the structure.



Fig. 5. The plants on the bridge

Consequently, the bridge's structural integrity had significantly deteriorated, and this was noted during a survey conducted in 2017. The bridge exhibits visible signs of joint degradation, with gaps between the masonry elements where the joints have failed. In some areas, the joints have completely fallen out, leaving behind noticeable gaps between adjacent elements (Fig. 6). This type of joint failure is typically caused by the long-term exposure of the bridge to various environmental factors such as moisture, temperature changes, and natural wear and tear.



Fig. 6. Degradation of the joints.

The survey identified several missing bricks from the voussoir and half of one pier that had crumbled (Fig. 7).

In addition, many of the stone blocks in the pier were significantly eroded, with some completely cracked, and many areas so degraded that they could be easily crumbled by hand (Fig. 8). This deterioration had weakened one of the bridge's supports to the point that it had lost its load-bearing capacity, further compromising the overall stability of the structure.



Fig. 7. The heavily damaged pillar.



Fig. 8. The cracked sandstone on the corner of a pillar.

The railway line run directly adjacent to the bridge, which meant that the structure was constantly exposed to vibrations and shock waves whenever trains passed over it. These vibrations had a destructive effect on the structural stability of the bridge and could accelerate the deterioration of the already weakened structure. Additionally, the railway embankment had reached the piers of the bridge. As a result, constant pressure was placed on the piers, which eventually led to a decrease in their load-bearing capacity. The combined effect of inadequate maintenance, vegetation growth, and the impact of the railway line had a serious impact on the structural integrity of the bridge.

Despite its deteriorating state, the bridge remained in use, connecting the two parts of the town. However, in 2020, disaster struck, and a total collapse of two of the openings occurred. The collapse was a result of years of neglect and a combination of environmental factors and construction faults.

### The process of collapse

The assumed static framework of arches consists of a curved rod that is fixed at both ends. Therefore, the structure becomes statically indeterminate. If cracks or joints appear in the structure, the indeterminacy of the

structure decreases. Such structures work excellently with up to three cracks. However, when the fourth crack appears, the structure becomes over-determinate, losing its stability, leading to collapse (Gilbert 2006, Gilbert & Melbourne 1994). The theoretical process of collapse is shown in Fig.9, which illustrates the joints that form cracks at the site.

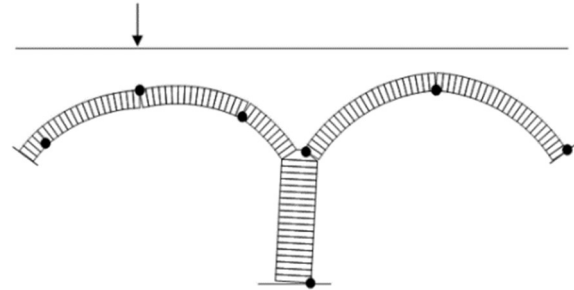


Fig. 9. The theoretical process of collapse (Gilbert and Melbourne, 1994)

Real bridge collapses are more complex than described above and do not always occur uniformly but rather result from the accumulation of multiple problems. Additionally, due to the spatial structure of bridges, collapses are typically irregular, and it is rare for a crack leading to joint formation to affect the entire width of the bridge. The complex process of collapse is evident in the case of the Piliscsaba bridge, which was in continuous use as a road bridge (Fig.10). The bridge suffered from half-sided collapse, which was likely due to missing structural elements caused by the erosion of the pillars, as described earlier.



Fig. 10. Destruction of the Piliscsaba bridge (www1)

### Results of the calculations

Firstly, the appropriate geometric data should be entered into the software. The load was applied as a concentrated axial force, and then the load was distributed over the extrados by the backfill. The required parameters for modelling are contained in the



following two tables. The Table 1 shows the properties of the sandstone, while the Table 2 lists the direct input parameters for modelling. The calculation requires compressive strength values, however, preliminary tests have shown that the strength parameter is not decisive in the calculation, as failure occurs due to loss of

stability. Therefore, the coefficient of friction between two blocks is the most sensitive parameter in the modelling process. However, measuring this value is not an easy task. According to Sadek et al., (2018), the coefficient of friction between blocks and mortar is approximately 0.5 under perfect conditions.

Table 1. Mechanical properties of the sandstone

Number of sample	Dry density [kg/m <sup>3</sup> ]	Ultrasonic pulse velocity [m/s] dry with plaster	Saturated density [kg/m <sup>3</sup> ]	Ultrasonic pulse velocity [m/s] saturated with plaster	Water absorption [%]
1	2159	3078	2269	3445	4,70%
2	2263	2809	2401	3609	4,61%

Table 2. used for modelling

Density backfill [kg/m <sup>3</sup> ]	Density masonry [kg/m <sup>3</sup> ]	Compressive strength [MPa]	Coefficient of friction
1800	2159	38	0,5

Parameters

The software is capable of considering minor structural defects, but the pier cannot be completely removed. The load-bearing capacity of the weakened structure was 86 kN. It is worth noting that as the structure deteriorates, the load-bearing capacity decreases significantly, Figure 11. show the collapse process. However, in cases where a significant part of the pillar is missing, it becomes challenging to accurately determine the load-bearing capacity, and only estimates can be made. Failure in masonry arch bridges due to weak pillars typically results failure mode with the collapse of

the weakened pillar. However, in cases where the weakened pillars are not considered in the model, the nature of the failure can be completely different. In such scenarios, failure occurs at the critical point of the arch where the load-carrying capacity is exceeded, resulting the collapse. It is essential to accurately model and consider all potential weak points of masonry arch bridges to ensure their safety and longevity. Therefore, it is essential to regularly inspect and maintain masonry arch bridges to ensure their safety and prolong their lifespan.

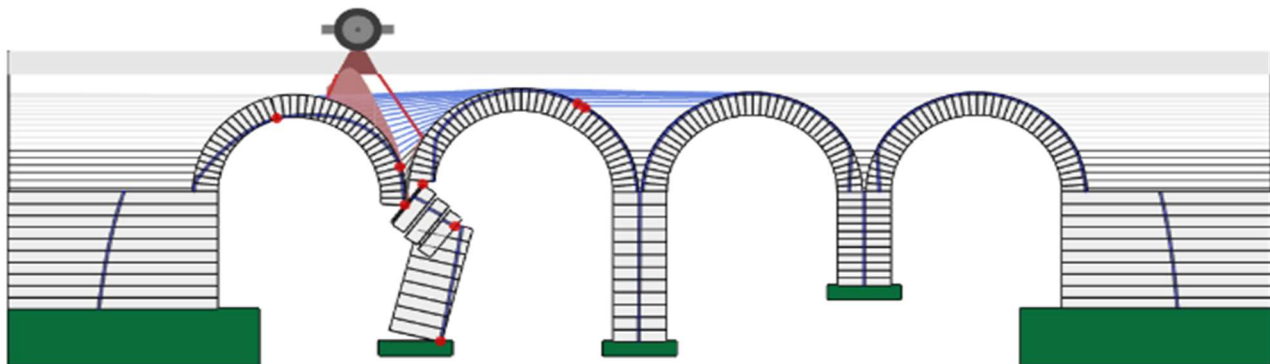


Fig. 11. failure process of the bridge, where the pier was weakened

### Lessons learned from the problem and discussion

The collapse of the bridge in Piliscsaba in 2020 was a tragic event that could have been avoided if proper maintenance and repair work had been carried out over the years. The structural damage that led to the collapse

of the bridge was a result of several factors, including neglect the environmental conditions, and construction faults. However, there were several preventive measures that could have been taken to avoid the collapse of the bridge.

Continuous monitoring, inspection, and maintenance of arch bridges are important tasks to prevent serious accidents. The condition of the bridge can be determined by a simple visual inspection; if no defects are visible (such as cracks, missing joints or elements, deformation, or vegetation), then it can be safely assumed that the structure will continue to function properly in the future.

The important steps to preserve its structural integrity, mainly consist of the following issues.

One important factor is vegetation management. As mentioned earlier, overgrowth of plants had entangled and covered the bridge, causing significant damage. Regular maintenance and management of the vegetation would have prevented it from growing uncontrollably, reducing the risk of roots penetrating the brickwork and causing further damage (Federal Highway Administration 2019, Ritzinger 2017).

Another important measure that could have been taken is water drainage management. Proper drainage management would have prevented water from accumulating and seeping into the masonry, which weakened the structure. The installation of drainage systems or regular cleaning of the existing drainage system could have helped to mitigate this issue.

The railway line should not have been located so close to the bridge. The constant vibrations from passing trains can weaken the structure of the bridge over time. The railway line was built after the bridge, the engineers could have taken the bridge's proximity into account and designed the track accordingly, with appropriate measures to prevent damage to the bridge (Wood et al., 2008).

One of the simplest and oldest interventions to address the problem of missing mortar joints is the re-filling of decayed or missing joints. The essence of the intervention is to restore the original connection at the vaults. This process does not require any special intervention and can be performed relatively quickly and easily. The method involves removing the worn or even disintegrated mortar joints from the structure, and then filling the voids with new mortar. This solution does not increase the load-bearing capacity of the structure, and after the repairs, the original load-bearing capacity can be assumed, provided there are no other types of damage (Mirza & Hussain 2019, Roca et al., 2015, Xuan & Krivenko 2018).

When there is advanced structural deterioration, it often happens that not only the mortar between the elements is damaged, but also the building blocks themselves. The blocks can turn out or fall out of the structure, and these shortcomings must be remedied by

replacing the elements. During the procedure, the location of the element to be replaced must be adequately cleaned, as plants and other things can settle in the gaps. After cleaning, the surrounding joints should be filled with the appropriate mortar in the manner described above, and the new building block should be inserted. If necessary, these should be supported until they solidify to ensure that they remain in the desired position (Bofoz & Mikułowski 2021, Binda & Saisi 2001).

Regular monitoring of the bridge's load-bearing capacity could have helped to identify any weaknesses or issues before they became critical. Building Information Modeling (BIM) would be a good solution for ensuring the safety of existing structures, as it can contain all the necessary information about a structure, and any future strengthening or modifications can be easily incorporated into the model. This would allow for more efficient monitoring and record-keeping of the structures. The application of BIM has already been successful in many historic buildings and is becoming increasingly popular today (Barazzetti et al., 2015).

Lastly, it is crucial to note that proper funding and resources allocated for the maintenance and repair of the bridge could have prevented its collapse. Neglecting critical repairs and reinforcements due to a lack of resources is a common issue for many infrastructure projects. Adequate funding would have allowed for prompt repairs and maintenance to be carried out, preventing the damage from worsening, and ultimately leading to the collapse of the bridge.

In conclusion, regular inspections and maintenance, vegetation management, water drainage management, regular monitoring of load-bearing capacity, and proper funding and resources allocation are all crucial factors that could have prevented the collapse of the bridge in Piliscsaba. It is important to prioritize the maintenance and repair of infrastructure projects to ensure their long-term durability and safety.

## **Conclusion**

The collapse of the Piliscsaba bridge serves as a cautionary tale about the importance of proper maintenance and preservation of historic infrastructure. Bridges, like many other types of infrastructure, are subject to the forces of nature and the passage of time. Without adequate maintenance and repairs, even the most robust and durable structures can deteriorate, eventually leading to their collapse.

One of the critical lessons that can be drawn from the case study of the Piliscsaba bridge is that early detection of structural problems is essential. Regular inspections and maintenance are necessary to ensure that structural problems are identified and addressed before they

escalate into more significant issues that can compromise the overall stability of the structure. In the case of the Piliscsaba bridge, the damage to the structure had been allowed to accumulate over a long period, which ultimately led to its collapse.

Another important lesson from this case study is the need to balance the preservation of historic structures with the need for modernization and upgrades. While it is essential to preserve historic infrastructure for future generations, it is equally important to ensure that these structures are equipped to handle modern traffic loads and other demands. Balancing these competing priorities requires careful planning and coordination among stakeholders, including preservationists, engineers, and policymakers (Rypkema 2019, Kourakis & Pournaras 2019).

**Acknowledgments:** The research reported in this paper is part of project no. BME-NVA-02, implemented with the support provided by the Ministry of Innovation and Technology of Hungary from the National Research, Development and Innovation Fund, financed under the TKP2021 funding scheme.

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The collapse of the Piliscsaba bridge underscores the importance of investing in infrastructure maintenance and repair. Bridges, roads, and other infrastructure are critical to the functioning of modern societies, and neglecting their upkeep can have significant economic and social costs. By investing in infrastructure maintenance and repair, governments can ensure that their communities are safe and resilient, while also preserving the historic structures that are part of their cultural heritage.

Finally, the case study of the Piliscsaba bridge highlights the importance of proper maintenance, preservation, and modernization of historic infrastructure. By learning from the mistakes made in the past, we can ensure that our infrastructure is safe, resilient, and able to meet the needs of future generations.

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## REVIEW OF THE IMPACT OF CLIMATE CHANGE IN THE DURABILITY OF ROOFING SLATES

V. Cárdenes<sup>1\*</sup>

(1) Geology Department, Oviedo University, Spain. [cardenesvictor@uniovi.es](mailto:cardenesvictor@uniovi.es)

**Summary:** One of the multiple effects of climate change is the alteration of the weathering resistance of building stones. The increase in temperature and atmospheric acid conditions is seriously affecting the integrity of buildings and monuments all around the world. Temperature increment leads to higher rates of salt crystallization and crack development induced by differential stresses, while acid conditions trigger dissolution and/or oxidation processes. As a common component of architectural heritage, slate is also exposed to the undesired effects of climate change, especially on its most popular use as roofing slate. This work reviews the physicochemical properties of different varieties of roofing slates, evaluating their potential response to the weathering conditions induced by climate change.

# The original ornamental stones and the conservation of the monumental cultural heritage

R. Bruno<sup>1</sup>

(20) DICAM-University of Bologna, Italy, [Roberto.bruno@unibo.it](mailto:Roberto.bruno@unibo.it)

**Summary:** *The world cultural heritage recognized by UNESCO is based on monumental assets characterized by the use of the stone. In many cases it is inevitable a replacement of the compromised stone elements. In Italy, the first country in the world for the number of cultural WHs, the maintenance of monuments calls for guaranteeing an extractive activity in support of extraordinary maintenance and restoration. A policy is needed that ensures the activity of specific quarries for the supply of the original materials. A conflict may arise between the need of conserving nature and cultural heritage. An artisanal extractive project, devoted to guarantee the maintenance of the cultural heritage, is easily sustainable. A cultural heritage conservation policy based on non-original rocks, is not sustainable because it irreparably damages the monument value. The conflict between the two conservation needs, natural and cultural heritage, call for supranational legislative interventions (EU, UNESCO).*

**Key words:** ornamental stones, cultural heritage, restoration, original material

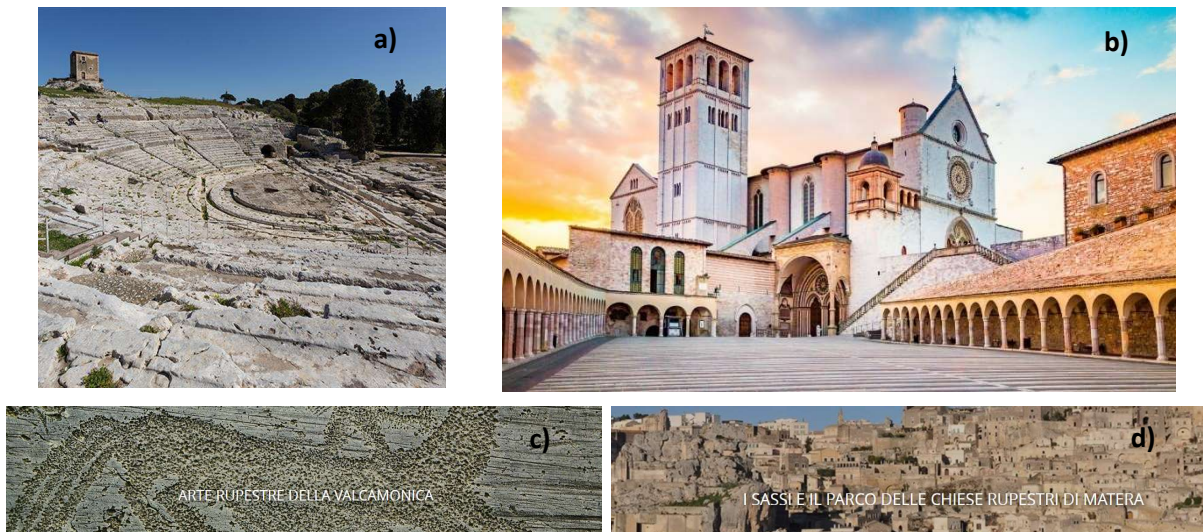


Fig. 1. Some Italian monuments, UNESCO Cultural World Heritage: a) The Greek Theatre of Syracuse; b) San Francesco complex Assisi; c) Valcamonica rock engravings; d) The Sassi of Matera.

## Cultural heritage is stone

Let us consider the UNESCO Worlds Heritage (WH), 468 over 547 are cultural heritage (Fig.1). And if we see Italy, the 1<sup>st</sup> country in terms of number of properties (58 over 547), 70% are cultural heritage properties (Fig.2).

The materials our monumental cultural heritage is made of is stone (Fig.3).

- Historical centres
- Palaces
- Churches
- ...

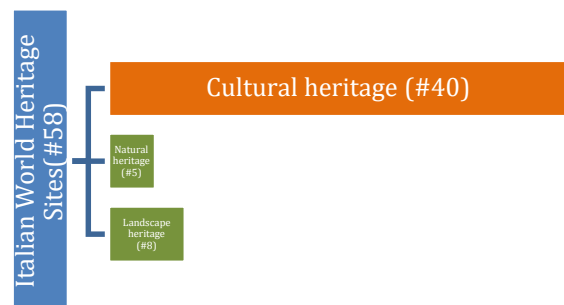


Fig. 2. UNESCO data.



Fig. 3. Historic centre of Florence and Trevi fountain, Rome.

But also, large gardens with a refined use of stone (Fig.4). The Italian cultural heritage is largely based on the rocks, obviously extracted from quarries.



Fig. 4. Monreale Cathedral.

Some rocks defy the centuries without too many problems thanks to their nature, the use that has been made of them and the climate. The travertine has been there for 20 centuries in Rome (Fig.5).

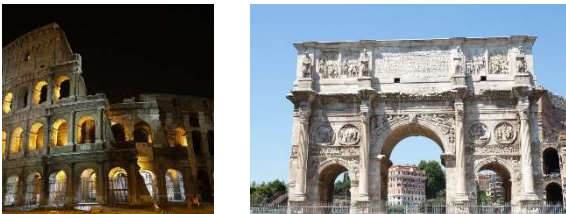


Fig. 5. Coliseum and Arco di Costantino, Rome.

Some rocks are more "sensitive", suffer from the weather. Many historic buildings of Bologna used the materials available in the surrounding area. Apart from clays for bricks, these are sandstones and gypsum, which have a greater tendency to degrade (Fig.6).



Fig. 6. The sandstone of Palazzo Bocchi, Bologna.

### The integrative restoration

The Torre degli Asinelli and the Garisenda can be considered the heart of the city of Bologna. Both buildings, dating back to the 11<sup>th</sup>-12<sup>th</sup> century, were

made of selenite, a raw gypsum extracted from the quarries of Monte Donato (Fig.7).



Fig. 7. The two towers of Bologna and the base of the Garisenda.

They need restoration, that is the preservation of cultural heritage requires obviously maintenance. And the next question is to understand what restoration is needed to preserve these rocks.

On ordinary and extraordinary maintenance, we must be pleased with our excellence achieved, internationally recognized. The University of Bologna is also at the forefront (Fig.8).



Fig. 8. Training in restoration of cultural heritage at the University of Bologna.

We must be pleased with the consolidated market of stone restoration (Fig.9).

But extraordinary maintenance with technological restoration interventions is often no longer enough. In many cases an integrative restoration, even if only partial, of the compromised stone elements is inevitable.



Fig. 9. Advertisements for stone restoration companies

The Investigations on the stability of the Garisenda tower (Fig.10) have highlighted that "...selenite, like every natural material, degrades above all in contact with heat and humidity" (Bologna24ore, 2022).

After micro-coring carried out to insert the steel ropes. the second phase of the works also envisages the replacement of stone elements in selenite.



Fig. 10. Degradation of the selenite of the Garisenda tower.

Where to get the selenite necessary for the restoration work on the base of the Garisenda tower which involves the replacement of stone elements? It is unacceptable to think of a restoration that uses rocks other than the "original" ones, even if "similar". It is now a mature awareness, even at the level of public administration.

### Original materials for an integrative restoration

The Piedmont Region, dedicates a section to Ornamental stones as evidence of local history and culture and publishes a census of Piedmontese decorative and building stones from Roman times to today, that "provides an interesting and complete overview of the historical and cultural significance of this type of material" (Fig.11).

Governo Italiano

REGIONE PIEMONTE

segui su [social media icons]

ricerca sul sito

L'Amministrazione		Tempi		Servizi		Bandi Enti Locali		PiemonteInforma		Piemonte da Vivere	
TRADITIONAL NAME	PETROGRAPHIC NAME	PLACE OF ORIGIN	LOCALIZED QUARRY	PERIOD	USES		SIGNIFICANT EXAMPLES				
Red Pantheo	granite	Belmonte, Valperga, Prascorsano	Yes	'900	Hotel Principi di Piemonte, Torino						
Green Cesana	ofical lime	Cesana Torinese	Yes	'900	Politecnico Torino						
Green Rochemolles	ofical lime	Bardonecchia	Yes	900	Stazione Ferroviaria Bardonecchia						
Gren Susa	ofical lime	Falcomagna (Bussoleno)	No	Middle Ages, 700	Fonte battesimale Cattedrale di Susa						
Marble of Pont	marble	Fondiglie (Pont Canavese)	No	'700 - '800	Statuaria sabauda						
Marble of Perrero o of Faetto	marble	Cima di Roccabianca	Yes	'500 - '600	Capitelli e basi delle colonne Santuario Consolata a Torino						
Marble of Salza of Pinerolo	marble	Rocca Corba	Yes	'800	Cancellata Piazzetta Reale a Torino						
Marble of Foresto	dolomitic marble	Foresto (Bussoleno)	Yes	Roman Period, - '700	Facciata del Duomo di Torino						
Marble of Chianocco	marble	Chianocco (Bussoleno)	Yes	Roman Period, - '700	Facciata di Palazzo Madama a Torino						
Breccia of Melezet	marble	Melezet (Bardonecchia)	No	'400 - '500, '900	Fonte battesimale chiesa di Beaulard						
Calcare	marly limestone	Marcorengo, Monte da Po, Lauriano	Yes	Roman Period, Middle Ages	Chiese romaniche del Monferrato						

Fig. 11. Piedmont Region, census of historical ornamental stone quarries no longer mined and examples of the use of these stones as decorative and building stones from Roman times to today. (Regione Piemonte, 2023).

Moreover, it focuses explicitly the restoration issue, by pointing out the need of an integrative restoration based only on original stones: "Only recently has the need and the possibility of finding, in limited but sufficient quantities for restoration, the material exactly identical to the original been taken into account, identifying the site of origin..." (Regione Piemonte, 2023).

This is correct, if we want that our cultural heritage remain "original". On the contrary, a conservation and restoration policy based on rocks that are "similar" to those to be worked on does not appear convincingly sustainable. The concept is largely intuitive and

apparently implemented by the legislation. But a little deeper analysis reveals that things are not exactly clear. First of all, in Italy this kind of regulation is the responsibility of the Regions, so in fact there is an absolutely inhomogeneous case series. Furthermore, the concept of "original material", even when expressly referred to, is rather nuanced.

This is the case of the UNESCO WH Sassi of Matera (Fig.12). Which materials to use for the extraordinary maintenance of the exteriors and interiors? The Zetema Foundation, which also merges the Italian Ministry of Culture and Matera Municipality, works in the field of protection, management and enhancement of cultural assets and activities of Lucanian territory, with a specific interest on their authenticity. It writes on its web-site: "The material constantly used for the residential constructions of the Sassi is only one, even if it is infinitely varied throughout the city: tuff" (La Città dell'uomo, 2020).



Fig. 12. View of the Sassi of Matera.

But there is no univocal trace concerning the obligation to use that material because it is not univocally identified. The "Management Plan" of Sassi di Matera WH by Municipality of Matera (Colonna et al, 2014) recommends the use of "similar" materials for restoration. For the "Regulation for the restoration and the management of properties included in the register of Cultural Heritage" of the Municipality of Matera, restoration means keeping the integrity of the materials. And the "tufo" (tuff) material is a calcarenite that can be exploited all over the same geological formation, even in quarries far away from the surrounding area, therefore characterised by locally different facies.

There is the interesting case of slate from the village of Lavagna (Lavagna in Italy is the name of the blackboard). This stone is mined in an artisanal way in underground (Fig.13). Ever in its easiest application, roof slate, the



problem of preservation of the cultural heritage, by



Fig. 13. Underground artisanal mining of Lavagna slate

preserving the original materials applies. In Roma the twin churches of Piazza del Popolo, both originally with slate roof from Lavagna, have been restored on 2013 and 2019 with two different materials, only one coming from the Lavagna quarries, giving rise to a strong polemics because of the visual dyschromia (Fig.14).



Fig. 14. The twin churches of Piazza del Popolo, Santa Maria in Montesanto e Santa Maria dei Miracoli, Rome

### What is meant by “original material”

Petrographically speaking there is no doubt what a material is used in a monument, but the same rock can come from spatially or temporally different geological formations. Coliseum is made by travertine, and travertine there is also in Turkey or USA. Any natural property is not spatially and temporally constant, so that different travertines are physically different, also aesthetically. And this is true even inside the same geological formation. Think of the Carrara basin, each quarry mines a marble with different aesthetic quality.

The correct interpretation of "original stone", strictly speaking, means a stone extracted from the same quarry of origin or from quarries belonging to the same geological district of origin, if there are no important aesthetic changes. This means that each monument must be linked to the specific quarry of origin.

There are some cases where the memory of the original quarry has faded. It is necessary just to resort to the same geological formation and to identify a quarry with an adequate material. It is the case of the basalt of the

cathedral of Orvieto. In particular, the side elevation and the interiors are characterized by a typical duotone given by the alternation of black and white, i.e. travertine and basalt (Fig.15).



Fig.12. Interiors of the Cathedral of Orvieto

A recent study published by researchers from the Department of Earth Sciences of the University of Perugia (Moroni et al., 2000) shows how the material used comes from a small area adjacent to Orvieto. In the aforementioned study, samples taken both from the external and internal walls of the Cathedral and from the quarries/areas bordering Orvieto were analysed. Eventually, a quarry was recognized for its material as "original", and their usability certified for the purposes of a restoration (Fig.16).



Fig. 13. Analysis of the production cycle of the basalt quarry authorized for the restoration of the Cathedral of Orvieto

### The policies for a sustainable restoration of monumental cultural heritage

Once proven that the original material exists and that it is potentially available, the public authority must guarantee the possibility of carrying out correctly the necessary integrative maintenance, i.e. it must guarantee that exist the supply of the original material.

Farsighted historical policies show us the way forward, as many examples demonstrate. Let us consider the case of the “la Veneranda Fabbrica del Duomo” in Milan (Fig.17). This is the historic body responsible for the conservation and enhancement of the Cathedral. Established in 1387 by Gian Galeazzo Visconti, Lord of Milan, for the design and construction of the monument, it has been working for over 630 years in the conservation and restoration of the Duomo 1387. He placed the Candoglia marble quarries at the disposal of

the institution, excluding any profit-making purpose (Fig.18).



Fig. 14. The Cathedral of Milan



Fig. 15. Access road to the Candoglia marble quarry and extraction of a marble block.

This is a particular case, but the strategy is correct, i.e. it is necessary to guarantee the activity of a minimum number of quarries of the material necessary for the integrative maintenance and replacement restoration of buildings and works whose cultural value is recognized. To implement such a strategy, each monumental building must be associated with an area or even a quarry from which to extract the certified material for its original restoration.

Starting from the fact that the original material is identified, possibly certified, two cases can occur: a) the quarry (extraction basin) is in production, so there are no supply problems; b) the quarry does not produce. In this second case there can be very different situations, such as:

- the quarry falls within a protected area, excluded from mining activity by the regional extraction activities plan (PAE);
- quarrying is possible, but uneconomical (for several possible causes);
- quarrying is possible, but in fact it is inhibited by social opposition, typically for environmental or disturbance reasons.

In this second case the public authority must intervene by choosing between two public interests, often considered opposite: 1) to guarantee the environmental and social sustainability 2) to guarantee the maintenance of the monumental cultural heritage. Each choice has costs and benefits, but a transparent decision, possibly non-emotional, must be made.

If a positive decision has been taken, the public authority must identify on the PAE the quarry (the basin) authorized to produce materials for the restoration and define the maximum production volumes. It is very probable that, both for reasons of economy and for

reasons of social acceptability, industrial production is not permitted. This could eventually require artisanal extraction plans.

In this case, economic support to production should be envisaged and some bodies such as universities could play a role. In fact, mining engineering could derive benefits from a direct assumption of the management of an artisanal production, both in terms of teaching and research activities (internships, stages, laboratory activities, experimentation fields; ...).

In summary, the constituent elements of an organic policy of integrative restoration with original materials on a national scale are the following:

- a) Identification of the original stones used through a survey on a municipality scale of the monumental cultural heritage, including relevant buildings.
- b) Locating and enhancement of quarries where the original stone originate and certification of origin.
- c) Relaunch in the PAE of the areas/quarries charged of the (non-industrial) production aimed at the restoration of cultural heritage.
- d) Just in case, limiting the extraction to the strictly limited quantities recognized as necessary for the due maintenance, as a strategic material.

#### Not starting from scratch

There have been and are many actions and initiatives useful for implementing such a policy, but so far, they have been isolated. So, it's not starting from scratch.

In terms of census stone-building, we can remember a couple of initiatives, as that one in Piedmont Region already introduced, and that in the town of Bologna. Here, Prof. Dal Monte of University of Bologna on 2004 made a geological-tourist map of "The Stones of Bologna - lithology of a city"(Dal Monte, 2004). A map identifies the stones used in the main buildings and monuments of the town (Fig.19). Also, with reference to a forgotten production area, it has been introduced the survey made for identifying the site origin the basalt used for the Orvieto cathedral.

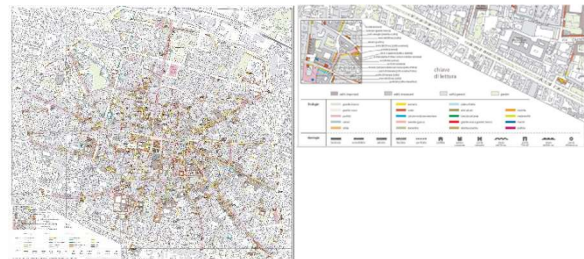


Fig. 16. The map "The Stones of Bologna - lithology of a city"

With reference to the localization and relaunch in the public PAE of some specific quarries (basins), we can cite the case of the Montovolo stone (and its stonemasonry

school). There are no active quarries here and the renovation must be carried out using boulders from cultivated fields. In fact, many historic buildings in and around Bologna use this special sandstone. As well as many sculptures, high relief engravings dating back to the Etruscan period (Fig.20).



Fig. 20. Example of use of Montovolo stone in the Bologna area, in buildings (Palazzo del Capitano della Montagna, Vergato, Rocchetta Mattei, Grizzana Morandi) and in archaeological artifacts (Sanctuary of the Blessed Virgin of Consolation, Montovolo Park).

While on the one hand there is a question of how to use the material, on the other it is a matter of reactivating old quarries. A short work done in Univ. of Bologna (Baietti, 2021) dealt with the “Possible reopening of the Cava de' Berardi”, a quarry to date crag for climbers. In the 25 years old PIAE 1996-2005, the references for an artisanal exploitation of the Montovolo stone were rough-hewn, but clearly outlined:

*“SPECIAL REQUIREMENTS: The activity will be managed with **artisanal criteria** and with methods appropriate to the context of environmental value: for this purpose, the maximum annual extractable quantity is set at 100 m<sup>3</sup> and the sandstone blocks can only be extracted with the use of wedges, pneumatic hammers o small explosive charges and without the use of earth moving machines or complex mining equipment (e.g. cutting ropes); for the transport means suitable for the local road must be used, which must not be widened, avoiding as far as possible the use of large trucks (over 150 quintals at full load).” (PIAE, 1996)*

### The inadequacy of a priori choices

It should be noted that the climate in Italy and partly in the EU is prejudicially opposed to the extractive industry. This is mainly due to the high increase of sensibility to nature conservation joint with a constant loss of culture about mining in Europe, as UE is highlighting more and more. In the media, the culture of conservation of natural heritage prevails, often declined together with another culture of conservation, that of cultural heritage. The conservation of natural and

cultural heritages can sometimes come into conflict, as in the case of the extraction of supplementary materials for restoration. And priorities must be established.

Italy is full of natural parks and environmental constraints, but it is also the 1<sup>st</sup> Cultural WH country, mainly based on stone buildings, churches, monuments. The environmental sensitivity and social acceptability of mining / quarrying often slows down or precludes the granting of permissions and is unfavourable to a motivated planning not only of industrial productions, but even of artisanal productions. The role of media is crucial.

Many extractive sites in Italy are in natural parks and it suffices little common sense to reconcile the two needs, that of production and that of nature preservation. Beyond the most evident example, that of the National Park of the Apuan Alps and the quarrying basin of Carrara, many examples can be cited. Sometimes, it suffices to continue excavation underground for limiting any environmental impact. A project that falls into the so-called "artisanal mining" is a fortiori sustainable if well oriented, by easily avoiding the most impacting events and environmental risks. And we are talking of an activity perfectly cut-out also to guarantee the supply of original materials for the correct maintenance of cultural heritage.

The current conflict, somewhat artificial, between the natural heritage and of the cultural heritage conservation, highlights the need to establish priorities, better if with specific legislative interventions of national (non-regional) level.

In Italy any decision about mining of whatever is delegated to the Regions, who make their specific PAEs and nowadays it is very difficult to harmonize the different approaches. This choice, which generates obvious risks from a strategic point of view in the case of Critical Raw Materials, is a losing approach even when it prevents integrative maintenance with the original materials. Some regions as Piedmont, Liguria, Toscana looks like quite aware and proactive in preserving the main area of historical production of ornamental stones. Other ignore the problem or counter any mining policy, even of the stone.

There is the feeling of the need of a joint, European analysis, able to set up a unique policy for the member states.

### Conclusions

It is necessary to develop a constructive attitude. Often our attention to the cultural heritage and the stones stops at the observation, study, and admiration of the work. About mining activities, today only radicalized ideological positions are gathered. Instead, we need to

look ahead and guarantee an extractive activity, even artisanal, to support an integrative restoration based only on original materials. On the other hand, what is the alternative restoration? Technological artificial solutions appear that are difficult to justify in terms of cultural heritage conservation (Fig.21).



Fig. 21. Advertising of artificial reproductions of local stones.

The stone sector must initiate or strengthen a collaboration with the monument restoration sector. It

is a question of making two sectors that are very distant from an industrial, cultural, and higher education point of view talk and cooperate. But this dialogue can be strategic alliance to highlight the need to have original materials for the defence of cultural to force the public authorities to take the consequent decisions.

We expect that Europe promotes a large work for identifying the sites who supplied the materials for building our monumental Cultural Heritage. This is propaedeutic to any correct integrative restoration and implies a capillary coordinated work at the local level for the census of all the buildings, bridges, ornaments that must be considered European Heritage and often WH. Moving from there, we expect concrete actions, as a support for a sustainable reopening of the original quarries, if closed.

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## TOP 12+1 ORNAMENTAL STONES FROM ROMANIA WITH NATIONAL AND GLOBAL HERITAGE VALUE

V. Cetean<sup>1,2,3</sup>, T. Filipciuc<sup>1</sup>, R. Fărnoagă<sup>1</sup>, E. Tudor<sup>1</sup>, D. Ion<sup>1</sup>, S. Lörincz<sup>1</sup>, G. Dincă<sup>1,3</sup>

(21) Geological Institute of Romania, email of the 1<sup>st</sup> author: [valentina.cetean@yahoo.com](mailto:valentina.cetean@yahoo.com)

(22) National Associations of Professionals in Geology and Mining

(23) Mineral Resources Expert Group, EuroGeoSurveys

**Summary:** *The activities carried out in recent years resulted in the preparation of the first national list of Romanian stones with national and global heritage value, in accordance with the criteria recently revised by the IUGS - Heritage Stone Subcommittee. From the total of 62 ornamental stones listed in the National Annex of the European standard EN 12440:2018 Natural stone - Denomination criteria, a number of 28 meet the requirements for stones of national heritage significance. Grouped by wider geological formations, historical and archaeological use to build the architectonic heritage of world sites, for emblematic buildings or in significant works, a "short" list of stones with heritage value was drawn up. These stones are: Măgura Călanului limestone, Rușchița marble, Moneasa breccia limestone, Alun marble, Vașcău polychrome limestone, Podeni sandstone limestone, Albești fossiliferous limestone, Geoagiu and Cărpiniș travertines, Viștea limestone, Măcin granite, Pietroasa-Deva trachyte, Codru Babadag limestone and Borsec travertine.*

**Key words:** *Romanian heritage stone, ornamental, scientific designation*

### GENERAL FRAME

The mining operations during the communist regime in the countries of Central and Eastern Europe were largely influenced by their chosen policy: self-sufficiency in mineral resources. The field of ornamental stones was no exception. Thus, at least for the period 1950-1990, the most important administrative buildings (including ministries, local councils and prefectures, universities, schools, hospitals, and courthouses, banking, electricity, telephony, and transport offices), buildings related to tourism (hotels, spas, cinemas, recreational areas) and even private buildings - were built using exclusively stones mined in Romania. The exceptions are insignificant as a percentage of the total, not exceeding 0.5-1%, according to the studies done by the research units [Cetean et al, 1998-2001].

Of course, the economic aspect was the most important criterion, the cost of the raw material to be imported and paid for in foreign currency being only part of the equation (a solution to be avoided or justified extremely rigorously, being accepted only in niche or national security situations).

The transport of ornamental stone from outside the country's borders certainly mattered at least in equal proportion, but it is quite possible that if this option would have been chosen, the preferred source would still have been the country with the greatest resources

even at present, namely China, with which Romania had close political and commercial relations.

The consequence of this apparently nationalistic policy was, however, a positive one, and ended by leaving its mark on all heritage buildings erected during this period. Thus, to the few quarries in organized exploitation known in the first half of the 20th century, were added those brought to light by prospecting work after 1960. The main focus was on perimeters with marble, as the most sought-after stone for use in ornamental elements, but also limestone and travertine with highly decorative characteristics and, lastly, granite and andesite. The most important of them in terms of extracted volume have operated for decades, but today some of them are closed, either because of the extremely high cost of exploitation for the specific conditions of the deposit, or because of their inclusion in protected areas or national reserves.

Out of the 131 ornamental rock quarries in Romania in the records of the National Agency for Mineral Resources and/or professional ones, 67 are included in the National Annex (no. A.2.18) of the European Standard EN 12440 [EN 12440:2008]. Most of them are in permanent or seasonal exploitation, the rest are in suspended activity due to the low feasibility of their exploitation in relation to the supply of imported rocks.

As no systematic review has been previously carried out regarding the stone resources in Romania with national

and global heritage value, in the framework of two national projects the available information and data have been re-analysed to determine how many of these stones meet the minimum criteria recently detailed by the IUGS - Heritage Stone Subcommittee, to be included in a list with scientific purposes. The main criteria applied were: their significance in the human culture (usage of the stones to build the architectural heritage of world sites or in significant works, in public or industrial projects), wide-ranging geographic application and ongoing availability.

The biggest challenge of the endeavour was the complete absence of freely accessible information regarding the stones used in public and/or heritage buildings of reference. If there were any information available about the construction itself, especially in the case of heritage buildings, at most the petrographic type of rock used was mentioned (e.g., limestone, marble, granite, basalt, etc.), and exceptionally the name of the stone was mentioned. Another obstacle was the fact that the vast majority of the old quarries changed their owner repeatedly, and their archives no longer exist (in the event that they might have contained some information regarding the place of application of the products made from that particular stone). The only notable exception is for the Ruşchița marble, which is the largest and oldest exploitation of ornamental stones in Romania, and which has recorded a significant number of historic buildings where this stone was used.

Thus, it was necessary to apply an indirect analysis criterion based on data from private professional archives [Cetean et al, 1998-2001], as well as the knowledge and experience of the specialists that authored the paper. Therefore, the databases held by Technical Committee 115 Natural Stone, published articles, reports, various technical presentations, technical expertise, etc., were analysed.

These were complemented by three decades of fieldwork, observing and recording the most well-known types of decorative stone used in important administrative or public buildings in Romania. In a subsequent stage, the construction period of these buildings will be determined, in order to document the minimum period of use of these rocks as thoroughly as possible. Although the approach is not easy, involving

multiple resources for documentation, it was the only applicable option.

#### ROMANIAN ORNAMENTAL STONES WITH HERITAGE VALUE

The first version of the list of Romanian ornamental stones with national and global heritage value includes a number of 25 areas where mining activities for extracting rocks have been carried out for at least the last half-century, to which we added 3 documented historical quarries:

- Măgura Călanului - pre-Roman limestone quarry whose activity stopped in the 2nd century AD - a source of stone for the Dacian fortresses in the Orăștie - Șureanu Mountains, UNESCO monuments;
- Bucova – Roman marble quarry from which stone was extracted for Ulpia Traiana Sarmizegetusa, the capital of Dacia (the ancient name for Romania) during the Roman occupation; it is currently inactive, but until two decades ago, marble was extracted from the same deposit, but from a perimeter neighbouring the former quarry;
- Pietroasa-Deva – an andesitic rock quarry (trachyte type), currently in operation; from areas historically used for some building elements of the cult sites at Sarmizegetusa Regia (the capital of the Dacian Kingdom), for a good part of the Roman epigraphic monuments from Micia (Vețel, Hunedoara County) and, somewhat more recently, for the construction of Deva Fortress, in the 8th century. Even though the historic extraction sites are not identical to the current quarry location, they are in close proximity and belong to the same volcanic body.

The most representative 12 types of stones were selected for this study (Table I), based on selection criteria that indicated the highest percentage, in addition to the most significant historical stone quarry used in monuments that are intrinsically linked to national identity.

On the structural map of Romania (Fig.1) it can be seen that the distribution of the perimeters is centred on the Transylvanian area, 9 perimeters out of 13, followed by Northern Dobrogea with 2 perimeters, respectively 1 perimeter in the Eastern Carpathians and 1 perimeter in the area of the Southern Carpathians.

Table I – List of the most 12+1 known Romanian ornamental stones with national and global heritage value

No.	Name of natural stone*	Petrological family/group**	Typical colour	Status	Coordinates	
					X	Y
1	Ruşchița	marble	pink, white-greyish, white-yellowish, grey	in exploitation	45.6462480	22.4057590

2	Moneasa	breccia marble limestone	Reddish, red to brownish, black, grey	in exploitation	46.4737580	22.2793560
3	Alun	marble	white-yellowish-grey	ceased	45.7054670	22.6816530
4	Vaşcău	limestone	polychrom; grey to dark grey, reddish or black	in exploitation	46.4690140	22.4253930
5	Podeni	sandstony limestone	grey-whitish	in exploitation	46.4371030	23.6282500
6	Albești	fossiliferous limestone	yellow-beige	in exploitation	45.3081569	25.0074577
7	Geoagiu & Cărpiniș	travertine	beige-yellowish to yellow-beige	in conservation; in exploitation	45.9315790	23.1854460
8	Viștea	limestone	yellow-beige	in exploitation	46.7988280	23.4650950
9	Măcin	granite	reddish-grey	in exploitation	45.2357038	28.2179546
10	Pietroasa-Deva	trachyte	grey	in exploitation	45.8700958	22.8706598
11	Codru Babadag	sandstony limestone	yellow-beige	in exploitation	44.8607970	28.6791229
12	Borsec	travertine	beige-yellowish	ceased	46.9651360	25.5699590
13	Măgura Călanului	oolithic and clastic limestone	cream, whitish, cream -yellowish	ceased, Pre-Roman quarry	45.7635790	23.0501571

\* According to EN 12440; \*\* EN 12670 & EN 12407

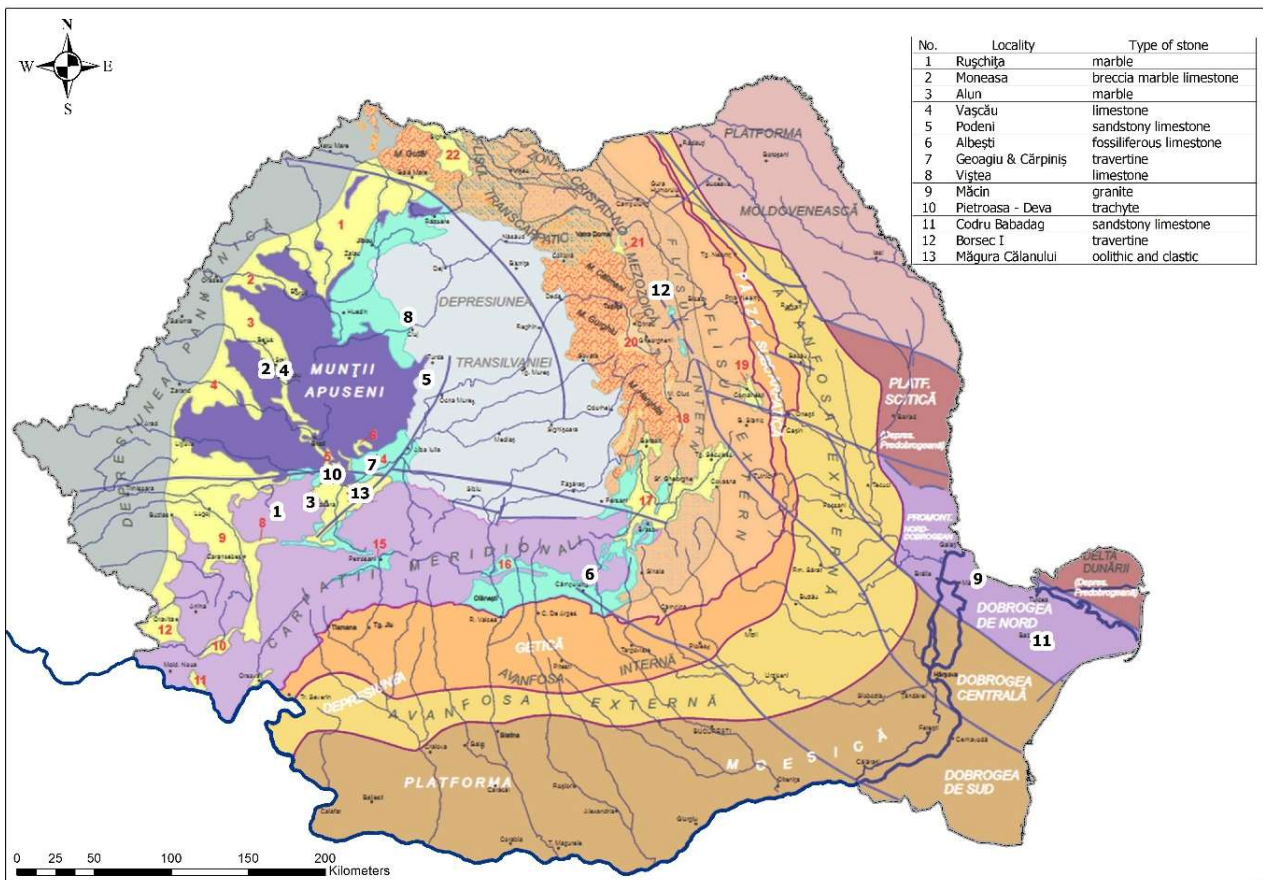


Fig.1 Positioning on the structural map of Romania of the most known ornamental stone with heritage value

The most iconic building of Romania's last years of communism is the Palace of the Parliament (Fig.2a). According to the World Records Academy, the Palace of the Parliament is the third largest administrative building for civil use in the world (with a floor area of 330,000 m<sup>2</sup>), the heaviest building in the world (2,550 m<sup>3</sup>), and the most expensive administrative building in the world (valued at 3 billion euros in 2006). It has 9 above-ground levels and another 9 underground levels, as well as approximately 1000 rooms.

The building was designed and constructed using exclusively Romanian raw materials. From the basic construction materials to the ornamental stones used on the exterior (horizontal and vertical cladding, decorative elements for terraces) and interior (floors, walls, pillars, stairs, balustrades, columns, general frames for openings, etc.), huge carpets, lighting fixtures, furniture, and other textile elements.



(a)



(b)

Fig. 2 (a) The Parliament Palace from Bucharest and outdoor surfaces are covered by approximately 750,000 sqm Podeni and Viştea limestone tiles and panels (Photo: Dan Mihai Bălănescu); (b) Podeni limestone, the main stone used for external cladding and terraces

The construction of this impressive building required an huge amount of stone, corresponding to the size of the building, the most important stones being also the best known for the Romanian reference architecture of the time (1980-1987): Podeni (Fig. 2b) and Viştea limestone for the exterior, and Ruşchiţa and Alun marbles as the preferred materials for the interiors (over 1,000,000 m<sup>3</sup>), Moneasa limestone, and Borsec, Cărpiniş, and Geoagiu travertine (Fig. 3) - already stones with national heritage value at the time of use.

The **Ruşchiţa marble** is the most famous and wide used Romanian ornamental stone. The old quarry is being operative since 1883 and developed by step-by-step expansion (Fig. 3), reaching 130 m depth [Cetean, 2013].



Fig. 3 The oldest organised Ruşchiţa marble quarry

Nowadays, the marble is extracted in other four quarries, allowing to the owners to introduce on market important volumes of this very beautiful marble showing unique ornamental characteristics: high crystallinity and medium size of crystals, good translucence and colours from white and grey to pink, usually with grey veins (Fig. 4).





Fig. 4 Detail of the stone floor along the main hall of the Romanian Palace of Parliament (Rușchița whitish marble, Moneasa reddish and black limestone, Caprioara beige – yellowish limestone)

The Rușchița marble presents good physical-mechanical properties and can be easily cut to size or processed for both indoor and outdoor applications: cladding, flooring, wall and ceiling finishes, stairs, solid masonry units or a very large range of architectural elements.

In Romania, in the last half of the 20th century, Rușchița marble was the main stone for cladding, flooring and architectural elements for a number that cannot be quantified of administrative, public or private buildings. Could be included in this extensive list administrative and diplomatic institutions, City Halls, schools, hotels, cathedrals and churches, shopping centres, airports, underground stations. The most known from the Bucharest capital are the Palace of the Parliament (Fig. 2a), National Library, Telephone Palace, National Bank or Museum of Contemporary Art from Bucharest. The quarry owner's testimony suggests that the marble extracted from the quarry was used in several notable buildings worldwide, including the Parliament buildings in Vienna (Austria) and Budapest (Hungary), the BBC centre in Mainz (Germany), the bathrooms of the Sultan's palace in Brunei, and numerous private buildings in Asia. The official documentation and technical information required to support these claims exist, but their historical applications have not been extensively documented in widely accessible sources, despite their commercial significance.

The most well-known and utilized variety of coloured limestone in Romania is the brecciated **limestone of Moneasa** (Arad County). Exploited regularly since the mid-20th century, Moneasa limestone has two defining characteristics that make it unique and beloved by architects of the period: its brecciated texture, and its predominant reddish colour, with shades ranging from

reddish-brown to pinkish-cream (Fig. 4), but also with a black or grey-black variety. Both colouristic varieties are of the allochemic, macrosparitic type, with a microcrystalline-granular structure consisting of an aggregate of diagenized and widely developed calcite crystals. The mass of the black-grey variety is penetrated by a branched and irregular system of fine fissures filled with a broadly crystallized calcite, with a sugary appearance and mosaic structure. The superior workability comes even now from its good compactness (even though its genesis in the area of brecciated carbonate deposits induces differentiated resistances depending on the colour characteristics) and the calcitic composition [Cetean et al, 1998-2001].

In terms of crystallinity - very fine - and translucency, the **Alun marble** (Hunedoara County) occupies the first place among the Romanian marbles. The "warm" appearance is given by the predominantly yellowish-white (ivory-like), pinkish-white, greyish-white colour, often with a ribbon-like appearance given by reddish-brown or greyish-green veins (Fig. 5). The stone structure is granoblastic, locally even pelitic, and the texture is massive. Microscopic analysis has shown that the marbles in the Alun deposit are composed of 96 - 9% micritic spar calcite, to which are added iron hydroxides in the form of pelicles, quartz up to 3%, muscovite and chlorite approx. 1%, bleached and opaque minerals up to 0.5-1% [Cetean et al, 1998-2001].

The **Vaşcău limestone** (Bihar County) is a rock with a polychrome appearance, with layers of different aspects generally following each other vertically. At the base of the carbonate deposits, there are grey limestones with white or yellowish-green spots and joints filled with calcite. Above them are red limestones with shaly argillites in thick beds of 1-1.5 metres with a monoclinic structure, followed by mottled grey limestones that give way to purple limestones and, at the top, pink, carnelian-white limestones and red breccias in beds with a monoclinic structure. The top layer consists of soil and clayey deluvium. The deposit is massive and tectonically unaffected [Cetean et al, 1998-2001].



Fig. 5 Detail of the stone floor from the Palace of Parliament, made by veined Alun marble and Moneasa reddish breccious limestone

The **Albești limestones** of Eocen age occurring in the area of the Albești village (Argeș County) were exploited for centuries in many quarries [Ilinca et al., 2022]. The useful limestony strata have a total thickness of about 25 m, composed by blueish grey limestones detachable in large blocks and containing coralline algae, porous limestones of yellowish-light grey to reddish colour (the richest in fossils, from large foraminifera as Nummulites, Operculina, Assilina, Discocyclus, molluscs, to echinoids, algae, fish teeth, etc. and greyish white limestones and containing large foraminifera (Nummulites) and fragments of mollusc shells. Taking into consideration the important scientific significance of the Eocene sequences, the Old Quarry was established in 1954 a protected area under the name of Albești Nummulitic Limestone Nature Reserve of geological and palaeontological interest.

Archaeological discoveries show that the oldest evidence of the use of the Albești Limestone is in the 2nd to 3rd centuries A.D., for the construction of a Roman camp. The use of the stone was continued in the Middle Ages (e.g., for the tombstone of an important representative of the local city, who died in 1300) and later on for use in buildings as churches and monasteries, public and private palaces, monuments, mansions, houses, old stone crosses placed mainly at crossroads etc. Up to now, the authors of the first comprehensive study about the heritage value of the Albești limestone [Ilinca et al., 2022] have identified 368 buildings and other constructions in which this stone was used, of which 204 are on the List of Historical Monuments of Romania. Some examples are: the National History Museum of Romania, Horezu Monastery [1693], Palace of the Ministry of Public Works [1910] from Bucharest (City Hall since 1948),

Curtea de Argeș Monastery [1526] (Fig.6), Stavropoleus Church [1724], the Monastery of the Three Hierarchs of Iași (Iași County) [1639], the Arch of Triumph monument [1922], the Peleş Castle (Sinaia town, Prahova County), the Central Market Halls (Ploiești city, Prahova County), Brâncoveanu Monastery (Brașov County), etc.).



Fig. 6 Curtea de Argeș Monastery, cladled by Albești limestone

The **travertines in the Geoagiu - Cărpiniș - Banpotoc** (Hunedoara County) area were formed by the deposition of excess carbonate material by the thermal springs that appeared on the lines of minimum resistance in the limestones and dolomites of the Rapolt crystalline island. Under the effect of their own weight, the travertines slid on the clay bed and were deposited in lower areas. Due to the inconsistent flow rates and their uniform distribution in space, the carbonate deposits have variable thicknesses and are discontinuous. In general, the travertine is non-homogeneous in direction and depth, with irregular intercalations of different colours and variable compactness. The rock's compactness in the deposit varies from compact to vacuolar and strongly brecciated. The structure of the travertines is of chemical precipitation, and the texture is vacuolar. The travertines are of the allochemic type with sparitic and microsparitic binder, aragonite macrosparitic travertines, travertines, sometimes with bioclasts or of the arenitic type. Their colour varies from cream to yellowish, yellowish-brown, and brown. The compact brown travertines have been described as the Banpotoc limestones [Cetean et al, 1998-2001].

The **Viștea limestone** is igneous, of allochemic type, and has a massive character, with cracks dividing it into blocks. Its colour is white-yellowish-cream, and on the surface, it becomes grayish-brown in fresh cracks due to the oxidation of organic substances. The fracture is irregular and the porous character of the limestones is obvious. The rock structure is post-depositional, fenestral type, with primary and secondary voids. Intraclasts are represented by remnants of carbonate

sediments undergoing lithification or compacted with an angular outline) and bioclasts (skeletons, crusts of crinoids, gastropods, brachiopods, foraminifera). Extrabasinal particles represent 20% of the total clasts. The binder is orthochemical and is represented by micritic and sparitic calcite (30-50% of the total) and sparitic (15-30 to 50-60% of the total calcite). Clay minerals have a proportion of 15-25% of the rock mass [Cetean et al, 1998-2001]. Like the Podeni limestone, it was intensively used locally, but the most important quantity was transported for the exterior cladding of the Palace of Parliament (Fig. 2a).

The **Măcin granites** (Tulcea County) are the oldest magmatic rocks from Romania, with a very beautiful aspect and good physical and mechanical characteristics (Table 3). The area of the Măcin Mountains is one of the most important granite resources for ornamental building industry, but the Măcin Mountains are a beautiful area for tourism, too and now included in a protected area. With the start of the regular and extensive exploitation of Macin granite at the beginning of the 19th century, master stonemasons were brought from Italy, Greece and Bulgaria. Thus, in addition to the raw or shaped stone blocks used in public buildings and houses in the area that is difficult to date, the production of tiles for cladding and flooring and kerbs, slabs and setts for external paving began. The granite was thus sent for many of the administrative buildings in Tulcea and Constanta counties, but also for other big cities in the country, especially for horizontal exterior and interior cladding [Cetean et al, 1998-2001].

The **Pietroasa-Deva andesites** (Hunedoara County) consist of a subvolcanic body of greyish andesites, sometimes altered and argillaceous. The deposit is in the form of a massive, stock-like body with a cover of altered and highly fractured andesites. The massive structure of the deposit is readily apparent in the current quarry opening, but also in its volcanic palaeoform, which is consistent with the relief. Microscopic analysis shows that the Magura andesites are amphibole (10-20%), pyroxene (20-30%) and biotite (2-5%) andesites [Cetean et al, 1998-2001]. The Pietroasa-Deva area (although not the present quarry site) has been proved to be one of the historical quarries on the Romanian territory [Barbu et. Al, 2021], source of stone for some building elements of the cult sites of Sarmizegetusa Regia, capital of the Dacian kingdom between the 1st century BC and the beginning of the 2nd century AD [Mârza, 1997]. Also, starting from the beginning of the 20<sup>th</sup> century, this stone was used for the construction of important administrative or public buildings in Bucharest, Cluj, Arad, Timisoara, Alba-Iulia, Budapest, Vienna or for many commemorative and funerary monuments and

architectural elements for private, public and religious buildings in the city and county.

**Codru Babadag limestones** have granular, microcrystalline and cryptocrystalline structures and organic and chemical precipitation textures. The representative type is gritty limestone, from which transitions are made to calcareous sandstones, while spongolite occurs as a bioconstructed rock in which the silica in the spicules is sometimes replaced by calcite [<https://eternitystone.ro/piatra-naturala/>]. The colours range is from yellowish-white to yellowish, yellowish-brown, grayish-yellowish and grayish-whitish. The stone quarry was inaugurated during the reign of King Charles I (the end of the 19th century) and operates with a special permit in a "site of community importance" and "special wildlife protection area".

The **Borsec travertine** presented variations in terms of thickness and quality in each of the quarries in operation. There are numerous karst phenomena encountered in the travertine mass, and the void volume is about 20%. The colour of the rock varies from white-yellowish to grey-slightly reddish yellowish, vacuolar, with the appearance of decayed wood. It was mined in two petrographic facies: micro-sparitic allochemic travertines and microsparitic allochemic travertines from calcite (88-90%) associated with quartz, muscovite and other minerals. Used extensively in public and administrative buildings, as an ornamental stone for horizontal and vertical cladding. The subway in Bucharest is one of the most cognoscible places, but also the Palace of the Parliament interior cladding. Currently the quarrying is stopped.

The **Măgura Călanului limestone** quarry from Hunedoara County (Fig. 7), probably the most spectacular ancient monuments of its kind outside the Greco-Roman world, was exploited only in the ancient period and is well preserved. It is considered to be the main source of stone for the ashlar blocks used at the monumental structures of Sarmizegetusa Regia, the capital of the Dacian Kingdom, and to the ensemble of fortresses and fortifications around it: Costești-Cetățuie, Costești-Blidaru (Fig. 8), Fețele Albe, Piatra Roșie, Bănița, from 1999 inscribed on the World Heritage List as part of the serial site "Dacian Fortresses in the Orăștie Mountains (Cetean et al., 2022.; Pețan, 2022)



Fig. 7 The western exploitation side of the Măgura Călanului historic limestone quarry

Although the limestone from Măgura Călanului has the least ornamental characteristics among the 13 stone types, the oolitic and clastic limestones from this historical quarry are the most important and relevant Romanian stone, presenting the highest and easy recognizable heritage value.



Fig. 8 The Costești - Blidaru fortress, one of the Dacian historic monuments erected by Măgura Călanului ashlar limestone blocks

#### INFORMATIVE QUALITATIVE CHARACTERISATION OF THE SELECTED HERITAGE STONES

The main physical-mechanical properties of the stones selected for their ornamental characteristics, durabilities in public or private works, historical or modern ones, of national and global heritage significance, are detailed in Table II, where:

*1 – Rușchița marble, 2 – Moneasa limestone, 3 – Alun marble, 4 – Vașcău limestone, 5 – Podeni limestone, 6 – Albești limestone, 7 - Geoagiu & Cărpiniș limestones, 8 – Viștea limestone, 9 – Măcin granite, 10 - Pietroasa-Deva trachyte, 11 - Codru Babadag limestone, 12 – Borsec travertine, 13 - Măgura Călanului limestone.*

Table II – Characteristic of the selected Romanian ornamental stone with heritage value

Characteristic	1	2	3	4	5	6*	7	8	9	10	11	12	13
Density [Kg /dm <sup>3</sup> ]	2.75	2.72-2.74	2.74	2.72-2.82	2.70		2.35-2.60	2.75	2.64-2.71	2.67			2.65
Apparent density [Kg /dm <sup>3</sup> ]	2.71	2.70	2.72	2.70-2.72	1.99		2.25-2.49	2.23	2.65	2.43	1.96-2.15	2.30	2.20-2.27
Total porosity [%]	0.37-0.74	0.50-0.75	0.53	0.37-3.5	18.10		9.4-16	18.9	0.22-0.25	9.0	9.66-22.54		4.82-14.10
Water absorption at normal pressure [%]	0.09-0.21	0.08 - 0.12	0.15	0.06-0.14	9.60		1.4-3.66	4.54	0.08-0.14	2.54-3.20	4.53-11.2	1.80	1.61-7.18
Compression strength [N/mm <sup>2</sup> ]	85-124	92 - 102	115	90-147	27.3		32-66	45	125-160	120			63
Flexural strength [MPa]	13.3-19.3						10.6		8.2-16				6.80

\* un-available free information

#### CONCLUSIONS

From a total of over 130 ornamental stones from Romania in the official lists, 28 of them were selected that fulfil the conditions of national and global heritage significance. A number of 12 quarries in activity or that worked at least in the 20th century were selected. To these was added the most important quarry from a historical point

of view, which has not worked since the 2nd century AD. The process of substantiating the dossier of each stone regarding its heritage value is ongoing only for a small part of them. With the exception of historically exploited quarries, the most challenging aspect remains the compilation of databases with the objectives where these stones were used and for which there are no documented evidences, involving substantial resources to be achieved.

**Acknowledgments:** This research was funded by the Ministry of Research, Innovation and Digitization through the projects: *RoQ-Stone - Romanian stone for construction – quality, cultural heritage value, scientific designation* (contract no. 28N/2019, project code PN 19-45-02-01) and *ProGeo-RO - Increasing climate resilience and environmental sustainability by knowing and monitoring of the Romanian geological heritage with international relevance* (contract no. 34N/2023, project code PN 23-39-03-01).

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# Wet and dry conditions influence on natural stone cladding performance with dowel-pin anchor system

V. Pires<sup>1\*</sup>, C. Lisci<sup>2,3</sup>, F. Sitzia<sup>2,3</sup>, J. Mirao<sup>2,3</sup>, T. Alves<sup>3</sup>, P. Faria<sup>4</sup>

(24) HERCULES Laboratory, Institute for Advanced Studies and Research, LEM | Laboratório de Mecânicos da Universidade de Évora, University of Évora, Évora (Portugal), e-mail: [vlcp@uevora.pt](mailto:vlcp@uevora.pt), web: <https://www.cienciavita.pt/en/FB15-B1D0-606A>, \*Corresponding author

(25) HERCULES Laboratory, Institute for Advanced Studies and Research, Geosciences Department, School of Sciences and Technology, University of Évora, Évora (Portugal)

(26) Geosciences Department, School of Sciences and Technology, University of Évora, Rua Romão Ramalho 59, 7000-671 Evora (Portugal)

(27) GEOBIOTEC and Geosciences Department, School of Sciences and Technology, University of Évora, Rua Romão Ramalho 59, 7000-671 Evora (Portugal)

**Summary:** *Natural stone cladding with dowel-pinned anchor systems are commonly used in building façades. Their loading behaviour and mechanical performance in wet and dry conditions is not fully understood, particularly the correlations between the pore structure and the anchorage breaking loads. To answer this concern, a series of breaking load tests on dowel-pinned anchors in both soaked and dry condition were carried out for two Portuguese limestones commonly settled in architecture. The load bearing capacity by analysing the rupture geometry and rupture angles was enquired. These data were co-related to the porous structure assessed by helium and mercury porosimetry, water absorption and SEM microstructural analyses. The results were found to generally link saturated stones with higher open porosity to lower anchorage breaking loads. The results of this research can improve the structural safety of stone claddings and will increase awareness of the importance of water absorption in their performance.*

**Key words:** *Natural stone from Portugal, Porosimetry, Façade, Anchor breaking load, Dowel-pin anchor*

## 1. Introduction

Natural stone cladding with dowel-pinned anchor systems has been commonly used in building façades. Still, their loading behaviour and mechanical performance in soaked conditions is not fully understood compared to the traditional available test results in dry conditions (20°C), particularly the correlations between the pore structure and the anchorage breaking loads. The focus on this study was on performing a series of loading tests on dowel-pinned anchors in both soaked/saturated and dry conditions. Experimental work was carried out on two Portuguese limestones commonly selected as cladding materials. The failure of the limestones and stainless-steel pins was determined based on their respective load-bearing capacities, which finally represented the breaking load. It is our intention that this research will be of particular interest for façade engineers and architects, helping particularly on: i) understanding structural safety of dowel-pinned claddings; ii) increase awareness on the importance of stone materials' water absorption in their performance in wet environmental conditions.

Natural stone is a building material with widespread use over time, mainly due to its robustness, durability and availability in a variety of colors and textures. If, for many centuries, it was practically the only structural material for enduring longstanding construction, today, with the appearance of new construction materials, especially reinforced concrete, its use has been progressively dedicated to cladding of building façades. Under this usage, technological developments have enabled the delivery of elements of natural stone cladding with ever larger dimensions and smaller thicknesses, contributing to economic competitiveness in its use, particularly when assessed in the complete lifecycle of construction. The progressive increase in stone slab size demands new requirements regarding the capacity to withstand forces, particularly wind and seismic ones and the attachment system, crucial in view of the potential damage that a detachment of a high weight slab can cause. These elements must therefore be the subject of a structural design to ensure an appropriate level of safety (de Sousa Camposinhos, 2014), (Hoigard & Scheffler, 2007).

When selecting a stone cladding material according to its mechanical performance, one of the most important properties is described by strength. In this situation, strength is typically determined through laboratorial destructive flexural (EN 12372), anchorage (EN 13364) or compressive tests (EN 1926). These methods are used to evaluate stone mechanical quality and estimate the maximum allowable load before failure and none of these European standards remark the possibility of performing the test in both saturated and dry conditions. Previous works on stone physical-mechanical properties that affect stone behaviour as a construction material have already been presented by several authors focusing on: i) salt crystallization effect on porous natural stone flexural and compressive strength (Benavente et al., 2004); ii) freeze-thaw weathering effect on stones flexural and compressive strength performance (Martínez-Martínez et al., 2013); iii) the effect of humidity in façade claddings (Pereira et al., 2018); iv) the interaction between rainwater and natural stone finishing in façades and floors (Sitzia et al., 2022); v) the influence of the pore system of sedimentary rocks in their decay during several aging tests (Molina et al., 2011); vi) long term changes and natural stone variability (Yates et al., 2012). Still, a great number of works related with stone façades performance are focused on stone masonry walls and heritage (Corradi et al., 2003), (García et al., 2012), (Ludovico-Marques et al., 2012), (Papamichos et al., n.d.) and physical-mechanical performance studies on natural stones employed as cladding materials in contemporaneous heritage and façades is less exploited.

Nevertheless, the authors have already dedicated their research to this topic focused on natural stone physical-mechanical performance to develop and improve stone selection indexes and criteria suitable for each application requirements (Simão et al., 2010), (Lisci, Pires, et al., 2022), (Lisci, Sitzia, et al., 2022), (Vera Pires et al., 2023), (V. Pires et al., 2014), (V. Pires et al., 2022), (V. Pires et al., 2011).

Among natural weathering agents, water is one of the most relevant. Materials in the built environment are also exposed to the changing effects of water and this fluid is an important medium for the transport and alteration effects of other agents such as salts, biological agents and other diverse substances that can be deposited from water (Alves et al., 2021). The influence of water content in the physical mechanical performance of natural stone and the correlations between several properties in saturated and dry conditions have been reported by several authors as summarized in Table I. Still, to the authors knowledge the comparison between saturated and dry anchorage rupture load for dowel-pin anchor systems has not been approached.

## 2. Materials

Two Portuguese limestones widely used in contemporaneous architectural and civil heritage projects selected for this study: CM and BL (Fig.1). CM limestone is a light-beige medium soft carbonate rock with recognizable depositional texture of sustained grain type. BL is a blue-greyish compact limestone rich in fossils and organic matter.

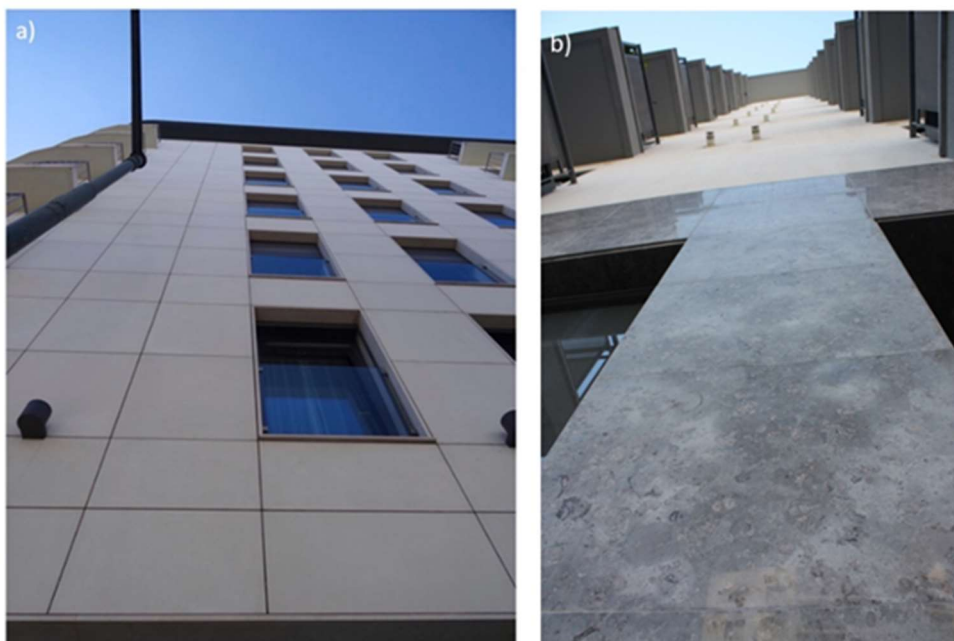


Fig.1 Examples of architectural uses of the selected lithotypes: CM (a) and BL (b) as cladding materials in building facades.

Table 1 A compilation of some of the most relevant studies and correlations made in saturated and dry conditions in natural stone material

Authors	Saturation State	Correlated properties	Stone type
Rabat, Á. Cano, M. Tomás, R. (Rabat et al., 2020)	Dry and saturated	Uniaxial compressive strength, density, ultrasonic wave velocities (Vp and Vs), dynamic and static elastic modulus	Calcarenites
Török, Á. Vásárhelyi, B. (Török & Vásárhelyi, 2010)	Dry and saturated	Apparent density, uniaxial compressive strength, ultrasonic wave velocity (Vp)	Travertine
Verstrynge, E. Adriaens, R. Elsen, J. Van Balen, K. (Verstrynge et al., 2014)	Dry and saturated	Porosity, apparent density and ultrasonic tests, compressive tests, Acoustic Emission (AE)-controlled creep tests and X-ray microfocus Computed Tomography (microCT) at different load levels.	Diestian and Brusselian ferruginous sandstone
Erguler, Z.A. Ulusay, R.(Erguler & Ulusay, 2009)	Dry and saturated	Porosity, water absorption, Uniaxial compressive strength, elastic modulus, needle penetration resistance (NPR)	Clay-bearing rocks from Turkey (bearing rocks such as claystone, mudstone, siltstone, marl and tuff)
Kim, E. Changani, H. (Kim & Changani, 2016)	Dry and saturated	Porosimetry, wave velocities (Vp and Vs), static, fast and dynamic loading compressive tests, Static loading indirect tensile tests (Brazilian method)	Red and Buff sandstone
Si, Wenpeng Di, Bangrang Wei, Jianxin Li, Qian (Si et al., 2016)	Dry and saturated	Ultrasonic P-wave and S-wave velocities	Artificial sandstone

### 3. Methods

Petrographic analyses were made with Hirox-01 digital microscope. Thin sections of 30 µm thickness were analysed to identify the mineralogical compositions, rock fabric, and fossils.

Limestones mineralogical composition was made by µXRD with a Bruker D8 Discover diffractometer (Bruker Company, AXS Karlsruhe, Germany) with CuKα radiation tube operating at 40 kV and 40 mA. The XRD peaks were measured between 2° to 75° 2θ, with 1s counting time per point. The Powder Diffraction Database (PDF-ICDD,

International Centre for Diffraction Data) using the Bruker EVA software was held to identify the crystalline phase.

SEM analyses were performed with a scanning electron microscope HITACHI S3700N. This technique was selected for microstructural analysis and to identify the differences between the limestones microtexture.

Apparent density and open porosity were determined according to EN 1936 standard in specimens with 50 mm side.



A Micrometric AccuPyc II 1345 Helyum pycnometer was used for the real ( $V_R$ ) and solid volume ( $V_S$ ) assessment and this result allowed to assess the real density ( $\rho_R$ ), closed porosity ( $\Phi_C$ ), total porosity ( $\Phi_T$ ) of each limestone, the selected experimental methodology was already employed in a previous research by some of the authors (Fabio et al., 2020).

Determination of pore size and open porosity in solid materials by mercury porosimetry was made according to ISO 15901-1:2016 standard. Tests were performed at the accredited *Laboratório de Ensaios, Desgaste de Materiais* (IPN LED&MAT).

Water absorption at atmospheric pressure ( $W_{abs}$ ) was determined according to EN 13755 and the saturation index (S.I) was assessed.

Anchor breaking load were determined for both limestones (CM and BL) in saturated and dry conditions.

For each limestones, three slabs with dimensions  $400 \times 200 \times 30$  mm<sup>3</sup>, containing cylindrical holes of 8 mm diameter and 35 mm depth in which a stainless-steel pin was fixed using the standard recommended cement were tested. Tests were conducted applying a load perpendicularly to the bedding planes and following EN 13364 standard. Rupture angles ( $\alpha^\circ$ ) from the detached cones were measured after each test. Tests were conducted in a Pegasil by Zipor universal press and using a load cell of 25 kN (Fig 2a).

Compressive strength ( $R_c$ ) was assessed saturated and wet conditions, in specimens with 50 mm side by uniaxial compression test in accordance with EN 1926 standard. The equipment used was a PEGASIL EL200 hydraulic press with a capacity of 1200 kN (Fig. 2b). Load was applied perpendicular to any stratification plane or discontinuities when evident.

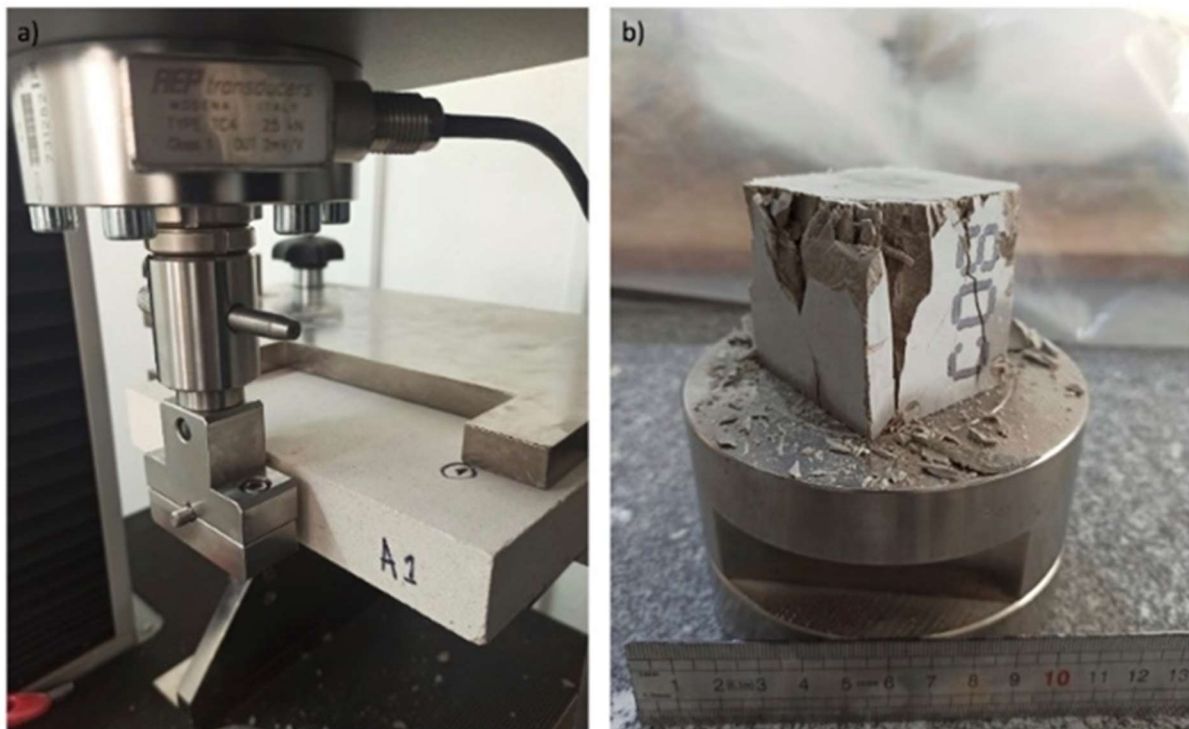


Fig.2 Experimental set-up used in the mechanical tests: a) anchor breaking load test in dowel-pin anchor system, b) uniaxial compression strength specimen after rupture

#### 4. Results and Discussion

##### 4.1. Stones petrographic and mineralogical composition

CM limestone is a light-beige medium soft carbonate rock with recognizable depositional texture of sustained grain type. 65% vol. of the rock consists of allochems such oololiths and peloids. The interstices are occupied by microcrystalline sparitic cement ( $\approx 10\%$  vol.). A  $\approx 15\%$  vol. consists of fossils and bioclasts (i.e., bivalves,

brachiopods, gastropods, calcareous algae etc.). According to Folk classification, the stone is characterized as bio-pel-oo-micrite (Fig.3 a), b) and c)). The XRD analysis confirm the 100% presence of calcite.

BL is a blue-greyish compact limestone characterized by  $> 90\%$  vol. of a micritic matrix containing few percentages of clay minerals ( $< 2\%$  vol.). Allochems of quartz in a range from 0.4% to 1.8% vol together with impurities of pyrite are present. The stone is marked by the abundant presence of organic matter up to 6% vol.

The bioclastic component is also relevant. According to Folk classification, the stone is classified as biomicrite (Fig.3 d, e) and f)). XRD analyses, beside calcite (~ 95%),

includes the presence of accessory minerals such as quartz (~ 3%) and clays (~ 2%).

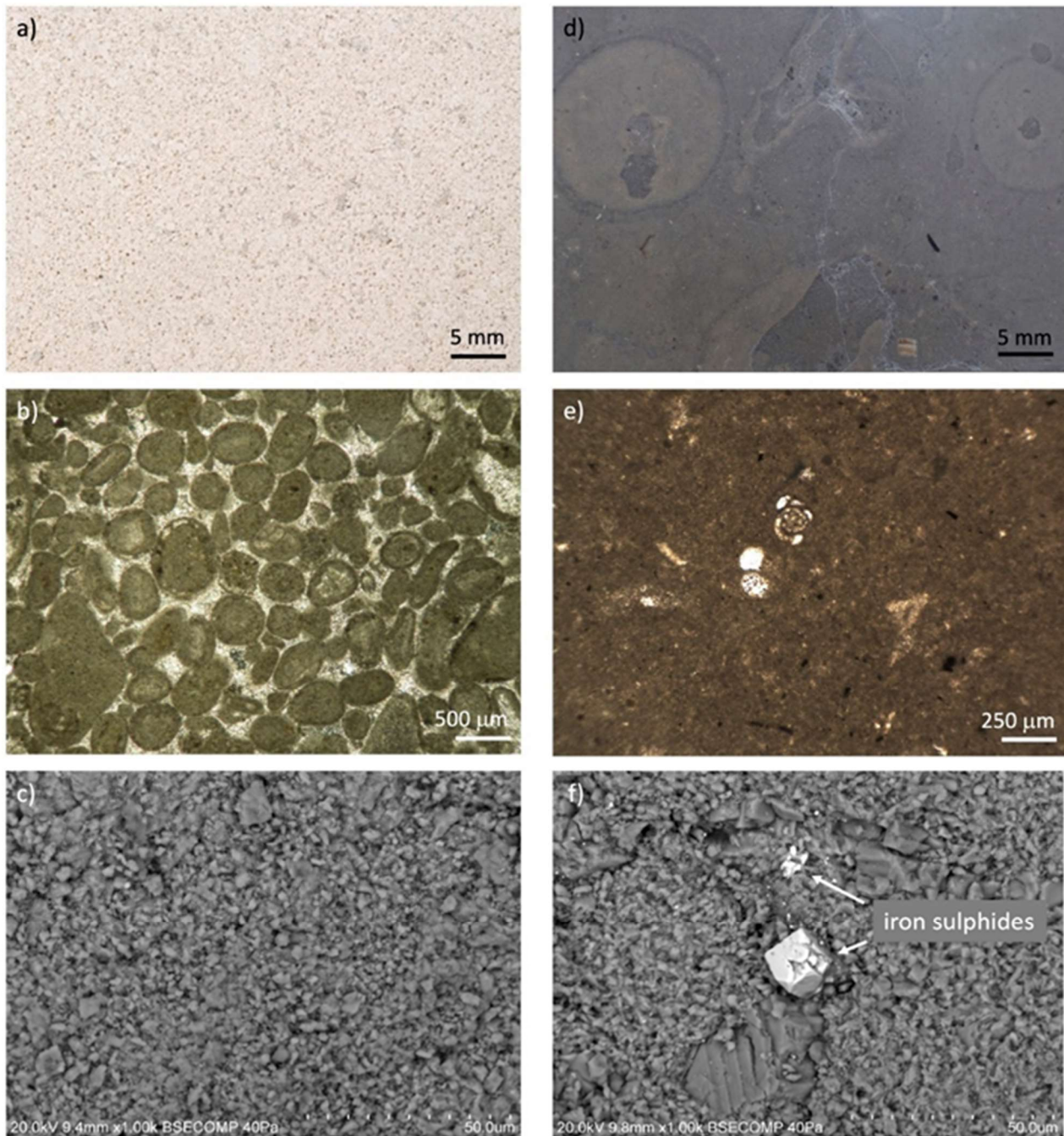


Fig.3 Building stones under study: a) and b): Macroscopic feature and image in thin section of CM, c) SEM image showing the limestone micro texture; d) and e): BL and its petrographic traits, f) SEM microtexture of BL limestone depicting the presence of iron sulphides.

#### 4.2. Water absorption, porosity & density

According to EN 1936 for dense, low porosity stones the differences between real and apparent density, as well as between open porosity and total porosity, can be assumed as very small. For these stones it is often

assumed that it is sufficient to determine the apparent density and the open porosity. Indeed, this is nearly accurate for BL limestone however, helium and mercury porosimetry were also selected to fully understand the water impact on these stone anchor and compression strength in both saturated and dry condition. Porosity in general is considered as one of the most important

physical characteristics of a stone not only for cladding but for also for flooring, masonry and countertops applications. As water is trapped within stone pores it might be responsible for dissolution, deterioration, growth of biological species, internal stress due to water freezing, crystallization and hydration of salts, among others (Mosquera et al., 2000).

Density, porosity, pore size diameter and water absorption at atmospheric pressure results are shown in Table II. Clear differences in the porous structure of the selected limestones are depicted: *i)* CM has averagely 91% more open pores than BL limestone according to the Helium porosimetry method (12.54 % and 1.09% respectively). This difference is consistent with the results achieved from EN 1936 method where CMN

open porosity is 86% higher than the one calculated for BL (8.43% and 1.21% correspondingly); *ii)* Apparent density results show higher differences in EN 1926 comparatively to the mercury porosimetry method. This is mainly because both methods have significant differences since in the EN 1936 specimens are soaked in water at lower vacuum pressures (0.2 Psi) and mercury porosimetry used a higher pressure of 0.5 Psi; *iv)* Average pore diameter expose a difference of 93% between CM (0.29  $\mu\text{m}$ ) and BL (0.02  $\mu\text{m}$ ). Implication that CM limestone is much more susceptible to the interaction with water; *v)* water absorption at atmospheric pressure also present relevant differences between CM and BL, indicating that CM absorbs 87% more water than BL.

Table II. Saturated and dry uniaxial compressive strength results for the selected limestones in which is seen the difference caused by saturation the stones performance.

Test method	Helium porosimetry (Fabio et al., 2020)				EN 1936		EN 13755	Mercury porosimetry ISO 15901-1:2016 standard		
	$\rho_R$ [g/cm <sup>3</sup> ]	$\Phi_{He}$ [%]	$\Phi_C$ [%]	$\Phi_T$ [%]	$\rho_{B\_H_2O}$ [g/cm <sup>3</sup> ]	$\Phi_{H_2O}$ [%]	$W_{abs}$ [%]	$P_{diam\_Hg}$ [ $\mu\text{m}$ ]	$\rho_{B\_Hg}$ [g/cm <sup>3</sup> ]	$\Phi_{Hg}$ [%]
CM	2.70 ± 0.01	12.54 ± 0.5	1.80 ± 0.42	14.33 ± 0.52	2.35 ± 0.03	8.43 ± 0.33	3.57 ± 0.15	0.29	2.70	18.0
BL	2.70 ± 0.01	1.09 ± 0.92	1.34 ± 0.36	2.43 ± 0.77	2.67 ± 0.02	1.21 ± 0.90	0.45 ± 0.34	0.02	2.60	2.20

$\rho_R$  = Real Density through helium porosimetry;  $\Phi_{He}$  = Helium open porosity;  $\Phi_C$  = Helium closed Porosity;  $\Phi_T$  = Helium total Porosity  
 $\rho_{B\_H_2O}$  = Apparent Density in water;  $\Phi_{H_2O}$  = Open Porosity in water;  $W_{abs}$  = Water absorption at atmospheric pressure;  $P_{diam\_Hg}$  = Average open pore diameter through mercury porosimetry;  $\rho_{B\_Hg}$  = Average apparent density through mercury porosimetry;  $\Phi_{Hg}$  = Average open porosity through mercury porosimetry

### 4.3. Saturated and Dry - Uniaxial Compression Strength

The data set present in Table II show the uniaxial compressive strength results in saturated and dry conditions. Results highlight a decrease of 28% in CM compressive strength from dry to saturated test condition. An increase in the variation coefficient was also detected. Authors believe that this is probably related to sampling issues. BL results, on the other way, present a different trend with an apparent increase in compression strength. Since BL is a low porous limestone, this result was interpreted as a comparable between dry and saturated conditions meaning that this stone uniaxial compressive strength is less susceptible of being altered by the presence of water at room temperature. However, it is important to highlight that, these results cannot affirm that BL will not be altered by the presence of water in the long term. Previous works have report issued in similar lithotypes linked to the

presence of clayed materials that altered the stones mechanical performance in a permanent and critical way (Simão et al., 2010), (V. Pires et al., 2022).

Similarly, to what was discussed in other studies, the open porosity and pore size diameters seems to be the prevailing control factors of durability and strength. Its influence is not equally important under dry and water saturated conditions (Török & Vásárhelyi, 2010).

Table II. Saturated and dry uniaxial compressive strength results for the selected limestones in which is seen the difference caused by saturation the stones performance.

Limestone	CM		BL	
	Dry	Saturated	Dry	Saturated
Specimen / Test condition				
Mean value	85	61	137	154
Standard deviation	13	15	20	56
VC (%)	15	24	15	36
Δ %	-28		12	

#### 4.4. Saturated and Dry - Anchor breaking load and rupture analysis

Saturated and dry anchor breaking load are shown in Table III. Similarly, to what was observed in compression strength, CM depicted a drop of 10% in anchor breaking load. The same trend was detected in BL. This drop similarity is, highly probably, linked this test method particularities which is also dependent on other factors besides stones heterogeneities and specimens' geometry. Anchor breaking load test is also dependent on the load transfer between the stainless-steel pin and this is conditioned by the quality of the pin placement through standardized cement. Since this operation is done manually when preparing the specimens, some uncertainty factor might mask the difference between CM and BL porous structure.

Anchor breaking load rupture angles examples in dry and saturated conditions are show in Fig.4. Results were as follows:  $38^{\circ} \pm 8^{\circ}$  (saturated) and  $30^{\circ} \pm 8^{\circ}$  (dry) in BL and

$35^{\circ} \pm 9^{\circ}$  (saturated) and  $29^{\circ} \pm 7^{\circ}$  (dry) in CM. Data seem to suggest higher rupture angle and smaller rupture areas in saturated conditions however breaking loads were lower in both tested stones. For this reason, anchor breaking should be determined through saturated and dry condition as one of the main characteristics studied when stone is applied in a ventilated façade (V. Pires et al., 2013), (V. Pires et al., 2011), (V. Pires et al., 2014) .

The dispersion observed on anchor breaking load test results might be considered high, but, taking into account the characteristics of brittle materials, like natural stones, and the scatter usually obtained in flexural, tensile and compression tests, the range of values is within the expected variations (Amaral, Guerra Rosa, et al., 2008), (V. Pires et al., 2013), (Amaral, Fernandes, et al., 2008).

Table III. Saturated and dry anchor breaking load results for the selected limestones in which is seen the difference caused by saturation the stones performance.

Limestone	CM		BL	
	Dry	Saturated	Dry	Saturated
Specimen / Test condition				
Mean value	1571,2	1421,4	2480,8	2243,54
Standard deviation	349,4	211,5	552,4	323,7
VC (%)	22	15	22	14
Δ %	- 10		- 10	

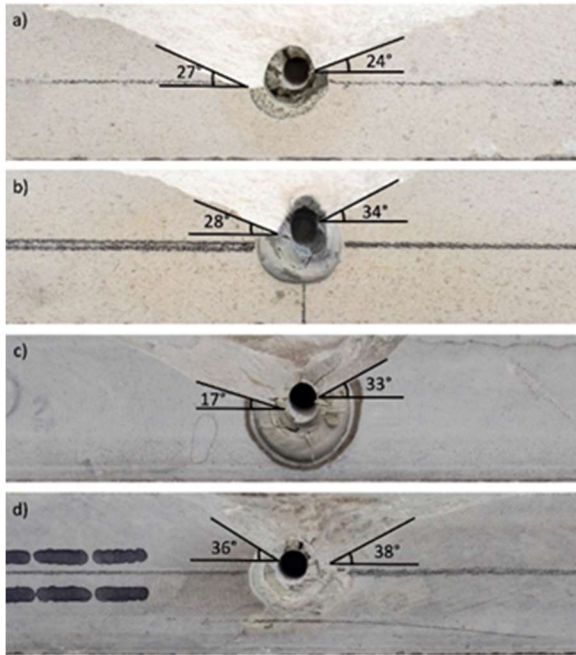


Fig.4 Anchor breaking load rupture angles in both dry and saturated conditions: a) and b) CM rupture angle measurement in both dry and saturated test conditions and c) and d) BL rupture angle measurement in both dry and saturated conditions.

## 5. Conclusions

A series of physical properties linked to natural stone porous structure, uniaxial compression strength tests and loading tests on dowel-pinned anchors in both soaked/saturated and dry conditions were carried out for two Portuguese limestones commonly selected as

**Funding and Acknowledgments:** Carla Lisici gratefully acknowledges FCT for the grant SFRH/BD/149699/2019 co-funded by the European Social Fund (ESF) and MEC national funds. The authors gratefully acknowledge the following funding sources: i) INOVSTONE4.0 (POCI-01-0247-FEDER-024535), co-financed by the European Union through the European Regional Development Fund (FEDER); ii) Fundação para a Ciência e Tecnologia (FCT) under projects UIDB/04449/2020 and UIDP/04449/2020—through HERCULES laboratory and European Community – Portugal; iii) Grant Contract number: Sustainable Stone by Portugal Call: 2021-C05i0101-02 – *agendas mobilizadoras para a reindustrialização – PRR*, Proposal: C632482988-00467016. Fabio Sitzia gratefully acknowledge the program Recursos Humanos Altamente Qualificados for the contract with Ref. ALT2059-2019-24. Vera Pires gratefully acknowledge the Contrato Programa entre FCT e a Universidade de Évora no âmbito do concurso estímulo ao emprego científico institucional 2018.

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cladding materials. Results show that the uniaxial compression strength depends on the stone porous structure. The load-bearing capacity is less dependent on the stones porous structure and the and it is also simultaneous associated to the strength of the steel pin and the rupture resistance of the dowel hole. More conclusions are given below:

a) Experimental uniaxial compression strength data suggest that BL is less dependent on water saturation, potentially suggesting that this stone performance is less affect by water. This result is only valid in isolated conditions and previous alteration studies on this lithotype and the presence of mineralogical impurities should be considered for suitable stone selection and cladding dimensioning.

b) CM limestone presented relevant compression and anchor breaking load drops when we compare dry and saturated results. This fact should be accounted in the cladding panel dimensioning when design the façade and can be overcome through stone slab thickness increment.

c) In general, the experimental results linked saturated stones with higher open porosity to lower anchorage breaking loads. The results of this research aimed improve the structural safety of several dowel-pinned stone claddings and the awareness increase of the importance of stone materials porous structure analysis in their performance and in stone façades cladding design.

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# CONTINUOUS ADDITIVE MANUFACTURING PROCESSES THROUGH THE USE OF LIME WASTE SLUDGE (Extrusion and Binder Jetting)

L. Gouveia<sup>1\*</sup>, A. Mateus<sup>1\*\*</sup>, F. Gaspar<sup>1\*\*\*</sup>, J. Fernandes<sup>2</sup>, L. Oliveira<sup>3</sup>, Alexandre Vieira<sup>5</sup>, D. Pereira<sup>4</sup>,

(28) Centre for rapid and sustainable product development - Polytechnic of Leiria, \*[liliana.gouveia@ipleiria.pt](mailto:liliana.gouveia@ipleiria.pt); \*\*[artur.mateus@ipleiria.pt](mailto:artur.mateus@ipleiria.pt); \*\*\* [florindo.gaspar@ipleiria.pt](mailto:florindo.gaspar@ipleiria.pt);

(29) MVC – Mármore de Alcobaça, Lda., [josefernandes@mvc.pt](mailto:josefernandes@mvc.pt);

(30) AMCubed, Lda, [luismso@amcubed.com](mailto:luismso@amcubed.com);

(31) HRV - Equipamentos de Processo, S.A., [diogo.pereira@hrv.pt](mailto:diogo.pereira@hrv.pt);

(32) Mármore Vigário, Lda

**Summary:** *Alongside technological development in the various sectors of industry, this research work has focused on the developing of processes for the recovery and to value limestone waste sludge derived from the extraction and transformation of ornamental rock, which are destined for landfill.*

*The present study evaluates solutions for the incorporation of these residues in the form of paste or powder, in continuous additive manufacturing processes, as aggregates on cement-based mixtures, thus attributing characteristics of a by-product, allowing the reduction of the residual volume, in the inclusion of design in pieces with added value, with applications in the sectors of Construction, Architecture, Landscaping and Consumer Goods.*

**Key words:** *3D printing, additive manufacturing, stone sludge, extrusion, binder jetting*

## 1. Introduction

The ornamental stone transformation activity has as its main processes the cutting and sawing of raw blocks from quarries, followed by various complementary finishing processes until obtaining the final product. All these transformation processes generate high amounts of waste, 52% in the form of fragments and 27% in the form of pastes, causing high costs for companies in this sector.

This study aims to develop a continuous digital manufacturing system (Binder Jetting and extrusion), with a differentiated configuration in the reuse of this calcium carbonate-based residue, for mass production with customization of complex geometric parts, creating design with added value. The main connection between these two processes lies in the initial state of the raw material, which may be powder or paste, interconnected with a binding material, which in the present study was the Portland cement.

### *Additive manufacturing process by Binder Jetting*

The additive manufacturing process by Binder Jetting, as the name implies, consists of depositing a binding agent on successive layers of raw material. In this process there is a powder reservoir that feeds the printing bed with the aid of a roller that evens out the layer. Printing

is carried out, layer by layer, with a head that deposits a binder in the zones of the defined geometry. The final piece is, in turn, obtained by joining all the layers of powder and the design printed by the binder. It should be noted that this process does not require support structures given the envelopment of the piece by the powder where the binder was not deposited.

### *Additive manufacturing process by extrusion*

Differentiated from the previous process, the additive manufacturing system by extrusion builds the object layer by layer, through a filament. This process is normally use cartesian or anthropomorphic systems, forming the piece vertically, by an extruder that deposits a bead in the shape of the intended design.

### *Continuous additive manufacturing process*

What these two technologies have in common is the problem of the useful area for printing being limited, disabling continuous mass production, that is, with the limitation of the printing area, each printed piece needs to start a new printing process again, causing constraints and delays in an industrial production line.

One of the solutions that this study presents, to overcome this constraint, is the construction of



equipment with continuous additive manufacturing processes, that is, this equipment incorporates the same technologies previously mentioned, with the particularity of the printing being theoretically infinite. The **displacement of the table** is carried out by a rail system that moves horizontally, with each new layer built. This system also means that there are no downtimes between different prints, since, after each printed piece, the finished pieces remain on the defined coordinates while the next print starts.

So that the deposition of the material can occur on the base, the plane of displacement of the extrusion head or binder is carried out on an inclined plane (45° angle), which results in an oblique printing of the geometries. In the case of extrusion printing, it occurs as shown in figure 1.

The method represented in figure 1, takes advantage of the supply of material by compressed air. The pneumatic circuit allows controlled pressure on the piston inside a cylindrical reservoir. This condition determines the flow of deposited material, which is subsequently refined with the final flow rate of the extruder during its deposition.

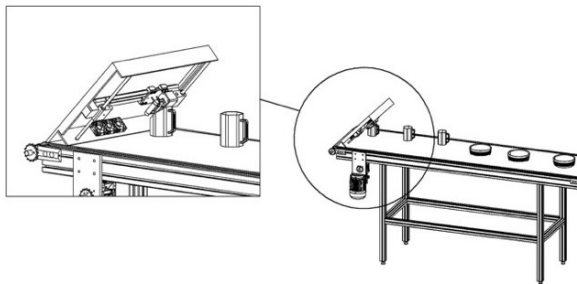


Fig. 1 – Schematic of continuous additive manufacturing process by extrusion.

This slightly inclined construction area allows the print head to reach higher speeds during movements, reducing part construction times, which, combined with the possibility of producing parts with theoretically infinite lengths, stands out from the systems already on the market.

In the case of equipment for continuous additive manufacturing processes by binder jetting, the device uses the same inclined setup, with the particularity that the printing chamber is closed, which allows the preservation of the powder bed, that is, in this production line, the parts after being manufactured remain safe and supported by the non-agglomerated powder until their final removal.

The method represented in figure 2, allows building physical models from a powdered material, connected through a liquid binder that is applied by the print heads. The pieces are later removed at the end of the production line from among the non-agglomerated powder. This end-of-line powder can always be reused for future prints.

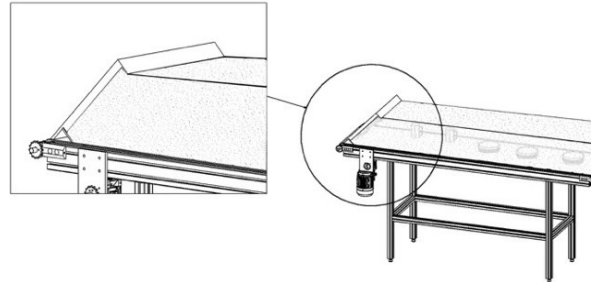


Fig. 2 - Schematic of continuous additive manufacturing process by binder jetting.

This equipment was designed with an inclined printing area of 900 x 900 mm and during the printer's construction project adjustments were necessary due to the chosen movement method for the printing axes. As the project progressed, we increased the scale of the printer from its original dimensions. We decided to use a moving rail system since it allows the printing bed to constantly advance without jeopardizing the stability of the printing in progress.



Fig. 3 – Construction of continuous additive manufacturing equipment

As shown in figure 3, the Z axis moves over the base, on horizontal rails, and accompanies the printing of the X and Y axes, allowing continuous pieces of large dimensions. In terms of length, we continue to have an enormous freedom of scale, as it is only necessary to add platforms with rails so that printed part can have the desired length.

The printheads are applied in the XY plane, and each one of them is destined for each printing process, extrusion and binder jetting.

In extrusion printing, the printer is fed from two cylindrical tanks, which allows to easily switch to the other after finishing the first one, using a valve system, making it possible to refill the empty tank. The material is pressed by compressed air that feeds the LDM WASP XL Extruder (figure 4), which prints the pieces with the final diameter defined by the extrusion nozzle. As long as the tanks are kept stocked with material, the printing will always be operational, being able to continuously print pieces in overlapping layers.

In printing by binder jetting, the feed is carried out from a deposit, above the print heads, which feeds the bed with powder, smoothing the layer in the direction of the y axis. With each layer, the print head draws a shape, repeating this process in all layers, until the piece is completed.

This equipment is in the process of completion, showing the following illustration:



Fig. 4 – Schematic representation of the printer: 1. Cylindrical deposits (material for extrusion); 2. LDM WASP XL extruder; 3. Binder printhead (printing by binder jetting); 4. Controller; 5. Print bed; 6. Layer smoothing roller (binder jetting)

## 2. Materials

### Raw stone material preparation process

The residual sludge of this industrial sector results mainly from operations of cutting, polishing and finishing of ornamental stone.

This process originates very fine stone particles, which, when mixed with water (an element that helps to lubricate the stone cutting blades and milling cutters), form a fluid mixture that flows by gravity through the industrial unit's drainage network to the water treatment station, where they end up sedimenting, allowing reuse of water in the cooling and lubrication systems of cutting equipment.



Fig. 5 – Decantation of stone particles and separation of water

The sedimented particles are compressed in a filter press, where volume and moisture content decrease. These sludge free of stone fragments are deposited in an isolated place, so that there is no cross contamination between the different types of material.



Fig. 6 – Discarding of sludge in landfill

To overcome this problem, and use this industrial waste, it is necessary to prepare the raw material in its isolated state, free of moisture content.



Fig. 7 – Raw material derived from landfills

These landfill-derived sludges have between 25% to 14% moisture. The drying of the sludge causes the agglomeration of the stone dust into lumps, which requires further disaggregation steps.

To this end, a drying line was built that facilitates the entire process of drying and separating particles from the raw material. This drying line consists of a disaggregation equipment, a vibrating chute with heating elements, a sieve, and a mixer.



Fig. 8 – Raw material drying line

The raw material is deposited in the disaggregation equipment, causing the material to break down and homogenize it into smaller particle size.

These particles are then conducted continuously, on a conveyor belt, to the vibrating chute.



Fig. 9 – Transport of the Deslumer to the vibrating chute

In the vibrating chute, the material moves slowly and continuously, drying along the chute, with the help of ceramic resistance panels and infrared radiation. This equipment uses vibration to facilitate the drying and movement of stone dust particles. At the end of the course of the equipment, there is a sieve net that separates the totally disaggregated particles from those that are still agglomerated. The stone powder that passes through the sieve is directed, through the conveyor belt, to a big-bag reservoir and the still agglomerated particles return to the starting point of the drying line.



Fig. 10 – Transport from the vibrating chute to the big-bag reservoir

This stored powder will then be placed in the mixing equipment, to be involved with the selected binder material.



Fig. 11 – Mixer for homogenization between stone powder and binder material

### Stone powder properties after processing

The inclination of the platform (printing bed) takes into account the action of gravity in the disposition of the powder and its friction angle. This slope value was determined from the angle of repose of the raw material, which in this case is the residual stone powder. This test was carried out by free dropping and stacking of the powder, and it was concluded that the maximum angle of repose of this material is in the 45%-50% range, as shown in figure 12.

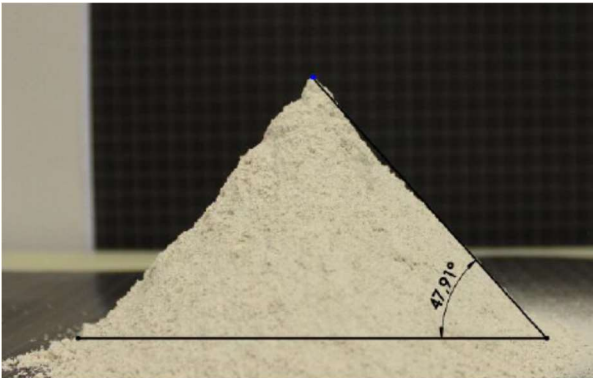


Fig. 12- Angle of repose of the raw dried stone powder.

According to the thermogravimetry test carried out, this raw material, dry stone powder, is equivalent to the expected chemical constitution of the original material – since it results from the processing of limestone and marble, it is expected that the stone powder composed essentially of calcium carbonate. This hypothesis is validated by the loss of ~44% of the mass in the temperature range between 600°C and 800°C, a value consistent with the theory for the decomposition of calcium carbonate (see figure 13). In other words, powdered limestone cannot be blended back together without a binding material.

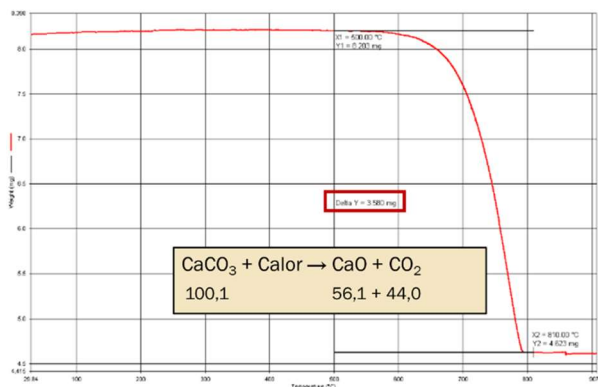


Fig. 13 - Thermogravimetry curve of stone powder and equation for the theoretical decomposition of calcium carbonate

The technological challenge posed here encompasses the selection and development of various materials for additive manufacturing, allowing for the optimization of the industrial process. In this sense, in order to maximize the use of stone powder, it is currently more feasible to apply cementitious binders. There are ongoing studies with other binding materials, both for paste and for powder, for better inclusion in the various additive manufacturing processes.

### Tests on the mixture between the stone powder and binder

For any of the additive manufacturing techniques, coupled with the production lines described above, it was essential to adapt the printing materials. This process made it possible to determine which were the best percentages of mixture between the stone powder and the cementitious binder material. Starting from preliminary tests, concentrations between 10% and 90% of dark cement were made. Of these, mixtures with cementitious binder, 30% was the selected percentage for testing. Tests were also carried out on specimens with admixtures with cement percentages of 30%, 50% and 70%.

In regard to the addition of admixtures, Viscocrete superplasticizer from Sika which regulates viscosity and facilitates extrusion. In this application, the admixture is added at a percentage of 1% in relation to the weight of cement in each specimen (Table I).

Name	Binder (L) g/%	Stone Powder g/%	Adjuvant (ViscoCrete) gm p/1%L	Water (A) g/%L	Moisture content in Stone Powder	Contraction
PC3_D	195g/30%	455g/70%	-----	90g/46%	16,1%	0,77%
PC3Ad	195g/30%	455g/70%	1,95g	104g/53%	13%	0,07%
PC5Ad	325g/50%	325g/50%	3,25g	116g/36%	13%	0,09%
PC7Ad	455g/70%	195g/30%	4,55g	135g/30%	13%	0,78%

Table I - Results of mixtures between stone powder and cement

During the mechanical bending tests, the specimens were loaded at a controlled displacement of 0.1mm/min, in a three-point system, with a spacing between the two support points of 100mm, as shown in figure 14.

The compression tests were carried out with a controlled displacement of 0.5mm/min, in a setup shown in figure 15, with two metal plates measuring 30

x 30 x 10 mm in contact with the test piece, which compressed equally the entire surface of the test piece.



Fig. 14 - Mechanical bending tests

In short, the base mixture of the specimens, PC3\_D, was the one that presented the worst results in the mechanical tests (figures 16 and 17). It was concluded that there was a reduction in resistance in the bending tests, even with the use of the admixture, contradicting the results of the compression tests, where there was a significant increase in resistance, affected by the percentages of binder used, regardless of the percentage of dark cement in each specimen.



Fig. 15 - Mechanical compression tests

However, it was concluded that the use of the adjuvant improves the results, with greater resistance to compression. In turn, the percentage of stone powder used does not cause major changes in the resistance of the material. That said, the best composition, and the one we intend to use for the case studies, will be PC3Ad or PC5Ad, which use the largest amount of stone dust, where we will be applying each one of them according to the case studies to be manufactured. If it is found that, during the printing tests, the material does not adapt to the circumstances, another composition with different binder values will be selected, such as PC7Ad.

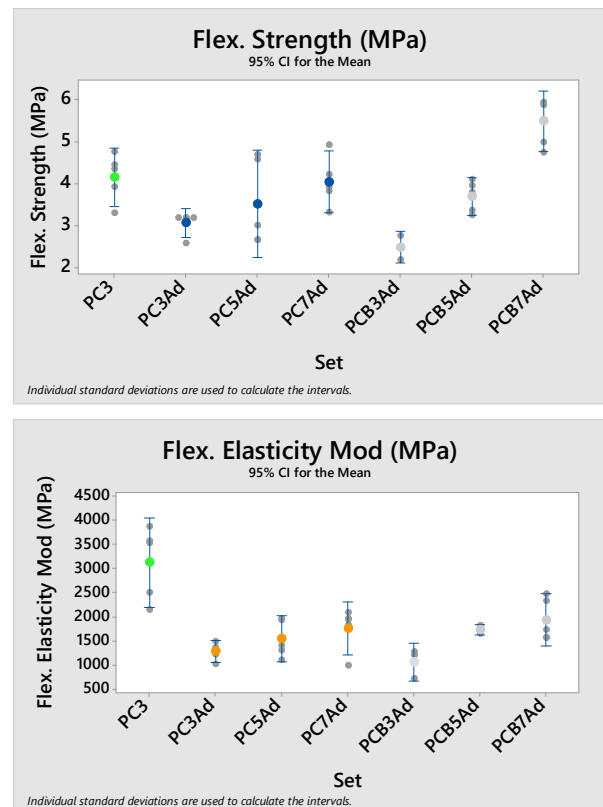


Fig. 16 - Summary of results of mechanical flexion tests

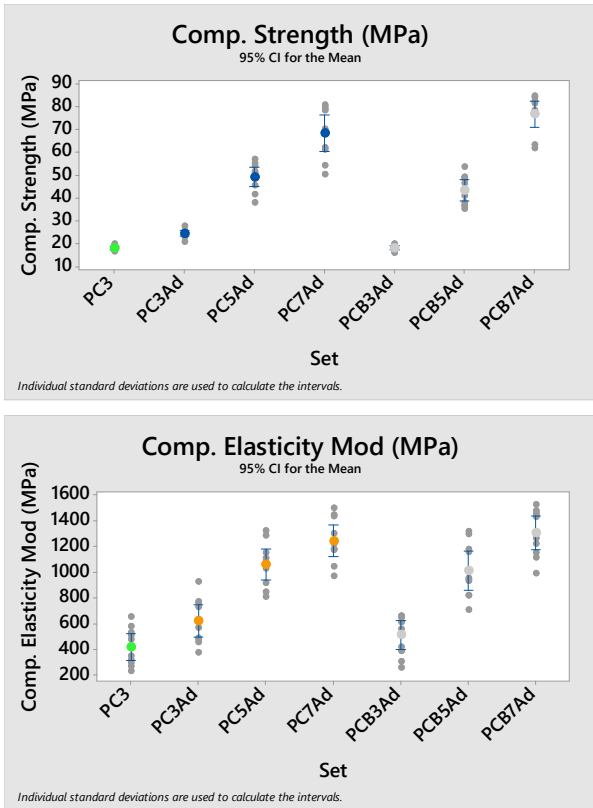


Fig. 17 - Summary of results of mechanical compression tests

### 3. Printing tests

#### Binder jetting

To carry out these tests, an equipment adapted for the production of specimens of different materials was used, in order to analyze the hardening processes of them. With it, we evaluate the connection between layers of the printed piece and the absorption of an aqueous element (water), to activate the hardening of the layers.

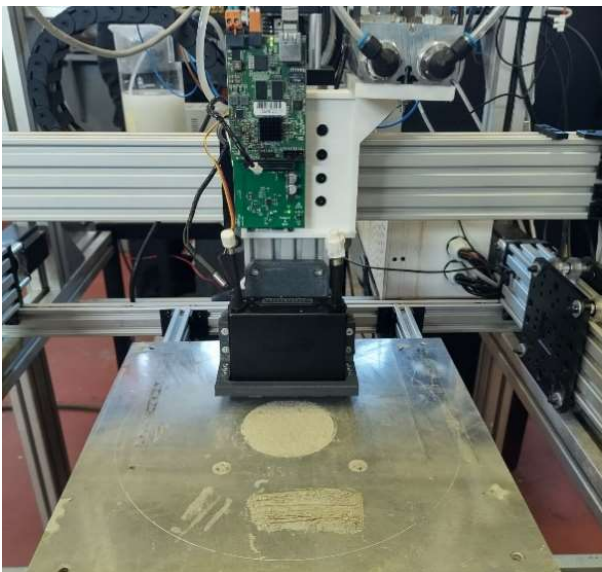


Fig. 18 - Equipment for testing various materials

This equipment consists of a vertical cylindrical deposit, printing horizontally, similar to the binder jetting printing method. This consists of: lowering the platform at each layer (1mm); place the PC3Ad mixture (70% stone powder + 30% cement) over the impression area; activate the roller that distributes the material evenly, thus forming a thin layer; the print head then deposits the aqueous binder (water in this case), causing it to activate the cement and agglomerate the stone dust. This process is repeated until the desired part is obtained. The specimens were removed from the platform after a period of 24 hours, this time was estimated based on the binding material.

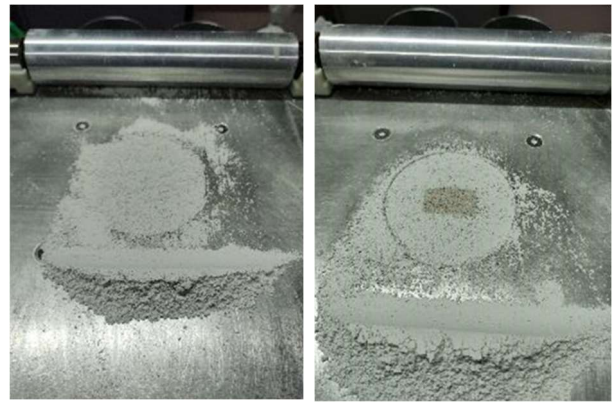


Fig. 19 - Printing by the binder jetting process with the PC3Ad mixture



Fig. 20 - Final result of one of the specimens printed by binder jetting

#### Extrusion printing

With these tests it was intended to evaluate the additive manufacturing process by extrusion. To this end, the testing process was carried out on a Delta WASP 2040 printer, equipped with the LDM WASP XL Extruder. This extruder is composed of a servomotor, a spindle, a feed chute and an extrusion nozzle. The material is fed through a cylindrical deposit, which contains, in this case, the PC5Ad mixture, where it is pushed by a piston with the help of compressed air. The material, when compressed, flows through the channel that feeds the extruder where it meets the spindle. The spindle moves according to the orders of the servomotor and the operating system. When all steps are in tune, such as air

pressure, print speed and material deposition; the material is deposited through an extrusion nozzle, thus making a continuous impression. These impressions were made with an 8mm nozzle.

#### 4. Conclusion

Both with the powder and with the paste of this by-product, we obtained favorable printing results, surpassing the final result both in its physical form and

in the resistance of each piece. These results demonstrate that the use of this type of mixture, both in the use of stone powder and in the use of the binder (cement), can result in the integration of products in several sectors, such as Construction, Architecture, Landscaping and Consumer Goods, reducing the direct impact that stone dust has on the ecosystem.

**Acknowledgments:** The authors gratefully acknowledge Fundação para a Ciência e a Tecnologia (FCT) through the support of CDRSP (Strategic Projects UIDB/04044/2020 and UIDP/04044/2020; and Associate Laboratory ARISE LA/P/0112/2020). This research was also funded by Portugal2020 (Centro2020) project “digitalSTONE – Direct Digital Manufacturing Approach for Mass Production of Tailored STONE Based High Value Products” (project n.º 039929).

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DGEG – Direcção Geral de Energia e Geologia (2020) - Informação Estatística da Indústria Extractiva n.º 22. December de 2020. Available at: <https://www.dgeg.gov.pt/pt/estatistica/geologia/publicacoes-estatisticas/estatisticas-da-industria-extrativa/>

# HERITAGE AND HISTORY OF THE MARBLE INDUSTRY

## 10 YEARS OF THE ESTREMOZ ANTICLINE STUDY (PORTUGAL)

Armando Quintas<sup>1\*</sup>, Carlos Filipe<sup>2</sup>

(1) Cidehus – Évora University / Cechap Studies Center (Portugal) \*armando.quintas@gmail.com

(2) Cechap Studies Center / School of Arts and Humanities – University of Lisbon (Portugal)

### Summary:

In Portugal, the largest marble reserve is located on the region of Alentejo, in the southern of the country. For centuries, the continuous exploration of these quarries, led to the creation of an industry. The transmission of know-how of its operation was associated with the evolution of production techniques and technologies, in order to satisfy the demands of the national and international markets of architecture and sculpture.

Since the Roman period (1st century), these marbles have been taken to different places, and in the last 150 years, with the intensification of merchandise circulation, they have been exported to many faraway countries, reaching the five continents.

The present text aims to show how the multidisciplinary study "Heritage and History of the Marble Industry" developed by the CECHAP Studies Center in cooperation with research centers from Portuguese Universities, has been contributing (since 2012) to the historical, heritage, social and economic knowledge of this industry.

**Key words:** Marble, Portugal, Alentejo, History, Industry

### 1 – Geological context and economic value

In the Alentejo region, in southern Portugal, there is a unique geological formation that extends throughout the municipalities of Sousel, Estremoz, Borba, Vila Viçosa and Alandroal, know as Estremoz Anticline. This deposit provides the country with its finest marbles, whose physical-mechanical characteristics and polychromatic proprieties have made the preferences of quarrymen in the last two millennia.<sup>1</sup>

In this context, it is not surprising that marbles of great beauty and varied colors have been extracted. Today, the predominant ones are white, pink, cream and grayish blue, which are current used for ornamentation in urban architecture (facades, walls, floors, stairs, balustrades, columns, moldings and coatings) as in funerary architecture and sculpture [Casal Moura:2007, 20-30]

In 2012, the reserves of these marbles were estimated around 51 million cubic meters, with a

commercial use of only 20% of the extracted material [Carvalho:2012]. Extraction, in turn, recorded in 2019-2020: 335,281 tons, being mainly destined to exportation, which reached in 2021 a gross value of one hundred and six million euros. The main markets were France, Saudi Arabia and China [D.G.E.G.:2021 and I.N.E.:2022].

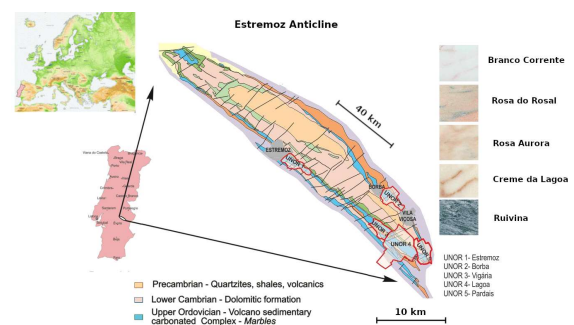


Fig. 1. The anticline of Alentejo Marbles. Source: Adapted from Cartografia Temática, 2008

<sup>1</sup>The Portuguese marble can be found in the Alentejo region, but the only one zone in operation in the last 30 years is the Anticline of Estremoz. We note that despite Alandroal and Sousel being included in this geological

formation, in these municipalities no stone is extracted for ornamental purposes.



According to data from 2019, as far as enterprises are concerned, there were in Central Alentejo, 45 for extraction and 57 for processing, with a turnover of 57.797.431€ and 33.507,795€. In terms of gross value added, the extraction companies reached 22.158.388€ and the processing companies reached 9.594.693€. Regarding human resources, there were 418 employees in the processing. No data are available for extraction, but it should not be too far from half of that number, due to the smaller quantity of quarries in operation today [Quintas:2022,270-271].

## 2 – The historical and heritage study of the Alentejo marbles

The relevance of a research in the social and human sciences about these marbles was due to the fact that they are widely represented in abundant technical literature (geology, economy, technology), but not in historical literature. A new field of study that allow us to understand the evolution and the importance of the cultural and social heritage of this industry.

For this reason, it was launched in 2012 by CECHAP (Vila Viçosa), a non-profit association dedicated to culture and science, the study PHIM – Heritage and History of the Marble Industry. The studies are developed in partnership with research units of Portuguese Universities as: Cidehus Centro Interdisciplinar de História, Culturas e Sociedades (University of Évora); IHC – Instituto de História Contemporânea (Universidade Nova de Lisboa); CIES – Centro de Investigação e Estudos de Sociologia (ISCTE – Instituto Universitário de Lisboa), ARTIS – Instituto de História de Arte (Universidade de Lisboa), IURIS – Instituto de Investigação Interdisciplinar em Direito (Universidade de Lisboa) and CEG – Centro de Estudos Globais (Universidade a Aberta).<sup>2</sup>

This is a multidisciplinary study that addresses different research lines focus on the survey, treatment and inventory of national and international archives, documents, audiovisual and archaeological sources. Covers the areas of geo-sciences; classical archaeology (roman empire - 1st century to the 6th century); industrial archaeology; technical processes and technological evolution; social and organizational aspects; history of art,

construction and urbanism; oral history with interviews of former industrial actors; mining and quarrying legislation; economics and entrepreneurship history; environmental and sustainability resources, and digital humanities with the processing of cartography and information on the creation of dedicated website and public database.

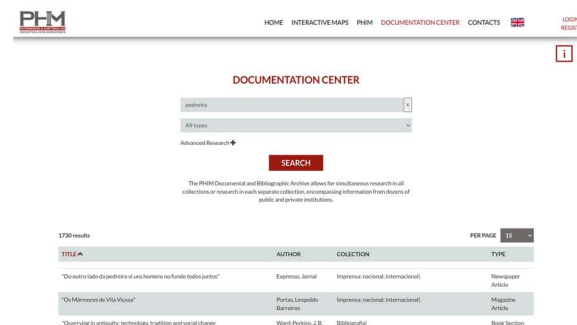


Fig. 2. The database from website project, 2022

All of them are related to their relationship with the territory in the various periods of history. It intends to contribute to a permanent update on the sustainability and use of this natural resource, playing a role in its cultural affirmation, throughout the promotion of industrial tourism.

It also seeks to contribute to a better performance in decisions about the organization of the territory, with emphasis on the mitigation of environmental problems in the context of the circular economy.

The funding for this study relies on CECHAP own resources, contracting support, research support from the partners, but mostly on the European Regional Development Fund (FEDER), which throughout the Alentejo programs, has already supported this project three times (in 2015,2019, and 2022)<sup>3</sup>

The research work continues, with new expected outputs as early as 2023-2024, namely in the research in the field of marble enterprises history, participation in academic and cultural forums, scientific articles, master's and doctoral dissertations and production of new heritage educational content.

## 3 – Outputs, transfer and dissemination of knowledge and marble industry promotion

In 2022, ten years of the study dedicated to the Alentejo marbles were completed, with broad and

<sup>2</sup> <https://www.marmore-cechap.pt/en/aboutUs>

<sup>3</sup> ALENT-08-0347-FEDER-002329; ALT20-08-2114-FEDER-000077; ALT20-08-2114-FEDER-000213.

significant results for the historiography of this economic activity, and a preparation to discuss the actual marble industry issues.

First, the outputs achieved with the scientific production: survey and inventory included in a database (more than twenty thousand records referring to the consulted documentation). Five monographs that targeted themes such as geology, history, economics, art history, architecture, companies, history of work and social organization, trade unions, tourism, and heritage [Alves:2015; Serrão et al.:2019; Alves et al.:2019; Carneiro et al.:2022; Matos et al.:2022].

This is the first global study dedicated to the ornamental stone sector in Portugal, covering, the chronological period from the 1st century until the present. In parallel, two heritage educational notebooks have already been produced – “Discovering White Gold”, dedicated to elementary and high school students. The booklets aim to sensitize young people to themes about geology, the mineral constitution and characteristics of marble, the industrial activity of extraction and processing, the landscape and the uses of marble in architecture. Besides these publications, about forty scientific have already been published, as well as the participation in more than sixty events, both national and international. Around two dozen scientific events (symposiums, workshops, congresses and exhibitions) on the subject of history and heritage of marble industry have also been organized.

Secondly, using the digital humanities, all the project's activity is available online, aggregating it throughout the dedicated portal to the study of the Heritage and History of Marble Industry.<sup>4</sup> In the website it is possible to find the production performed<sup>5</sup>, have access to a set of Georeferenced data, to a 360 degrees images, published news and access to the repository of information in the documentation centre. In the same dynamic, an online game (for young students) with questions and answers entitled “Ouro Branco – the challenge” was launched in 2022.<sup>6</sup>

Third, and as result of the production and dissemination developed, the study team, became part of two networks. First the NEREMA – International Network on Research on Marble and

Decorative Stones, composed by members from Spain, France, Italy, Belgium, Germany, Denmark and Netherlands. Second, a network on extractive landscape research with members from Spain and Italy.

In this way it has been possible to understand the evolution of this industry and its historical and territorial dynamics, to increase knowledge about its actors (entrepreneurs, workers, technicians and engineers), to understand the impact of its foreign trade since 1850, to know the markets and exports destinations and the problems manifested in the industry, both in its internal trade and in its impacts on the quarries territory [Quintas:2020;93-116 and Filipe:2015,57-93].

On the other hand, the continuous research on these marbles has allowed the team of this study to follow the present dynamics of this industry and to understand the current situation. The team, therefore, is geared to public discussion, promoting reflection with the various economic agents and politicians, in order to change the mentalities and draw attention to real opportunities for this industry, taking into account the current problems.

And to this aim, it has been participating in several forums and integrating a consortium of companies, public and private institutions, and other associative entities<sup>7</sup>. One of the discussed problems is the excessive waste, in order of 80% of extracted marble, that ending up in the impacting heaps. Their multiplication over dozens of years has been disturbing the landscape, the agricultural areas, water lines, access to properties and even the industrial spaces. But their rubble is not deprived of possible uses and therefore should be considered an asset with economic value of greater interest. Therefore, it is necessary to develop a strategy that aggregates research and innovation oriented to technology transfer processes for the creation of new ornamental products, the use of rubble for ceramics, lime, mitigation of heavy metal pollution (by using geomaterials), and also for the production of stone paper from marble calcium carbonate. Another identified problem concerns the industrial uses of water uses in extraction and processing, The most part, from aquifers, as well a sludge that is originated by sawdust and that is not stored in

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4 [www.marmore-cechap.pt/en](http://www.marmore-cechap.pt/en)

5 [www.marmore-cechap.pt/en/publications](http://www.marmore-cechap.pt/en/publications)

6 <https://ourobranco.pt/login>

7 Consortium Stone Cast Portugal – Pedra como pilar para o futuro. [http://lp.boldint.com/stone\\_cast](http://lp.boldint.com/stone_cast)

properly protected spaces, polluting surrounding agricultural land and contaminating water lines because don't have required treatment. In addition, it must be decided what to do with the hundreds of large and deepness abandoned quarries, in order to prevent new accidents. In the 2018 the National Road 255 between Borba and Vila Viçosa collapsed resulting in five deaths<sup>8</sup>. Until few months there was constraints and dangers for the circulation on other roads, as was the case of Vila Viçosa-Alandroal and the entrance in city of Estremoz<sup>9</sup>. And the situation of Vila Viçosa – Bencatel remains with various problems in different sections of that road. [Quintas:2022]



Fig. 3. Abandoned quarries. Source: Marble Route, 2022

In the field of industrial tourism, the marble route (managed by CECHAP) offers a series of routes throughout industry, heritage and landscape, in which marble is the central element<sup>10</sup>. This a cultural product of an industrial nature, emerged from an academic project, that turned in a commercial activity [Filipe et al.:2014]. The itinerary most sought after by tourists is the one dedicated to visit the quarries with the enjoyment of their landscapes changed by the industry<sup>11</sup>. The marble

route is a window on the territory, growing and evolving with the transfer of knowledge from the scientific study about history and heritage of marbles. Only with a permanent and systematized research is possible to create, innovate and improve the offer of industrial tourism, allowing to guide a privileged knowledge about the evolution of the territory in terms of landscape, economic and social role that marble represents. The testimony evidenced by the visitors at the end of their visits is gratifying. The marble route already reached more than 50 nationalities, a significant part of the visitors are professionals of stone, like architects and engineers.

#### 4 – Final considerations

The marble study has already evolved beyond history itself, as it has already posed a series of current and pertinent questions about this industry. In this sense, the study begins to focus on environmental problems and land management. By this way it has contributed with awareness-raising actions, resulting from the transfer of knowledge, carried by the CECHAP team, seeking to disseminate good practices to be introduced, refocusing the importance of this mineral reserve on the international scene and seeking solutions to existing issues by seeing them as an opportunity.

In practical terms, in addition to the published scientific production, the experience acquired with this study made possible, to increase the practice and development of integrated industrial tourism in marble sector, and enable the CECHAP team to participate in the discussions about the importance of this sector and its territory, as an agent of civil society.

It is a territorial study model that can be transferred to other geographies and other ornamental stone productions, such as limestone's, granites or slates.

**Acknowledgments:** This work is funded by national funds through the Foundation for Science and Technology under the project UIDB/00057/2020

<sup>8</sup> [https://pt.wikipedia.org/wiki/Trag%C3%A9dia\\_de\\_Borba](https://pt.wikipedia.org/wiki/Trag%C3%A9dia_de_Borba)

<sup>9</sup> <https://rr.sapo.pt/noticia/pais/2022/07/13/estremoz-encerra-estrada-devido-a-proximidade-de-pedreira/291966/>

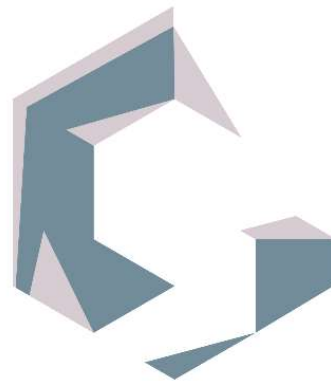
<sup>10</sup> <https://rotadomarmoreae.com/en>

Registered in IGAC – Propriedade Intelectual nº 3855/2011; INPI – Propriedade Industrial nº 177/2012; Turismo de Portugal – RNAAT nº 145/2014; EUIPO – *European Union Intellectual Property Office* nº 017947184.

<sup>11</sup> <http://rotadomarmoreae.com/en/visit/2/tour-dos-marmores-em-vila-vicosa>

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**GLOBALSTONE**  
c o n g r e s s 2 0 2 3

**Wednesday, June 21<sup>st</sup>**  
**PRESENTATIONS**

## **Limestone processing sludge as a secondary raw material for innovative earthenware manufacturing – The LIFE4STONE study case**

Regina Santos, CTCV – Technological Center for Ceramics and Glass, [regina@ctcv.pt](mailto:regina@ctcv.pt)

Joana Salgado, CTCV – Technological Center for Ceramics and Glass, [joana.salgado@ctcv.pt](mailto:joana.salgado@ctcv.pt)

António Silva, CTCV – Technological Center for Ceramics and Glass, [antonio.silva@ctcv.pt](mailto:antonio.silva@ctcv.pt)

Waste production represents one of the main constraints of the natural stone industrial sector. In Portugal, this sector is responsible for the production of around 200 commercial types of rocks (granites, limestones, marbles, slates and schists, among others). The low recoveries attained in most quarries and processing plants, with values ranging between approximately 20 to 40%, has led to the accumulation of large amounts of mineral waste, namely very fine particles resulting from the stone cutting and sawing that, when mixed with the processing water, originate a residual sludge.

Companies, business associations, universities and also research and innovation centers, like CTCV, have been increasingly engaged in the characterization and assessment of this type of waste materials aiming its incorporation into other types of materials and/or products that feed various industries, such as ceramics and thus replacing primary raw materials, additives and/or fillers, among others, on a circular economy approach.

The LIFE4STONE project is an example of a stone-ceramic industrial symbiosis study case. Its objective is to find environmentally sustainable solutions for the sludge valorization as secondary raw material in the ceramic sector, contributing to its circularity and categorization as a new mineral resource that may be used in some ceramic manufacturing processes, being focused in the incorporation of carbonate sludge in ceramic compositions for decorative earthenware products, with a strong positioning in Portugal and with a high export capacity.

The results of the incorporation tests performed in LIFE4STONE are very promising once demonstrated that the substitution of a calcite raw material by the carbonate sludge in calcitic earthenware ceramic compositions does not compromise its technological performance, suggesting the future evaluation and classification of this waste as a secondary raw material.

The main technological and performance characteristics were not compromised and some in fact reinforced the quality of the end products, as it was possible to verify that: (1) The linear shrinkage after drying is slightly higher in the 100% sludge compositions, with no significant changes being verified in the 50% sludge specimens; (2) Linear shrinkage after firing is similar for all compositions, though slightly higher for the 50% sludge; (3) The flexural strength values are also similar, without a clear trend since it remains within the respective margins of error; (4) Water absorption is roughly in the same order of magnitude in the compositions with sludge when compared to the reference.

These tests, complemented with characterization data, prove their technical viability on a laboratory scale, since there are no significant differences between the reference composition and those with a substitution of 50 and 100 % of calcite by the mineral waste.

Keywords: natural stone, processing sludge; waste recovery; circular economy; secondary raw materials; ceramics.

# CIRCULARITY PATHWAYS TO THE MATERIAL RECOVERY FROM BEGE BAHIA DIMENSION STONE

C.M. Souza<sup>1\*</sup>, E.S.C Freitas<sup>2</sup>, L.H. Xavier<sup>3</sup>, R.C. Ribeiro<sup>4</sup>

(33) Caroline Martins de Souza, \*carolinemartins.ambiental@gmail.com

(34) Emmanuelle S. Carvalho Freitas,

(35) Lúcia Helena Xavier,

(36) Roberto Carlos da Conceição Ribeiro

**Summary:** Brazil is one of the world's largest producers of ornamental stones, with an annual production of approximately eight million tons. The large amount of waste generated has become one of the biggest problems in the stone sector. The reduction and reuse of industrial waste generated during exploration and processing is one of the major challenges to mitigate environmental impacts. In this way, the circular economy encompasses both the recovery of products, components and materials through reuse and recycling. The objective of this work is to present preliminary suggestions such as operational routes and strategies to improve the circularity of the materials, aiming at the production of low-cost composites that can be used as piezoelectric floors, having about 80% of Bege Bahia marble waste, which treats It uses a low hardness limestone that is easy to cut and process; and its residues, generally, present ultrafine granulometry.

**Key words:** circular economy 1, Bege Bahia 2, piezoelectric 3

## 1. Introduction

### 1.1 Bege Bahia dimension stone

The stone commercially known as Bege Bahia marble in the dimension stone sector is abundant in the Salitre River region, which is characterized by the Caatinga formation, and comes from alterations of formation limestone. Its extraction is mainly concentrated in the city of Ourolândia, with mining, processing, and beneficiation activities (Figure I).

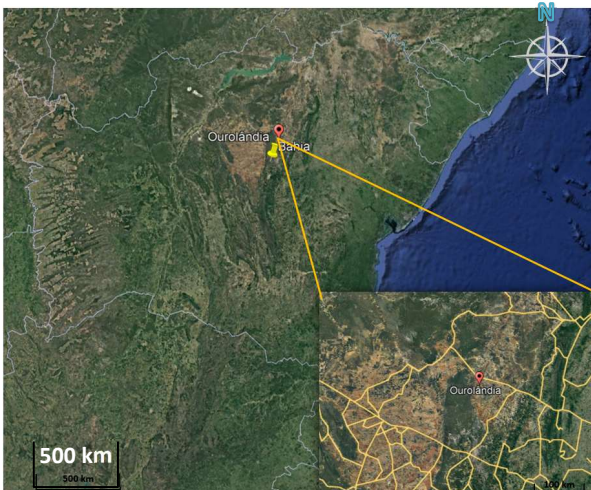


Fig. I. Location map of Ourolândia/ BA (GoogleEarth,2023)

According ABIROCHAS (2019), Brazil is one of the world's biggest producers of ornamental stones, with an annual production of approximately eight million tons, ranking among the top five global producers with a global

participation of 5.4%, With a revenue of \$1.34 billion, surpassing the historical record of \$1.30 billion registered in 2013. There were found 78 existing processes in the Mining Registry and Sigmime/DNPM (2023) of Marble mining companies in Ourolandia/BA, according to Figure II.

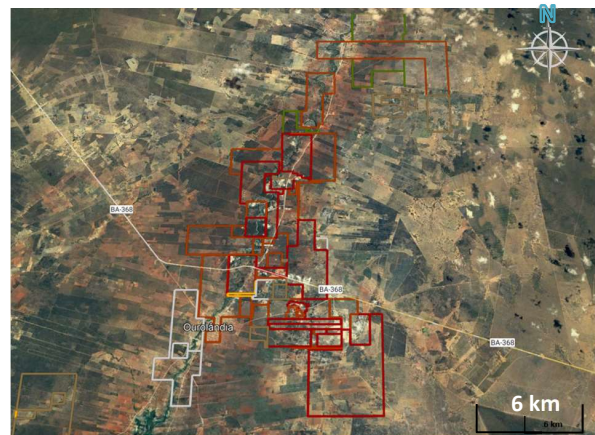


Fig. II. Area of Marble mining companies in Ourolandia / BA (GoogleEarth,2023)

The Brazilian industrial park is mainly composed of approximately 1,600 conventional cutting machines. Along with this production, a large amount of waste is generated. In the case of Bege Bahia reject, no abrasives are detected, as the process is essentially done with diamond-edged machines, which facilitates its use as a mineral filler (Ribeiro et al., 2012). Only about 30% of the extracted blocks from the mines are utilized, generating a large volume of waste.

The large amount of waste generated has become one of the biggest problems in the stone sector, as the rejects are usually disposed in piles occupying large areas, together with the mining areas or are destined to landfills that are already saturated (Figure III) and may be subject to landslides and sediment transport by rainwater to watercourses, causing siltation, increased turbidity, and suspended particles. The reduction and reuse of industrial waste generated during the exploration and process is one of the major challenges to mitigate environmental impacts (Carmo,2020).



Figure III: Bege Bahia waste

### 1.2 The Circular Economy

The current linear economic and production model causes numerous negative impacts on the environment, due to the extraction of massive quantities of natural resources, resulting in a high generation of waste. The circular economy suggests a transformation of this linear model into a cyclical model where waste is reintegrated into the production chain (Freitas, 2022), adding value to a material that only has associated expenses. The principles of circularity establish requirements for valuing recovery routes based on sustainability and the generation of secondary material (Velenturf et al.,2021). This way, the circular economy encompasses both the recovery of products, components, and materials through reuse and recycling, presenting solutions for the identification and valorization of secondary materials, including waste treatment as a secondary resource. (Ellen MacArthur Foundation).

### 1.3 Utilization of Bege Bahia waste

The resources from dimension stones processing are an interesting source of material that meets the technical requirements for applications in different areas such as flooring, architectural composition elements, decoration, furniture, and others. The use of mineral fillers in the polymer industry aims to reduce costs for the sector. With the improvement of the use of these techniques, it is observed that these fillers could enable important transformations in the properties of polymer materials, such as density control, improvement in optical effects, thermal expansion control, flame retardation, modifications, electrical resistance, and magnetic susceptibility, as well as improvement of mechanical properties, such as hardness and tear resistance (Ribeiro et al, 2012).

According to (Ribeiro et al, 2012), the chemical analysis of Bege Bahia waste indicated that the main element in the residue is calcium (48.85%), which is naturally found as calcium carbonate, and that silica (5.13%) and iron oxides (0.34%) are extremely low, characterizing the high quality of the residue for application as a mineral filler. Bege Bahia marble sawmill residues generally have granulometry ultrafine and low levels of silica and iron, making it with high potential for application as a mineral filler, since there is no need for high costs with its beneficiation. (Vidal et al., 2009).

### 1.4 The use of the 3D Printer in the reuse of Bege Bahia waste

Technological advances have contributed to the manufacturing of different materials using 3D printers, which use overlapped layers of polymer resin. Therefore, taking advantage of this technology to generate piezoelectric floors could provide a response to the saturation of landfills, promoting circularity through the recovery of secondary waste and energy generation in pedestrian areas.

This energy could be used for low-consumption food devices such as lighting and security cameras, noting dependence on the electrical grid and wanting to reduce greenhouse gas emissions.

The incorporation of residues in the polymeric matrix could also contribute to the reduction of the cost of the material, since the residue would serve as filler, replacing part of the polymeric resin without compromising the final properties of the material (.

Several studies such as (Cholleti,2018) admired that piezoelectric materials can generate electrical energy from mechanical stresses, such as pressure and vibration. When a piezoelectric material is subjected to mechanical stress, it generates an electrical potential difference on its surface. Thus, by using 3D printing



technology to create piezoelectric floors with waste from Bege Bahia, it is possible to capture the energy of pedestrians' steps and convert it into electrical energy.

## 2. Objective

The objective of this work is to present preliminary suggestions such as operational routes and strategies to improve the circularity of the materials, aiming at the production of low-cost composites that can be used as piezoelectric floors having as 80% of Bege Bahia marble waste, transforming it into a potential solution to circularity.

## 3. Materials and Methods

The methodological approach sought, in an exploratory way, to collect and analysis secondary data. A literature review by authors in the area to consolidate aspects related to the use of secondary materials as a productive institution and primary data provided by local union representatives and the Associação de Mármore Bege Bahia (ASSOBEGE)

### 3.1 Materials

The waste used in this work comes from the mining of Bege Bahia marble, in the city of Orolândia – BA. Polypropylene has a melt index of 1.5g/10min and a density of 0.903 g.cm<sup>-3</sup>

### 3.2 Composite processing

The processing consisted, in a first step of mixing polypropylene with limestone residue, and the composites were processed with 10, 20, 30, 40 and 50% by mass. Then, the mixture was extruded in a twin-screw extruder, model DCT 20. The final shape of the specimens was obtained by the Injector Battenfeld Plus 35 machine in Table I.

Table I: Percentage of waste in each composite

BB01	BB02	BB03	BB04	BB05	BB06
0%	10%	20%	30%	40%	50%

### 3.3 Chemical Analysis of the Waste

The determination of the composition of the residue was carried out by the coordination of mineral analysis (COAM) of CETEM.

### 3.4 Determination of Specific Mass

The specific mass of the composites was determined according to the ASTM D792-13 (2013) standard.

### 3.5 Mechanical Behaviour

The tensile test was performed using an Emic mechanical testing machine, according to ASTM D 638

(2010). The impact test carried out using the Izod test, according to ASTM D 256 – 05 (1993). The bending test was carried out using an Emic universal mechanical testing machine, in accordance with the ASTM D 790(1984).

### 3.6 Determining the Waste Class

The dangerousness of a waste is classified according to its physical, chemical, or infectious-contagious properties, which may present a risk to human health and the environment, when the waste is handled or disposed of improperly. The NBR 10.004/04 (2004) was used to classify Bege Bahia marble waste and composites into class I or II, based on their dangerousness and inertia. The residue was submitted to solubilization, and leaching tests and the resulting extracts were submitted to chemical analysis. The same procedure was performed with the composites. The results were compared with the maximum limits established in Annexes G and H of the standard.

### 3.7 Electric circuit generation

It will be necessary to carry out a future study for the construction phase of the electrical circuit for the piezoelectric floors.

## 4. Results and discussions

### 4.1 Characterization of Composites

#### Determination of Specific Mass

The specific mass values obtained for pure PP (0%) were around 0.9 g.mL<sup>-1</sup>. It was also observed that there is little variation in mass specific with the percentage increase of residues, because with 10 and 20% the value of the specific mass was 1 g. mL<sup>-1</sup> and with 30, 40 and 50% the specific mass increased to 1.1 g.mL<sup>-1</sup>.

### 4.2 Chemical Analysis of the Waste

The evaluation of the residue by FRX determined contents of 43.5% of CaO, about 8% of MgO, 0.5% of Al<sub>2</sub>O<sub>3</sub>, 5.4% SiO<sub>2</sub> and about 43% loss on ignition since it is a limestone calcite.

### 4.3 Mechanical Behaviour

According to the tensile tests it is possible to obtain some parameters. The first to be analysed will be the Yield Stress of the material, as can be seen in Fig. IV. The yield stress is the maximum stress that the material supports still in the elastic regime of deformation. That way, it appears that the presence of this load is responsible for making the composites support less tension. In Fig. V it is observed that the specific deformation in the rupture of the free polypropylene of mineral load is high, reaching values around 300% and with the addition of waste mechanical stabilization of

the material is verified, since the specific deformation decreases gradually reaching values around 10%.

In Fig. VI, the modulus of elasticity of the composites can be verified. Young's modulus or Modulus of Elasticity is a mechanical parameter that provides a measure of the

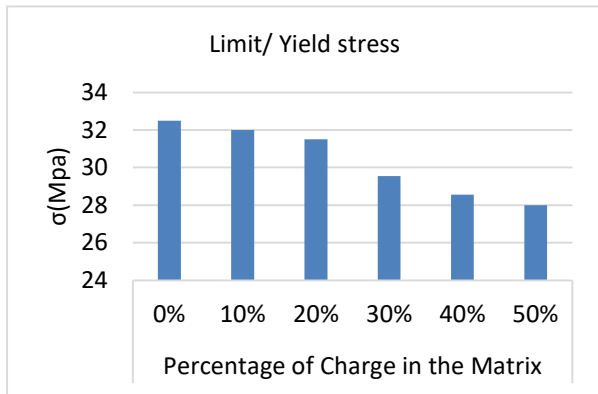


Figure IV: Yield stress

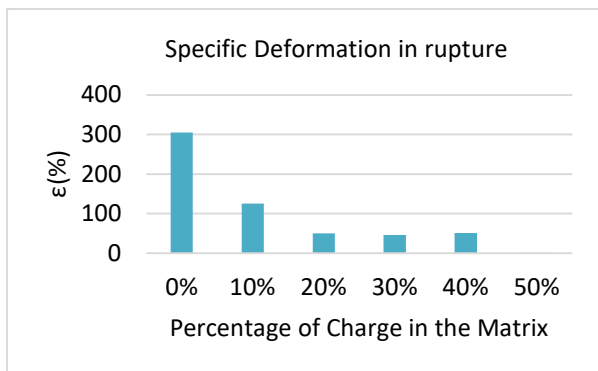


Figure V: Specific Deformation in rupture

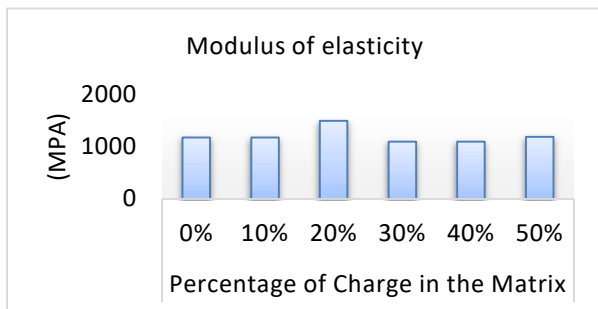


Figure VI: Modulus of elasticity

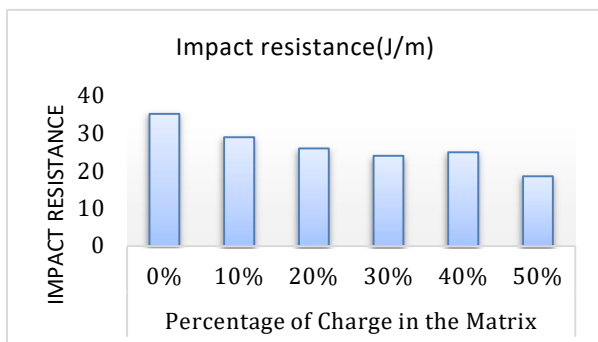


Figure VII: Impact resistance

stiffness of a solid stuff. In Fig. VII, corresponding to the Izod Impact test, it is also verified that the addition of the residue is responsible for the mechanical stabilization of the material, since with the addition of load the values of resistance to impact remain around 20 J/m.

After adding the residue to the PP matrix to form the composite and this composite being leached and solubilized, chemical analysis results did not indicate solubilization or leaching of any of its elements, classifying the material as Class II B – non-hazardous and inert.

### 5. Conclusion

It can be concluded that Bege Bahia limestone waste can be used as mineral filler in the production of polypropylene composite, reaching 50% by mass, capable of generating a floor.

In addition, it is possible to verify that the increase in load does not change its specific mass, the composite has high resistance and is not dangerous. This material showed good results that can be directly related to the durability of the product and great potential for piezoelectric use.

In general, the use of waste from Bege Bahia in the production of piezoelectric floors represents a sustainable solution for the management of waste generated in the stone sector, in addition to contributing to the generation of clean energy in urban environments.

For future work, it will be necessary to carry out the study of the assignment of piezoelectric tablets to form a system of 2 floor plates containing the Bege Bahia residues.

#### 4.4 Verification of dangerousness

**Acknowledgments:** The authors thank the to CNPq for financial support, to Cetem and INT for infrastructure, and Marcelli Conceição.

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# Research on how to recycle side blocks in granite factories

## To change the wastes into useful products

Eng. Firas Aljedi

(1) Eng. Firas Aljedi, firasaljedi@hotmail.com

**Summary:** *stone Processing plants in general give a lot of waste materials and this include all types of stone factories such as limestone, Marble, Basalt, Granite, Slate, and Etc. as we need these products for our civil building the wastes will increase more and more daily, and these wastes will effect the environment in a-negative manor. This study is focusing on side blocks coming from granite factories. This research aims to save the environment by converting waste materials into useful products.*

**Key words:** *Granite wastes, Marble wastes, products from waste, useful products from stone waste,*

### General body of the manuscript:

We had studied granite factories with accurate calculations on how to decrease the wastes' quantity that is being produced by companies, in fact, these wastes are harmful to the environment. Nevertheless, we can change these waste materials to be useful for everyone and decrease the pollution rate. Those calculations are represented in three steps:

1. Machines that produce wastes during granite processing.
2. Waste quantity that a factory produces.
3. Useful solutions for these wastes.

Let's proceed and learn about how to convert wastes into something useful.

- 1) We have studied the factory which uses the machines that produce wastes during granite processing and are categorized into different sections as the following:

#### 1-a) Gangsaw department:

1-a-1) 04 Gangsaw- Gaspari Menotti (size 480-450 Jumbo Matic).

#### 1-b) diamond wire cutting:

1-b-1) 03 single wire saw, Pellegrini.

1-b-2) 02 multiwire, 70 wires.

#### 1-c) block cutters:

1-c-1) 03 block cutters, pedrini 56 discs.

1-c-2) 01 full tile line.

1-c-3) 03 single disc cutters, Chinese type

#### 1-d) Polishing Machine:

1-d-1) Gaspari Menotti with 22 heads and 06 sectors.

#### 1-e) Bridge cutters:

1-e-1) 04 bridge cutters from GMM company Tecna type.

1-e-2) 07 bridge cutters from GMM company Axia full type.

#### 1-f) small cutters:

1-f-1) 02 small cutters from SNIC company for squaring the tiles

1-f-2) 02 small cutters from SNIC company for tiles details and squaring

#### 1-f) Edges' detailing machine

1-f-1) 01 edge detailing from Marmo Meccanica with 08 heads

#### 1-g) flaming machines

1-g-1) 01 flaming machine from Pellegrini company with 02 torches



Figure.1 Gang saw machine



Figure. 4 Block cutter single disk



Figure. 2 Block cutter



Figure.5 polishing Machine

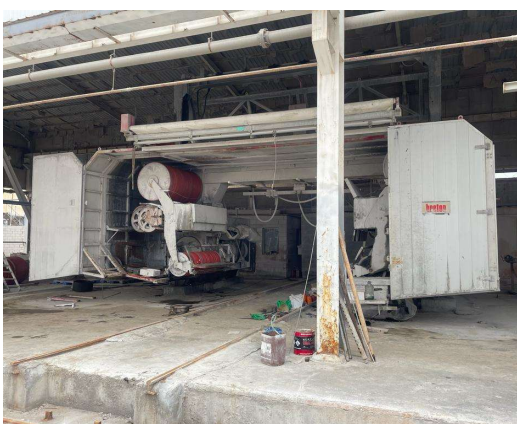


Figure. 3 Multiwire saw

2) Wastes quantity that factories produce. I've done some accurate calculations for the amount of wastes from the automatic press mud with the following details :

2-a) the factory has 3 press mud machines with the size 100\*100\*4.5 cm \*20 plates give 19 waste biscuits

2-a-1) the size of one biscuit: 100\*100\*4.5 cm

2-a-2) the weight of one biscuit is Kg: 65 Kg

2-a-3) one set of mud wastes give  $19 \times 65 = 1.24$  Ton.

2-a-4) 3 stations of press mud give average wastes by day after checking in daily for a three months = 37.2 ton.

2-a-5) wastes in one year in Kg:  $37.2 \text{ ton} \times 300 = 11160 \text{ ton/year}$

2-a-6) wastes in one year in  $\text{m}^3 = (11160 \text{ ton} \times 0.855) / 1.24 \text{ ton} = 7965 \text{ m}^3/\text{year}$

2-b) wastes coming from blocks:

2-b-1) wastes coming from a Gangsaw after cutting blocks in a month is:  $8*4*3*2 = 192$  side slabs/month

2-b-2) wastes coming from a Multi wire after cutting blocks in a month is:  $40*2*1*2 = 160$  side slabs/month

2-b-3) wastes coming from Block cutters after cutting blocks in a month is:  $10*3*3*1 = 90$  slabs bottom/month

2-b-4) the size of slabs approximately =  $310*190 = 5.89m^2$

2-b-5) monthly total of side slabs =  $5.89*(192+160+90) = 2603.38 m^2$

2-b-6) the thickness of the side slabs starts from 4 to 10, which gives the average thickness of 7cm

2-b-7) monthly total of wastes =  $2603.38 m^2*0.07 = 182.24 m^3/month$

2-b-8) wastes from factory in one year =  $182.24 *12 = 2186.88 m^3/year$

2-b-9) wastes in one year in ton=  $2186.88 m^3*3=6560.64 ton/year$

2-c) the wastes from cutting the slabs and tiles equals to 15% of the total production in  $m^2$ :

2-c-1)  $64000*0.15 = 9600 m^2$

2-c-2) we can calculate the wastes in basic of 2cm thickness:

Wastes in one month in  $m^3$ :  $9600 m^2*0.02 = 192m^3/month$

2-c-3) wastes in one year in  $m^3$ :  $192*12 = 2304m^3/ year$

2-c-4) wastes in one year in Kg:  $2304 *3 = 6912ton$

2-d) total wastes from factories every year is

Table 1: total wastes per year:

Section	Material	Ton	$m^3$
A	Sludge	11160	7965
B	Side Slabs	6560.64	2186.88
C	Small Tile	6912	2304
<b>Total</b>		<b>24632.64</b>	<b>12455.88</b>



Figure. 6 Side Blocks



Figure. 7 Gangsaw Trolley with side slabs



Figure. 8 Tiles Wastages



Figure. 9 Sludge wastages

- 3) Last but not least, the solutions for these wastages material as its or with minimum processing requirement.
  - 3-a) sludge wastes:
    - 3-a-1) you can test the sludge in a granite interlock and it would give you better results than the normal interlock.
    - 3-a-2) we can add the sludge with normal cement mixture for cement block and the product will give the same good results of that as a normal cement which consists of sand and cement gravel.
  - 3-b) side slabs



Figure. 10 Single wire wastages

3-b-1) side slabs are also used in open areas such as floors and it gives good and strong results.

3-b-2) use a splitting machine to cut the slabs into cubes in the sizes of 10\*10(3-10) up to 40\*40 (3-10) cm and then you could use it at the entrance of heavy-duty roads.

3-b-3) those side slabs can also be used in different ways in landscapes and sizable gardens.

3-b-4) these slabs can be used to cover up the slopes and gives a good result that is better than concretes, especially in roads and inside the plants.

3-b-5) these slabs are also used in making carb stone by cutting and removing the leftovers using a machine and make the necessary details.



Figure. 11 machine for dressing side slabs



Figure. 13 side slabs cubes

3-c) small tiles.

3-c-1) use a strong pressing stamp machine for granite to create and give numerous designs that attracts one's eye

3-c-2) use the small tiles in construction as a spacer under the steel, that will give a good result

3-c-3) some small broken tiles can be used in landscapes to give a unique style.

3-c-4) when using the small tiles as they are, it is called a crazy cut which can be placed in many areas.



Figure. 14 Stamped Granite



Figure. 12 Granite interlock



Figure. 15 Curb stone from Side slabs





*Figure. 16 Landscape from side blocks*

#### **Title and authors**

Eng. Firas Al Jedi, Civil Engineer of Tishreen University, Syrian Arab Republic. Since 1993 he is specialist in Managing and Constructing Engineering Projects. Firas Aljedi is an expert in stone field and worked as director and consultant for many companies in Syria and Saudi Arabia, developing stone quarries, worked with big Italian Companies to develop granite and marble processing machines, and working with German Company to produce useful products from wastage and harmful elements brought from Marble & Granite Factories.

#### **Acknowledgements:**

Masa-study and information as per P.I.N.O.1010042247

Masa-StraBe 2, D-56626 Andernach/Rhein, Germany, tel- +49-(0)2632-92 92-0

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# SOAPSTONE – WAYS RECYCLING WASTE ROCK AND MINERALS

S. Leinonen<sup>1\*</sup>

(37) Seppo Leinonen, Geological Survey of Finland (GTK), Neulaniementie 5, Kuopio, Finland, \*[seppo.leinonen@gtk.fi](mailto:seppo.leinonen@gtk.fi)

**Summary:** Soapstone is known as a dimension stone also a unique material in heat retaining fireplaces. It is easy to cut and carve due to the softness. As usual in block extraction, non-usable rock corresponds generally over 50 % or even more of quarried total. Cutting also small and irregular shaped boulders and utilization of minerals expand the possibilities of recycling quarry and processing wastes. Both the main minerals, talc and magnesite are common in industrial uses, talc as a filler in papers, plastics, and ceramics, while majority of magnesite as a refractory material, also applied in magnesium chemical industry and preparation of flame-retardants depending on the temperature of heat treatment. Evaluation of recycling potential bases on technical properties measured for 11 soapstone from eastern Finland. Beneficiation test was carried out for a one flaked talc containing type.

**Key words:** Soapstone, technical properties, waste rock, recycling, talc, magnesite, beneficiation

## 1. Introduction

Soapstone is defined in prEN12670 Natural stone – Terminology as a 30-70 % talc composing rock and commercially as a rock consisting of talc, carbonate, chlorite, and serpentine (technical committee CEN/TC 246 Natural stones). Geologically soapstone is a metamorphic, hydrated, and carbonated ultramafic rock, occurring usually in Archean and Proterozoic schist belts, also in young mountain regions (Naldrett, 1966; Evans and Trommsdorff, 1970; Pinheiro & Nilson, 2000). Formations are identified and classified by chemical composition also according to adjacent lithological associations (Thayer, 1990; Sarkar et al., 2018). Depending on partial pressure of carbon dioxide in metamorphic fluid, also other minerals among talc-carbonate are apparent, like serpentine, olivine, tremolite and anthophyllite.

Historically soapstone has been applied as a cook ware, also to make sculptures, for example in Norway, dated as old as late Bronze Age to pre-Roman Iron Age ≈ 2200-2400 BP (Grenne et al., 2017). It has been also used in constructions and ornaments, one of the most known is Nidaros Cathedral in Trondheim, Norway (built in years 1070-1300), Storemyr et al., 2010 and temples of Hoysala architecture in India during the 13<sup>th</sup>-14<sup>th</sup> centuries. In Finland soapstone was popular in architecture of Art Nouveau and national romanticism (1895-1920), Hannula & Salonen, 2007. At present, structures are mostly made by slabs as outer coverings, and indoor flooring also elements in fireplaces (Fig. 1 and 2). Soapstone is acoustically dead on the stairs and floors, not slippery when wet in the bathrooms.

*Fig. 1. Soapstone from Juurikkaniemi formation, in municipality of Kuhmo, in eastern Finland. Honed surface finishing, scale 50 mm.*



## 2. Objectives

The aim of this study was to compare the most common technical properties of various eastern Finnish soapstone to find the basis for increasing the use of waste rock as a dimension stone, also in heat retaining fireplaces. In addition to utilizing small and irregular boulders, there has also been an idea to evaluate the suitability of the main minerals in industrial applications - talc as a filler mineral and magnesite in refractories.

Technical tests were available for 11 soapstone, detailed mineral composition and bench-scale beneficiation try-outs for one flaked talc containing magnesite type species. Because of the softness of soapstone careful handling is necessary in saw cutting and moving the semifinished slabs along processing lines. Even if the enriching of talc and the magnesite are feasible by common flotation procedures, the concentrates should be pure enough for industrial uses. Criteria for recyclability as dimension stone were strength and hardness, exploitation as a mineral the nature of talc and magnesite as well the mass recovery in conventional flotation, and purifying techniques for concentrations.



Fig. 2. Soapstone fireplace, model Käpykolo (photo by Tulikivi).

### 3. Research methods

The strength of soapstone was measured by uniaxial compression test EN 1926, flexural strength according to EN 12372 and point load test using method EN 13364 in the laboratory of Suomen Kivikeskus, Juuka. Density and porosity measures are with the standard EN 1936. Tests were applied for 11 soapstone types quarried in eastern Finland (Fig. 3 and 4). Analytical methods for mineral composition and for characterizing minerals species were microscope, mineral liberation analyser (MLA), field emission scanning electron microscopy (FE-SEM) and microprobe in Espoo and Outokumpu laboratories of Geological Survey of Finland (GTK). Beneficiation properties for a flaked talc containing soapstone were obtained by bench-scale flotation in GTK's pilot plant, Outokumpu. Chemical analyses were performed by Eurofins Labtium Oy with X-ray fluorescence (XRF), carbon with combustion technique (Leco-method).

### 4. Results

Silica content of whole rock chemical composition is typically very low in soapstone, here 45.35 wt.% SiO<sub>2</sub>

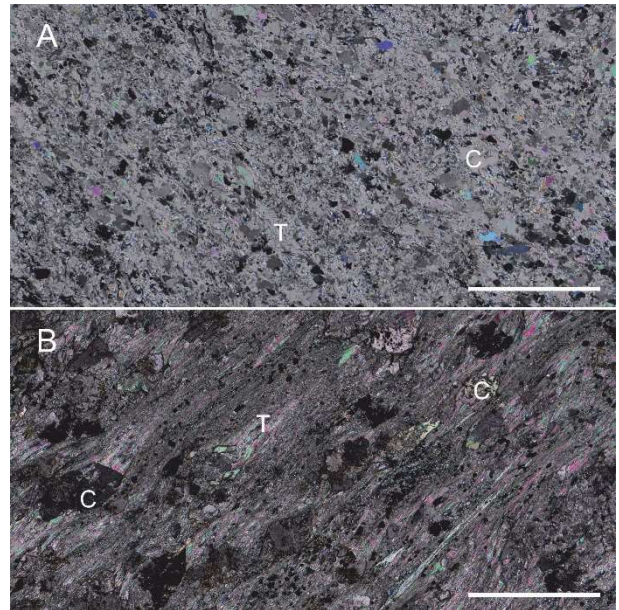


Fig. 3. Microscopic images of talc-magnesite soapstone. A) An even grained, non-oriented texture and B) soapstone with coarse carbonate crystals and a distinct foliation (T=talc and C=carbonate), scale 10 mm.

in average, min 35.04 and max 53.14 (7 of 12 samples have below 45 wt.%), while magnesium showed relatively high values, ranging from 34.16 to 45.67 wt.% MgO (in average 39.36), Table I (oxide-% are calculated as volatile free). Iron content was about 12.00 wt.% Fe<sub>2</sub>O<sub>3</sub>, aluminium and calcium averagely 2.00 Al<sub>2</sub>O<sub>3</sub> and 1.00 CaO respectively. Titanium and manganese were in

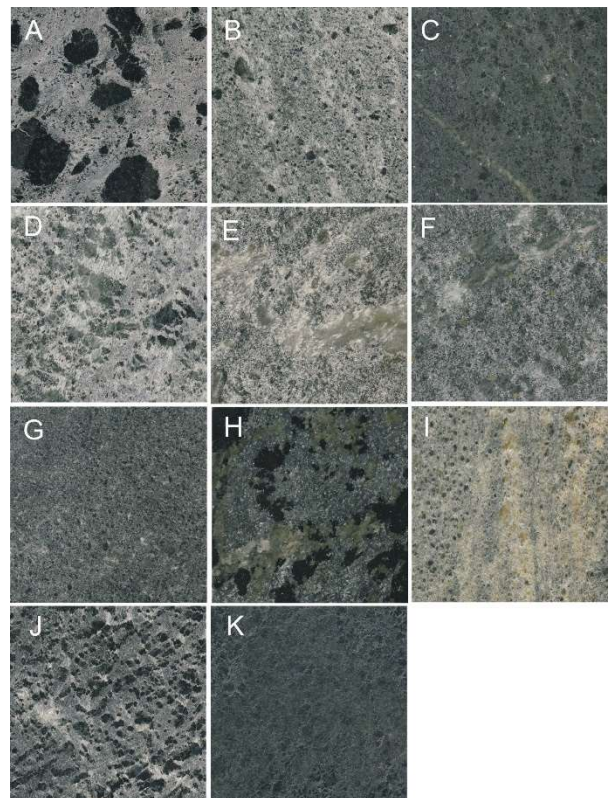


Fig. 4. Macroscopic textures of soapstone with honed surfaces. Talc-magnesite soapstone, sample in image H includes also serpentine. All samples represent commercial soapstone from eastern Finland.

minor amounts, while sodium and potash only in tracing extends. Whole rock chemical analyses of main components and some minor elements were by XRF (X-ray fluorescence spectrometry), Philips Axios WD-XRF equipment, pulverized sample and the prepare a push button, method 175X. Samples were crushed with a jaw crusher (jaws from Mn steel) and ground in a hardened carbon steel grinding vessel. C in carbonates, and non-carbonate carbon were measured with Leco-analyser (a combusting technique with infrared detector). Carbon dioxide (fixed in carbonates) varied between 10.33 and 23.50 wt.% CO<sub>2</sub> (in average 17.53).

Table I. Whole rock chemical composition by XRF, 12 soapstone from eastern Finland (wt.%, volatile free), b.d.l.=below the detection limit.

No.	SiO <sub>2</sub>	TiO <sub>2</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	CaO	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	Sum
1	47.20	0.067	34.16	1.749	16.56	0.139	0.134	b.d.l.	b.d.l.	100
2	42.97	0.029	40.19	0.666	13.07	0.195	2.881	b.d.l.	b.d.l.	100
3	44.85	0.120	41.84	2.624	10.29	0.102	0.175	b.d.l.	b.d.l.	100
4	43.25	0.115	41.22	2.466	9.89	0.144	2.908	0.010	b.d.l.	100
5	44.14	0.044	37.77	0.817	16.66	0.167	0.405	b.d.l.	b.d.l.	100
6	49.02	0.059	39.75	1.374	9.53	0.079	0.171	b.d.l.	0.016	100
7	43.79	0.062	45.67	1.307	8.77	0.119	0.286	b.d.l.	b.d.l.	100
8	53.14	0.034	37.66	1.677	7.38	0.040	0.060	0.008	b.d.l.	100
9	35.04	0.092	37.17	0.694	23.50	0.173	3.226	b.d.l.	0.105	100
10	46.47	0.065	41.51	1.774	9.84	0.218	0.115	0.013	b.d.l.	100
11	50.89	0.169	34.93	3.547	8.06	0.056	2.329	b.d.l.	0.016	100
12	43.47	b.d.l.	40.45	2.045	13.74	0.127	0.169	b.d.l.	b.d.l.	100

Talc, magnesite, and chlorite were over 80 % of mineral composition of 12 examined soapstone (80.60-98.30) of which talc is the most abundant, the mean value 50.04 wt.%, variation from 42.77 to 61.38. Magnesite appeared at an average 30.28 wt.% (16.43-42.87), while the total carbonate content with dolomite showed some higher values (21.27-47.13). The average of chlorite was 8.45 wt.% and oxides 6.02. Occasional accessories were serpentine and sulphides, dolomite varied from zero to as highest 12.58 wt.% (Table II). The most common oxides were magnetite and chromite, while the most abundant sulphides were pentlandite, pyrite and pyrrhotite (completely absent in 4 of 12 analyses).

Table II. Main minerals of 12 soapstone, measures by MLA. Abbreviations: Tlc, talc; Mgs, magnesite; Dol, dolomite; Cal, calcite; Srp, serpentine; Amp, amphibole; Sulp, sulphide; Othe, others.

No.	Tlc	Mgs	Dol	Cal	Srp	Chl	Amp	Sulp	Oxid	Othe	Sum
1	46.84	21.22	0.05	0.00	0.19	12.54	0.00	0.46	18.68	0.02	100
2	47.64	36.44	1.29	0.00	0.46	3.26	0.00	0.28	10.24	0.38	100
3	56.97	32.19	0.11	0.00	0.24	6.10	0.00	0.52	3.85	0.02	100
4	46.95	36.67	10.46	0.00	1.21	1.69	0.00	0.05	2.97	0.00	100
5	61.38	21.61	2.22	0.00	0.27	10.07	0.00	0.00	4.41	0.04	100
6	57.56	23.30	2.75	0.00	0.35	1.41	0.00	0.01	14.58	0.03	100
7	43.73	34.45	5.27	0.00	0.21	11.29	0.00	0.00	5.03	0.02	100
8	43.68	27.92	0.00	0.00	17.40	10.54	0.00	0.39	0.07	0.00	100
9	52.76	29.34	0.01	0.00	0.47	11.07	0.00	0.00	5.53	0.81	100
10	50.97	40.85	0.00	0.00	1.12	1.75	0.00	0.20	3.92	1.18	100
11	49.25	42.87	0.66	0.00	0.62	6.18	0.00	0.00	0.42	0.00	100
12	42.77	16.43	12.58	0.04	0.00	25.48	0.00	0.06	2.50	0.13	100
Avg	50.04	30.28	2.95	0.00	1.88	8.45	0.00	0.16	6.02	0.22	
min	42.77	16.43	0.00	0.00	0.00	1.41	0.00	0.00	0.07	0.00	
max	61.38	42.87	12.58	0.04	17.40	25.48	0.00	0.52	18.68	1.18	

The mineral liberation analyser (MLA) determinations were done with a Quanta 600 ESEM device in GTK's

Outokumpu mineralogical laboratory. The equipment included a scanning electron microscope and an energy-dispersive X-ray spectrometer. The software produced by JKTech was used for quantitative measurements. The samples were analysed automatically with the XMOD-STD point calculation method. An average of 24,000 points were measured from each sample.

#### 4.1 Technical tests

Density of the studied samples varied from 2800 to 3130 kg/m<sup>3</sup> (average of 12 values 2949), EN Standard 1936 and water absorption 0.05-0.42 % (0.17), EN13755 (Leinonen, 2013). There was a relatively large variation between the highest compressive strength 51.5 Mpa compared the lowest value 18.3. The most strength soapstone showed nearly 3 times higher value, whereas comparing flexural strength there was more than a 5-fold difference between the weakest and the strongest structural type (Fig. 5). Breaking load test (EN 13364) also displayed the same kind of strength measures than with compressive and flexural strength experiments, i.e., over 3 times higher result compared to the lowest values (Table III).

Strength of soapstone relates mostly on small grain size, grade of foliation, and mineral composition. The most strength species include less sheeted structured soft talc (1 Mohs) and chlorite (2-2.5) than the weakest while both carbonate and serpentine increase the strength.

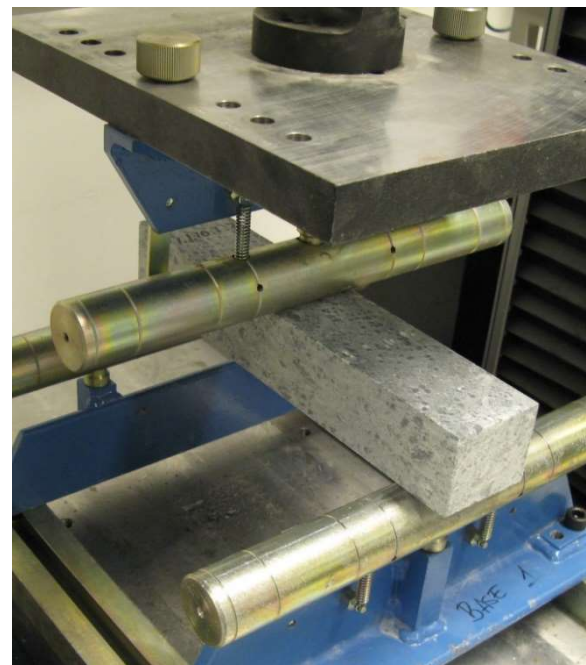


Fig. 5. Test of flexural strength for a carbonate spotted soapstone.

Thermal properties were also in interest of the testing project, the average of heat capacity for 11 samples was 1490 J/kgK in room temperature and that of thermal conductivity 7.63 W/mK. The same properties for a dense fire brick are lower 880 J/kgK and 1.40 W/mK.

Table III. Technical properties of soapstone, samples from Eastern Finland. Tests according to EN-standards.

No.	Apparent porosity	Density	Compressive strength	Flexural strength	Breaking load
	%	kg/m <sup>3</sup>	Mpa	Mpa	F (N)
	EN 1936	EN 1936	EN 1926	EN 12372	EN 13364
1	0.39 %	3080	23.5	7.7	1800
2	0.47 %	3130	24.1	6.4	1450
3	0.36 %	2970	38.9	13.5	1950
4	0.58 %	2970	30.9	11.7	1350
5	0.25 %	2910	45.1	11.5	2150
6	0.47 %	2910	20.4	9.2	1450
7	0.80 %	2890	18.3	7.6	1300
8	0.38 %	2800	51.5	15.4	3800
9	0.56 %	2920	21.1	3.5	1600
10	0.30 %	2950	49.2	12.0	2350
11	1.32 %	2910	18.5	2.9	1100
min-max	19 %	89 %	36 %	19 %	29 %
Avg	0.53 %	2949	31.1	9.2	1 846
Std	0.30 %	90.5	12.8	4.0	751.8
Min	0.25 %	2800	18.3	2.9	1100
Max	1.32 %	3130	51.5	15.4	3800

#### 4.2 Beneficiation tests

Beneficiation tests were carried out for Haapponen soapstone. The location of the deposit is in the municipality of Suomussalmi, eastern Finland. The main chemical elements of 20 samples with bulk rock XRF analyses were SiO<sub>2</sub>, MgO, Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> (Table IV). The mineral composition includes talc, magnesite, and chlorite, also in less amounts of dolomite, magnetite, and chromite (Table V). The grain size varies 0.2-20 mm, coarsest grains are magnesite carbonate. Talc occurs in flaked (0.2-1 mm) and small grained (<< 0.2) particle sizes, the texture displays various schistose types. Talc has recrystallized in metamorphic and deformation events, coarse flakes occurring typically beside carbonate crystals (Fig. 6).

According to the microprobe analyses (4 thin sections, 45 measures), the chemistry of talc is relatively constant, but can be grouped to a high-iron 2.55-3.81 wt.%, FeO and a low-iron 0.67-2.12 compositions (Table VI).

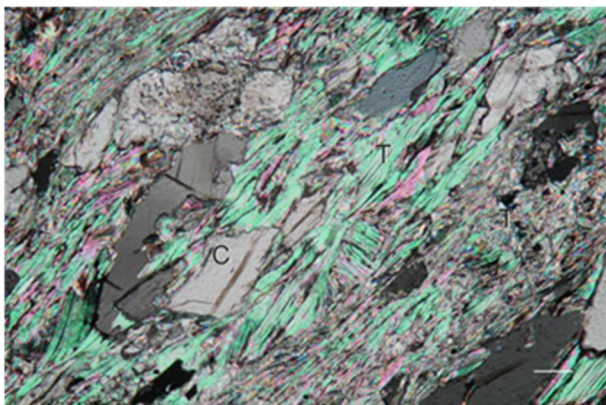


Fig. 6. Microscope image of Haapponen soapstone. Sample RH93 29.23-29.39, T=Talc and C=carbonate, scale 0.1 mm.

Magnesite also shows an iron-poor 5.96 wt.% (2.00-7.08) and an iron-rich type 13.44, (9.00-15.30), 3 thin sections and 81 analyses (Table VII).

The microprobe analyser was Cameca SX100, accelerating voltage was 15 keV with a beam current of 20 nA and a beam size of 5 µm.

Table IV. Chemical elements by XRF whole rock analyses (wt.%) of Haapponen soapstone (20 samples), S and C (wt.%) by Leco-method, loss of ignition (LOI) was measured after combusted in a muffle furnace at 950 °C.

No.	SiO <sub>2</sub>	MgO	Ni	LOI	Al <sub>2</sub> O <sub>3</sub>	Fe	S	C
1	36.80	30.30	0.22	19.53	2.15	5.84	0.15	4.76
2	32.90	33.00	0.22	24.05	1.33	5.32	0.01	5.99
3	31.30	34.00	0.23	25.12	0.98	5.20	0.02	6.25
4	32.50	32.50	0.21	22.80	1.47	6.17	0.01	5.82
5	33.30	31.30	0.21	22.50	1.52	6.86	0.03	6.19
6	31.50	34.90	0.22	24.12	0.99	5.16	0.03	6.32
7	32.50	33.10	0.23	24.23	1.30	5.30	0.12	6.41
8	33.60	31.20	0.21	22.98	2.08	6.07	0.03	6.21
9	32.20	33.20	0.21	25.22	1.22	5.02	0.02	6.54
10	32.10	33.40	0.23	25.53	1.05	4.87	0.01	6.75
11	38.40	31.00	0.21	20.38	1.30	4.69	0.08	5.26
12	35.30	27.80	0.18	21.17	2.27	6.04	0.06	5.12
13	31.40	30.10	0.11	23.77	1.27	8.00	0.05	6.22
14	31.40	32.30	0.21	24.70	1.12	5.32	0.12	6.47
15	31.90	34.50	0.23	24.53	0.92	5.16	0.06	6.50
16	30.00	33.90	0.19	24.20	1.03	6.79	0.26	6.42
17	34.80	31.90	0.20	22.64	1.20	5.35	0.07	6.76
18	32.50	34.50	0.22	22.98	1.54	5.20	0.03	5.72
19	31.40	33.20	0.19	24.25	1.30	5.94	0.10	6.71
20	33.40	32.20	0.21	22.86	1.72	6.02	0.04	5.86
Avg	32.96	32.42	0.21	23.38	1.39	5.72	0.07	6.11
min	30.00	27.80	0.11	19.53	0.92	4.69	0.01	4.76
max	38.40	34.90	0.23	25.53	2.27	8.00	0.26	6.76
STD	2.03	1.76	0.03	1.60	0.39	0.80	0.06	0.55
CV	6.15 %	5.43 %	12.93 %	6.82 %	28.42 %	14.07 %	94.51 %	9.02 %

Table V. Mineral compositions of Haapponen soapstone (wt.%), 20 drill core samples. MLA analyses.

No.	Tlc	Mgs	Chl	Dol	Magn	Chr	Sulp	Othe	Sum
1	52.39	34.11	9.46	1.25	2.17	0.47	0.00	0.15	100
2	54.71	35.36	8.04	0.15	1.15	0.22	0.00	0.37	100
3	53.94	36.23	7.63	0.09	1.89	0.11	0.00	0.11	100
4	53.87	33.13	8.02	2.84	1.38	0.57	0.00	0.19	100
5	56.31	31.32	6.55	4.93	0.01	0.43	0.29	0.16	100
6	59.33	23.06	11.91	4.36	0.03	0.66	0.44	0.21	100
7	54.84	36.22	6.38	0.11	2.35	0.07	0.00	0.03	100
8	54.36	33.63	9.17	0.64	1.78	0.24	0.00	0.18	100
9	53.19	34.89	7.65	1.06	3.01	0.12	0.00	0.08	100
10	54.81	34.39	6.44	0.36	2.98	0.22	0.55	0.25	100
11	54.38	29.34	9.30	2.53	3.87	0.44	0.00	0.14	100
12	61.26	29.01	6.74	2.62	0.00	0.28	0.02	0.07	100
13	65.89	22.67	7.41	3.21	0.35	0.19	0.14	0.14	100
14	51.15	37.18	6.41	0.68	4.15	0.31	0.00	0.12	100
15	53.32	31.82	10.30	0.51	3.94	0.06	0.00	0.05	100
16	52.81	38.15	7.69	1.10	0.00	0.02	0.10	0.13	100
17	53.74	35.89	8.18	1.33	0.04	0.29	0.41	0.12	100
18	52.05	33.64	12.26	1.09	0.53	0.26	0.00	0.17	100
19	56.90	30.86	9.95	0.70	1.35	0.14	0.00	0.10	100
20	56.80	17.88	13.40	11.44	0.03	0.08	0.05	0.32	100
Avg	55.30	31.94	8.64	2.05	1.55	0.26	0.10	0.15	
Min	51.15	17.88	6.38	0.09	0.00	0.02	0.00	0.03	
Max	65.89	38.15	13.40	11.44	4.15	0.66	0.55	0.37	
STD	3.47	5.31	2.06	2.62	1.45	0.18	0.17	0.08	
CV	6.3 %	16.6 %	23.8 %	127.8 %	93.6 %	68.4 %	174.8 %	54.7 %	

Talc concentrates of 20 enriching tests were quite pure, grading averagely about 95 wt.% talc (min-max, 92.0-96.9). Recoveries after 3 cleaning flotation varied 50-64 %, in average 56 (Fig. 7). Brightness was 69.7 % (ISO R457), after micronizing that much higher 83.0 (Korhonen & Lavikko, 2018), Table VIII. Recoveries of magnesite varied from about 95 % in the rougher flotation wastes up to 98 when cleaning stages included.

According to the test 2, 94.5 % of the total magnesite ended to the enrichment sand in the rougher flotation, 5.5 to the talc concentrate. Magnesite content was relatively high 75.34 wt.%, while after the three cleaning stages included, significantly that lower, the average of all 20 flotation tests 47.33 wt.% (the range of variation 27-58 wt.%). Other minerals in magnesite waste of the test 2 were chlorite 11.37 wt.%, talc 9.86, dolomite 1.75 and magnetite 1.68.

Table VI. Mineral chemistry of talc, Haaponen deposit, 45 microprobe analyses.

	SiO <sub>2</sub>	MgO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	MnO	CaO	N <sub>2</sub> O	K <sub>2</sub> O	NiO
Avg	62.80	29.21	0.01	0.19	2.52	0.01	0.01	0.00	0.01	0.43
Min	59.47	27.67	0.00	0.00	0.67	0.00	0.00	0.00	0.00	0.30
Max	64.60	31.22	0.06	2.10	3.81	0.04	0.04	0.02	0.05	0.54
STD	1.05	0.93	0.01	0.38	0.83	0.01	0.01	0.01	0.01	0.10
CV	1.7 %	3.2 %	134.5 %	206.3 %	32.9 %	155.7 %	91.0 %	246.2 %	111.7 %	23.6 %
Count	45	45	23	43	45	17	31	7	26	43

Table VII. Mineral chemistry of magnesite, Haaponen deposit soapstone, 81 microprobe analyses.

	MgO	FeO	MnO	CaO	SrO	BaO
Avg	39.49	9.75	0.25	0.08	0.02	0.01
Min	34.15	2.00	0.01	0.01	0.00	0.00
Max	44.02	15.66	0.41	0.66	0.07	0.07
STD	5.34	3.94	0.07	0.08	0.02	0.01
CV	13.5 %	40.4 %	26.4 %	89.7 %	130.0 %	216.6 %

Black oxides, typically magnetite affects mostly to the quality of talc concentrate, especially decreasing the brightness and the whiteness. The amount of magnetite in talc concentrates was generally low, 0.00-1.50 wt.%, detected values in 16 concentrates of 20, in average 0.36 wt.%. About 85 % of the magnetite ended to the enrichment sands. Sulphides were rare, analysed in 4 of 20 concentrates, in average 0.004 wt.%. (0.000-0.070).



Fig. 7. Cleaning stage of bench-scale flotation test of Haaponen talc-magnesite rock (soapstone). Enriching tasks were in GTK's laboratory.

Mineral composition of enrichment sands for final concentrates were measured by MLA, while that of

rougher flotations were calculated from the whole rock chemical analyses. Brightness of commercial talc for a filler grade of papers and plastics varies commonly 80 % to 96 %, magnesite raw materials, applied in refractory materials, are high in magnesia, 95 wt.% MgO.

Table VIII. The main results of the 20 flotation tests. In addition to the mass recovery, there are also values for talc content and recovery, loss of ignition, i.e., mass-% of total volatiles (LOI), ISO brightness, D50 particle size, silicon oxide and iron by XRF-analyses.

	Mass wt. %	Talc wt. %	Talc Rec. %	LOI wt. %	SiO <sub>2</sub> wt. %	Fe wt. %	ISO R457	D50 μm
Avg	32.7	95.3	56.4	5.7	59.3	2.5	69.7	59.5
Med	32.2	95.6	55.9	5.7	59.5	2.4	69.8	60.3
Min	27.9	92.0	49.5	5.4	57.3	1.8	62.9	45.7
Max	37.3	96.9	64.0	6.3	60.1	3.3	74.3	68.4
STD	2.4	1.3	4.2	0.2	0.6	0.4	2.6	6.4
CV	7.4 %	1.3 %	7.4 %	3.4 %	1.1 %	18.2 %	3.7 %	10.7 %

## 5. Discussion

Soapstone have generally a schistose structure which along it tends to break down causing losses both in quarrying and processing. Extraction is typically done by a chain saw, primary slabs are cut by a blade saw or by a diamond wire cutting. The most strength types are preferred in dimension stone processing and in end-product manufacturing, like fireplaces. Strength is especially emphasized when recycling small, wasted boulders (Fig. 8). According to the technical tests the most suitable structural types of the studied soapstone species for a product manufacturing (C, E, G, H and K in Fig. 4, page 2) have over 2-folds higher compressive strength (51.5-30.9 Mpa) and 2-4 folds higher flexural strength (15.4-11.5) than the schistose types, 24.1-18.3 and 7.7-2.9 respectively. The most strength textures have also less porosity than the weakest types (Table III, page 4).

Mohs hardness, calculated from mineral composition varies from 2.09 M to 2.79 M, an average 2.50. The highest values are measured from those which include plenty of carbonate, the hardness of magnesite is 3.5-4.5 M (talc 1 M). The hardest ones, in the Mosh scale contains also relatively high percentages of magnetite, 20-10 wt.% (hardness of magnetite 5.5-6.5 M).

Abrasion resistance (hardness) by Big Wheel abrasion test (EN 14157) relates quite well to the mineral composition, and those, which display below average measures, i.e., the hardest soapstone species (C, D, F, H and J in Fig. 4, page 2) contain averagely 30 % more carbonates (coarse grains) and 56 % more chlorite than the less abrasive. Hard magnetite occurs typically within magnesite and in association with chlorite.

When developing the utilization of small irregular shaped boulders (a non-usable rock) by the saw cut, the product design should be made basically for thicker and smaller structural elements than the most common slab

dimensions, 600x300x60 mm. Aggregated products, like for environment decorations and sauna stones, are able to be processed from wide range of the strength, however, taking into account that the most schistose soapstone tend to turn as thin pieces when crushed and form also more fines than the less foliated types.



*Fig. 8. Small irregular shaped chain saw cut soapstone boulders in Kivikangas quarry, the municipality of Suomussalmi in Eastern Finland.*

The froth flotation is the most used technique to produce talc concentrate from a talc-magnesite rocks, i.e., soapstone ore types. Talc is commonly the primary valuable mineral, but the utilization of magnesite has also been studied (Pressacco et al., 2010). Haaponen deposit, located in Eastern Finland, is classified as a soft soapstone but after all it can be also used for construction and to manufacturing fireplaces.

Cause of relatively coarse-grained talc there was an interest in carry out bench-scale froth flotation for a preliminary estimate if the Haaponen soapstone is also suitable for talc and magnesite raw materials. The frother (Brennfroth 90) dosage was 60 g/t in rougher flotation and 10 g/t in 1<sup>st</sup> talc cleaning stage and 5 g/t in the 2<sup>nd</sup> talc cleaning stage (third without frother). All the flotation stages were performed in natural pH 8-9. Grinding fineness was about 80 % < 90 µm (Korhonen & Lavikko, 2018). Brightness of the talc concentrate of this study was quite good 69.7 % (D50 46-68 µm), ISO R457 and able to be improved up 83 by an additional grinding of final concentrate, D50 14-20 µm (even more bright by fining down to less than 10 µm particle sizes). It has been also tested dry and wet magnetic separation techniques (Abeyasinghe, 1996) to upgrade both whiteness and

brightness also by leaching with hydrochloric acids (Ahmed, 2011).

According to the froth floatation studies, relatively high magnesite content was reached in the waste of the rougher flotation 75 wt.% (the mass recovery was relatively high 95 %). Cleaning stages for the talc increased the recovery of magnesite up to 98 % but at the expense of a poorer concentration. Most of the impurities, talc and chlorite are cleanable from magnesite sand by common beneficiation techniques, while most of the iron occurs in the magnesite lattice (around 10 %) which can be accordingly removed, at least decreased in a large extend with a chlorine roasting (Pressacco et al., 2010).

Talc is a filler mineral in various materials paper, plastic, paint etc., while magnesite applied commonly as calcined caustic magnesia in industrial materials, flue gas desulphurization and in refractories of steel and glass industries. Fused magnesia is applied in refractory and electrical insulating materials. High recoveries of talc and magnesite are also critical factors in addition the to the requirement of purity of the concentrations when evaluating the profitability of waste rock recycling.

## **6. Conclusion**

At present dimension stone processing of soapstone is designed only for big blocks. Based on the circular economy concept, soapstone industries should also have manufacturing lines and product collections able to utilize small irregular shaped blocks which thus far ends to waste rock piles. Because of the soft nature, saw cutting to the elements of any dimensions prefers hard and solid structured types. This study revealed that there is large variation in strength among commercial soapstone in eastern Finland (18.3-51.5 Mpa), of which the most tight-structured ones have about 50 % higher values than the average. Quarry wastes, which are not reasonable to be recycled to slabs or tiles could be crushed to the environmental products and the sauna stones, while fines formed during aggregate production are usable for example in porcelain items. Beneficiation tests show that main minerals, talc, and magnesite can be enriched to own phases by various flotation procedures. Talc was obtained in high mass recoveries (averagely 32.7 wt.%) and purity (talc in final product 95.3 wt.%), but still one of the challenges is to reach sufficiently pure magnesite (95 wt.%) from waste fraction. According to the results of the technical studies there are possibilities to increase waste-based soapstone production and utilization of main minerals in industrial application, measured both in volume and by turnover. Available raw material developing circular economy, the yearly amount of soapstone quarry waste (0.15 Mt) counts on 15 % of talc ore mining in Finland.

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## TRANSITION TO A GREENER INDUSTRY THROUGH CIRCULAR ECONOMY PROCESSES THROUGH ADDITIVE MANUFACTURING

Elena López Martínez / [elena.lopez@ctmarmol.es](mailto:elena.lopez@ctmarmol.es)

Francisco Javier Fernández Cortés / [javier.fernandez@ctmarmol.es](mailto:javier.fernandez@ctmarmol.es)

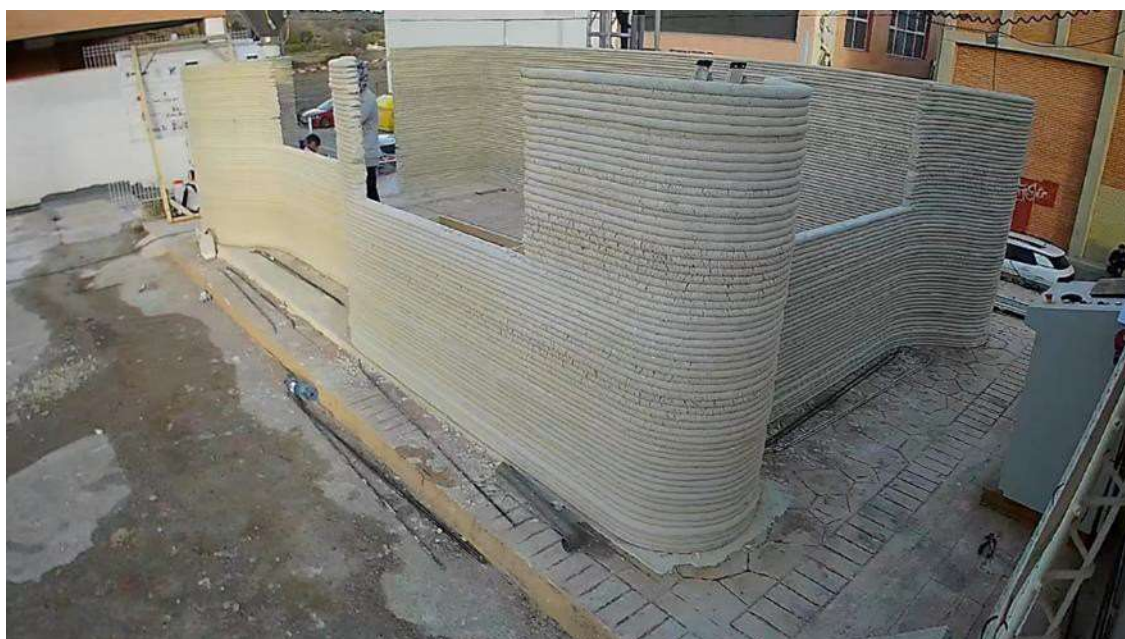
The abandonment or inadequate management of the waste produced by the extractive industry can affect the landscape, soil and vegetation; or in certain cases to aquatic ecosystems and human health. In addition, in some cases, structural failures in the facilities in the event of breakage, could affect people and property. The collection and treatment of this waste enables the quality recycling of the materials that make it up and allows its use in other uses, such as construction materials. All this entails a saving of raw materials and the conservation of natural resources.

That is why, among the technological challenges posed by the materials consumed in the construction sector, the need for a continuous innovation effort stands out in order to increasingly achieve products with greater added value, with new designs and better characteristics, but in line with a sustainable development model that reduces the consumption of resources and the generation of waste. In this sense, the circular economy constitutes a great opportunity in business, social and environmental terms.



The Technological Center for Marble, Stone and Materials has developed this project, incorporating Additive Manufacturing technology to process marble sludge waste and plaster waste, reinforcing them with glass fiber waste to obtain commercial prefabricated products.

The project will contribute to reducing the consumption of mineral resources in the construction sector, to reduce the generation of waste and to manufacture new products with technologies included in Industry 4.0 such as Additive Manufacturing, generating significant environmental, social and economic benefits.



Within the framework of this project, the characterization and adaptation of industrial waste has been carried out to subsequently develop different formulations with the aim of achieving optimal mechanical behavior, either to be used in ornamental elements or to meet structural requirements. All this, through Additive Manufacturing technology, that is, 3D printing.

All this work has resulted in what has been called 'Green Concrete', a material made from waste, specifically 40% by weight of waste, which means that they are not taken to the dump and that implies less consumption of energy and less CO<sub>2</sub> emissions, among other advantages. As a demonstrative example, the 3D printing of a habitable structure has been carried out in the Francisco Fernández Torralba sports complex in Caravaca de la Cruz in collaboration with the Building School of the University of Valencia.

## Towards Sustainability Goals in the Portuguese Ornamental Stone Industry's Supply Chain 4.0

Isabel Duarte de Almeida<sup>1</sup>, Agostinho da Silva<sup>2,3</sup>, Carlos Rabadão<sup>2,3</sup>

(38) Instituto Universitário de Lisboa (ISCTE-IUL), IBS, Lisboa, Portugal \*[isabel.cristina.almeida@iscte-iul.pt](mailto:isabel.cristina.almeida@iscte-iul.pt)

(39) ESTG, School of Technology and Management, Polytechnic of Leiria, Portugal

(40) CIIC, Computer Science and Communication Research Centre, Leiria, Portugal

**Summary:** *In the age of Industry 4.0 (I4.0), the advent of new digital disruptions has brought about a positive impact on supply chains (SC). This has caught the attention of both researchers and practitioners, who are now exploring the potential of these disruptions, especially in relation to sustainability. However, despite this growing interest, the research on this subject remains in its early stages and lacks cohesion, with limited collaboration between academia and policymakers. Thus, the aim of this paper is to investigate how these technologies can redefine company processes and SC in the Portuguese ornamental stone (POS) industry. The primary goal is to identify novel capabilities within the SCOR model that can contribute to sustainable performance and facilitate the achievement of the 17 Sustainable Development Goals (SDG)..*

**Keywords:** *Ornamental Stone; Industry 4.0; Supply chain 4.0; Sustainability; SDGs*

### Introduction

The pace of raw material demand worldwide has surged in recent decades. This growth is driven by the rapid industrialization of emerging economies and excessive material consumption in developed nations. The interconnected global SC are vital in meeting our daily needs in every single society, as almost everything we rely on goes through them. However, due to the vastness of these transactions, there is a cause for concern, prompting the global community to recognize the urgent need for practical solutions. Climate change data indicates that global warming is projected to increase (IPCC, 2018), while social issues have been exacerbated, especially with the arrival of the COVID pandemic and the adverse effects of globalization. Also, Russia's war on Ukraine has severely disrupted global markets, exposing vulnerabilities to the supply of raw materials necessary for industrial production and fuelling inflation.

The growth of construction, manufacturing, and energy industries depends heavily on mineral resources, making them valuable economic assets in the regions where they are discovered. Portugal has a rich and complex geology, resulting in substantial mineral wealth, including numerous deposits of ore, industrial minerals, and ornamental stones (OS). Many small and medium-sized enterprises (SMEs) are actively exploring these resources, focusing on the quality and diversity of base ornamental stones, as well as precious metals like lithium. These mineral resources and OS are

currently being exploited, transformed into finished product and delivered local and worldwide through SC. However, the lack of effective land-use policies to safeguard the access of the extractive industry to these places poses a significant risk to current and future production. This jeopardizes the contribution of this sector to the national economy and the supply of future generations, making it imperative that action is taken.

Across the globe, both governments and businesses have taken decisive action in reply to the pressing issue of sustainable development. They have committed to fulfilling the United Nations' 2030 agenda, which outlines 17 critical goals for achieving sustainable development. The agenda underscores the importance of collaboration and coordination among diverse stakeholders and across SCs to achieve a balance between economic, environmental, and social dimensions, as highlighted by Luthra and Mangla (2018).

The OS industry's supply chain (OS-SC) involves multiple stages, from quarrying and extraction to the final distribution of refined stone products. Quarrying is followed by processing, where stones undergo cutting, polishing, and finishing to enhance their quality and appearance. Processed stones are then categorized based on various aesthetic factors and stored in warehouses. From there, stones are sold to distributors, wholesalers, and retailers, who play crucial roles in sourcing, processing, and selling the products. Challenges in the SC include responsible sourcing, sustainable practices, fair

labour, and efficient transportation logistics. In addition, technological advancements, such as digital platforms and online marketplaces, have also influenced the industry. Overall, the SC in the OS industry is a complex network that continues to evolve based on sustainability concerns, technological advancements, and changing consumer preferences. To tackle the sustainability challenge, OS-SC, as the overall SC, should consider transitioning to a digital ecosystem. This shift will help in creating resilience and developing the necessary abilities to steer the OS-SC towards sustainability strategically. (Cañas et al., 2020).

The fourth industrial revolution, also called I4.0, has ushered in game-changing technologies such as IoT, Blockchain, robotics, and artificial intelligence (AI) (Almeida & Silva, 2020). The idea of exploring the effects of I4.0 technologies on sustainability performance has become increasingly relevant

complexities involved in redesigning OS-SC, assess the potential of I4.0 technologies in enhancing sustainability performance across the set of SDGs, and highlight sustainability capabilities to uncover the interplay between ecological, social, and economic dimensions within the SCOR model processes. A preliminary framework is presented to visualize the potential connections between SC 4.0 capabilities, sustainability dimensions, and SDGs.

## **Theoretical Background**

### *-Sustainability and Innovation in the POS Industry*

It is an established fact that the traditional linear industrial economy operates by extracting natural resources, converting them into goods, distributing them, and ultimately discarding them as waste (Ashby, 2015). The cost of resources has historically been declining in real terms, providing little incentive to conserve them throughout the 19th and 20th centuries. However, since the turn of the 21st century, this trend has been reversed, and the cost of materials and energy has steadily increased, putting immense pressure on SC due to increasing demand from rapidly growing economies. For an economy to be sustainable, it needs equitable patterns of production and consumption. Amid increasing competition between industries, it's crucial to adopt a practical approach that optimizes production costs, enhances quality, and reduces lead times. Organizations have recently been focusing on creating practical strategies to boost

since the adoption of the 2030 Agenda, opening a vast field for researchers and practitioners to explore the impact of I4.0 technologies on sustainability performance (Sachs et al., 2019). One crucial aspect of sustainability is traceability, which has been emphasized by the UN Global Compact and Business for Social Responsibility (BSR), with Blockchain technology expected to play a vital role in achieving this goal (UN & BSR, 2014; Khanfar et al., 2021).

There have been efforts to create a sustainable OS-SC using I4.0 technology and to achieve the UN's SDGs. However, these efforts haven't shown significant results yet. The current literature lacks comprehensive frameworks that address the intersection between SC 4.0 capabilities, sustainability's triple bottom line, and the 17 SDGs. To bridge this gap, this research aims to address the

their performance, accuracy, reliability, decision-making abilities, control, and flexibility (Silva & Almeida, 2020). Furthermore, the manufacturing sector has been incorporating closed resource cycles into its I4.0s to support sustainable operations management (Russo et al., 2019).

Our daily lives heavily rely on mineral extraction and processing products, which are crucial for various industries such as construction, pharmaceuticals, and cosmetics (Azapagic, 2004). While it is expected that the stock of POS suitable for building, decorative construction, or artistic purposes will remain economically extractable for the next 500 years (Carvalho et al., 2018), continuous mining and large-scale surface stone extraction have resulted in environmental, economic, and social sustainability issues that pose challenges to the POS sector. As a result, weighing the impacts of their actions on the environment, economy, and society has become a crucial consideration for the POS industry, rather than just focusing on profit and economic efficiency.

The extractive industry plays a crucial role in boosting the economy, but its resource-intensive practices have raised concerns about its long-term sustainability in today's environmentally conscious era (Careddu et al., 2019). The OS industry faces significant sustainability challenges, including extractive waste, waste from stone processing plants, and issues with critical and secondary raw materials (Kulczycka & Dziobek, 2021). Failing to

adhere to legal and environmental standards could result in serious ecological and safety issues for businesses. The OS industry's environmental impact is significant and visible, especially in vulnerable areas. This impact can lead to habitat destruction, loss of fertile land, and damage to historic archaeological sites. Waste management is also a significant issue for the stone industry. Therefore, companies are responsible for protecting the environment and ensuring natural resource sustainability. As stone is a natural product with varying characteristics, manufacturing may require discarding some parts, which must be done individually. In countries with developed OS industries like Portugal, waste from natural stone processing plants creates environmental and economic problems if not adequately managed. Additionally, customers' growing environmental concerns and the adoption of transparency processes in the value chain (Mehta & Chahal, 2021) signal the need for a globally connected network of companies in the OS industry.

In today's building and construction industry, there is a noticeable rise in the use of sustainable OS products (Liu et al., 2022). Customers who desire modern designs incorporated in point-of-sale displays are willing to pay more for eco-friendly options. Stone's natural and long-lasting appearance and minimal environmental and social impact make it an attractive choice. Companies must adapt to changing customer preferences to remain competitive and satisfy their clientele. Therefore, they need to find ways to meet these demands quickly and efficiently. However, it is impossible to eliminate material scraps in the POS production chain completely. Therefore, the most effective solution is to reuse materials that cannot be sold as OS (Moreira et al., 2022). A triple bottom line strategy is essential for preserving existing resources instead of acquiring new ones. This approach requires active maintenance and waste reduction measures. To achieve this, all stakeholders in OS-SC must collaborate and generate ideas to extend natural resources' useful life and facilitate easy reuse. Government-funded initiatives for waste management and social behaviour change programs can also be helpful.

Therefore, a comprehensive approach focusing on stakeholders' well-being, concerns about resource depletion, waste disposal, industry members' survival, and market communities' economic issues

is necessary to ensure sustainability in the POS industry.

#### *-Bridging Sustainability & Industry 4.0 to Supply Chain 4.0 in Ornamental Stone sector*

The fourth industrial revolution involves integrating modern smart technologies to automate traditional industrial operations. According to the literature, I4.0 technologies can reduce transactional costs, increase business value creation, enhance flow transparency, and decrease carbon emissions (Fernando et al., 2021). I4.0 pertains to the current trend in automation technologies in manufacturing, including the POS industry.

In a factory setting that incorporates I4.0 technology, machines work together in a network to create flexible and intuitive systems that can quickly respond to human input, and feedback gathered through interactions with objects during the manufacturing process (Faller & Feldmüller, 2015). Many POS companies have already switched to I4.0 to achieve production flexibility and offer mass customization to their customers (Won & Park, 2020). However, the digital transformation of manufacturing and production processes is an inevitable reality. This represents a new era in the industrial value chain organization and control. To remain sustainable and competitive, businesses must fully embrace I4.0's mobilizing effects (Caiado et al., 2022). This will lead to the creation of the next generation of factories that leverage Cyber-Physical Systems (CPS) (Lee & Lim, 2021) to support production, resulting in "Smart Factories for the Internet of Things" (also known as "digital factories"). Smart factories aim to improve efficiency, reduce waste, and increase productivity. Innovative methods come with risks, but they can be reduced by using performance simulation tools that suggest potential scenarios during production and real-time physical experimentation (Diao & Sum, 2022). To adopt this innovative industry paradigm, POS companies need to restructure their production process; however, there is no universal standard for adopting I4.0 as every case is unique and dependent on the organization's maturity. An inter-operable global SC should have shared knowledge of I4.0-related concepts and digital technologies aligned with specific conditions, regardless of geographical region (Rosário & Dias, 2022).

The mobilization and technological change in the POS sector align with two Sustainable Development Goals: SDG9 (Industry, Innovation, and Infrastructure) and SDG12 (Responsible Consumption and Production). This approach emphasizes the importance of sustainable industrial policy, I4.0 technologies, digital transition, and new skills development to foster European competitiveness of the sector. According to Silva and Almeida (2020), the integration of these elements is key to achieving this goal. The upcoming industrial mindset focuses on sustainability as a design principle. The priority is to extend products and materials' lifespan while maximizing their value. To achieve this, all companies in the industry sector need to follow a common agenda accepted by stakeholders along the SC (Frazao, 2019). The adoption of a unified framework that enforces decisions based on sustainability's triple bottom-line principles, the SDGs, and the globalization background is, therefore, crucial.

The implementation of I4.0 has revolutionized SC management by creating an integrated ecosystem fuelled by advanced technologies (Javaid et al., 2022). The POS industry is starting to adopt some of the most researched and widely used technologies, such as blockchain, the internet of things, big data, cloud computing, robotics, sensors, 3D printing, and AI. (Silva & Almeida, 2020; Frazao, 2019). Known as drivers of change, these technologies support sustainability at every level (Centobelli et al., 2020). Supply Chain Council, a global non-profit organisation, has developed a process reference model called SCOR (Supply Chain Operations Reference) to provide a comprehensive and standardised set of definitions and best practices for managing and optimising SC. It provides a common language and structure for understanding and evaluating different aspects of the SC, which helps organisations analyse and improve their SC performance (APICS, 2017).

SCOR framework is designed to simplify the language used to describe SC management by categorizing the following six process categories: plan, source, make, deliver, return, and enable. The latest version of the framework recognizes I4.0 technologies and consider them as emerging practices (SCC, 2017). These technologies have the potential to drive process excellence across all six levels of the SCOR Model (Table 1), transforming traditional SC into connected ecosystems enabled by seamless planning and execution systems, visible

logistics operations, autonomous transactions, smart procurement and storage, efficient and timely spare part management, and advanced big data analytics (Ivanov et al., 2019). SCOR framework fits in OS-SC management.

Table 1. SC4.0 process quality criteria built on SCOR model processes

SCOR Model Processes Process	Process excellence criteria in Supply Chain 4.0
Plan (involves activities related to developing a supply chain strategy, including demand and supply planning, resource allocation, and capacity planning)	Modal optimization Efficient and lean planning Improved sales and operations planning Accurate forecasting
Source (focuses on activities related to sourcing and procurement, including supplier selection, negotiations, and supplier relationship management)	Optimized & efficient sourcing Long standing Supplier relationship Sustainable and responsible sourcing Supplier diversity and development Improved supplier's collaboration
Make (encompasses activities related to manufacturing and production, such as production scheduling, quality management, and maintenance)	Lean manufacturing Efficient waste management and disposal Improved manufacturing quality Efficient resource management
Delivery (involves activities related to order fulfillment and delivery, including order management, logistics, transportation, and customer service)	Transport optimization Efficient packing, pick & put-away Optimized loading Sustainable transportation modes Automated administrative tasks Traceable transactions/ products
Return (deals with activities related to reverse logistics, product returns, and managing customer complaints or product recalls)	Electronic returns tracking Traceable transactions/ products Efficient returns management
Enable (comprises processes associated with SC management such as business rules, facilities performance, data resources, contracts, compliance, and risk management)	Continuous improvement and measurement

Blockchain is one of the most innovative technologies. The block architecture of Blockchain is able of storing and transmitting data with the added benefits of traceability, privacy, security, and transparency (Habib et al., 2022). Smart contracts (self-executing and ensuring trust between parties) are a key feature of the Blockchain. They reduce repetitive tasks, save transactional and administrative costs, and add value to contracts (Christidis & Devetsikiotis, 2016). Blockchain technology is particularly useful in SC management, measuring carbon footprint, product traceability, and product authenticity, as per Zkik et al. (2022) and Ayan & Güner (2022) explain. It also benefits the environment and society, such as supporting job creation, facilitating humanitarian SC, and improving environmental goals like water sustainability (Zhao et al., 2019). In addition to IoT, Blockchain, RFID tags, or connected sensors are emerging technologies with interoperability capabilities (Zhao et al., 2019). Keeping devices and actuators linked to the internet is crucial in today's world (Ayan & Güner, 2022). Mainly in smart industries, warehouses, and transportation, physical systems continuously transmit a large amount of data to the network. Real-time data generated by IoT helps organizations manage waste, recycle, reuse, recover, and remanufacture, resulting in economic and environmental benefits (Cañas et al., 2020; Silva & Almeida, 2020).

I4.0 are also significantly impacted by AI and advanced analytics. In addition to allowing companies to anticipate demand, predictive analytics also helps them avoid delays, surplus production, and inefficient loading (Matenga et al., 2022). Moreover, robotics and intelligent autonomous vehicles consistently perform repetitive tasks (Phuyal et al., 2020). Arunmozhi et al. (2022) report that they are extensively used in warehouses and distribution centres, leading to increased efficiency and sustainability benefits,

production capacity and schedule maintenance to minimise disruptions.

Additionally, I4.0 technologies can be used to select suitable suppliers and coordinate operations among partners. This is done using Blockchain technology to share data across SCs and coordinate operations among partners in the I4.0 environment. This system is complex and data-rich, with data analytics playing a crucial role in evaluating and optimising performance. As a result, companies can gain



Figure 1. Driving SCOR processes towards the 17 SDGs in POS industry's 4.0 Ecosystem SC

including emissions reductions, cost savings, and social welfare improvements.

*- Pathways to Sustainability and 17 SDGs in POS industry's 4.0 Ecosystem Supply Chain*

The concept of SC 4.0 involves an interconnected ecosystem where all stakeholders work together to achieve resilience, innovation, and sustainability in pursuit of SDG 9 (Javaid et al., 2022). This approach uses I4.0 technologies to optimise design, management, and planning. However, there are challenges and research issues to be addressed. Real-time market intelligence is needed to predict future demand and understand customer behaviour. Data analytics can help minimise stock inventory while maximising customer service levels. AI and machine learning can be utilised to allocate

valuable insights through data-driven SC analytics and make optimal decisions to gain a competitive edge.

To achieve the SDGs in the POS industry, implementing a 4.0 ecosystem in SC must be considered. This is a process that needs to describe the activities associated with integrating and enabling SC strategies. These include the creation of and management of business rules; performance management through continuous improvement; managing data, information, and SC technology; human resources management; contracts and agreements management; network design, regulatory and compliance management; risk management, Environment, Social, and Governance (ESG) management; enterprise business planning, segmentation creation and management; and circular SC management.

Using the SCOR model will provide an integrated, simultaneous, and connected framework for evaluating and improving performance across OS-SC levels. Moreover, integrating the drivers of sustainable SC 4.0, the SCOR model can be used, step by step (following the Table 1 six SCOR framework processes) to find categories of novel capabilities that will facilitate sustainable performance and 17 SDGs achievement (Figure 1).

A SCOR process begins with Planning. A streamlined and responsive planning process is enabled by Industry 4.0 technologies, which facilitate trust, transparency, and stakeholder collaboration. As a result of real-time collaboration, companies can maintain close relationships with their suppliers and monitor their compliance with environmental and social standards (Mondejar et al., 2021). As a result, SDGs 10 and 3 are achieved. As a result of this collaboration, progress towards the SDGs will be enhanced, eventually leading to the achievement of SDG 17.

SCs can gather verified information about suppliers by incorporating these technologies into their Sourcing processes, including their working conditions, contractual terms, and environmental impact. With blockchain technology, for example, suppliers can verify and authenticate labels and certifications (White, 2017). SC partners are more likely to collaborate with local businesses and trust new suppliers when they can access information about their suppliers' economic, environmental, and social conditions (Kshetri, 2022). SDG 8 is therefore achieved as a result. Furthermore, using AI to optimise routes and loads reduces costs and increases environmental benefits.

The use of smart Manufacturing in the POS industry creates a triple win by boosting production, improving worker safety, and reducing resource consumption, waste, and emissions (Birkel & Müller, 2021; Hofmann & Rüscher, 2017). In addition, blockchain technology simplifies reporting procedures and provides reliable data on emissions, social conditions, and governance (ESG) statistics to stakeholders (Kopyto et al., 2020). As a result of this process, SDGs 12, 13, 14, 15, 3, and 5 can be achieved significantly.

Deliveries are the last mile, and they require an ecosystem that promotes sustainable practices, rethinks asset utilization, and utilizes data. Figliozzi and Jennings (2020) states that electric and

autonomous machines and vehicles for manufacturing processes or transport are crucial to driving sustainability. Further, AI can reduce costs and improve the environment by optimizing routes and loads at the last mile.

SCs are challenged by Driver scarcity. To address this issue, technology enables gamified training programs that attract young workers. Simulators provide novice drivers with a safe environment for practising operating large machinery. Investing in effective training systems (Chaim et al., 2018) contributes to higher quality education, decent work, and economic development (SDGs 8 and 4).

Collaborating with customers is critical to achieving sustainable Returns. In the era of Industry 4.0, processes can be streamlined, and transaction tracking can be more efficient (Yadav et al., 2020). Using Big Data and AI will provide customers with insight regarding optimal timing and drop-off points, reducing energy consumption, emissions, and costs (SDGs 12 and 13).

Lastly, the Enabling process enhances evaluation, allows constant improvement, and synchronizes operations. As a result of Process 6, the company strives to identify, reduce, and eliminate suboptimal processes to meet its goals and initiatives (SDGs 17 and 9), as Ferreira et al. (2021) explained

### **Final Remarks**

This research delves into how OS-SC processes can be transformed by I4.0 technologies. It emphasizes the importance of understanding these technologies and their characteristics to effectively integrate them and pursue the Sustainable Development Goals (SDGs). The study draws on literature to connect Industry 4.0 technologies' capabilities to SCOR processes and evaluate their impact on the SDGs. While these technologies bring significant advantages to sustainability, the UN's agenda has interconnected goals that necessitate collaboration among stakeholders and synchronization of supply chains to drive systemic change. Advancements in technology can result in industrial growth, which can raise greenhouse gas emissions and exacerbate social inequalities. Thus, investments in technology should be accompanied by redesigning business models, employee training, and increasing stakeholder participation. To achieve the SDGs, it is fundamental to transform the OS sector and its SC and use sustainable technologies. This paper offers research perspectives on SC 4.0's



potential in meeting all SDGs. It suggests redesigning OS-SC by integrating appropriate technologies, monitoring, and involving all stakeholders. Future research should focus on empirical work to investigate how SC 4.0 capabilities can impact the SDG agenda. Additionally, research

can be expanded to examine SDGs not directly linked to technological advancements, such as SDGs 1, 2, 6, and 16. Data-driven approaches are necessary to analyse and evaluate the impact of Industry 4.0 technologies on the SDG agenda.

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## Circular economy in Stone: industrial symbioses with ceramics

M.I.A. Almeida<sup>1\*</sup>, A.O. Amado<sup>2</sup>, C.C. Surname<sup>3</sup>

(41) CTCV - Centro Tecnológico da Cerâmica e do Vidro, \*[marisa@ctcv.pt](mailto:marisa@ctcv.pt)

(42) CTCV - Centro Tecnológico da Cerâmica e do Vidro,

(43) Affiliation of the 3rd author

### Summary:

*Promoting the efficiency and sustainability of natural stone extraction and transformation processes is particularly relevant when the objective is to promote the management of natural resources, as the materials exploited are non-renewable resources (finite materials). In addition, these processes are likely to produce large amounts of waste, considered of low value, which are normally sent to landfills (and their environmental problems.) In this way, the production of large amounts of waste by the natural stone has given rise to continuous research on new technologies and applications, in order to promote new synergies and industrial symbioses that advance towards the circular economy.*

*The circular economy in stone sector is a sustainable and innovative economic model, that includes the use of this waste through its incorporation in the production of ceramic products, with a view to reducing the percentage of raw materials in the composition of ceramics and energy consumption, thus promoting synergies between different industrial sectors. This article presents case studies with industrial symbioses.*

**Key words:** natural stone, circular economy, synergies, ceramics industry,

### Introduction

The ornamental stone sector is very important to the country's economy, being a strongly exporting sector and recognized as one of the main players worldwide.

Promoting the efficiency and sustainability of natural stone extraction and transformation processes is particularly relevant when the objective is to promote the management of natural resources, as the materials exploited are non-renewable resources (finite materials). In addition, these processes are likely to produce large amounts of waste, considered of low value, which are normally sent to landfills. This practice however entails risks, namely, the increase in occupation of the territory and an environmental problem. In this sense, sustainability policies and measures have emerged, in order to contemplate the economic, social and environmental aspects in an integrated way and simultaneously align with the 17 Sustainable Development Goals (SDGs) of the United Nations, particularly Goal 12: Production and Consumption Sustainable, which refers to achieving sustainable management and efficient use of natural resources.

In this way, the production of large amounts of waste by the natural stone industry has given rise to continuous research in the search for new technologies and applications, in order to promote new synergies and industrial symbioses that advance towards the circular economy.

The circular economy is a sustainable and innovative economic model, that must be a viable alternative that organizations should look for, offering different mechanisms for creating value with respect for the environment.

To ensure a circular economy that calls for the reuse, repair, renovation and recycling of materials and products associated with it is growing. In this way, the value of products must be preserved for as long as possible and the waste generated by companies can be used in the economy as secondary raw materials. According to the portuguese Decree-Law No. 102-D/2020, of December 10, one must first opt for an attempt to prevent and reduce these substances, followed by preparation for their reuse, subsequent recycling or other types of recovery and its disposal, as the last step.

### Ornamental stone sector

The ornamental stone industry is one of the oldest sectors in the stone industry with great interest and potential, constituting the sector with the highest added value, whose product is essentially for export, the sector produces a wide range of products, especially for the construction industry, such as wall and flooring materials, as well as large blocks for various functions from statues to tombstones, among others. This sector encompasses two types of economic activities: extraction and transformation.

The extraction activity encompasses the extraction of ornamental stone masses in locations favorable to their

occurrence, mostly underground exploration. The transformation activity has as its main processes the cutting and sizing of stones to desired dimensions, followed by various polishing and finishing processes.

The stone extraction industry generates large volumes of waste (inert natural materials), and the way in which these wastes are treated directly affects the impacts generated in this industry, as well as the environmental performance of the materials produced. The waste generated in the transformation industry of ornamental stones is mainly created in the cutting and sawing phases, which generate solid particles when cutting the stone and sludge whenever water is used to facilitate the process and sedimentation of the particles occurs.

In this sense, the prevention of waste production and the promotion of its reuse and recycling in order to extend its life cycle and develop circular economy strategies should increasingly be a priority in the stone sector, considering that the waste generated in this sector is one of the largest sources of waste in the European Union (EU), which is mainly characterized by materials removed from the surface to allow exploitation, stone waste, and residues generated during stone extraction and transformation activities.

In recent time, we have been witnessing that the ornamental stone sector has been increasingly concerned about the environmental issues arising from its exploration and production. Thus, the search for alternatives for the waste associated with this industry is a topic that is increasingly being explored by companies in the sector.

The stone powder that occurs in the extraction process, namely in drilling, blasting, loading, transportation and unloading of materials, and in the process of transforming the rock, such as polishing and finishing, in the customization of the final product, is an inert residue with a fine fraction that can be used in paving of roads and as a binding material in civil construction works.

The sludge resulting from cutting operations employing water, which aids in cleaning, cooling and lubricating the process, is a mixture of water and fine stone particles, which when subjected to sedimentation settle forming a sludge (Fig.1). This sludge can be used as a raw material in the manufacturing processes of other products and construction processes such as roads paving, foundation material or even as a binder in civil construction works.



*Fig.1. Sludge resulting from sedimentation.*

In the process of transforming the stone, there are also stone remnants/trimmings due to adjustments made to the characteristics of the stone block to meet the needs of the cutting machinery and the specificities of the final product to be designed, generating waste such as defective or broken material and sawn plate edges without any use (Fig.2), thus constituting inert waste, to the extent that they have a composition equal to that of the marketable product.

These waste materials are reused whenever possible to manufacture other products of different dimensions and are also recovered and directed towards the construction industry. The trimmings that are not recoverable and reusable are often directed towards the landscaping recovery of quarries.



*Fig.2. Stone wastes and byproducts.*

### **Incorporation studies**

The production of large quantities of waste by the ornamental stone industry has led to continuous research for new technologies and applications, in order to promote new industrial synergies and symbiosis that advance towards a circular economy with the use of these materials for the same or other applications.

Thus, several studies have been developed to evaluate the feasibility of incorporating waste from the stone sector in civil construction (cements, concretes, mortars), public works, ceramic and glass products and agriculture to regulate soil acidity.

These studies aim to evaluate the quantities to be incorporated to ensure the specific properties of each product according to its usual technical performance (without waste). In this way, they try to solve the problems related to this type of waste, promoting industrial symbiosis and resource valorization, promoting eco-innovation and circular economy strategies.

In ceramic products, the sludge can be used as a substitute for feldspar, sand and some of the clay minerals needed for ceramic composition and in glass silica sand can be replaced. In concrete, sludge can function as a substitute for a percentage of cement. The stone remnants can replace cement in the manufacture of concrete and mortars.

Valuing waste from the natural stone industry involves incorporating it into production processes in different industrial sectors, being ceramics industry a potential destination for waste from the Ornamental Stones sector. The use of this waste through its incorporation in the production of ceramic products has been widely explored, with a view to reducing the percentage of raw materials in the composition of ceramics and energy consumption, thus promoting synergies between different industrial sectors and always guaranteeing the specific properties of each product according to its usual performance.

Ceramics have been shown to be a potential industry for the destination of waste from the ornamental stone sector.

The Centro Tecnológico da Cerâmica e do Vidro (CTCV) which is an organization that supports the industry and Centro de Tecnologia e Inovação (CTI), has developed several studies that have shown great promises with the substitution of natural raw materials replaced by waste from other industrial sectors (secondary raw materials).

As part of the SIAC CTCV-2021 project – Reinforcement of activity for Indústria 4.0, Circular Economy and Energy Efficiency, CTCV has developed a website with Centro de Recursos Economia Circular (<https://www.ctcv.pt/economiacircular/>), where a series of best practices and examples of the application of some circular economy strategies applicable to various sub-sectors of the ceramic industry are listed, national and international examples. In this Resource Center there is an example of waste from the extractive industry.

Some of the studies realized in CTCV were developed with waste from the extractive and processing industry of natural stone, namely sludge resulting from cutting and sawing, in which the possibility of mitigating or even preventing the environmental impacts generated by these materials at their end of life was confirmed, contributing to the development of the circular economy in the ornamental stone sector.

It was confirmed that the ceramic industry is a strong candidate for the final destination of waste from the production of ornamental stone, which depending on the composition of the waste can be incorporated into different types of ceramic paste for the production of different products.

In the studies carried out at CTCV work was done on the incorporation of granite and limestone slurry for the production of bricks, and marble sludge for the production of ceramic tiles.

The sludge used in various studies were subjected to chemical analysis and respective leaching tests, according to the methodology indicated in the current legislation and the results obtained allowed for the classification of those sludge as “inert”. Thus, we can infer that their incorporation into other materials does not pose risks to the environment.

The laboratory characterization carried out at CTCV facilities consisted of tests after drying and firing in ceramic compositions without waste and compositions with different percentages of sludge. In these tests, parameters such as green-dry and dry-fired shrinkage, mechanical strength in dry and fired conditions, water absorption after firing and evaluation of efflorescence, texture, color and detection of defects were determined.

From the results obtained after drying and firing of the various compositions tested it can be concluded that the sludge from the processing of stone has the potential to be industrially incorporated into the ceramic sector, namely in the construction sector, in bricks and flooring (ceramic tiles).

The studies carried out allowed to verify that the technological characteristics of ceramic pastes, namely drying and firing parameters do not undergo significant changes when composition have small percentages of granite sludge (2 to 15%), limestone (1%) and marble (5 to 10%), demonstrating the feasibility of incorporating this type of waste into the ceramic industry.

## Conclusions

Despite its size, Portugal has considerable diversity of ornamental stone deposits and is internationally recognized for the quality and performance of its products from this sector.

However, the production of ornamental stone presents some associated constraints, mainly the high production of waste during the extraction and transformation processes.

The valorization of these waste materials should involve their incorporation into production processes of different industrial sectors, promoting new industrial

synergies and symbioses, and minimizing the exploitation of virgin raw materials.

This circular economy strategy requires a “new” vision on the key success factors throughout the product value chain, from its conception to its end-of-life, in a life cycle approach, and using robust tools such as Life Cycle Assessment to perceive critical points and areas for performance improvement.

**Acknowledgments:** The authors can express their appreciation or acknowledge to support and help their received on their work etc.

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# PROPOSAL OF A ROAD MAP FOR THE SUSTAINABILITY OF NATURAL STONE

P.Marone<sup>1</sup>

<sup>1</sup>Paolo Marone, IS.I.M., International Marble Institute Director, [paolo.marone@stonetechtraining.it](mailto:paolo.marone@stonetechtraining.it)

## Summary:

### PART I : “NATURALMENTE SOSTENIBILE”

“MANIFESTO” OF THE SUSTAINABILITY OF NATURALLY AUTHENTIC STONE

(by PNA – PIETRA NATURALE AUTENTICA with the technical support CONFINDUSTRIA MARMOMACCHINE, IS.I.M. and ITALIA CIRCOLARE)

### PART II: “ZEROSTONE WASTE / END OF STONE WASTE”

APPLIED RESEARCH AND GOOD PRACTICES FOR WIDE USE OF NEW TECHNOLOGIES AND NEW ADDITIVES FOR THE RECOVERY OF SOLID DERIVATIVES AND PROCESSING SLUDGE OF SUSTAINABLE NATURAL STONE

(by IS.I.M., TEKSPED and others for CE Project “LIFE”)

### PART III: GLOBALSTONE-E-CULTURE AND E-LEARNING

PROPOSAL FOR THE CREATION OF A CULTURAL AND INFORMATION PLATFORM

TO SPREAD KNOWLEDGE, TECHNOLOGIES AND ARTS OF THE GLOBAL (SUSTAINABLE) STONE

(as IS.I.M. proposal of discussion during next GLOBAL STONE Congress in 2023)

Key words: PNA/Naturally authentic stone, sustainability of the stone, zero stone waste, end of stone waste, e-culture of global stone.

## PART I - MANIFESTO OF THE SUSTAINABILITY OF NATURALLY AUTHENTIC STONE

### 1. NATURALLY TOWARDS THREE DIMENSIONS OF ENVIRONMENTAL, ECONOMIC AND SOCIAL SUSTAINABILITY

Resistance, protection, functionality, safety, well-being, prestige, and beauty are the questions that man has always asked to natural stone.

Man has always been like this who built villages and cities, roads and bridges, gardens and aqueducts, public and religious buildings, monuments and works of art.

We immediately distinguish the authentic natural stone that is extracted from quarries from the non-authentic artificial stone or artificial agglomerate following an industrial process and the addition of additives, cements, resins.

All the "voluntary human geography" bears witness to a millenary tradition of planning, creativity, use, craftsmanship and processing techniques of authentic natural stone: a material that expresses the historically defined relationship between the natural and anthropic environment, between nature and culture, according to principles of continuity, harmony and economy.

Hard stones and soft stones, light stones and dark stones, stratified stones and massive stones, porous stones and compact stones, light weight stones and heavy stones, monochromatic stones and multi-coloured stones, represent a universe of natural geodiversity that has contributed to structuring and designing our landscape.

Now that the balance of this relationship, perceived as a need and a duty, is once again at the center of the most advanced reflection of the culture of designing and building, authentic natural stone recovers its leading role among the building materials available, as one of the most suitable to interpret the current and urgent demands of sustainability and environmental protection.

According to United Nations estimates, between now and 2050 more than two-thirds of the global population will live in cities. Already today, 60% of total energy consumption and 70% of emissions is linked to "urban consumption".

For this reason, the quality of buildings is one of the decisive factors in the fight against the climate crisis.

Today authentic natural stone can serve the creation of an urban ecosystem with a reduced environmental impact that is comfortable, durable, hospitable and beautiful.



We repeat and sculpt these words in our minds:  
Authentic sustainable natural stone. PNAS

At the dawn of the third millennium, sustainability is the new question to which the Italian sector of the authentically natural stone sector intends to offer answers and solutions. Among the most dynamic of Made in Italy, the Italian stone and techno-marble industry sector closed 2021 with a turnover of over 4 billion euros, a value of exports of almost 3 billion and an annual trade balance of 2.5 billion, thus returning to pre-pandemic levels, thanks above all to the driving effect of exports. With over 3,200 companies, the sector employs a total of around 34,000 people, demonstrating solid economic and employment stability even in times of crisis.

A long history behind it and know-how handed down from generation to generation allow Italian stone companies to offer the national and international market sought-after and coveted products for their high physical and aesthetic qualities.

The PNA Network - Authentic Natural Stone, which brings together the most representative companies in the national techno-marble supply chain made up of Producers and Processors of Marble, Granite and Natural Stones in general, Manufacturers of Machines, Plants, Tools and Complementary Products, Institutions Private, intends to enhance the contents of economic, social, environmental sustainability, the circular economy of Authentic Natural Stone and its positive impact on the entire supply chain from building design to the well-being of living and its leading role in the transition process ecological.

## **2. NATURALLY SUSTAINABLE TO ACHIEVE THE SUSTAINABLE DEVELOPMENT GOALS (SDGS) OF THE UN 2030 AGENDA (2015 PARIS AGREEMENT)**

Through the UN Agenda 2030, the international community has adopted a charter of commitments and concrete and shared objectives to promote sustainable development on a global level.

Energy efficiency, reduction of CO2 emissions, optimization of water resources, circular economy, protection of biodiversity, dignity of man and of work, social and territorial cohesion are the fundamental pillars on which all actions are based.

Italian companies in the natural stone sector intend to participate effectively in achieving the sustainability objectives in relation to their specific field of activity:

### SDG 12 - Responsible consumption and production

Extract only what is needed, plan the cultivation of the quarries and do it in full respect of the environment, increasingly refine the processing technologies, develop good circular economy practices to wisely use the

ornamental material, and in general all the extracted material; take care of the landscape of the extraction sites during the cultivation phase; recover mining sites once decommissioned.

These are the ambitious objectives that PNA intends to achieve in order to define the conscious and responsible production model of natural stone.

In the quarry, the machinery must only be equipped with non-polluting and silent engines, which use sustainable and renewable energy from photovoltaics, wind turbines and river turbines. All powder residues not released into the air or with the risk of polluting the environment but collected and recycled entirely.

### SGD 11 - Inclusive, safe, resilient and sustainable cities and human settlements

Natural stone by virtue of its durability, thermal performance, absence of harmful emissions, even during processing, the possibility of recycling and recovery at the end of its life, its use for draining floors also for the good management of rainwater, of its resistance to external agents and its unique beauty helps to contain the environmental impact of urban contexts and to improve the quality of life of the communities that live there.

### SDG 8 - Decent work and economic growth

A safe working environment and responsible management of critical issues represent the fundamental conditions for achieving the well-being of all employees in the stone sector, committed to ensuring the necessary safety, welfare, inclusion and contrast measures against discrimination of any kind, training and continuous updating, also on issues related to reducing the environmental impact of the entire supply chain. The extracted materials will above all have to be transformed into slabs and manufactured articles in the industrial districts bordering the quarries and create jobs and local economies, then transported and exported as semi-finished and/or finished products to outlet markets. The export of blocks will be reduced to the minimum necessary.

### SDG 13 - Climate action

With a concentration of CO2 in the atmosphere 48% higher than in the pre-industrial era, actively counteracting climate change is the most important challenge in terms of sustainable development. The use of natural stone in construction, guaranteeing the energy efficiency of the building, the hygrometric balance, the healthiness of the living and working environments and respect for biodiversity, represents a concrete action for the benefit of the mitigation of climate-changing emissions. Mitigation that is also recorded in the extraction cycle of authentic natural stone which is characterized by significantly lower CO2 emissions than those generated by other "non-natural" building materials.

The architects will have to design floors and walls considering the colors and the thermal and physical characteristics of the minerals contained in each stone and the mortars and adhesives will have to be weighted on the basis of waste recovery and the chemical-physical compatibility of the system formed by stone, adhesive and screed/wall.

### SDG 17 Partnership for Goals

Promote knowledge of the characteristics and uses of the different lithological varieties by involving and uniting the world academic world, make available an information exchange platform, with user interaction and meta-language, for the growth of the culture of sustainability of natural stone, encourage the research and innovation in the stone sector, promoting the dissemination of good circular economy practices of waste are the demanding challenges common to all companies in the sector. Addressing them in a synergistic and coordinated way through the participation of all the players in the production chain and in the world of design and construction has been the priority objective of PNA and PNAS since its establishment.

Our industry needs to be accountable and active in the process of defending our planet from its enemies. Work better, work the stone better.

### **3. NATURALLY SUSTAINABLE TOWARDS THE MEASUREMENT OF ESG CRITERIA**

The commitment of companies to sustainable development is described and measured through ESG criteria, non-financial parameters that enhance company performance from an environmental point of view (Environmental), respect for people and communities (Social) and good management and administration (Governance).

Defining corporate identity according to the principles of environmental, economic and social sustainability corresponds to a broader model of Corporate Social Responsibility (CSR) which includes the ability to associate profit with the creation of measurable social value.

Reducing waste, pollution, CO2 emissions, guaranteeing equal opportunities, health, safety and people's needs, correct remuneration, fighting corruption and structuring one's core business, introducing these principles into corporate assets, they represent as many strategic choices that describe a company that is permanently oriented towards growth.

The envisaged obligation of non-financial reporting, the new European Taxonomy which classifies eco-sustainable activities towards the ecological transition, together with the ESG ranking which help to define the economic value of organizations as well, constitute the new competitive factors of companies, which in this way they become more attractive for consumers, stakeholders and investors.

The authentic natural stone sector intends to deal with ESG factors in terms of energy and water consumption, the circular economy of by-products and waste, the well-being of workers, the positive effects for communities.

Energy consumption: the extraction and processing of authentically natural stone, using more and more energy from renewable sources and adopting highly energy-efficient Made in Italy technologies, is progressively reducing its Carbon Footprint.

Water consumption: optimizing the use of water resources and adequately treating the quantities necessary for processing the stone sector, reusing wastewater as much as possible and purifying the water released into the environment, are sustainability objectives for the entire sector. Reducing the consumption of blue water more and more, making the most of green water and containing, if not eliminating, the discharge of gray water are concrete actions that stone companies have already been implementing for years, especially if equipped with modern and efficient systems made in Italy.

The reduction of waste/derivatives: the residual materials produced by the extraction and processing of stone, their management and the reduction of their environmental and hydrogeological impact, represent a new competitive factor and sustainable development for the entire stone sector. Following the principles of the circular economy, research and technological innovation have been directed towards the enhancement of the different types of waste and by-products to make them become secondary and secondary raw materials.

The reduction of the cutting thicknesses: in addition and in line with the objectives of the previous points, the reduction of the thicknesses of the cutting tools combined with more performing cutting methods in order to favor the development of innovative technologies which reduce the cutting thicknesses, the reduction of cutting waste kg/m<sup>2</sup> and reduction of energy consumption (renewable) kWatt/m<sup>2</sup> of surface of cut stone material.

The environmental restoration of extraction sites: designing the management of the quarry according to its recovery and subsequent renaturalization or enhancement interventions allows for the recovery of abandoned sites more effectively, reducing the environmental impact of the entire extraction activity.

Safety and protection at work: adopting measures to protect workers in the sector and their physical and mental health, implementing company welfare initiatives and training and continuous updating of skills, means actively promoting well-being as an integral factor of the company organization of businesses. Technologies with remote controls and Industry 4.0 are now present in most of the Italian stone industries.

Positive impact on local communities: enhancing the quality of the material extracted and transformed in its nearby industrial districts, combined with increasingly technologically advanced processing know-how in the wake of respect for a centuries-old tradition, means being active witnesses of a truly spread throughout the territory and to grow around the stone defined as a factor of economic, social and cultural development a process of cohesion and identity capable of involving the whole community.

#### **4. NATURALLY SUSTAINABLE TOWARDS ENVIRONMENTAL REGENERATION**

The forced location of the production activity is the main characteristic of stone companies. A feature that entails the need to contain the environmental and landscape impact on the places where natural stone is grown.

Competing with sustainable development for a company in the sector therefore means not only complying with the regulations, increasingly precise and binding, which regulate mining activity, but also managing production according to an identified and shared principle of collective environmental responsibility, which puts at the center is respect for places and communities, with a view to their essential recovery and regeneration.

If the two relationships between global stone extracted and ornamental stone and between created waste and recycled waste contribute to defining the sustainability of stone production, with the aim of reducing waste and reusing it as a secondary raw material, so the recovery of sites of quarry, once the production is completed, it represents the true closing of the circle.

Furthermore, it is the duty of the stone sector, even during the quarries' cultivation phase, to make the access roads, the landscape of the quarries and the surrounding area in general pleasant and comfortable.

The main objective is to restore the balance of the ecosystem, but there is no shortage of positive experiences also for the recovery of disused quarries intended for different public uses, such as the construction of natural amphitheatres, exhibition venues or sites that can preserve and pass on the memory of local industrial archeology and help the other productive traditions of the place.

Listening to and involving citizens and local institutions in regeneration projects regarding the methods and goals to be achieved represents an indispensable action of cohesion with positive effects on local communities, with a view to corporate social responsibility that enhances its action in a structured way social.

#### **5. NATURALLY SUSTAINABLE TOWARDS INNOVATION, RESEARCH AND DEVELOPMENT. THE DEFINITION OF A SECTORAL AVERAGE LCA**

Knowing the environmental impact of a product and its

effects on human health during its entire life cycle, which goes from production with all its phases to transport, use and disposal, today represents a decisive factor for make conscious and responsible choices in a logic of truly sustainable development.

The Life Cycle Assessment (LCA) is the study that examines and measures the interactions of a product (good or service) with the environment, from the analysis of the raw materials to the final disposal, thus restoring its overall environmental footprint and also allowing comparative analyzes with other similar products, based on shared and certified standards and parameters.

To promote ever more in-depth knowledge of the environmental footprint of natural stones, as an indispensable fact in the most qualified and sustainable design and construction choices, PNA has commissioned the Polytechnic of Turin to prepare an LCA analysis of the sector average of the product "slabs of authentic natural stone" and, in anticipation, the consequent EPD (Environmental Product Declaration), in accordance with the guidelines relating to stone products.

The definition of the boundary of the LCA analysis system involves the identification of the phases and processes to be included in the analysis; in its traditional conception, the entire life cycle is considered, starting from the acquisition of raw materials up to the management at the end of the useful life including the phases of manufacture, distribution and use, up to the end of life (whether demolition and/or disposal and/or recycling according to a cradle to grave approach).

In the specific case, given the peculiarities of the life cycle of the PNA, we will proceed with the analysis "from cradle to gate" which ends with the finished product deposited on the yard, ready for the destination market.

The provision of the LCA analysis allows authentic natural stone companies to increase all their competitive factors in the name of certified sustainability.

As an intrinsically circular and fully recoverable material (both for waste from the upstream production process and for downstream demolition materials), natural stone can also be analyzed by arriving at a "cradle to cradle" LCA), capable of restoring its nature as a product with "never end of life", because it is 100% and almost infinitely reusable.

#### **6. NATURALLY SUSTAINABLE TOWARDS THE CIRCULAR ECONOMY OF WASTE**

Trying to minimize material waste during cultivation and processing is the first form of respect for natural stone. For the entire stone sector, ZERO STONE WASTE or END OF STONE WASTE is a concrete goal that can be achieved in a short time.

The scraps that the sector produces every year in the form of stone elements of various sizes - such as shapeless or

defective blocks in the quarries, crusts of blocks after sawing, pottery, fragments and dusts deriving from processing sludge - are only partially used in processes recycled as a secondary raw material (MPS), as well as having very high economic and environmental costs.

The challenge of the circular economy in the stone sector is to enhance all waste and promote an ever wider use of it, working in symbiosis with other industrial realities, to the point of contributing to a short circular supply chain of natural stones with positive employment effects on the territories of production, cultivation and extraction.

The marketing of by-products, waste products and derived natural stone products, in addition to reducing economic and environmental costs, frees up additional resources to invest again in research and development, according to a logic of virtuous circularity.

The green transition of the stone sector involves the need to implement processes with a high technological innovation content, thanks to research projects that see private companies and universities as protagonists, but above all it can and must be based on the impetus and synergistic strategy of associations, consortia and institutes capable of gathering the excellence of the Italian stone industry and ferrying it towards ever greater sustainability.

The ZERO STONE WASTE project, coordinated by the International Marble Institute (I.S.I.M.), following the European directive 2008/98/CE of the End of Waste and adapting to the recent European and Italian regulatory changes, is aimed at the recovery and use of derivatives and waste from the processing of ornamental stones through the development of suitable process methodologies and specific production technologies that can be acquired by companies in the sector. The ZERO STONE WASTE (ZSW) promotes the almost zero reduction of the costs of conferring waste to the point of proposing new primary secondary materials (MPS) for the building industry, all declarable as CAM due to the high content of recycled and recovered stone by-products.

Each company, based on the types of waste it produces, can find the ad hoc answer for its circular production in the technologies developed by the ZSW project.

The ZERO STONE WASTE project was recently supported by the European Commission (EC) within the LIFE Program and involves a group of Italian companies producing technologies, chemical additives, stone materials which are coordinated by the International Marble Institute (I.S.I.M.) and by the responsible office of the EC.

## **7. NATURALLY SUSTAINABLE TOWARDS GREEN STANDARDS OF DESIGN AND CONSTRUCTION**

In Europe, buildings and the construction sector are responsible for 36% of annual CO<sub>2</sub> emissions, 40% of energy consumption, 50% of raw material extractions and

21% of drinking water.

In sustainable building and green building, which are increasingly establishing themselves, natural stone plays a leading role, as a material that allows the creation of a real urban ecosystem of high environmental quality, in which they can interact in a functional way and positive natural matrices, semi-natural matrices and human intervention.

The main guarantees that natural stone offers are:

**Respect for biodiversity:** paving walkways in gardens and open spaces with natural stones can protect and create specific areas of biodiversity. Using stone for urban roofing guarantees a high quality of the urban environment.

**Control of the quantity of rainwater:** authentically natural stones can give rise to partially permeable paving which allows to minimize or mitigate the surface runoff of rainwater.

**Reduction of heat islands:** the various types of natural stone expertly used in external floors, building roofs and car parks are able to minimize the impact on the surrounding microclimate.

**Maximization of energy savings in building management:** the use of authentically natural stones, due to the characteristics of the different minerals they contain, can help reduce energy consumption for cooling and heating buildings, with the consequent reduction of CO<sub>2</sub> emissions.

**Extension of the life cycle of the building heritage:** authentically natural stones are "eternal", if used appropriately with respect to the weather conditions and their functional characteristics and subjected to simple maintenance thanks to modern surface treatment and restoration techniques. In fact, their use makes it possible to preserve resources, preserve cultural heritage and reduce waste.

**Possibility of recovery/recycling at the end of its life:** the scraps of authentically natural stones deriving from demolition and construction, originating secondary raw materials of excellent quality, can be reused both as ornamental coatings, after a simple surface treatment, and for the production of sands and gravels.

For this reason, an authentically natural stone slab, once certified with EPD (Environmental Product Declaration), can contribute to the sustainability rating of buildings based on the main and most advanced international programs: LEED, BREEAM, ENERGY STAR, GREEN STAR, HQE, ITHACA.

The use of natural stones, allowing to reduce the quantity of building materials for construction and demolition, to save raw materials and energy and to contain CO<sub>2</sub> emissions, can also meet the sustainability requirements required of building materials according to the

Environmental Criteria Minimums (CAM) of the building of the Public Administration.

## **8. NATURALLY SUSTAINABLE TOWARDS WELL-BEING, HEALTH AND SAFETY OF LIVING AND WORK ENVIRONMENTS**

The increasingly widespread environmental sensitivity, when it meets the most advanced culture of design and construction, is oriented towards the choice of natural materials, as a guarantee of living comfort in the name of respect for the environment and saving energy and resources.

Indeed, the intrinsic qualities of authentically natural stone contain all those properties which, depending on the lithological varieties and the required functions, promote the safety, well-being and beauty of the spaces in which they are used.

The thermal properties of the stone material make it possible to save energy for cooling and heating the rooms, while contributing to the hygrometric balance of the rooms; the almost total absence of harmful emissions is a guarantee of health; the sound-absorbing capacities help to counteract the increasingly widespread noise pollution in cities; the durability and resistance to wear and tear over time make natural stone an eternal material that is increasingly easy to maintain, thanks also to modern processing and polishing techniques.

Without forgetting that these characteristics of authentically natural stones are combined with the principles of the circular economy, for the lengthening of the life of the buildings that use it, for the urban regeneration and the contrast to the degradation of the common spaces, and for the recovery, also philological, of the valuable architectural assets that use it.

Choosing marble, granite, travertine, sandstone and quartzite in the light of knowledge of their material and functional characteristics means building a stable, welcoming and hospitable urban landscape, being the repository of the social, historical and cultural values of the community that inhabits it and delivering to future generations the memory of today's architecture, in its more classic or more challenging and innovative design solutions.

## **9. NATURALLY SUSTAINABLE FOR THE DEVELOPMENT OF MADE IN ITALY (and further countries)**

In our country, famous all over the world for the harmony of the natural and urban landscape and for the elegant and refined lifestyle, natural stone has been the first "material" where functional and aesthetic principles meet.

Natural stone can also combine the qualities of resistance, uniqueness, aesthetic performance and non-perishability with the potential linked to technological innovations that can be put at the service of planning, architectural

and design ideas and choices based on quality, well-being and beauty.

This is the added value that authentically natural stones bring to the world: their unique identity, their wonderful materiality and at the same time Italian know-how and know-how, interpreted in the light of the most up-to-date processing techniques and craftsmanship and industrial ability of Italian companies.

A know-how that since the Renaissance workshops has developed during construction, "working" and developing an Italian method of enhancing the stone that the whole world recognizes us.

From precious marbles for interior furnishings to stones more suitable for outdoor use and for the redevelopment and decoration of urban environments, the story of Italian quality, and its regional and local specificities, passes through the export of authentically natural stones.

With an international market aimed at more than 140 countries and which sees the United States, Germany, Switzerland, the United Kingdom, France, but also the United Arab Emirates and China among its main customers, the stone sector is the ideal testimonial of the synthesis of Made in Italy, which concerns precious materials known all over the world and our way of working them, the result of an ancient tradition and the most advanced research and applied technological innovation, as demonstrated by the sector's involvement in the creation of Technical and Research Centers, as part of international cooperation agreements.

## **10. NATURALLY SUSTAINABLE TOWARDS CORPORATE RESPONSIBILITY IN A NEW ECONOMY OF RELATIONSHIPS**

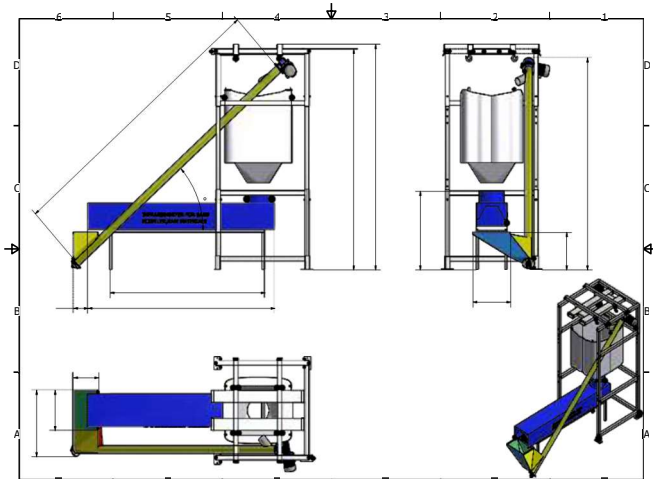
The promotion of the culture of use and processing of natural stones in the construction and design sector is the mission of the Authentic Natural Stone Business Network, as a project that brings together the major companies in the sector.

In addition to the already present organization in production districts, on a local and regional basis, the aggregation of companies in the stone sector in a national network thus becomes a decisive competitive factor in proposing a governance model for the entire sector, dealing with the most urgent needs of sustainable development and the achievement of the objectives defined by the ecological transition.

In fact, PNA contributes to promoting and spreading the culture of authentically natural stone among architects, designers and technicians in the sector, so that the stone material can increasingly be included in the solutions offered to clients, and together it builds a table of proposals, research and definition of the sustainable development objectives of the sector, in a logic of continuous improvement for the reduction of the

environmental impact and an ever greater cohesion with the territories and communities.

PNA, responding to the objectives of SDG 17 which identifies in the partnership by objectives the engine for the competitive and truly sustainable development of the sector, intends to make its contribution to the contents of economic, social, environmental sustainability, to the adoption of ESG criteria, to research and development,



technological research, the enhancement of the unique and unrepeatable characteristics of natural stone, the reduction of the environmental impact of production and processing, the promotion and dissemination of the circular culture of stone waste as a systemic and guiding vision capable of identifying them as economic resource and form of material and energy saving. Naturally together with stone.

## PART II - ZERO STONE WASTE PROJECT and SOLUTION / END OF STONE WASTE

### 1 – COMPLETE CHAIN SUPPLY

The ZSW solution, consists of a series of equipment and technologies: GEF; GEA; GEM\_DRY; GEM\_WET; GEC, each of them performing a specific phase on the stone waste treatment process. This solution, named ZSW, has been designed starting from the former prototypes developed in the past and is ready to be developed in real conditions and built at industrial scale. Depending on the different waste streams and of the value-added materials to produce, the ZSW is built, by the partners ISIM, TEKSPED and PENTACHEM, in different configurations, including one or more of the following equipment interconnected:

- GEF: Geo eco-filler line;
- GEA: Stone-smashing crusher for aggregate line;
- GEM: Global Eco-mixer line (types: dry and wet);
- GEC: Global Eco-cellular concrete mixer & pump.

GEF equipment is the first component of the production

line. It allows to produce the eco-filler starting from slurries.

GEA consists of an adapted crusher for grinding stone scraps and big-size blocks to crush them producing powder which will feed the GEF.

GEM add to the filler different products (cement, additives) to prepare several types of high value pre-mixed powders (dry) or pre-mixed mousse (wet) with different characteristics, depending on the aggregates added. Powders can be stored in bags or feed the GEC to produce directly on-site different type of products, such as lightweight concrete or concrete based on polymers. "Mousses" can be stored in

silo or plastic bags or feed the GEC. GEC can produce directly on-site different type of products, such as lightweight concrete or concrete based. The ZSW is a small dimension plant which could be designed with two different configurations:

- Fixed, to be installed in a stable site and appropriate when wastes are produced continuously;
- Mobile, which can be transported on a 40" container for temporary works or for reaching several sites.

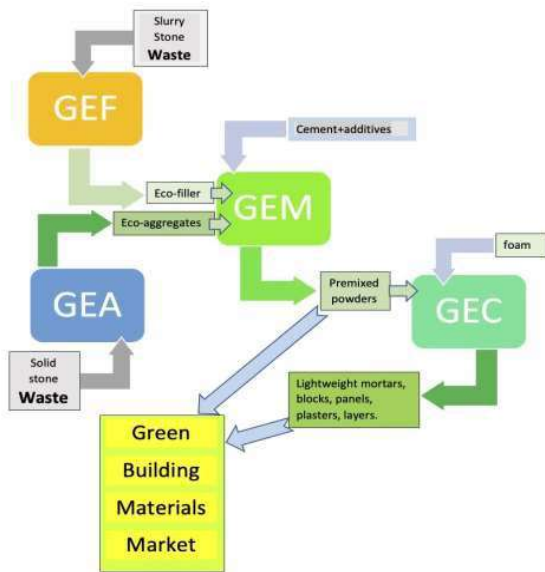
TEKSPED is the responsible of the manufacturing of the plants which will be used for each plant.

A technical description of the components of the ZSW solution presented below.

#### GEF: sludge/powder recycling into eco-filler

This equipment, which is the core of the technology, extracts fine particles from sludge and powders through a sieving below 500 micron and reduces the humidity below 0.5%. In addition, the iron content is removed by a magnetic roller. Sludge wastes can be treated after a period of 5 to 7 days for natural evaporation of water. By means of a magnetic iron-separator the iron is separated upgrading the quality of the eco-filler.

GEF equipment could work with a wide range of waste flow, ranging from 1 to 5 tons per hour of sludge depending on humidity and mesh. According to the plans, the GEFs which will be build and used for the project will treat an average of 4 tons/h of sludge and will be able to produce around 3 tons/h of high-quality eco-filler.



additives will be mixed with eco- filler (between 30 and 75% of the mixture) to create valuable products ready to be used, such as mortars, adhesives, stucco or other construction pre-mixed products.

The GEM equipment can produce between 3 and 5 tons/hour of pre- mixed powders or mousse depending on the initial conditions of the eco- filler. For the project, the goal is to produce 4 tons/h of pre-mixed powders with high characteristics from an input with at least 60% of eco-filler and eco-aggregates.

### GEF line

It follows a scheme of a Mini-GEF, the smallest version of the equipment, which is used for laboratory trials:

The new version of GEF which is developed for the project includes an IoT device capable to automatically check the quality of the powder, to record their characteristics to a centralized control unit, and to command some actuators from remote.

### GEA: production of eco stone aggregates

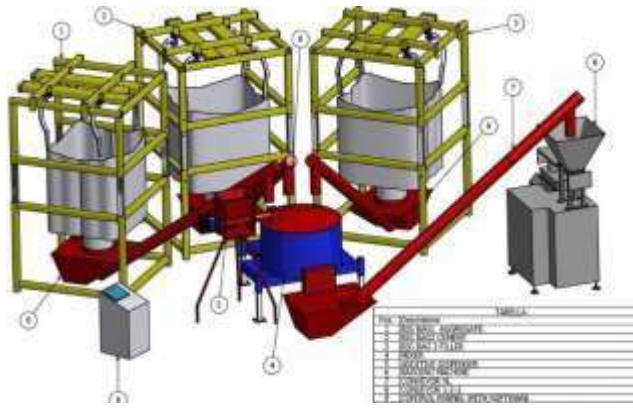
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GEA is an optional crushing equipment that can grindstone blocks and scraps in order to produce powders (particle size below 0.5mm) which could feed the GEF (for producing eco-filler) or GEM (as an inert component of the pre-mixed). That equipment will be purchased on the market and adapted to be integrated into the production line as a component of the plant.



### GEM: production of pre-mixed powders

The eco-filler produced by GEF is carried to a downstream mixer line (GEM), in which cement, aggregates and



### GEC: Concrete production and pumping

GEC is a special equipment for transforming the pre-mixed powders into lightweight cellular concrete (with a density between 400 and 1000 kg/m<sup>3</sup>), which can be used in-situ to produce lightweight artefacts or pumped into wall for covering and insulation. GEC could be used in DRY way: (small or big) reusable bags of pre-mixed powder (cement + eco sands / aggregates + eco filler + additives) are mixed with foam to produce standard or light concretes/mortars/precast.

TEKSPED (with the scientific supervision of ISIM and PENTACHEM) can currently modifying the equipment for using directly WET sludges for preparing mousse or ready concrete, without treating sludges with GEF. It can allow to uses the

filler and water of the slurry at practically no cost, only by adding a 0.5% of a special fluidifying agent and new generation additives. The innovation lies in the direct use of the mud from marble/stone processing. This new process is innovative application of the first ZSW LIFE project.

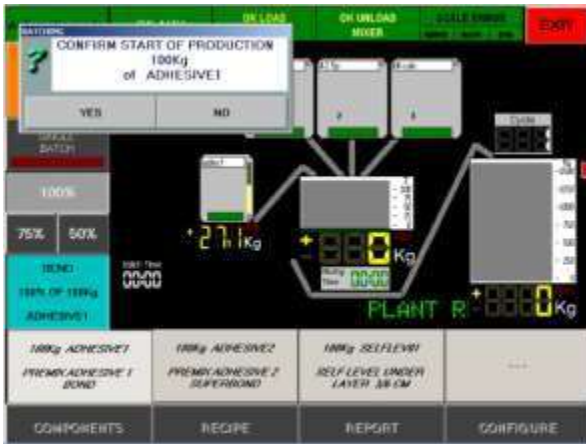
The standard production of the GEC equipment ranges from 2.5 to 6 m<sup>3</sup>/h, the equipment developed for the project is planned to have an output of lightweight cellular concrete of 4 m<sup>3</sup>/h, which could be used to produce lightweight artefact or artificial engineered stones.

The working conditions of ZSW production line and the quality of products will be managed by a specifically developed software application: the Plant Management System (PMS).



Example of user interface.





Example of the control interface of the plant.

The plant operator will interact and interface with the plant through the system console locally installed and, during the project, a new IoT Remote Control System (RE-PMS) will be developed and integrated as optional part of the solution. This system will allow a supervisor to monitor one or more plants from a distant site, as well as receive alarms or warnings, apply emergency procedures, modify some parameters, etc.

The innovation introduced by the ZSW plants could be summarized as follows:

- The GEF equipment is innovative as there is no other manufacturers, at this moment, offering a similar smart device, so flexible, friendly and easy to install in any stone industry. GEF offers features which could be at present obtained only by using more than one device which a stronger environmental impact, too;
- The GEM equipment is innovative as for its reduced dimension, and for the sophisticated electronic controls, it offers the possibility to create pre-mixed materials with a small investment, affordable also by small industries;
- The innovation carried by GEC is the capability of producing different types of light and standard concretes/mortars by using CAM recycled pre-mixed only. That feature is the result of years of trials conducted by ISIM and TEKSPED and we have no evidence of competitor able to use "second" materials for producing the high-quality products as the one produced by our GEC.

Moreover, a new GEC model could produce light concretes/mortars by directly using wet sludges, avoiding the employ of the GEF for producing the dried eco-filler. That option could be used mainly

when the user is close to the stone wastes. For the "standard" market it will need to produce dry, bagged pre-mixed mortars/adhesives, using dry eco-fillers as described above.

We could also emphasize that the most significant innovation of the offered ZSW solution is that in integrate a series of very efficient and effective equipment in one small PRODUCTION LINE. The solution could be offered at affordable prices in different configuration (fixed or mobile, including one or more equipment) for answering to the need of recycling the waste at Km0, as innovative alternative to a "centralized" treatment facility which will require heavy and expensive transportation of the stone wastes.

The evidence of recycling some stone waste is very limited and Marmomacchine states that its associated (the Italian stone quarries and industries) are searching easy and affordable solutions for setting up circular economy practices.

Moreover, an additional technical innovation comes from the availability of the Remote-Control System, an IoT device which transforms ZSW into an industry 4.0 compliant plant.

Wet solution of GEM for silicious/granite waste will be also very healthy for the workers.

## 2 - SCALE AND OUTPUT OF THE PROJECT

Different ZSW plants will be developed, installed and evaluated during the project LIFE, both including all equipment (GEA, GEF, GEM and GEC). Both plants are designed for producing an average of 4 ton/h of recycled "CAM/ECO" materials (light concretes or light artefacts for construction) for a total of no. 2 x 4 ton x 10 hours = 80 tons per hours= 80.000 kg per day (approx. 20% of local cement and only 2% to 4% of Italian additives).

A fixed-configuration plant could be installed in each processing plant stone-processing to produce pre-mixed powders and for testing the possibility of produce some type of concrete artefacts.

One plant investment as Italian technology is approx. €. 150.000,00. Probably some part of the plant could be built in local countries.

### **PART III - GLOBAL STONE-E-CULTURE AND E-LEARNING**

The idea of IS.I.M., International Marble Institute, after Covid pandemic long stop, is to

work for a proposal for the creation of a cultural and information platform to spread knowledge, technologies and arts of the GLOBAL (SUSTAINABLE) STONE permanent community, where to concentrate the publication, the reports, the tests, the manual, the books of our sector.

Through the realization of this platform and the actions described in Part I and Part II will be really possible to start to create, to improve and to develop the culture of the SUSTAINABILITY OF NATURALLY AUTHENTIC STONE.

Acknowledgments: The author can express his special appreciation and acknowledge for the support and the help received on this presentation to FLAVIO MARABELLI, PAOLO MARCESINI, CLAUDIA CECCARELLI, MICHELE ALLOCCA, PASQUALE BUONANNO, ANDREA NARRA, NURIA CASTRO, CESARE FERRARI and for the immense patient of LUIS LOPES. The author had the possibility and opportunity to WORK for PNA project (PIETRA NATURALE AUTENTICA) and for its MANIFESTO in 2022 and thanks to this work was possible to prepare this presentation for the next GLOBAL STONE CONGRESS. Another special thanks to European Commission (CE) and to FLORIANO TOSATO for the project LIFE, unique opportunity to develop the chain of ZERO STONE WASTE.

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[www.naturalstoneisbetter.com](http://www.naturalstoneisbetter.com)

## Natural Stones from Portugal – Proposal for harmonizing Commercial Names

J. Carvalho<sup>1\*</sup>, C. Carvalho<sup>1</sup>, L. Lopes<sup>2</sup>, A. Silva<sup>3</sup>, C. Santos,<sup>2</sup> L. Sousa<sup>5</sup>, R. Martins<sup>2</sup>, C. Marques<sup>4</sup>

(44) LNEG – National Laboratory for Energy and Geology, \*[jorge.carvalho@lneg.pt](mailto:jorge.carvalho@lneg.pt)

(45) UÉVORA – University of Évora and ICT – Earth Sciences Institute

(46) CTCV – Technological Centre for Ceramics and Glass,

(47) ASSIMAGRA – Portuguese Association of the Mineral Resources Industry,

(48) UTAD – University of Trás-os-Montes and Alto Douro and CGEO - Geosciences Center, University of Coimbra

**Summary:** *Over the years, the Portuguese Geological Survey – LNEG, has been dedicated to the characterization of Portuguese natural stones. Today, a newly updated LNEG web portal contains information on 149 natural stones, which are known by their unique trade names. Many of these names integrate the EN 12440 - Natural Stone - Denomination Criteria standard, but others do not. Furthermore, there many other names used to identify natural stones that are traded today and that do not integrate the LNEG web Portal or the EN 12440. This work presents a harmonization proposal for the commercial denomination of the Portuguese natural stones. It is a working basis for a final list that must be submitted to ISO/Technical Committee 118 – Natural Stones.*

**Key words:** *natural stones, commercial names, denomination criteria, harmonization, European standard*

### Introduction

In the early 80's of the past century, LNEG – National Laboratory for Energy and Geology (Portuguese Geological Survey), through its predecessor public institutions, began to characterize and catalogue the natural stones quarried in Portugal. This initial work resulted in the first Portuguese Ornamental Stones Catalogue, published in 4 volumes between 1983 and 1994, and 8 supplementary technical sheets edited between 1995 and 2000. At the beginning of the 21<sup>st</sup> century, the 1<sup>st</sup> computerized version of the Catalogue began to be developed and a Portuguese Ornamental Stones Portal was included in LNEG's website. It was updated with a new version in 2021 (<https://geoportal.lneg.pt/en/databases/rop/>), encompassing all the information contained in the catalogue, in addition to new functionalities of which the “search by features” stands out. Over the years, some new stones have been added to the Portal, which currently contains information regarding 149 commercial names.

Meanwhile, based on data from both LNEG's Ornamental Stones Catalogue and Portal, other printed and online catalogues have emerged with the aim of disseminating and promoting the Portuguese natural stones (e.g., <http://www.primeirapedra.com/stones/> and <https://stonebyportugal.com>), most of them endorsed by ASSIMAGRA, one of the Portuguese industrial associations dedicated to mineral resources.

The criteria determining the inclusion of stones with varied commercial names were not always the same, both in the Catalogue and in the web portals. It depended mainly on the commercial interests of the extractive companies, since they provided the samples for testing and also named their own stones. New commercial names were often given to stones with a name already well established in the market. Furthermore, natural variability contributed to this disorganization. Legal questions are also a key-point, since some commercial names are registered trademarks.

Today there is awareness that a unique type of stone can be traded under different commercial names, despite what the rational should be: assigning a unique commercial name to each type of stone. Indeed, in the world of construction materials, well-known traditional commercial names are often considered as more relevant quality certificates than stone scientific names or technological tests.

In this context of building materials, many of the names of the Portuguese natural stones are part of the EN 12440 – Natural Stone - Denomination Criteria. This European standard provides a list of names under which most EU stones are known and should be used when trading them. However, the Portuguese list in EN 12440 is strongly outdated, no longer matching what is currently published in the LNEG's Natural Stones Portal,

nor with the natural stones that are being quarried and traded nowadays.

This paper aims to disclose the work carried out to harmonize the nomenclature of Portuguese natural stones. The list of commercial names presented here is a working basis for a final list to be submitted to ISO/Technical Committee 118 – Natural Stones, to be analysed, voted and hopefully accepted to be included in the next revision of EN 12440. Some names may be included or excluded from the list, depending on the methodological criteria that are still being worked on.

## Methodology

A small working group took the task of harmonizing the names of the Portuguese natural stones. It integrates staff from the entities that have been most dedicated to this issue over recent years, namely LNEG – National Laboratory for Energy and Geology, ASSIMAGRA - Portuguese Association of the Mineral Resources Industry, CTCV - Technological Centre for Ceramics and Glass, and the universities of Évora and Trás-os-Montes and Alto Douro. The adopted methodology consisted of consecutive decision-making actions.

The first decision was the adoption of the denomination criteria set out in EN 12440, *i.e.*, that the commercial name must be accompanied by the stones' petrological family/group (according to EN 12407 and EN 12670), typical colour and its place of origin. There are no petrographic studies allowing the correct determination of the petrological family/group of all stones currently marketed, which should be a factor for their exclusion from a list of stones to be included in EN12440. However, the reality is that they are being commercialized, which is why, at least temporarily, they continue to be part of the list that is now presented. The colour of each stone was chosen from the list of 20 colours presented in Table I. The list tries to avoid the use of fancy colour names, and was recently defined within the framework of the Eurolithos project (Carvalho & Heldal, 2020).

Table I – List of colour names for natural stones

Natural Stone Colours	
Beige	Grey-green
Black	Light grey
Black-green	Light pink
Blue	Multi-coloured
Blue-grey	Pink
Brown	Red
Dark grey	Reddish brown
Green	White
Grey	White-black
Grey-brown	Yellow

A second decision was to exclude the stones considered as heritage stones, *i.e.*, those that are no longer being exploited, nor are they expected to be in the short/medium term for several reasons (lack of mineral reserves, land-use planning or environmental issues, etc.).

Thirdly, it was agreed to include in the new list all the stones for which production is expected to continue on a regular basis over the next few years, despite not yet being included in the Portuguese Ornamental Stones Portal.

Finally, considering the three main types of natural stones in Portugal – granites, limestones and marbles – some specific decisions were taken. It is usual that each granite, limestone or marble quarrying site produces more than one type of stone, which is due to chromatic and/or textural variations in the mineral deposit. These unique aesthetic features may result from surface weathering processes in granites, or different sedimentary layers in limestones, or particular metamorphic processes in the marbles case, amongst others.

The chromatic and/or textural variations may result from significant mineralogical and microstructural differences, which will necessarily have repercussions on their physical-mechanical characteristics. On the other hand, the stones can differ chromatically and/or texturally without showing significant differences in their physical-mechanical characteristics. In all these cases, the decision was to consider them different natural stones with specific commercial names.

Another common case is that of adjacent quarries exploiting the same stone type, which is traded with different commercial names due to individual marketing options. For these cases it was decided to adopt the name already established in the market, or the most consensual one; the remaining being taken alternatively, up to a maximum of two.

## Results

The result of the work carried out with all the commercial names known by the work team for Portuguese natural stones is the list of names presented in Table II. It is organized according to the main groups of natural stones and comprises a total of 160 different natural stone varieties, where 92 correspond to commercial granites, 27 to limestones, 29 to marbles, 11 to slates and one migmatite belonging to the miscellaneous group of natural stones. The total surpasses the current number of stones in the LNEG's Ornamental Stones Portal, because the inclusion of each new type of stone in this Portal

depends, above all, on the willingness of stone companies to provide the specimens for the necessary analysis and tests.

## Conclusions

Natural Stone Declarations of Performance contain the natural stones' commercial name, place of origin and relevant characteristics for the products to which they relate. Updating the current trade names of Portuguese natural stones has strong commercial implications

because it can contribute to mitigate the harmful effects of some existing disorder. At the same time, we hope that an updated list of Portuguese natural stones in EN 12440 standard may push companies to better comply with it.

Table II – Projected list of Portuguese natural stone commercial names for the next revision of the EN 12440.

Commercial Name	Alternative 1	Alternative 2	Colour	Group	Lithology	Municipality
AMARELO ALPENDORADA			yellow	Granite	Granite	Marco de Canaveses
AMARELO ARIZ			yellow	Granite	Granite	Moimenta da Beira
AMARELO AZÊVO			yellow	Granite	Granite	Pinhel
AMARELO BOTICAS			yellow	Granite	Granite	Boticas
AMARELO CARDIELOS	Amarelo Samonde		yellow	Granite	Granite	Nogueira
AMARELO CARRAZEDA	Amarelo Samorinha		Yellow	Granite	Granite	Carrazeda de Ansiães
AMARELO CASTRO DAIRE			yellow	Granite	Granite	Castro Daire
AMARELO CAVEZ	Amarelo Vitória	Amarelo Moimenta	yellow	Granite	Granite	Cabeceiras de Basto
AMARELO FIGUEIRA			yellow	Granite	Granite	Figueira de Castelo Rodrigo
AMARELO FONTE ARCADIA			yellow	Granite	Granite	Sernancelhe
AMARELO GALHO			yellow	Granite	Granite	Vieira do Minho
AMARELO GONDOMAR	Ouro Velho		yellow	Granite	Granite	Guimarães
AMARELO JARMELO SÃO PEDRO	Amarelo Pínzio		yellow	Granite	Granite	Guarda
AMARELO LAPA			yellow	Granite	Granite	Aguiar da Beira
AMARELO LUZELOS	Amarelo Pardo		yellow	Granite	Granite	Carrazeda de Ansiães
AMARELO MACIEIRA	Amarelo Beirão		yellow	Granite	Granite	Sernancelhe
AMARELO MANGUALDE	Amarelo Corvaceira		yellow	Granite	Granite	Mangualde
AMARELO MONDIM			yellow	Granite	Granite	Mondim de Basto
AMARELO PENAFIEL			yellow	Granite	Granite	Penafiel
AMARELO PONTE DE LIMA	Amarelo Limiano	Amarelo Estorãos	yellow	Granite	Granite	Ponte de Lima
AMARELO SÃO DOMINGOS	Amarelo Penas Covas		yellow	Granite	Granite	Montalegre
AMARELO SÃO MARTINHO			yellow	Granite	Granite	Monção
AMARELO SOAJO			yellow	Granite	Granite	Arcos de Valdevez
AMARELO SOUSELO			yellow	Granite	Granite	Cinfães
AMARELO TALHADAS			yellow	Granite	Granite	Sever do Vouga
AMARELO TRIGO	Amarelo Penedono	Amarelo Estrela	yellow	Granite	Granite	Penedono
AMARELO VALPAÇOS			yellow	Granite	Granite	Valpaços
AMARELO VILA REAL	Amarelo Real	Amarelo Torre	yellow	Granite	Granite	Sabrosa, Vila Real, Vila Pouca de Aguiar
AMARELO VIMIEIRO			yellow	Granite	Granite	Arraiolos
AZUL AIRÃO			blue-grey	Granite	Granite	Guimarães
AZUL BOTICAS			blue-grey	Granite	Granite	Boticas
AZUL CASTRO DAIRE	Cinzento Castro Daire		blue-grey	Granite	Granite	Castro Daire

Commercial Name	Alternative 1	Alternative 2	Colour	Group	Lithology	Municipality
AZUL GALHO			blue-grey	Granite	Granite	Vieira do Minho
AZUL GONDOMAR	Cinza Gondomar	Cinza Guimarães	blue-grey	Granite	Granite	Guimarães
AZUL LEIJAL			blue-grey	Granite	Granite	Montalegre
AZUL LIMAGA			blue-grey	Granite	Granite	Trancoso
AZUL PONTE DE LIMA	Cinza Limiano	Cinzento Ponte de Lima	blue-grey	Granite	Granite	Ponte de Lima
AZUL TRAGAL	Cinzento Tragal	Cinza Tragal	blue-grey	Granite	Granite	Mogadouro
AZUL TRANSMONTANO	Azul Cristal Transmontano	Azul Águas Frias	blue-grey	Granite	Granite	Chaves
AZUL VALPAÇOS			blue-grey	Granite	Granite	Valpaços
BRANCO AGUIAR	Branco Micaela		light-grey	Granite	Granite	Aguiar da Beira
BRANCO ALMEIDA			light-grey	Granite	Granite	Almeida
BRANCO CANDOSO	Branco Vila Flor	Cinzento Carrazeda	light grey	Granite	Granite	Vila Flor
BRANCO CARAVELA			light grey	Granite	Granite	Castelo de Vide
BRANCO CORAL	Coral White	Branco Miguel	light grey	Granite	Granite	Viseu
BRANCO LAPA			light grey	Granite	Granite	Nelas
BRANCO MACIEIRA	Branco Diamante		light grey	Granite	Granite	Sernacelhe
BRANCO PÉROLA	Branco Atlântico		light grey	Granite	Granite	Monção
BRANCO VILA REAL	Branco Real		light grey	Granite	Granite	Sabrosa, Vila Real, Vila Pouca de Aguiar
BRANCO VIMIEIRO	Silver White	Kristal White	light grey	Granite	Granite	Arraiolos
CASAS DA SERRA			yellow	Granite	Granite	Boticas
CINZA CLARO SÃO DOMINGOS			grey	Granite	Granite	Montalegre
CINZA ESCURO BRAGA	Azul Palmeira	Cinza Lagido	dark grey	Granite	Granite	Braga
CINZA FINO			grey-brown	Granite	Granite	Pinhel
CINZA LAPA			grey	Granite	Granite	Aguiar da Beira
CINZA PÓVOA			grey	Granite	Granite	Póvoa de Lanhoso
CINZA RIO DE MEL			grey	Granite	Granite	Trancoso
CINZALA	Cinzento Sta. Eulália		grey	Granite	Granite	Elvas
CINZENTO ALPENDORADA	Azul Alpendorada	Cinzento Claro	grey	Granite	Granite	Marco de Canaveses
CINZENTO ANTAS	Antas Grise	Grigio Aveiro	grey	Granite	Granite	Penalva do Castelo
CINZENTO ARIZ	Cinza Ariz	Zira Grey	grey	Granite	Granite	Moimenta da Beira
CINZENTO ARRONCHES	Cinza Arronches		grey	Granite	Granite	Arronches
CINZENTO CARAMULO			grey	Granite	Granite	Tondela
CINZENTO CARRAZEDA			grey	Granite	Granite	Carrazeda de Ansiães
CINZENTO CINFÃES	Cinzento Montemuro		grey	Granite	Granite	Cinfães
CINZENTO ESCURO RORIZ			dark grey	Granite	Granodiorite	Santo Tirso, Paços de Ferreira
CINZENTO ÉVORA	Branco-Preto Barrocal	Cinza Évora	grey	Granite	Granodiorite	Évora
CINZENTO FORNOS			grey	Granite	Granite	Fornos de Algodres
CINZENTO JARMELO SÃO PEDRO	Cinzento Arrifana		grey	Granite	Granite	Guarda
CINZENTO MANGUALDE	Cinzento Corvaceira		grey	Granite	Granite	Mangualde
CINZENTO PENAFIEL	Cinza Penafiel	Azul Penafiel	grey	Granite	Granite	Penafiel
CINZENTO PENALVA	Cinzento Esmolfe		grey	Granite	Granite	Penalva do Castelo
CINZENTO PÉROLA			grey	Granite	Granite	Penedono
CINZENTO PINHEL	Branco Ricão		grey	Granite	Granite	Pinhel

Commercial Name	Alternative 1	Alternative 2	Colour	Group	Lithology	Municipality
CINZENTO SÁTÃO	Silva Fine Grey		grey	Granite	Granite	Satão
CINZENTO TELÕES	Cinza Telões		grey	Granite	Granite	Vila Pouca de Aguiar
FAVACO	Cinzento Favaco	Preto Favaco	dark grey	Granite	Diorite	Elvas
GRAN PERLA	Perla Cinzento		grey	Granite	Granite	Cabeceiras de Basto
JANÉ	Gneisse Chainça	Cinza Alagoa	blue-grey	Granite	Gneiss	Castelo de Vide
MACHEADO CASTRO DAIRE			yellow	Granite	Granite	Castro Daire
OTELO	Lages	Pinheiro	grey	Granite	Granite	Marco de Canaveses
PEDRAS SALGADAS	Cinzento Claro Pedras Salgadas		grey	Granite	Granite	Vila Pouca de Aguiar
PRETO ARRONCHES	Gabrodiorito Arronches		dark grey	Granite	Diorite	Arronches
PRETO ODIVELAS	Gabro Odivelas		black	Granite	Gabbro	Ferreira do Alentejo
PRETO SANTA COMBA	Negro Santa Comba		dark grey	Granite	Granodiorite	Vila Nova de Foz Côa
ROSA ARRONCHES			pink	Granite	Granite	Arronches
ROSA MONÇÃO	Rosa Minho	Crema Júlia	pink	Granite	Granite	Monção, Valença
ROSA MONFORTE	Forte Rosa	Rosa D. João	pink	Granite	Granite	Monforte
ROSA REBOLEIRO	Salmon Reboleiro	Salmon Imperial	pink	Granite	Episyenite	Trancoso
ROSA SANTA EULÁLIA			pink	Granite	Granite	Elvas
SIENTO MONCHIQUE	Cinzento Monchique	St. Louis	grey-brown	Granite	Nepheline syenite	Monchique
SPI ALPALHÃO	Azul Alpalhão	Cinza Alpalhão	grey	Granite	Granite	Nisa
ABANCADO	Lioz Abancado		beige	Limestone	Limestone	Sintra
ALPININA	Alpinina Clara	Topázio	beige	Limestone	Limestone	Porto de Mós, Ourém, Santarém, Alcanena
AMARELO NEGRAIS			yellow	Limestone	Limestone	Sintra
ANÇÃ	Pedra Ançã	Calcário Ançã	white	Limestone	Limestone	Cantanhede
AZUL CADOIÇO	Vidraço Portela Azul		blue-grey	Limestone	Limestone	Alcobaça
AZUL FERREIRAS	Lagos Blue	Vidraço Escarpão	blue-grey	Limestone	Limestone	Albufeira
AZUL MOLEANOS	Moleanos Azul		blue-grey	Limestone	Limestone	Alcobaça
AZUL VALVERDE	Azul Mónica		blue-grey	Limestone	Limestone	Santarém
BRANCO VALONGO			beige	Limestone	Limestone	Rio Maior
BRECHA SANTO ANTÓNIO	Brecha Marítima		grey-green	Limestone	Limestone	Porto de Mós
BRECHA TAVIRA	Brecha Vermelha	Brecha Tavira Vermelha	reddish brown	Limestone	Limestone	São Brás de Alportel
CREME FÁTIMA	Crema Casal	Faticrema	beige	Limestone	Limestone	Ourém
ENCARNADÃO	Encarnado Lameiras	Encarnado Pedra Furada	red	Limestone	Limestone	Sintra
LAGOS GOLD			beige	Limestone	Limestone	Albufeira
LIOZ	Lioz Pero Pinheiro	Lioz Azulino	beige	Limestone	Limestone	Sintra
MOCA CREME	Moca Crema Clássico	Moca Crema Grão Grosso	beige	Limestone	Limestone	Santarém
MOCA PERLINA			beige	Limestone	Limestone	Santarém
MOCA RELVINHA	Relvinha	Moca Crema Grão Fino	beige	Limestone	Limestone	Santarém
MOLEANOS	Vidraço Moleanos	Crema Moleanos	beige	Limestone	Limestone	Alcobaça
MOLEANOS RIJO			beige	Limestone	Limestone	Alcobaça
SEMI-RIJO ARRIMAL	Salgueira	Branco Mar	beige	Limestone	Limestone	Porto de Mós, Rio Maior
SEMI-RIJO CABEÇA VEADA			beige	Limestone	Limestone	Porto de Mós, Santarém

Commercial Name	Alternative 1	Alternative 2	Colour	Group	Lithology	Municipality
SEMI-RIJO CODAÇAL	Rosal		beige	Limestone	Limestone	Porto de Mós
ST. FLORIENT ROSE			light pink	Limestone	Limestone	Sintra
VIDRAÇO ATAÍJA AZUL			blue-grey	Limestone	Limestone	Alcobaça
VIDRAÇO ATAÍJA CREME			beige	Limestone	Limestone	Alcobaça
VIDRAÇO PORTELA CREME			beige	Limestone	Limestone	Alcobaça
AZUL LAGOA	Azul Guerra		grey	Marble	Marble	Vila Viçosa
BRANCO BORBA	Branco Venado Borba	Branco Vergado Borba	white	Marble	Marble	Borba
BRANCO ESTREMOZ	Branco Venado Estremoz	Branco Vergado Estremoz	white	Marble	Marble	Estremoz
BRANCO LAGOA	Branco Venado Lagoa	Branco Vergado Lagoa	white	Marble	Marble	Vila Viçosa
BRANCO PARDAIS	Branco Venado Pardais	Branco Vergado Pardais	white	Marble	Marble	Vila Viçosa
BRANCO ROSADO ESTREMOZ	Branco Rosado		white	Marble	Marble	Estremoz
BRANCO VENADO POÇO BRAVO			white	Marble	Marble	Borba
BRANCO VIGÁRIA	Branco Venado Vigária	Branco Vergado Vigária	white	Marble	Marble	Vila Viçosa
CREME BORBA	Creme Venado Borba	Creme Vergado Borba	beige	Marble	Marble	Borba
CREME MOURO			beige	Marble	Marble	Borba
CREME ESTREMOZ	Creme Venado Estremoz	Creme Vergado Estremoz	beige	Marble	Marble	Estremoz
CREME LAGOA	Creme Venado Lagoa	Creme Vergado Lagoa	beige	Marble	Marble	Vila Viçosa
CREME PARDAIS	Creme Venado Pardais	Creme Vergado Pardais	beige	Marble	Marble	Vila Viçosa
CREME ROSADO PARDAIS	Creme Rosado Fonte da Moura		light pink	Marble	Marble	Vila Viçosa
CREME VIGÁRIA	Creme Venado Vigária	Creme Vergado Vigária	beige	Marble	Marble	Vila Viçosa
FICALHO			multi-coloured	Marble	Marble	Serpa
PELE DE TIGRE			white-black	Marble	Marble	Borba
ROSA BORBA	Rosa Venado Borba	Rosa Vergado Borba	pink	Marble	Marble	Borba
ROSA ESTREMOZ	Rosa Venado Estremoz	Rosa Vergado Estremoz	pink	Marble	Marble	Estremoz
ROSA LAGOA	Rosa Venado Lagoa	Rosa Vergado Lagoa	light pink	Marble	Marble	Vila Viçosa
ROSA PARDAIS	Rosa Venado Pardais	Rosa Vergado Pardais	pink	Marble	Marble	Vila Viçosa
ROSA PORTUGAL	Rosa Aurora	Rosa Puro	pink	Marble	Marble	Borba
ROSA VENADO POÇO BRAVO			pink	Marble	Marble	Borba
ROSA VIGÁRIA	Rosa Venado Vigária	Rosa Vergado Vigária	pink	Marble	Marble	Vila Viçosa
RUIVINA	Ruivina Escuro		dark grey	Marble	Marble	Borba
RUIVINA CLARO	Ruivina Fonte da Moura		grey	Marble	Marble	Borba
TRIGACHES	Cinzento Anegrado Trigaches	Cinzento Claro Trigaches	grey	Marble	Marble	Beja
VERDE SERPA	Verde Atlântico		green	Marble	Marble	Serpa
VERDE VIANA	Verde Sampaio	Verde Viana Cristal	green	Marble	Marble	Viana do Alentejo



Commercial Name	Alternative 1	Alternative 2	Colour	Group	Lithology	Municipality
BLACK IN THE SKY			dark brown	Miscellaneous	Migmatite	Vila Pouca de Aguiar
ARDÓSIA CANELAS			grey-brown	Slate	Slate	Arouca
ARDÓSIA VALONGO	Lousa Valongo		dark grey	Slate	Slate	Valongo
PEDRA NEGRA FRADES			dark grey	Slate	Slate	Oliveira de Frades
QUARTZITO MURÇA	Xisto Vales		grey	Slate	Quartzite	Murça, Valpaços
XISTO BARRANCOS			grey-green	Slate	Phyllite	Barrancos
XISTO DONELO DO DOURO			black	Slate	Phyllite	Sabrosa
XISTO MOURÃO			blue	Slate	Phyllite	Mourão
XISTO NEGRO FOZ CÔA	Xisto Poio	Xisto Poio Azulado	black	Slate	Quartzphyllite	Vila Nova Foz Cõa
XISTO OXIDADO FOZ CÔA	Xisto Poio Amarelado		brown	Slate	Quartzphyllite	Vila Nova Foz Cõa
XISTO TANHA			dark grey	Slate	Phyllite	Peso da Régua
XISTO TARRASTAL			grey	Slate	Phyllite	Covilhã, Arganil

**Acknowledgments:** This work was supported by the Portuguese Foundation for Science and Technology (FCT) in the framework of the Strategic Funding, with UIDB/00073/2020 project.

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# SEARCHING FOR MULTIFUNCTIONAL PRODUCTS FOR PROTECTION OF NATURAL STONES

M. Rucha<sup>1\*</sup>, B. Sena da Fonseca<sup>2</sup>, A.P. Ferreira Pinto<sup>1</sup>, S. Piçarra<sup>2,3</sup>

(49) CERIS, Department of Civil Engineering, Architecture and Georesources, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa | Portugal, \*madalena.rucha@tecnico.ulisboa.pt

(50) Centro de Química Estrutural, Institute of Molecular Sciences, Departamento de Engenharia Química, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa | Portugal

(51) Escola Superior de Tecnologia do Barreiro, Instituto Politécnico de Setúbal, Lavradio 2839-001, Portugal

**Summary:** Natural stone is subject to various degradation phenomena caused either by water (eg. rain) and human factors (eg. graffiti). Therefore, the application of multifunction products, such as the ones exhibiting hydrophobic and oleophobic properties, is important to protect natural stone against damaging phenomena as well as to improve their proprieties. The aim of this paper is to study the applicability and potential efficacy of three laboratorial-developed products to protect stone against different phenomena. A very porous variety of stone was used for this purpose. The application of the products resulted in a reduction of the water absorption capacity of the samples, however, the chemical design of the products still needs to be improved concerning their capability to improve the behaviour of the stone against low surface tension liquids and their tendency to change the colour of the stone surfaces.

**Key words:** natural stone, multifunctional product, hydrophobic, oleophobic.

## 1. Introduction

Natural stones are present in a wide range of modern and old constructions, including built heritage, and have various functions and purposes (cladding, paving, sculptures, etc.).

In most cases, water is the main source of problems since it triggers various degradation phenomena, especially in porous stones. It can easily penetrate within the pores, transport soluble salts and dissolve some stone minerals, when combined with certain pollutants (Doehne & A. Prince, 2010), Figure 1.



Figure 1 – Salt crystals probably by capillary action in limestone in Convent of São Francisco (Alenquer).

Moreover, the presence of water has an important role in the biocolonization of natural stones (and on their natural bioreceptivity) which can affect their surfaces and be responsible for further degradation processes, Figure 2. Therefore, preventing water from entering the

pores of the stones will reduce the incidence of biocolonization, among others.



Figure 2 – Biocolonization in granite in Guarda Cathedral.

In addition to these “natural” mechanisms of stone degradation, great attention is being currently devoted to graffiti vandalism, as many stone surfaces from buildings, monuments and sculptures are being damaged. Sprays and other types of markers containing low surface tension solvents can permeate within the stone materials obliging the use of very intrusive cleaning methods. Graffiti, like other degradation factors, jeopardize the service life of the stone elements and their aesthetic and architectural meaning, Figure 3.

A possible solution to simultaneously deal with both problems is through the application of multifunctional, namely, products able to confer both hydrophobic and oleophobic properties to the stone surfaces. The application of these products in natural stones is interesting for the ornamental stone sector (i), as part of the processing and transformation process – acting as

preventive and active treatment – and for the construction sector (ii) when the natural stone is already in service. In both cases, the products aim to improve the natural performance of the stone elements against damaging agents, by enhancing their natural durability and reducing the periodicity of maintenance actions.



Figure 3 – Graffiti in the urban environment.

The hydrophobic and oleophobic properties are obtained by reducing the interfacial tensions between the liquids and stone surfaces. Hydrophobicity can be achieved by using molecules containing non-polar chains pointing towards the exterior of the stone surfaces, while oleophobic properties are commonly conferred by fluorinated compounds.

This work briefly introduces the principles of hydrophobic and oleophobic surfaces and discusses some laboratorial experiments performed to develop and test new products designed to confer these two functions. A sol-gel chemistry was followed, where polyethylsilicate and different organosilanes with or without fluorinated groups were employed. The behaviour and potential effectiveness of the products in very porous stones were assessed.

## 2. Brief introduction to hydrophobic and oleophobic surfaces

A material is said to be hydrophobic when its surface shows lack of affinity with water and hydrophilic in the opposite case (Law, 2014).

This property is evaluated through the static contact angle ( $\theta_s$ ) of a liquid on a solid surface (Parvate et al., 2020). A hydrophilic surface shows  $\theta_s < 90^\circ$  and a hydrophobic  $90^\circ < \theta_s < 150^\circ$ . In case the static contact angle is very large ( $\theta_s > 150^\circ$ ) the surface is classified as superhydrophobic, Figure 4 (Aslanidou et al., 2018).

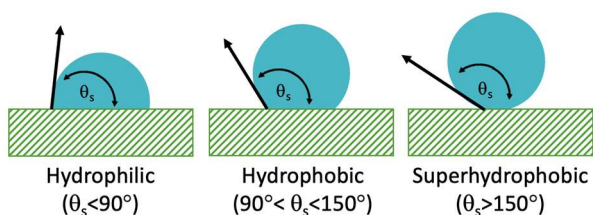


Figure 4 – Hydrophobic properties in static contact angle.

Hydrophobic surfaces are not necessarily water repellent, since water repellent surfaces implies that the water drop slides from the surface if it is tilted up to a maximum of  $10^\circ$  (Aslanidou et al., 2018). The sliding angle,  $\theta_t$ , is thus used to assess the water repellence of the surfaces.

Surfaces with multiscale roughness, such as the leaf of Lotus, are known for being superhydrophobic and self-cleaning (Parvate et al., 2020; Samaha et al., 2012).

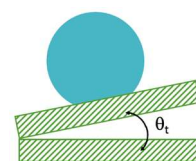


Figure 5 – Hydrophobic properties in tilt contact angle.

Oleophobic surfaces are similarly defined to hydrophobic surfaces, but instead of water (high surface tension liquid) these surfaces lack strong affinity with low surface tension liquids, such as oils or solvents present in graffiti.

Hydrophobic and oleophobic surfaces must show low surface energies between the surfaces and the water or oil molecules, respectively. The most common strategies to produce such surfaces consist of attaching stable non-polar molecules at the surfaces and to create a multiscale roughness, responsible for the so-called lotus leaf effect that can lead to superhydrophobic surfaces with self-cleaning properties (Parvate et al., 2020; Samaha et al., 2012). To induce hydrophobicity and oleophobicity, our strategy was to introduce polysiloxanes chains  $((\text{CH}_3)_3\text{Si}-[\text{O}-\text{Si}(\text{CH}_2)_2-]_n\text{O}-\text{Si}(\text{CH}_3)_3)$  and two distinct ORMOSILs containing non-polar lateral chains (either alkyl -  $(\text{CH}_2)_n-$  or fluorinated -  $(\text{CF}_2)_n-$ ) in the multipurpose product formulation (Parvate et al., 2020).

Hydrophobic and oleophobic products to protect stone surfaces must be easy to apply, stable to chemical agents, without excessively altering the visual appearance or water vapor permeability (Cappelletti & Fermo, 2016).

## 3. Materials and methods

### 3.1. Development of the products

Three products were developed in the laboratory following a sol-gel process, Table I. Small amounts of organosilanes having potential to impart hydrophobic/oleophobic characteristics to a base alkoxy silane mixture were added (Table II):

(i) poly(dimethylsiloxane) (PDMS) chain widely used to obtain hydrophobic and oleophobic properties, because provides low surface energy (Kapridaki & Maravelaki-Kalaitzaki, 2013);

- (ii) (ii) hexadecyltrimethoxysilane (HDTMS), an ORMOSIL with a long alkyl chain (with sixteen -CH<sub>2</sub>- nonpolar unities), which is expected to confer hydrophobic properties;
- (iii) (iii) 1H,1H,2H,2H-Perfluorodecyltriethoxysilane (PFDTES) which is a fluorinated compound, containing ten -CF<sub>2</sub>- unities.

Table I – Molar ratio of the developed products.

	Base alkoxy silane	H <sub>2</sub> O	HCl	Addition
PDMS				0.01PDMS
HDTMS	1 (D40)	0.15	7.88x10 <sup>-3</sup>	0.01HDTMS
PFDTES				0.01PFDTES

Table II – Representation of the organosilanes added to the base mixture.

PDMS	HDTMS	PFDTES

### 3.2. Stone

Silves sandstone (SS) was the natural stone variety chosen for the application of the products developed in the laboratory on samples of 20x20x30mm, Figure 6.



Figure 6 – Silves sandstone sample.

This stone has very high porosity with open porosity around 26.4±0.7% and is a red variety with fine grain, mainly composed of quartz and in small amounts of hematite and clay/micas minerals (Sena da Fonseca et al., 2022).

### 3.3. Treatment of the stone and assessment methods

After preparation of the products, the viscosity of each product was determined with a sine-wave vibroviscometer SV-10. The visual observation of the application of the product on the stone allows the penetration ability of the stone to be assessed by recording the evolution of the capillary fringe.

The samples were treated by applying each product by capillarity absorption for 3 hours and the amount of product absorbed was determined using Equation 1.

$$\text{Absorbed product} = \frac{m_f - m_i}{S} [kg/m^2] \quad (1)$$

Where:

$m_f$  - mass of the sample before application [kg];

$m_i$  - mass of the sample after application [kg];

S - area of the sample in contact with the product [m<sup>2</sup>].

Then, the samples were placed to age in a temperature and relative humidity (20±5°C and 70±10% R.H.) controlled dry chamber for 2 months.

The evolution of the capillary fringe during the application of the product to the stone was visually assessed and used to evaluate the penetration ability of each product.

The assessment of the hydrophobic and oleophobic effectiveness of the products was performed by measuring the static contact angle with water and oil drops on the stone surfaces, respectively following to the Young-Laplace fitting model. The water repellence of the surfaces was assessed by tilting the samples with a drop of the liquids on the top. The tilting angle was registered when the droplet started to slide. These measurements were performed on untreated samples and on samples treated with the developed products.

Colour measurements of the stone surfaces were performed before and after the treatments with a colorimeter (KangGuang WSD-3A) and using the CIELab system. In this system, the L\* coordinate represents the brightness of the colour (black - 0; white - 100), a\* the coordinate reflecting the red and green content (red (+a\*); green (-a\*)) and b\* the coordinate reflecting the yellow and blue content (yellow (+b\*) and blue (-b\*)). To understand the impact of the treatments on the stone surfaces, the total colour variation ( $\Delta E^*$ ) was evaluated according to Equation 2. The variation of each coordinate is given by the difference of the measurements of untreated samples (UT) and the treated samples (T) (e.g.  $\Delta L^* = L^*_{UT} - L^*_T$ ).

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (2)$$

The impact of the treatments on the original water absorption by capillarity of the stones was assessed by the tests described in RILEM 25-PEM.

## 4. Results and discussion

Dynamic viscosity of the products was measured after the preparation of the products at 20°C and are

presented in Table III. All values are quite similar, with PFDTES presenting a slightly higher value than the others (5.4 mPa.s).

Table III – Characterization of the products properties: dynamic viscosity, capillary fringe coefficients, absorbed product and curing time.

	PDMS	HDTMS	PFDTES
Dynamic viscosity (mPa.s)	5.1	5.1	5.4
Evolution of capillary fringe (mm/s <sup>0.5</sup> )	0.7	0.7	0.6
Absorbed product (kg/m <sup>2</sup> )	5.4	5.6	5.5
Curing time (month)	2	2	2

The formulated products penetrated the entire depth of the samples (30 mm) and with equivalent penetrations rates (observed by the capillary fringe on the lateral surfaces of the samples), Table III. Although deep penetrations were achieved, protective treatments should act at the surface, thus, penetration up to 30 mm might not be required (Rodrigues & Grossi, 2007).

The values of the absorbed product in each sample are very similar as well, Table III, and higher than the reported at the products datasheet of the multifunctional commercial products MasterProtect and ESTEL 1100 (of 0.3 kg/m<sup>2</sup> and 0.5 kg/m<sup>2</sup>, respectively) (C.T.S. s.r.l., 2015; Master Builders Solutions España, 2013). The difference in absorbed product between the developed and commercial products is predominantly due to the depth of application.

The mass stabilization occurred after 2 months for all the developed products. After this period, the potential effectiveness of the treatments was evaluated.

The static contact angle with water droplets in the treated samples is presented in Figure 7. The contact angle value in the untreated samples was not possible to be measured, as the water droplet was immediately absorbed by the stone sample ( $\theta_s=0^\circ$ ).

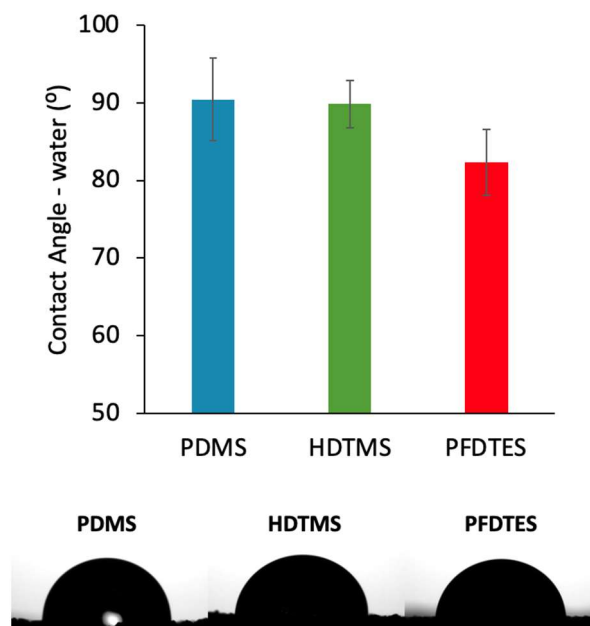


Figure 7 – Static contact angle of treated samples.

The surfaces treated with PDMS and HDTMS products lay in the transition between hydrophilic and hydrophobic ranges, but the stone treated with the PFDTES product presents a lower value, Figure 7. Although the fluorinated chains do improve the water repellence of the treated surface (that fully absorbed water droplets without treatment), was not enough to reach the needed 90° static contact angle to the hydrophobic classification.

The tilt contact angle with water droplets was performed and the samples treated with the products show angles larger than 10°, indicating that the treated surfaces were not water repellent (Kapridaki & Maravelaki-Kalaitzaki, 2013).

The static oil contact angle was performed on untreated and treated samples, being the oil droplets immediately absorbed by the stone surfaces in both situations, although at a slower rate in the treated stones. Nevertheless, none of the stone surfaces can be considered oleophobic.

A capillary water absorption test was further performed for 48 hours, Figure 8 - a), demonstrating that the application of the products does constitute barriers to water entry, since the untreated sample absorbed 4.5 kg/m<sup>2</sup> of water, while the treated samples less than 0.3 kg/m<sup>2</sup>. Thus, the products reduce the absorption of water by the samples near 15.

By comparing the capillary water absorption of the treated samples, it was found that PFTES was the organosilane that reduced the water absorption by capillarity the most, Figure 8 - b).

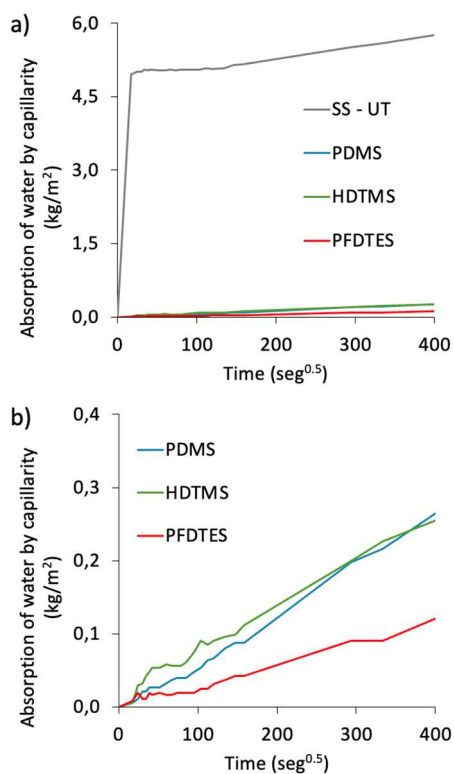


Figure 8 – Absorption of water by capillarity: Untreated (SS-UT) and treated samples (PDMS, HDTMS and PFDTES).

PDMS and HDTMS products have a very similar behaviour concerning both static contact angles and water absorption by capillarity, which may be associated to the chemical structure of these additives, both composed by non-polar chains without halogens.

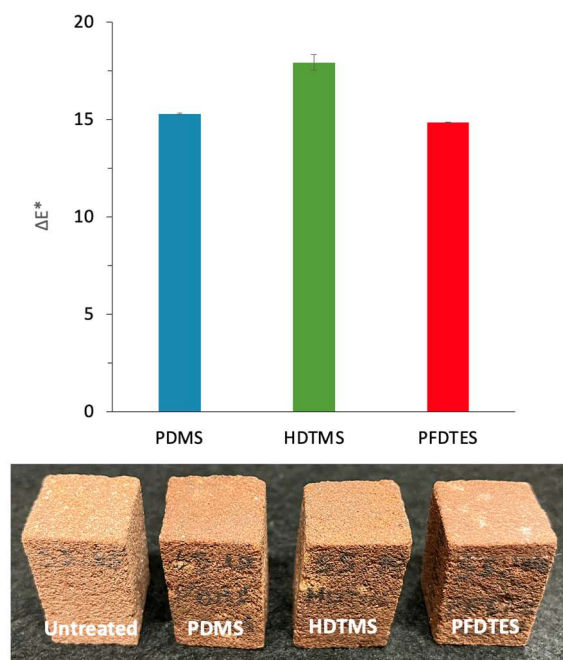


Figure 9 – Total colour variation and visual images of untreated and treated samples.

Figure 9 shows the total colour difference of the treated samples compared to the untreated samples as well as the visual appearance of the untreated and treated samples.

The total colour differences values for all the products are around 15, well above the value of 5 that is considered the limit for human naked eye detection (Rainer Sasse, 2001; Rodrigues & Grossi, 2007).

## 5. Conclusion

The tested treatments with the three laboratorial-developed products demonstrated good ability to penetrate into the stone, showing appropriate viscosities values and high measured penetration rates. However, the drying times are long (~2 months) which could be a drawback for the ornamental stone sector.

The application of any of the developed products significantly increased the water resistant properties of the stones, creating a barrier effect that strongly decreased the water uptake through the treated surfaces. The products containing PDMS and HDTMS are nearly hydrophobic (with static contact angles of 90°). Although PFDTES formulation showed a lower static water contact angle, it conducted to the best results in terms of water absorption by capillarity (the lowest value), thus making the lower hydrophobic effect at the surface being somehow compensated by the lowest water uptake by the treated stone. The high values of penetration depth may tell that a significant part of the alkyl / fluorinated chains are also located inside the pores (and not only at the stone surface). This paves the way for new approaches.

Although other assessments are required for definite conclusions, the results indicate that there is a potential improvement of the stone regarding its natural susceptibility to damaging mechanisms involving water.

Major improvements on the stone cleanliness (water repellence) and behaviour against low surface tension liquids were not observed, while the total colour variation changes are considered high ( $\Delta E^* > 5$ ). Future work will focus on the improvement of these limitations by increasing the concentration of the organosilanes in the products and/or follow other strategy for their incorporation in the base alkoxy silane product and use other application method/criteria to reduce colour alteration.

**Acknowledgments:** The authors acknowledge Fundação para a Ciência e Tecnologia (FCT) for funding the Project GreenMAP: Green multi-action products for the sustainable conservation of historic porous building stones (PTDC/ECI-EGC/2519/2020), CQE (UIDB/00100/2020 and UIDP/00100/2020), IMS (LA/P/0056/2020) and CERIS (UIDB/04625/2020). The author B. Sena da Fonseca received individual support from FCT (2020.04185.CEECIND).

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# The Portuguese natural stone georeferencing approach through the Stone4.0 Age project

A. Silva<sup>1\*</sup>, C. Claro<sup>2</sup>, N. Cristo<sup>2</sup>, C. Marques<sup>2</sup>

(52) CTCV – Technological Centre for Ceramics and Glass, \*[antonio.silva@ctcv.pt](mailto:antonio.silva@ctcv.pt)

(53) ASSIMAGRA – Portuguese Association of the Mineral Resources Industry

**Summary:** *The major goal of the Stone4.0 Age project's "Stone DNA" activity was to accurately correspond to each occurrence of any type of Portuguese natural stone (place of geographical and geological origin), to the distinctive characteristics towards a match of its commercial / trade name. The most representative quarrying clusters were identified and georeferenced, considering a 6-step methodology. From a web server database, 686 extraction sites were validated, resulting in about 150 to 250 commercial types. Actually, in the EN 12440:2017 (Natural stone – Denomination criteria), there are a total of 190 commercial types and in this activity, 146 have been clearly identified, corresponding to 133 normative sorts and therefore, this plainly justifies the need of data harmonization along with the main natural stone sector stakeholders. Another output is a new technical sheet, which include an up-to-date dataset and a location map that is set to be published in the StoneByPORTUGAL platform.*

**Key words:** *natural stone, occurrence, georeferencing, data harmonization*

## Introduction

The recent Stone4.0 Age clustering and collaborative project (2020-2022) was strategic for the promotion of digital transformation activities in SMEs of the Portuguese natural stone sector focusing the reinforcement of the StoneByPORTUGAL brand. In this context, the idea of a resourceful georeferencing methodology emerged from the "Stone DNA" work package, which contributed to the identification of potential sites for additional exploration and to assess the diversity of natural stone quarried out in the Portuguese mainland territory.

This work package highlighted the importance of updating and harmonizing knowledge about the key characteristics of the Portuguese natural stone. During the last decades, there have been significant changes in the sector, both in the extraction and processing activities. On the one hand, there was a decrease in the absolute number of dedicated companies and, on the other hand, the increase of its export capacity.

In order to further boost the exports, which is extremely important to ensure its competitiveness and economic viability, some of these PMEs, together with ASSIMAGRA and other scientific and technological partners, have actively sought to find innovative solutions that enable new business models, new services, new experiences and other ways to enhance automation and digitization of industrial processes.

In this specific case and in a simple and systematic way, it was intended to correspond / match to each natural

stone occurrence, *i.e.*, the place of geographic and geological origin, the respective distinctive features coupled with their commercial nomenclature. As foreseen in the "Stone DNA" activity, the most representative quarrying clusters for the main applications (indoor and outdoor floor and wall covering, decorative and special pieces, statuary, funeral art, among others) were identified and georeferenced.

This activity consisted in the development of a georeferencing methodology for the Portuguese natural stone occurrences, more precisely, by means of vector tools and also raster and geospatial databases integrated into a GIS platform. The GIS software used for this purpose was QGIS, an official project of the Open Source Geospatial Foundation (OSGeo).

The main objective was the elaboration of refreshed technical sheets containing the main petrological, geochemical and technological (physical-mechanical) signatures, not ignoring further info about the mining district of origin and the normative and/or commercial nomenclature.

## Methodology

The most representative extraction locations or major quarrying clusters of Portuguese natural stone were identified and georeferenced by a 6-step methodology:

(1) Consultation of various sources of information and the reference bibliography on the Portuguese natural



stone sector (e.g., books, articles, atlases, websites, etc.);

(2) Design and development of a GIS project;

(3) Extensive examination of the GIS geospatial data for its integration, analysis and further interpretation of gathered and processed information (e.g., shapefiles, rasters, WMS, WFS, aerial imagery, etc.);

(4) Analysis, treatment and processing of the georeferenced datasets regarding quarrying and exploration, but also the stakeholders' consultation towards the preparation of location maps;

(5) Design and elaboration of the StoneByPORTUGAL technical sheet template;

(6) Filling in the necessary information in the technical sheets for the commercial types on the ASSIMAGRA's StoneByPORTUGAL platform (stonebyportugal.com).

The tasks related to no. 3 and no. 4 are described below:

(i) Data acquisition through a Web Feature Service (WFS) server of the Portuguese Directorate-General of Energy and Geology (DGEG) and subsequent transformation into an Esri shapefile – .shp format;

(ii) Generation of the polygons centroid points and the respective XY coordinates in the ETRS89/PT-TM06 spatial reference system;

(iii) Analysis and photointerpretation of geospatial data via *Google Earth*, *Bing Maps*, among others.

(iv) Data discrimination by lithological and commercial types by several means (e.g., literature, ASSIMAGRA, the National Laboratory for Energy and Geology – LNEG and directly from the extraction and processing companies).

## Results

As of late 2022, in the DGEG's WFS source database, there are a total of 1401 administrative licenses, of which 844 correspond to the effective quarrying of natural stone. The remaining 557 are intended for the direct production of aggregates that feed other various industries (Fig. 1).

From the initial database of 844 entries, the following groups of licences were removed:

(a) 98 expired or formally inactive;

(b) 41 redundant;

(c) 19 deleted after final validation.

So, only considering natural stone quarrying, 686 were validated, resulting in about 150 to 250 commercial types, although some are known to be redundant.

A total of 686 entries remain in the "Stone DNA" activity database, that is to say, those that were validated and that may well have a recognised commercial name in the Portuguese natural stone market, along with others yet to be established and/or harmonized according to the EN 12440:2017 (Natural stone – Denomination criteria).

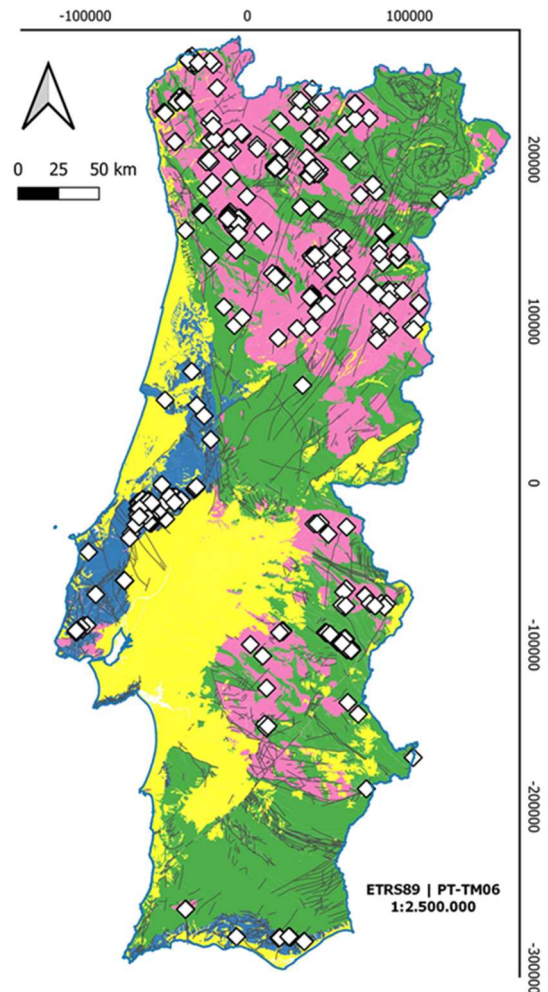


Fig. 1. Simplified geological map of mainland Portugal showing the location of the validated quarrying sites / mining districts of origin; the colours discriminate the major lithologies: i) green-metamorphic; ii) pink-igneous; iii) blue-Mesozoic sedimentary and; iv) yellow-Cenozoic unconsolidated sedimentary cover.

In fact, there are a total of 190 commercial normative types and for the purposes of the Stone4.0 Age project, 146 commercial types have been clearly identified, corresponding to a total of 133 normative sorts listed in the EN 12440:2017.

The valid quarries / licenses with an established trade / commercial name (and a small part to be established)

in the Portuguese natural stone market, were subdivided by rock types, without disregarding its main groups (metamorphic, sedimentary, igneous) and according to their nomenclatures (commercial *versus* normative).

The number of quarries is subdivided as follows by lithology:

- 20 slate-phyllite-schist: (10 commercial, 5 normative, 1 unknown);
- 26 limestone breccia (4 commercial, 4 normative);
- 171 limestone (41 commercial, 35 normative);
- 1 gabbrodiorite (unknown);
- 1 orthogneiss (1 commercial, 1 normative);
- 315 granite (87 commercial, 51 normative, 20 unknown);
- 8 granodiorite (3 commercial, 2 normative);
- 137 marble (15 commercial, 40 normative);
- 4 quartz diorite (1 commercial, 1 normative);
- 2 nepheline syenite (1 commercial, 1 normative);
- 1 limestone tuff (1 commercial, 1 normative).

To summarize, there are a total of 119 mining districts of origin, which are chiefly distributed as quarrying clusters of various sizes but also unique extraction sites.

In the majority of rock types, their commercial names exceed the normative ones. The real exception is the marble due to the reproduction of toponomy sorts like white/creme/rose from some locations in the Estremoz anticline region. It is also noteworthy the fact that some granites don't have any commercial or trade name attached.

ASSIMAGRA is currently addressing these subjects along with its partners for the purpose of harmonizing a georeferencing methodology and the vast and disperse natural stone nomenclature, by a recently created working group that include specialists from LNEG, CTCV, ASSIMAGRA and the universities of Évora and Trás-os-Montes e Alto Douro, thus the EN 12440 may be updated during 2023.

Technical sheets in the StoneByPORTUGAL platform include a new dataset and a simplified map that georeferences the origin of each type of natural stone (Figs. 2 and 3).

## Conclusions

The georeferencing tasks and detailed characterization of each type of natural Stone enabled its discrimination, considering different quarrying clusters, and culminated in the elaboration of a comprehensive technical sheet, thus allowing the comparison between various locations, simplifying and optimizing a selection process to the possible application intended by end-users, such as the professionals dedicated to architecture, engineering, design and decoration, *etc.*

In addition to the scientific knowledge enhanced by this project, the resulting outputs will allow the creation and access of content that promotes these Portuguese materials to prescribers, since this characterization constitutes a portfolio of stones "Made in Portugal" with reference to their textural characteristics and physical-mechanical properties and therefore, being the StonebyPORTUGAL brand through its online platform ([stonebyportugal.com](http://stonebyportugal.com)) aggregator of all the information, available soon to the general public.

Fig. 2. StoneByPORTUGAL technical sheet template

**Propriedades Físico-Mecânicas**  
Physico-Mechanical Properties

Densidade aparente (EN 1936) Apparent density

Porosidade aberta (EN 1936) Open porosity

Absorção de água (EN 13755) Water absorption

Resistência à compressão (EN 1926) Compressive strength

Resistência à flexão (EN 1316) Flexural strength

Resistência à compressão após gelo/dégelo (EN 12371)  
Compressive strength after frost resistance

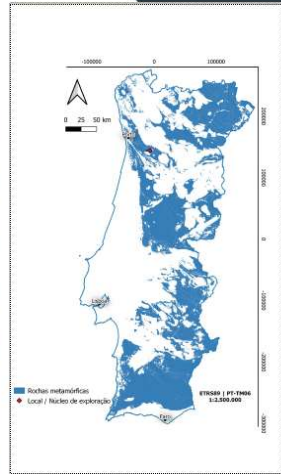
Resistência à abrasão (EN 1457) Abrasion resistance

Energia de rutura (EN 1458) Rupture energy

Coefficiente de dilatação térmica linear (EN 14581)  
Linear thermal expansion coefficient

Análise Química (% peso) Chemical Analysis (w. %)

SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	Outros Others
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Núcleo de exploração Mining district of origin

Ocorrência geográfica Occurrence

Fig. 3. StoneByPORTUGAL technical sheet template showing an example of a natural stone occurrence location map; blue-metamorphic.

**Acknowledgments:** The authors are thankful to the National and European entities that finance CTCV as a Technology and Innovation Centre – CTI. A very special gratefulness to ASSIMAGRA’s associates that actively participated in the Stone4.0 Age project and that continuously boost Portugal’s Natural Stone Industry.

# INVESTIGATION OF THE EFFECTS OF FIRE ON STONE MATERIALS: THE RIO DE JANEIRO CASE

R. E. C. Silva<sup>1</sup>; N. F. Castro<sup>1</sup>; B. F. C. Piacesi<sup>2</sup>; R. C. C. Ribeiro<sup>1</sup> & J.M.S. Santos<sup>3</sup>

(54) Researchers LACON/CETEM, \*rosanacoppede@gmail.com

(55) Chemistry Engineering student, UFRJ

(56) Geology student, UFRJ

## **Summary:**

This paper discusses the need for in-depth studies to understand the damage caused by fires on heritage stones in Brazil, particularly on stony monuments. The article highlights two common types of heritage stones found in Rio de Janeiro, Facoidal gneiss and Leptinito gneiss, and describes the changes in some properties when exposed to high temperatures.

For the Facoidal gneiss, the article compares the analysis results of actual samples exposed to fire to those obtained in fresh samples. For the Leptinito gneiss, cubic samples were subjected to high temperatures using oven-based techniques at varying temperatures, residence times, and cooling methods.

The results showed that the fired Facoidal gneiss' colour changed by superficial deposits and oil and a reddening zone close to the surface. Cracks, fractures, and surface hardness reduction were other results of this gneiss. Meanwhile, the Leptinito gneiss exposure to high temperatures in a muffle furnace resulted in various modifications, such as the red staining and whitening of the samples, biotite and garnet colour changes, cracking, and fracturing, with consequent p-waves velocity and surface hardness drop and porosity increase. Non-penetrative fractures at 800°C became penetrative at higher temperatures, and the minerals underwent transformations leading to a volumetric increase and activation of foliation planes.

Overall, the study highlights the importance of in-depth studies to guide restoration actions after fire damage to heritage stones.

**Keywords:** *fire damage, heritage stones, high temperature, Facoidal gneiss, Leptinito gneiss.*

## **1. Introduction**

Temperature variation is responsible for rocks' structural changes, the development of pre-existing microcracks and new cracks formation (Freire-Lista et al., 2016). As the density and geometry of cracks and pores are parameters that control the rocks' physicomaterial properties, the damage generated by fire on building stones can affect the construction stability, which is especially relevant for built heritage (Dionísio, 2007; Gomez-Heras et al., 2009; Vázquez et al., 2022). In Rio de Janeiro, events such as the accidental one that heavily damaged the National Museum in 2018 and the vandalic fire that affected the Monument to Pedro Álvares Cabral in 2021 put on evidence the lack of studies regarding the effects of fire on stones in Brazil. To understand the final situation of the stones after the fires and to guide restoration actions, in-depth studies are necessary (Martinho & Dionísio, 2020).

The National Museum, established on June 6, 1818, by D. João VI, is a university museum with an academic and scientific profile that focuses on producing and disseminating knowledge in natural and anthropological

sciences. The National Museum headquarters occupies the former residence of Emperor D. Pedro II, the Quinta da Boa Vista Palace, which became a 'hollow' bicentennial building and lost more than 80% of its collections after six hours fire. Beyond the impressiveness of its collections, including the oldest human fossil ever found in Brazil, called "Luzia", among 22 million items, the institution poses a tremendous national significance and responsibility as the oldest museum of the country and witness of its political, historical and scientific evolution (Duarte, 2022). The most representative building heritage stones of Rio de Janeiro (Castro et al., 2020; 2022), known as Facoidal gneiss and Leptinito gneiss, were used at National Museum. The first serves as basement blocks and dressing of the main façade and columns; both gneisses as doors and window frames (SAMN, 2021).

Figure 1 shows the aspect of some of those elements after the fire.



Figure 1: National Museum rocks after fire.

The Monument to Pedro Álvares Cabral (Figure 2) was fired during a protest against the modification of the indigenous law. Flammable material (rubber tyres and oil) was ignited on the Monument's pedestal base. The Monument was commissioned as part of the fourth centenary of the Discovery of Brazil commemorations, celebrated in 1900. The work by Rodolfo Bernardelli has a 10-meter pedestal in Facoidal gneiss over which bronze sculptures of three important historical figures stand: Pedro Álvares Cabral, commander of the fleet that arrived in Brazil in 1500; Pero Vaz de Caminha, clerk of the fleet; and Friar Henrique de Coimbra, chaplain and celebrant of the first mass (Tabet & Puma, 1983).



Figure 2: a) Monument aspect the day after fire and b) Detail of the damage. Photos by Pedro Dias.

The monument to Pedro Álvares Cabral is located at Largo da Glória, next to the Nossa Senhora da Glória do Outeiro Church, as shown in figure 3.



Figure 3: Location of the monument in Largo da Glória. Source: Google Earth, 2023.

Facoidal Gneiss (here identified as FAC) is a Neoproterozoic orthogneiss, composed of K-feldspar mega crystals usually deformed, looking like eyes (augen), surrounded by biotite lamellae arranged in a quartz-feldspar and biotite-rich matrix. Its colour varies from pink to greyish. It is essentially composed of K-feldspar (+microcline) (35%), quartz (30%), plagioclase (20%), and biotite (10%), and may contain garnet, muscovite, and other accessory minerals. Microcline can constitute more than 50% of samples with larger crystals (Castro et al., 2020).

Leptinito gneiss (LEP) is a Neoproterozoic orthoderivated garnet leucogneiss, of fine-to-medium grain size, with foliation defined by biotite lamellae and elongated quartz/feldspar grains. It is composed of quartz (30–50%), microcline (15–30%), plagioclase (15–20%), biotite (2–5%) and garnet (1–8%). Colour and textural aspects of this gneiss present variations, but the white tone (yellow when oxidised) facies, with uniform and parallel foliation, is the most common (Castro et al., 2022).

There are no studies dealing with the impacts of fire on these stones, which are the most representative on the built heritage of Rio de Janeiro.

## 2. Objectives

The main objective is to observe how the fire and the temperature decrease after the extinction affect two common heritage stones of Rio de Janeiro, Facoidal gneiss and Leptinito gneiss, describing changes in their physicomechanical properties.

## 3. Methodology

For built heritage is not possible to use samples of the specific rock, but in the case of the monument to Pedro Álvares Cabral For built heritage is not possible to use samples of the

specific rock, but in the case of the monument to Pedro Álvares Cabral, the City Hall of Rio de Janeiro provided two scales of FAC that came loose due to the fire: one external, directly exposed to the fire, and one from the immediate inner layer (Fig. 4).

The external piece was cut in three parts for observation in a stereoscopic magnifying glass (*Carl Zeiss*), and then the soot, fumes and ash were scraped off for future analysis. X-ray diffraction analysis (XRD), scanning electron microscopy (SEM, *Hitachi TM3030Plus*) with an EDS detector (*Bruker Quantax 70*), bulk density, apparent porosity, and water absorption (ABNT NBR 15845-2), Leeb surface hardness - HLD (*Equotip 3, Proceq*), p-waves ultrasonic velocity - UPV (*PUNDIT PL-200, Proceq*), and colour (*CieLab, Spectroguide Sphere Gloss, BYK Gardner*) were determined and compared to those obtained in fresh samples from reference data from CETEM (Fig. 4a).

Twenty-four cubic samples of LEP were extracted from a block donated for academic studies by the São Bento Monastery (Fig. 4b). They were heated in a muffle furnace at three different temperatures (800, 1,000, 1,200°C), four residence times (1, 2, 3, and 4 hours), and two different cooling methods, air and cold water.



Figure 4: Samples in this study. a) Monument pieces of FAC and b) Cubic samples of LEP.

The laboratory analyses of some petrophysical properties of the LEP (stereoscopic magnifying glass, bulk density, porosity, water absorption, hardness, p-waves ultrasonic velocity, and colour) were conducted before and after exposure to high temperatures.

The UPV and HLD results were compared to the reference data from CETEM. Those references were obtained from samples extracted from the cores of demolition blocks, as there are no active quarries to sample fresh rock. Notwithstanding, the reference materials' petrographic analysis and technological properties were compatible with those of similar fresh rocks.

#### 4. Results and Discussion

Cracking and colour changes were found in this study as related by Chakrabarti et al. (1996), Sippel et al. (2007), Vazquez et al. (2016), Nemeth et al. (2021), and other studies summarized in the reviews of Martinho and Dionisio (2020), Leroy et al. (2021), and Sciarretta et al. (2021). The main effects of high temperatures exposition of LEP and FAC are colour changes, cracks and fractures development and mineral loosing.

##### Stereoscopic magnifying glass

The visible effects of fire in the FAC of the monument are related to cracking (fractures), exfoliation, loss of material (rounding off the edges), black deposits (fumes and ashes), and staining (oil), as seen in Figure 2.

The FAC sample exposed to the fire (Fig. 5) presents colour changes and cracking. Regarding colour, it exhibits the three distinct zones described by Gomez-Heras (2006), from the surface to the inner part: superficial deposits of fumes and ashes (Fig. 5a), a thermal oxidation reddish zone (Fig. 5b) and the standard rock colour. Large feldspar crystals show cracks (Fig. 5c). The inner part shows some staining on the front but no apparent alterations on the back.

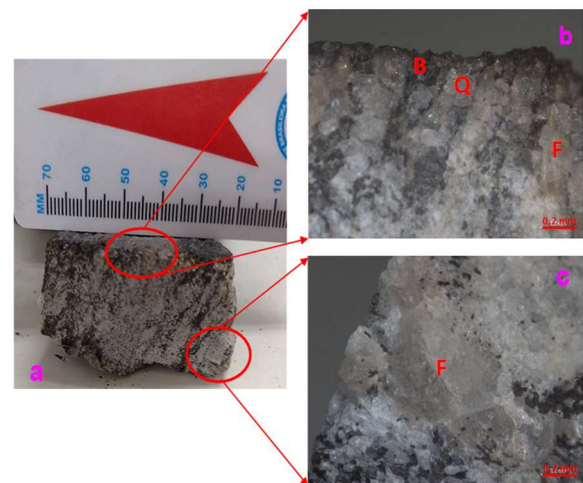


Figure 5. Normal cut of the FAC fired surface: fumes and ash external deposits (a); thermal oxidation zone (b); and feldspar cracking (c). B: biotite, Q: quartz and F: feldspar.

In the samples of LEP subjected to 800 °C, the rock dilated, but when it cooled, it was contracted, and it was possible to see some loosing at the contacts. The rocks that stayed for four hours at this temperature show increased cracks development. The colour of the biotite changed to golden. The specimens' colour changed to reddish, by thermal oxidation of iron-bearing minerals and whiter, probably due to the intense quartz fracturing (Sippel et al., 2007; Vazquez et al., 2016; Sciarretta et al., 2021). The water-cooled samples developed more cracks than the air-cooled ones, and all the specimens' surfaces were rough.

The crack density and extension increased largely in the LEP samples subjected to 1,000 °C. Biotite expanded and changed colour to golden, and garnets became grey-coloured. The red colour is more intense in the air-cooled specimens than in those cooled with water; the latter are whiter. Cracks are visible on the specimen's surface, mainly following the foliation planes, but some fractures prone to material detachment also appeared, with more intensity on the air-cooled samples. Surface roughening and small crystals loosening could be observed.

After cooling the samples heated at 1,200 °C, the damage is evident in all the specimens, although the apparent state of the air-cooled ones is the worse. Fractures are enlarged following the foliation planes or not, with areas showing material losses. Several fractures and minerals losing were also observed in the water-cooled samples, but their fractures were not as wide as those developed in the air-cooled specimens showing a higher apparent stiffness reduction. Air-cooled samples were also redder than the water-cooled ones that looked white.

Figure 6 illustrates the transformation of the LEP specimen heated to 1,200 °C for two hours and cooled with water, and Figure 7 illustrates the LEP sample after three hours of heating and air cooling.

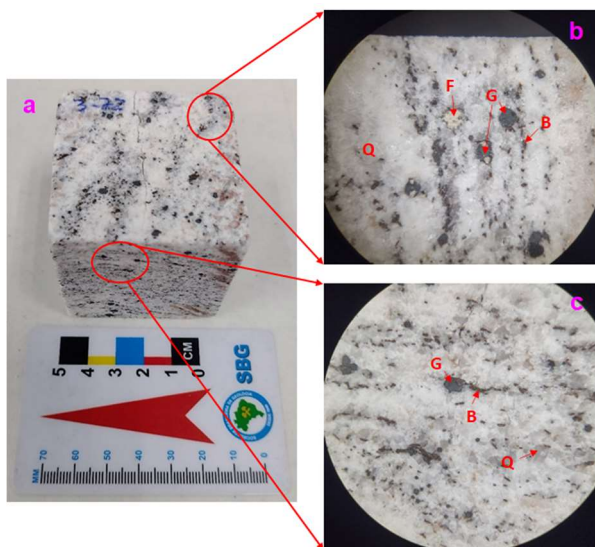


Figure 6: Change in mineral aspects in LEP subjected to 1200°C and cooled with water: a) Cracking, b) and c) garnets became grey and biotite golden. G: garnet, B: biotite, Q: quartz and F: feldspar.

The cracking process seemed to slow down in the samples cooled with water, and the fractures were less wide than in the air-cooled samples. Although quenching should be more damaging due to thermal shock (Chakrabarti et al., 1996; Pires et al., 2014; Shao et al., 2014), this was not observed in this experiment. In this specific case, the reason could be mineralogical

variations within the samples of the same block, as some destructive fractures developed in the more reddish specimens, which is also an indication of the suffered damage (Vazquez et al., 2016; Nemeth et al., 2021). Further investigations on the mineral structure and texture of the samples will be needed. The water-cooled specimens' colour was almost white, which following Sippel et al. (2007), could be due to the intense fracturing of quartz.

Figure 7 illustrates the damages observed on the LEP specimen cooled naturally after three hours in the muffle furnace at 1,200 °C: destructive fractures developed, material losses, individual mineral expansion and intense reddish colour.

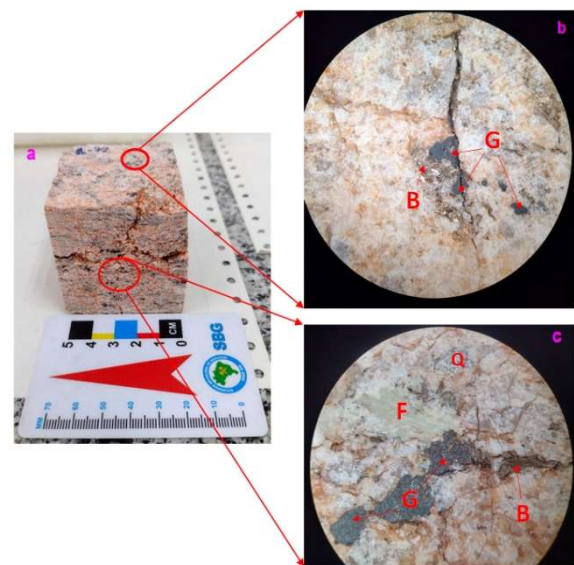


Figure 7: Changes in LEP aspects subjected to 1200 °C and cooled naturally: a) Rupture of the sample, b) and c) garnets became gray and biotite golden and enhance the cracking.

Complementing the petrography observations of FAC, the results of the X-ray diffraction mineralogical composition analyses identified quartz, k-feldspar (sodic albite), microcline, k-feldspar (calcic albite), sanidine, sodium aluminium silicate hydrate, anorthite and biotite. The presence of two kinds of albite, sanidine and anorthite, indicates a very high temperature in the fire (Haldar, 2020). Sanidine is typical of igneous rocks and forms between 650-800 °C.

Figure 8 shows micro-fissures propagation in FAC: intragranular fissures (quartz-quartz and feldspar-feldspar grain) and intergranular cracks (quartz-feldspar contacts).

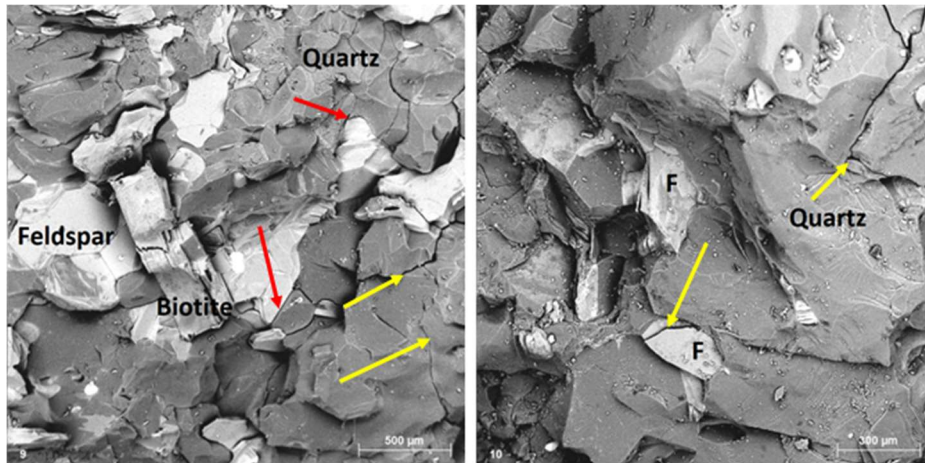


Figure 8: SEM images of intragranular (yellow arrows) and intergranular (red arrows) microfractures of FAC. F: feldspar.

### Colour

Figure 9 shows the differences in colourimetric data of LEP samples in the CIEL\*a\*b\* system. All the samples, after heating, had a positive shift of a\* (red to green), and most of them also of b\* (blue to yellow). This displacement of the a\* and b\* parameters is visible by the reddish staining of the samples by the thermal oxidation of iron-bearing minerals (Dionisio et al., 2021, Németh et al., 2021). The higher shift for the 1,000 and

1,200 °C groups after air cooling, as explained before, is clearly illustrated in Figure 9, as it is the observed whitening of the samples. The figure also shows no apparent differences in colour variation between the cooling methods at 800 °C. There was a high increase of the L\* parameter (black to white) from 1,000 °C and above, which, together with the a\* and b\* variations, lead to total colour variations ( $\Delta E^*$ ) of 4,9 to 11,1, clearly perceivable by the human eye.

Colour Differences after heating treatments - Leptinito gneiss

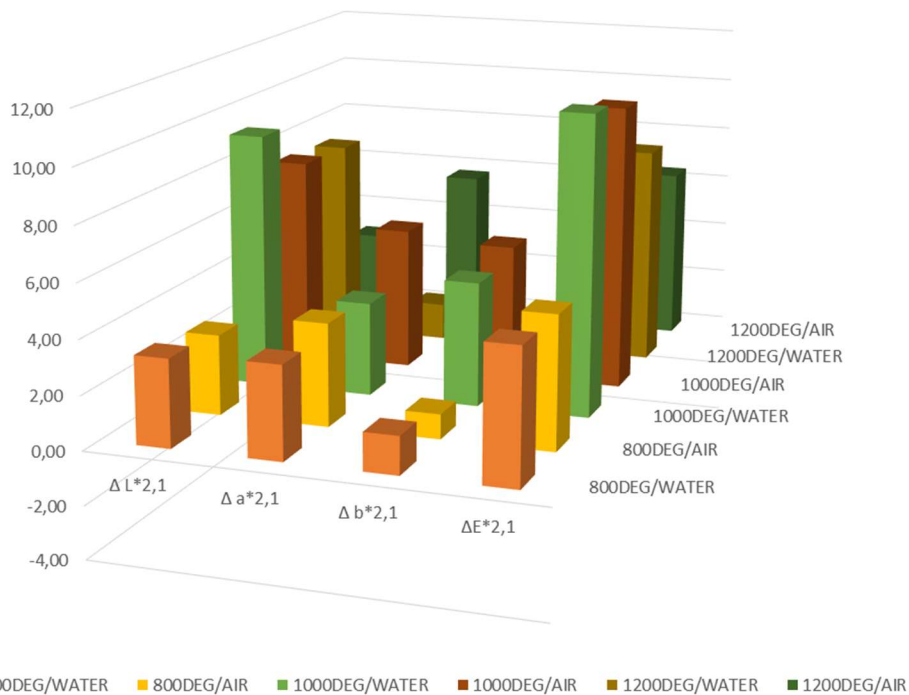


Figure 9: Averaged colourimetric variations of LEP samples after heating, CIE L\*a\*b\* system.



### Bulk density, apparent porosity and water absorption

As expected, the apparent porosity of all the samples increased (Leroy et al., 2021). This increase was higher for the LEP than for the FAC, which could be due to the unlike heating conditions (real fire and oven heating) and mineralogical composition and structure.

LEP regular and parallel foliation offers weakness planes where some fractures developed with temperature. It has higher quartz content than FAC. Quartz content is a critical parameter for the fire decay on silicate stones, having volumetric expansion much higher than other rock-forming minerals and anisotropic behaviour regarding linear thermal expansion (Winkler, 1997). Quartz thermal expansion grows with increasing temperatures and is twice as high perpendicular to the c axis, producing ever-growing thermal strains inside the stone, up to 573 °C, when thermal strains stabilize (Sciarretta et al., 2021). At temperatures beyond 573 °C, such as the ones in this experiment, the transition from low-to-high quartz changing the crystalline arrangement entails a huge expansion producing eye-naked visible cracks, as observed by Nemeth et al. (2021). Silicate rocks' residual strains (after cooling) are high if heated at more than 700 °C when the stones expand through the crack's growth and intergranular cracks formation (Sippel et al., 2007). LEP mineral composition and arrangement may have affected the observed thermal decay. It has high quartz content and, probably, higher thermal expansion perpendicular to foliation than parallel, as the mylonitic orthogneiss studied by Sippel et al. (2007). Cracks were observed in grain boundaries, as shown in Figure 10.

The FAC samples did not show as much cracking as the laboratory-heated LEP samples. More study is needed to assess the damage on those samples properly, but its distinct structure and texture may help it to be more resistant to thermal damage than the Leptinito, as it is to natural weathering. With lower quartz content than the LEP, quartz crystals in FAC are distributed in the biotite-rich matrix that may accommodate the thermal expansion of those crystals (Vazquez et al., 2022). Also, Lima et al. (2002) measured the thermal linear expansion coefficient of 61 Brazilian granitic building stones and found that the thermal linear expansion coefficient of rocks with 5% more quartz content was 16% higher, and that coefficient was also 2% to 6% higher for medium grain size than for coarse grain size rocks.

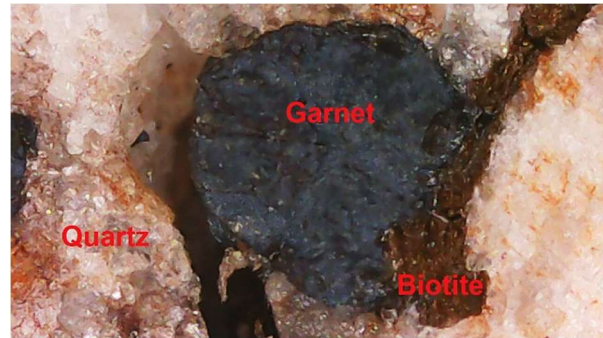


Figure 10: Detail of garnet-quartz contact with a fracture and garnet-biotite-quartz without fracture. Sample of LEP subjected to 1200°C for two hours and cooled naturally.

The time of permanence in the oven is another factor of cracking increase. The longer in the oven, the more penetrative the fractures.

Bulk density determination of the FAC scales resulted in 2,632 kg/m<sup>3</sup> (external) and 2,620 kg/m<sup>3</sup> (internal), slightly lower than the reference value (2,692 kg/m<sup>3</sup>), which cannot be attributed just to the fire as the stone's condition before the fire is unknown.

For the LEP, the bulk density decreased with increasing temperature: from initial 2,623 ± 9 kg/m<sup>3</sup> to 2,529 ± 18 kg/m<sup>3</sup> (800 °C), 2,492 ± 13 kg/m<sup>3</sup> (1,000 °C) and 2,385 ± 23 kg/m<sup>3</sup> (1,000 °C). These results reflect the specimens' volume increase due to the cracks and fractures' development (Nemeth et al., 2021). In this work, it was not possible to analyse differences in bulk density decrease regarding the oven residence time of the specimens or the cooling method, as only one specimen was used for each testing condition (Figure 11).

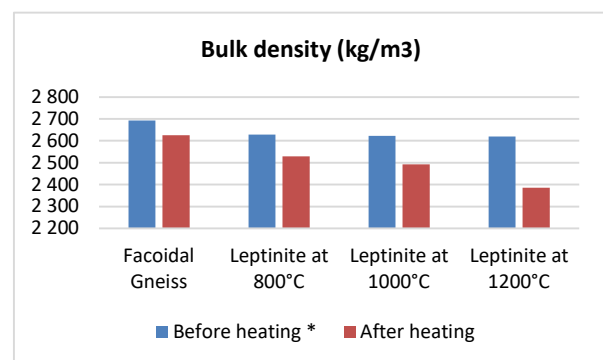


Figure 11: Bulk density of the samples before and after heating. \*FAC reference value from Castro et al., 2020.

LEP apparent porosity after high temperatures exposure increased by around 500% (800 °C), 700% (1,000 °C) and 900% (1,200 °C). Water absorption at atmospheric pressure, directly related to porosity, followed the same trend (Figure 12). The higher the temperature, the more the microcracking, cracking and fracturing, as indicated in the stereoscopic magnifying glass description. The FAC specimens detached from the monument had higher porosity than the reference value for this

lithotype, although, as explained before, the alteration degree of this stone before the fire is not known.

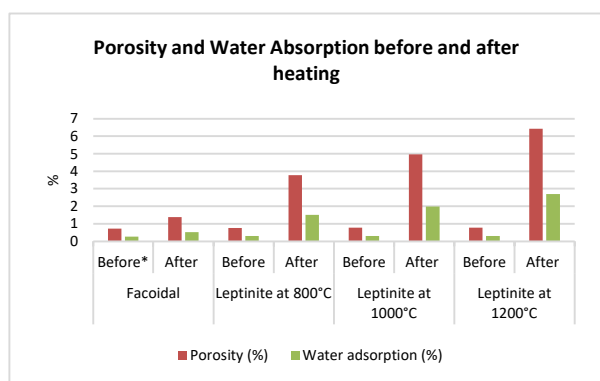


Figure 12: Porosity and water adsorption of the samples before and after heating. \*FAC reference values from Castro et al. (2020).

### Surface hardness

The Leeb surface hardness reference value for FAC is  $690 \pm 110$  HLD. The fired monument sample measurements (20) resulted in  $331 \pm 153$  (exterior scale) and  $428 \pm 90$  (inner scale). For the LEP samples, the surface hardness value dropped from  $802 \pm 22$  to  $331 \pm 76$  HLD after heating, a more than 50% reduction of this parameter. Németh et al. (2021) also verified a 60% reduction in rebound hardness value after a 750 °C heating treatment. The obtained individual hardness values did not allow the identification of any significant difference between groups by residence time or cooling method.

### Ultrasonic wave velocity

The ultrasonic wave velocity (UPV, p-wave) for reference FAC and LEP is around 4,000 m/s. The effect of cracking for both the fired FAC and the heated LEP specimens was depicted by the low UPVs measured: 500 m/s parallel to the foliation and 600 m/s orthogonal to the foliation for FAC, and 800 m/s parallel to the foliation and 600 m/s orthogonal to the foliation for LEP.

## 5. Conclusions

The fire exposure of FAC in the monument to Pedro Álvares Cabral at Largo da Glória generated some damages. Macroscopically, they are reflected in visible

cracking (fractures), exfoliation, loss of material (rounding off the edges), deposits (fumes and ashes), and staining (oil). In the samples studied in the laboratory, it was possible to identify intragranular and intergranular cracks in SEM-EDS images. The bulk density was slightly lower than that of sound rock, and porosity and water adsorption were a little higher but with similar values for the external and internal pieces. The XRD identification of sanidine points to a high temperature of the fire. This stone's condition before the fire is unknown, so that further investigations will be necessary.

The LEP exposure to high temperatures in the muffle furnace resulted in intense colour changes for all the specimens. All samples were red-stained by iron oxidation, at least to some extent, and whitened. Those colour changes were more pronounced as temperature increased. It was also observed that air-cooled samples were redder than water-cooled samples, which became whiter.

Cracks' density and intensity grew as the temperature rose: intragranular and intergranular cracks were observed in all the samples, and visible fractures developed at 1000 °C and 1,200 °C.

Biotite had a stewed appearance, and its colour turned golden. The garnets' colour became dark grey, and some crystals broke up at the maximum temperature. Quartz was intensely fractured and even appeared vitrified.

The mineral transformations and differences in thermal expansion caused a volumetric increase and initiated cracks in the stone, activating cleavage and foliation planes. The bulk density decreased by up to 10%, and porosity and water absorption increased by 900%. Surface hardness was reduced to less than half and the p-wave velocity to one-fifth, meaning that after a fire, those stones may endanger the stability of heritage buildings if they have a structural function.

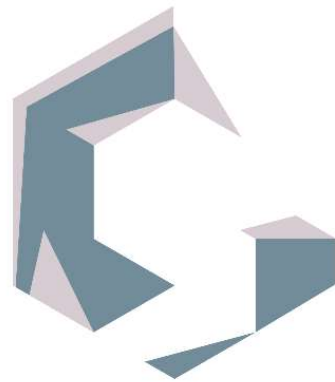
Other analyses will be carried on so this work can serve as a basis to prepare better a future study of the National Museum gneisses decay state.

**Acknowledgments:** The authors would like to express their appreciation to Vera Dias (City Hall of Rio de Janeiro) and to the São Bento Monastery for the samples, and Pedro Dias for the pictures.

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**GLOBALSTONE**  
c o n g r e s s 2 0 2 3

**Thursday, June 22<sup>nd</sup>**  
**PRESENTATIONS**

## BIM-BASED PLATFORM FOR ORNAMENTAL STONE SUPPLY CHAIN

Ferreira, Irene<sup>1\*</sup>; Reis, Gonçalo<sup>1</sup>; Almeida, Nuno<sup>1</sup>; Vieira, Carlos<sup>1</sup>; Silva, A.<sup>2,3</sup>; Rabadão, Carlos<sup>3</sup>;

(57) STREAM Consulting Lda, Edifício AOC Business Center Azabucho, Pousos, 2410-021 Leiria, Portugal, [\\*irene.ferreira@streamconsulting.pt](mailto:irene.ferreira@streamconsulting.pt)

(58) CIIC – Computer Science and Communication Research Centre, Polytechnic Institute of Leiria, Building C – Campus 2, Morro do Lena – Alto do Vieiro, 2411-901 Leiria

(59) MARE, Marine and Environmental Sciences Centre, Polytechnic Institute of Leiria, Portugal

**Summary:** *Building Information Modelling (BIM) technology improves architectural and infrastructure projects by digitizing their processes throughout their life cycle stages, such as design, construction, management, monitoring, and operation (Z. Wu, 2017). Despite the effort, much remains to be done for the current use of BIM in the construction industry chain. This gap can be significant in speeding up the decision-making system in awarding materials and services inherent to the construction of buildings (Xiao Lia, 2017) (SmartMarket, 2009). Therefore, the development of a digital and collaborative platform, BLM Stone, aims to bring BIM technology to the Portuguese Ornamental Stone sector. Through the BLM, the quarries and factories can directly connect to architects and engineers in a digital, collaborative, safe and user-friendly platform.*

**Keywords:** *BIM, Library, Marketplace, E-commerce, Web, Application*

### 1. Introduction:

Building Information Modeling (BIM) technology improves architectural and infrastructure projects by digitizing their processes throughout their life cycle stages, such as design, construction, management, monitoring, and operation (Jorge Collao, 2021). Despite the effort, much still needs to be done for the current use of BIM in the construction industry chain (Vilas-Boas et al., 2019). This gap can be critical to speed up the decision-making system in awarding materials and services inherent to the construction of buildings (Z. Wu, 2017) (Xiao Lia, 2017). Note that this process directly impacts the costs and deadlines for building execution. This process lacks efficiency and collaboration where the relevant information is stored and managed on different platforms inherent to the various stakeholders, typically geographically distant (Silva, Rabadão, et al., 2020). In this context, BIM, with recognized advantages in combining objects, attributes, and production characteristics, could be very relevant to support decision-making and collaborative work and thus increase the efficiency of the entire process. Of these processes, the acquisition of finished ornamental stone for assembly on-site stands out (Silva, Dionisio, et al., 2020). Therefore, the development of a digital platform, called BLM for Stone platform, aims to bring BIM technology to the Portuguese ornamental stone sector, allowing to put extraction companies and stone transformers in direct contact with architects and engineers, responsible for designing and construction works, in a digital, centralized, safe and easily accessible, which is the focus of the present work. Its main innovation features include, amongst others:

- Exploring interoperability mechanisms covers interfaces with proprietary formats, using Industry Foundation Classes (IFC) as an open BIM standard, as an indispensable source of information between all

procurement intervenient of ornamental stone customized products. Note that, despite all the efforts and improvements in IFC-enabled interoperability of various BIM platforms, there is still a way to go to provide a satisfactory level for industry users (SmartMarket, 2009), namely information loss and misrepresentation, resulting in model exchange being considered unreliable;

- Select and align customers' orders based on technical product specifications (e.g. materials, finishes, dimensions, delivery times) directly read from IFC files;
- Establish supplier performance indicators to direct client orders to suitable suppliers and speed up the entire procurement process;
- Simplify and speed up the ornamental stone supply chain procurement process.

The availability of online sales mechanisms is assumed today as a competitive factor in the business environment. This is also true for the ornamental stone supply chain. Then, one adds online sales mechanisms to centralize the offer of sale of all types of stone products, i.e. standard and customized (typically made by order). In that case, it guarantees the creation of a tool of greater added value that allows customers and suppliers greater speed of the business and a more simplified process.

This approach is like what has been done for the automotive, aerospace or simply international goods transport sector, which support their orders in centralizing platforms where worldwide customers seek to make available their needs and, consequently, any worldwide entity can answer to their orders faster. The BLM Stone platform follows this principle of operation and serves as an e-commerce platform and a facilitating platform to centralize ornamental stone product orders/sales. This concept is based on BIM information to ensure the correct order specification and,

consequently, manufacture the desired product in a more simplified and fast process.

## 2. BLM Platform description

The BLM Stone platform - BIM Library and Marketplace is an online application that supports the commercialization of ornamental stone products for the construction sector, among others. This application aims to provide and speed up the purchase of a very significant set of standards and customized ornamental stone products, whose dimensional and physical characterization, amongst other important order information, is in an IFC file. Thus, it is possible that any entity involved in the construction process, which needs these kinds of products, supports their supply process through BLM Stone platform. Since the application needs high performance and a fast user experience, it was developed based on single-page application technology for programming two distinct frameworks. For the frontend strand, the Nuxt framework for vue.js and the API platform uses a framework for Ruby, called Ruby on Rails.

### 2.1. Frontend application

Nuxt.js is a very intuitive Vue structure that solves the problem of structuring Vue.js projects, allowing the creation of applications with frontend architecture ready for companies. The resources used in this Nuxt technology are already structured according to architectural patterns to create enterprise applications (Kinsta.com, 2022). This type of site does not require any external content source, and the content is already embedded in the HTML, resulting in a dynamic page with dynamically updated objects. This structure is called a single-page application (SPA) (Kinsta.com, 2022).

On this platform, the client application used the technology of Nuxt.js, an open-source framework based on Vue.js, to create server-side web applications (SSR) and client-side web applications (CSR). It provides an optimized architecture for developing universal (also known as isomorphic) applications that can be rendered on the server or web browser. Nuxt.js includes routing, state management, page building and rendering, and support for CSS preprocessing, static file generation, and more. It uses the "configuration convention" concept to help developers build applications quickly with a clear and consistent framework. With Nuxt, developers can build scalable, efficient, and high-performance web applications, allowing users to experience a faster and more responsive user interface. It is often used in e-commerce, media, SaaS, mobile apps, and other web apps.

#### 2.1.1. Why Nuxt.js

The BLM Stone platform was developed using one of the most widely used frameworks for building SPA applications. In this, the Nuxt framework for Vue.js was

used. This framework allows the development of client applications more quickly and ensures the high performance of the final application. The choice of this tool was due to the popularity of the technology in the SPA application development market, the ease of incorporating built-in features, the ease of use of API endpoint mechanisms, the use of detailed error messages, as well as the existence of very detailed documentation (LogRocket Frontend Analytics, 2022).

#### 2.1.2. How it works

Nuxt.js works like a server-side structure when a user visits a website. If server-side rendering is enabled, requests are rendered on the server each time the user requests a page, so a server must be able to serve the page on each request.

These are the main actions and methods used in Nuxt.js (Kinsta.com, 2022):

- i. `nuxtServerInit` (Action): This is the first lifecycle hook on the server side if Vuex storage is enabled. It is a Vuex action that is called only on the server side to pre-populate the store and finally can be used to dispatch other actions in the Vuex store;
- ii. `validate()` (Function): The validate method is called before rendering page components. It is helpful to validate the dynamic parameters of a page component;
- iii. The `asyncData()` method is used to fetch and render the data on the server side, while the `fetch()` method populates the repository before rendering the page.

For example, when a Nuxt.js site receives an initial visit to the page, it calls the `nuxtServerInit` action to update the Vuex states (if defined in the Vuex repository). Otherwise, if `nuxtServerInit` is not defined, it will move to a different stage. It then looks for middleware in that order. First, it checks the `nuxt.config.js` file for any global middleware. If it is set, it runs before checking for middleware on the layout pages. Finally, it will run the individual page middleware, including the children of the page. After running all the middleware in order, it will verify that the route is dynamic and execute the `validate()` method created and validated (Kinsta.com, 2022). It then calls the page's `asyncData()` method to fetch and render data on the server side before calling the `fetch()` method to populate the client-side Vuex storage. At this point, the page should have all the data and content necessary to display a suitable Web page. The page is then rendered by Nuxt.js engine and shown to the visitor, completing the process.

One of the reasons Nuxt.js is very fast in performance is because of the code splitting feature, where a route receives a single JavaScript file with only the code needed to execute that route, thereby reducing the size of the app (Kinsta.com, 2022). This code-splitting feature uses the Webpack configuration bundled in when generating a static site version. This development approach requires using an external API for data

collection and displaying it on the client side. Most JavaScript frameworks like React.js, Vue.js, Angular, and Ember.js are single-page app frameworks.

## 2.2. API interface

For a large part of the e-commerce applications currently available on the WEB, the complexity of the business model makes it necessary to create an application distinct from the display layer, an API, to contemplate all the complexity of this model. This separation of business model rules from the frontend allows for greater flexibility in application layer changes and scalability for other technologies. An API stands for application programming interface—a software intermediary allowing two applications to talk. In this case, this API is responsible for receiving requests from the client application, processing them, requesting the necessary information from the data storage systems and returning the desired result to the client entity. In this application, an API was developed in Ruby, using the Ruby on Rails framework, a back-end that is programmatically accessible by late from the client application in Vue.js. Instead of using Rails to generate HTML code communicating with the server through forms and links, this API acts as a control mechanism between the frontend and data access. When the client application needs access to data, it forwards to the API a particular request, and it is the API that, through the business rules, requests from the database the desired data, processes, and returns them to the client application in the form of a JSON file.

### 2.2.1. Why Rails

Among the various languages used for developing API applications for the online store, in the 2022 Stack Overflow report, Ruby on Rails presents itself as one of the most widely used languages (Aglowiditsolution.com, 2022). The choice of this use is due to the accumulated knowledge of the team of developers in this language, as well as the following motivations:

- i. Development mode: Rails applications come with intelligent patterns for development, making development enjoyable without compromising production time performance;
- ii. Logging: Rails applications log all requests with a level of detail appropriate for the current mode. Rails logs in development include information about the request environment, database queries, and basic performance information;
- iii. Security: Rails detects and thwarts IP spoofing attacks and consciously handles cryptographic signatures from timing attacks;
- iv. Parameter parsing: Rails decodes the JSON and makes it available in params. Nested URL-encoded parameters can also be used;

- v. Conditional GETs: Rails handles GET (ETag and Last-Modified) conditional processing request headers and returns the correct response headers and status code;
- vi. HEAD requests: Rails will transparently convert HEAD requests to GET requests and return only the headers in the output. This makes HEAD work reliably across all Rails APIs (Rybyonrails.org).

### 2.2.2. How it works

This web application framework includes all the features to create database-backed web applications according to the Model-View-Controller (MVC) pattern. MVC divides an application into three tiers: Model, View, and Controller, each with a specific responsibility, as shown in Fig. 1.

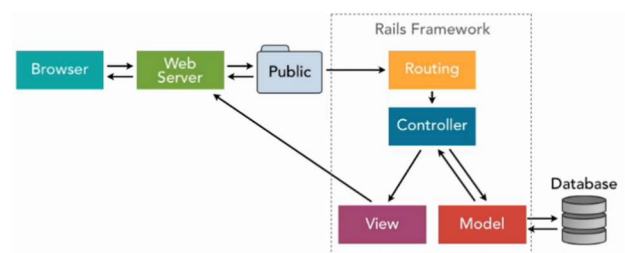


Fig. 1 Rails architecture (TK)

### 2.3. API development

This application was developed according to Fig. 2

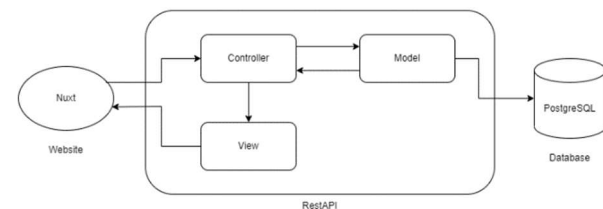


Fig. 2 Schematic representation of the platform ecosystem

Considering the previous structural elements, an API was built to store and access the transaction of data from a client application and the database. It uses 16 controllers, adequately configured to promote access control, data access and data return in JSON format. To this end, specific views related to the various controllers were created to allow the availability of this data through endpoints of the API itself (routes). A specific route is created for each of them to allow communication between the client platform and the API (Rubyonrails.org). The main objective for this API is to be an application layer that will be responsible for resolving the requests from the client application and getting or storing information in the database. Although it is in this application that the business logic roles are implemented, throw authentication roles, database access restrictions are needed to ensure the correct data storage usage.

## 2.4. Client application development

The development of the BLM Stone platform stemmed from the need to include BIM technology in the commercialization process of ornamental stone products. A preliminary stage, i.e. requirements identification, was carried out for that purpose. The functional requirements resulted from the identification of functionalities with the potential for the valorization of the sales process of these products, as well as the identification of functionalities collected from the experience of the suppliers of these products. This survey resulted in the following main features:

- i. Database for BIM files;
- ii. Centralizing platform for commercialization of ornamental stone products;
- iii. Commercialization of standard products;
- iv. Quote for customized products;
- v. Use of IFC files with drawing/technical information of the product;
- vi. Bank of suppliers by type of material and existing finishes;
- vii. Notifications of new customized product requests.

## 2.5. Validation tests

During the implementation process of the BLM Stone marketing platform, a set of tests were developed for both the API and the client platform to ensure that the functionalities implemented followed the intended specifications. For that purpose, the Postman platform was used as a tool for creating and managing the tests (Fig. 3).

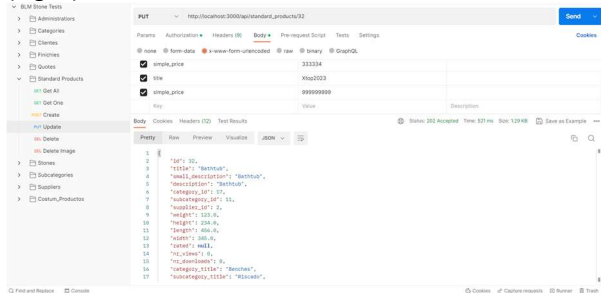


Fig. 3 Postman application where the tests were created and managed

Then, as for the client platform, the verification tests of the implementation of the functionalities result from the use of the Postman platform, as well as from the use of the platform by end users.

## 3. BLM Stone application

BLM Stone platform, apart from the usual mechanism of dissemination and sale of ornamental stone products, presents specific functionalities for the use of BIM files to maximize the availability of technical and commercial information about the product to be purchased/manufactured. Thus, regarding the use of

BIM files, it presents itself as a library of IFC files necessary for architects and engineers in civil construction to access technical information about standard ornamental stone products. Still, it is essential to highlight that, in a disruptive way, this application also supports customized products by submitting an IFC file with all the technical characteristics of a specific ornamental stone product/order (Fig. 4).

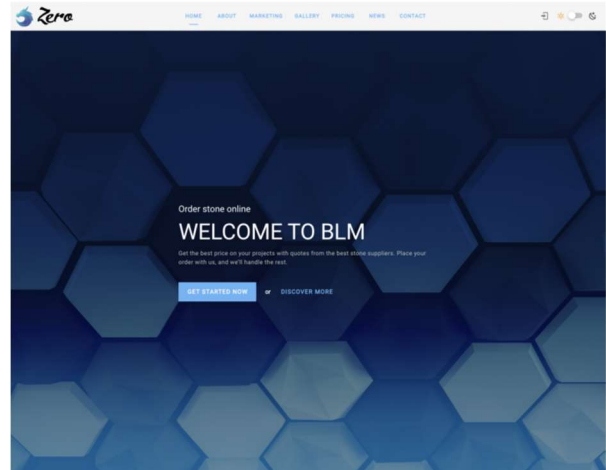


Fig. 4 Uploaded page for an IFC file

Thus, after submitting a customized product order, the BLM Stone application requests the IFC file and the description of some technical information regarding the customized product needed for the supplied quote. With this information, the BLM platform presents a pre-selected set of potential suppliers, in the case of standard products, or notifies the potential suppliers (i.e. whose production characteristics answer order specifications) to respond to these orders through an IFC file. Fig. 5 represents the workflow of quotation of a customized product.



Fig. 5. Request for quotation flow for customized products

In Fig. 6, it is possible to observe the menu produced by BLM platform regarding the quote list for one generic supplier of the customized product after receiving the notification to reply to that quote.

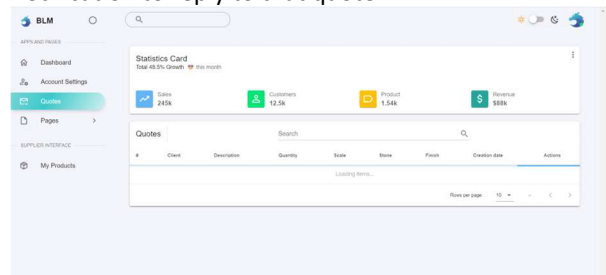


Fig. 6 Quote list of customized products

Then, all the proposals made by potential suppliers will be sent to that customer after the client accepts one of those proposals, and that specific order can be



produced. Aiming to help suppliers process, they can access a dashboard section that allows adding, removing, editing descriptions, enabling discounts, and other product information (Fig. 7).

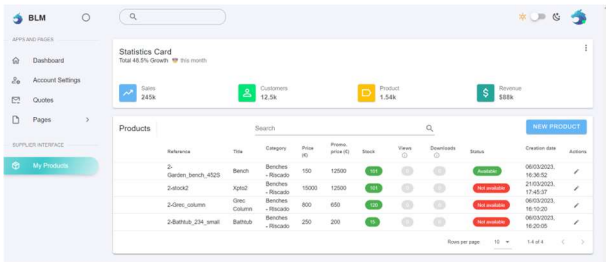


Fig. 7 Suppliers standard products panel

Regarding BLM platform's main objective, other features were implemented, namely:

- i. Display of ornamental stone products in a marketplace: On this feature, the user can see the details of all standard products available in the shop and the respective IFC file to download. This allows the customers to simulate the product in their architecture project;
- ii. Supplier management of standard products: All users with the role of supplier can access a specific area that allows managing their own standard products;
- iii. Management of products in purchasing areas: The client can access an area that contains the information on his active orders and other historical orders;
- iv. IFC Files Library: This library contains an IFC file available on a standard product. All clients can download those files and use them on their projects;
- v. IFC reader to display the file object: This feature is used to ask for a quote on a customized product. The application allows the user to include a specific IFC file and display it on a view component that allows the user to see the object uploaded;
- vi. User authentication: As other web applications, to create security levels and to allow or restrict some activities to users, this platform has three levels of restrictions (one for each type of user (client, supplier and administrator)), according to Fig.8 hierarchy;

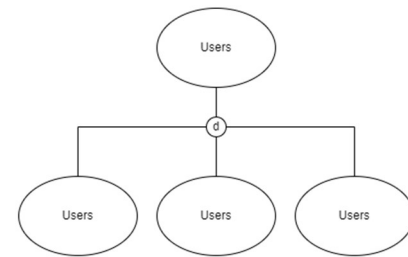


Fig. 8 User database hierarchy

- vii. Supplier notification based on IFC information: This feature consists of reading an IFC file uploaded by a client and automatically getting information from that file. Information like the type of stones, dimensions, and colour, among others, will allow us to identify and automatically notify a group of suppliers capable of doing that specific order.

#### 4. Conclusions and Future Work

As mentioned, much remains to be done for the current use of BIM in the construction industry chain, specifically for the ornamental stone supply chain. Regarding this challenge, the work previously carried out through BLM Stone platform development consists of a first step, namely, as an e-commerce application and a platform that connects customers and suppliers of the ornamental stone chain. However, there are still essential functionalities to be achieved BLM stone platform aims to bring BIM technology to the Portuguese mineral resources sector in a digital, centralized, safe and easily accessible. Thus, future platform developments will focus on creating an automated supplier filter and interoperability with other platforms. To ensure that costumers get the best solution for their needs, the platform must be capable of suggesting the best suppliers. To do that, suppliers must be ranked using an algorithm based on multiple indicators, such as item availability, time of delivery, and user reviews, among others. To keep the platform's database up to date regarding crucial information for its operation, it must be able to connect with already existing platforms. This will allow keeping information continuously updated on things such as type of material and finishes, among others.

#### Acknowledgements:

This work was financially supported by Project INOVMINERAL 4.0.: INOVMINERAL 4.0 - TECNOLOGIAS AVANÇADAS E SOFTWARE PARA OS RECURSOS MINERAIS (SI I&DT - Mobilizing Programs, POCI-01-0247-FEDER-046083) in the scope of Portugal 2020, co-funded by FEDER (Fundo Europeu de Desenvolvimento Regional) under the framework of POCI (Programa Operacional Competitividade e Internacionalização).

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A. Silva<sup>1\*</sup>, A.J. Cardoso<sup>2</sup>

(1) CISE - Covilhã, Portugal; CEFAGE-UE, Évora, Portugal; MARE, Leiria, Portugal, \*a.silva@zipor.com

(2) CISE - University of Beira Interior, Covilhã, Portugal

### Summary

*The technological intensification behind the modern supply chains is pushing companies to the extent of their critical mass and investment capabilities, struggling to access the market. Based on small and medium companies (SMEs) and integrated into the architecture, engineering, and construction (AEC) supply chain, the ornamental stone (OS) sector is relevant to the Portuguese economy. Through the BIM generalization at a global level, the AEC supply chain becomes fully digital, pushing, therefore, the manufacturing companies like the OS companies to meet digital requirements and challenges, from which arises the following question: What are the challenges for stone companies in BIM-oriented supply chains? Through a quantitative survey, the companies' concerns were evaluated on a representative sample of the Portuguese stone sector. From this methodology, the main challenges to succeed in BIM-oriented supply chains converge to efficiency, scale, and customer satisfaction.*

**Key words:** building information modelling, stone, supply chains, AEC, BIM

### 1. Problematic

In times of uncertainty and geopolitical games, the drift towards increasingly digital and global supply chains poses additional challenges for companies. In this regard, the digital transformation of manufacturing SMEs is often referred to by scholars (Hara et al., 2016) and institutions (European Commission, 2020) as essential for their survival. Connecting customers directly to providers (Haleem et al., 2018) and globally rated by customers (Medberg & Grönroos, 2020), the manufacturing SMEs' access to the digital supply chains is often referred to as requiring a market orientation mindset (Bicen et al., 2021).

### **BIM-oriented Supply Chains (BIM.SC)**

Architecture, engineering, and construction (AEC) have witnessed an increasing interest in using the concept of building information modelling (BIM) in conjunction with sustainability principles (Jalaei & Jade, 2015).

BIM technology can usually be summarised as the creation of a digital model of the construction (Silva, Rabadão, et al., 2020a). Although they differ, BIM maturity is often confused with BIM dimensions. While maturity refers to the ability level of the supply chain exchanges BIM information, the BIM dimensions refer to the different types of data linked to a model. By creating data dimensions for a construction component in BIM, the designer better understands the construction in a complete way, the work stages, how each component

must be delivered, the budget, and how the project must be maintained, among other aspects. These dimensions contribute to better management and data improvement toward project optimization. From the basic three dimensions, the state-of-the-art BIM evolved to more sophisticated ones, and nothing prevents new dimensions from being included in the future. In this regard, BIM keeps evolving permanently.

### **Challenges for Stone Companies in BIM.SC**

Stone has always been an essential component of building construction, and therefore, the ornamental stone (OS) companies integrate the AEC supply chains. Mainly for small and medium enterprises (SMEs), the OS sector is relevant in some European countries. In Portugal, for example, the OS sector represents more than 16,600 direct jobs, being one of the foremost private job generators in inland regions. Despite the recent AEC crises, this sector has recorded sustained growth in exports over the last two decades, placing Portugal as the eighth country in the international trade of OS and the second country in international trade per capita worldwide.

Procurement occurs during the designing phase in a BIM-oriented supply chain (BIM.SC). BIM impacts the stone elements as to any other material used in AEC since this technology creates information models for any element added to the digital model during the designing phase. Sharing graphical and non-graphical information stored in a database, each solid element added has

several BIM dimensions, making the model as authentic as possible and pulling the procurement upwards. From this arises the following question: What are the challenges for stone companies in BIM-oriented supply chains?

To find a possible answer to this question, it can be assumed that each BIM dimension links to the AEC supply chain, which brings specific concerns to the stakeholders. The OS company's concerns are evaluated in BIM.SC to reach the research objective.

As a methodological procedure to reach this objective, along this research, (1) each BIM dimension linkage to the AEC supply chain will be identified, then (2) the companies' concerns will be evaluated through a quantitative survey on a representative sample of the Portuguese stone sector, from which, (3) the OS companies in BIM.CS will be inferred.

## **2. Linking BIM Dimensions to AEC Supply Chain**

Looking at BIM from an architectural drawing tool view, it differs from CAD since it uses solid blocs instead of lines and curves. Moreover, BIM is referred to as the enabler of the digital AEC supply chain (Barazzetti et al., 2015). Furthermore, in BIM, the construction elements have multiple dimensions (Corry et al., 2015) comprising all phases of a construction process (Karan & Irizarry, 2015).

Each BIM dimension acts as a layer. A complementary element of information such as cost, accuracy, sustainability, and compatibility, among other information (Sacks et al., 2010). By creating and using these dimensions, one can better understand the planning of the work to the management of the undertaking sustainably (Corry et al., 2015). In this regard, details such as the graphic representation of safety and sustainability can be added to the project through the elements' dimensions (Lu et al., 2015), enabling the collaboration among AEC supply chain actors throughout the whole cycle of the built elements and project (Cheung et al., 2012). In this regard, it was elaborated the guidelines for a structured questionnaire based on the following questions:

2.1. Concerns on third BIM Dimension (3D.BIM - model, solid blocks interface & designing): (1) the operations shifting to digital? (2) the digital shopfloor technologies acquisition? (3) new skills admission? and (4) the real-time collaboration BIM users - providers?

2.2. Concerns on fourth BIM Dimension (4D.BIM - time, planning times & processes construction: In BIM.SC, what is the level of concerns for OS companies regarding (1) the scale for projects regardless of their dimension?

(2) the short lead times? and (3) the in-time delivery according to the market demands?

2.3. Concerns on fourth BIM Dimension (5D.BIM - cost, planning, estimation, means and economics): In BIM.SC, what is the level of concerns for ornamental stone companies regarding (1) the competitive prices? and (3) the zero defects (quality)?

2.4. Concerns on fourth BIM Dimension (6D.BIM - sustainability, analysis of thermal performance and efficiency energetic): (1) the low carbon footprint in processes & products (ecological products)? (2) the green label products? (3) the product's recyclability? and (4) innovative energy-efficient products?

2.5. Concerns on fourth BIM Dimension (7D.BIM - maintenance, operation approach & maintenance): In BIM.SC, what is the level of concerns for stone companies regarding (1) product maintenance? (2) customer response time? Furthermore, (3) incorporating the valuable service?

2.6. Concerns on fourth BIM Dimension (8D.BIM - safety, emergency plans and prevent security issues): (1) the comfortable elements? Furthermore, (2) the capability to improve products to prevent security issues?

## **3. Application Field and Quantitative Survey**

As stated above, the OS sector is relevant to the Portuguese economy, which since 2011, recorded an average annual growth turnover rate of 5.13% and revealed a low turbulence rate. This sector includes both upstream activities and downstream activities. Although the contribution of upstream activities is very significant in terms of turnover, the contribution to national wealth implies to rise in the value chain of the products exported, tasks inherent to companies whose activities are primarily downstream, which may or may not also have quarries.

### ***Population and Sample***

This sector has companies with multiple CAE (Economic Activity Classification), which makes analysis based on official data difficult. In this sense, for this research purpose, a direct countability at the level of mainland Portugal was carried out, accounting for 2.551 companies operating either upstream, downstream, or both activities, spread over eighteen Portuguese Districts.

This population analysis found that almost 70% of these companies are micro-companies or craft workshops with at least three workers. These micro-companies are far from taking advantage of the digitalization global shifting in the short or medium term. Moreover, these micro-companies exclusively provide a very close

market while using something other than computerized technologies. As these micro-companies do not fit the purposes of this research, they may be out of the population of this research.

Therefore, the population has nine hundred and ninety-four OS companies, employing more than three persons and using at least one computerized machine on the shop floor. From this population, the interviews were conducted with a sample of six-hundred and seventy companies, representing 67.4% of the population. This sample representativeness is further reinforced if considering the companies turnover (89.3% of the population), the exports (95.9% of the population), and the direct jobs (88.5% of population).

To carry out the survey, structured questionnaire guidelines were followed. The answers followed a five-point Likert scale, rating the concerns as "very high," "high," "moderate," "low," and "very low." All these companies were interviewed face-to-face from February 2022 to February 2023, and the results were recorded in an Excel file.

### ***3D.BIM Concerns Evaluation***

The interoperability is related to the ability of two or more systems to exchange data. Low interoperability in the AEC supply chain represents one of the most substantial reasons for budget slippages. Studies point to figures of billions of dollars spent annually (HM Government, 2011). Operate in BIM.SC in AEC also means to realize that interactivity in real-time collaboration with BIM users will become the natural way to sell. In other words, a significant component of the sales force will move to collaborative work with BIM operators. The companies' answers to the questions related to solid blocks interface and designing issues reveal that 65.4% of the respondents evaluate as "very high" or "high," the concerns related to real-time BIM collaboration and 59.6% the concerns related to digital shopfloor technologies acquisition (Table 2). Moreover, the concerns analysis found that 57.3% of OS companies consider that 3D.BIM highly impacts their activities.

### ***4D.BIM Concerns Evaluation***

Information related to schedule represents the fourth dimension (4D.BIM) (Robeznieks, 2009) while reducing execution time and eliminating schedule slippages is one of the objectives pointed out by the rulers for making BIM mandatory in AEC (HM Government, 2015). This information associated with the objects allows users to schedule and visualize the evolution of the building activities throughout the life cycle (Han & Golparvar-Fard, 2015). The result is a more efficient detection of conflicts and management of those conflicts or changes

that may occur during the construction and maintenance of a building throughout its life cycle. The 4D.BIM tools include processing the information collected on site, visualizing impacts, and informing stakeholders involved in the construction or warning of risks. This information can be integrated with project management applications, bringing benefits to users in terms of project plan optimization. Builders and manufacturers can use 4D.BIM to optimize their construction activities and coordinate different teams.

To integrate a BIM.SC implies the capability to respond quickly and provide products according to market demands. Moreover, this capability must be ensured regardless of the project dimension. That is, companies must have quick lead times either in small or large-scale projects. In this regard, from the answers to the questions related to planning times and constructive processes (4D.BIM), it was found that 70.8% of the respondents evaluate as "very high" or "high" the concerns related to capacity for projects regardless of its dimension (scale) and 69.2% to the concerns related to the in-time delivery according to the market demands (Table 3). Moreover, the analysis of the concerns found that 64,5% of the OS companies consider that 4D.BIM highly impacts their activities.

### ***5D.BIM Concerns Evaluation***

In BIM.SC contexts, the companies make their products available through open-access web libraries while costs and taxation become transparent in AEC (Race, 2013). By integrating the cost information with quantities obtained from 3D.BIM and 4D.BIM, the BIM technology, estimates the cost structure and cash-flow maps, making them available, thus replacing traditional Excel spreadsheets (Smith, 2014a). Moreover, through the information associated with the 5D.BIM, it is possible to make predictability of cost changes arising from changes in the type of materials, equipment, or labour (Lu et al., 2015), providing management methods and cost analysis in different scenarios and the respective impacts of each change. In this regard, from the answers to the questions related to planning, estimation of, means, and economic (5D.BIM), it was found that 68.2% of the respondents evaluate "very high" or "high" concerns competitive prices at a global level and 63.7% the concerns on zero tolerance to defects (quality) (Table 4). Moreover, the analysis of the concerns found that 65,9% of the OS companies consider that 5D.BIM highly impacts their activities.

### ***6D.BIM Concerns Evaluation***

In BIM, information related to sustainability represents the sixth dimension of the object. 6D.BIM information in BIM platforms also allows, in the design phase, to obtain

information on energy consumption estimates and the ecological footprint of the building during its entire life cycle, including its demolition.

In BIM.SC in AEC, product sustainability determines global competitiveness (Lyytinen et al., 2007). Incorporating sustainability components into the BIM model generates 6D models, which enable designers to meet carbon targets for specific project elements and validate the design decisions accordingly or test and compare different options (Smith, 2014a). Furthermore, the 6D.BIM also makes it possible to incorporate information gathered on site from measuring and verifying sustainability information during construction and better processes for choosing high-performance facilities. In this regard, from the answers to the questions related to sustainability analysis of thermal performance and energy efficiency, it was found that 63.1% of the respondents evaluate as "very high" or "high" the concerns on low carbon footprint in processes and products, and 61.9%, the concerns on energy-efficient products (Table 5). Moreover, the analysis of the concerns found that 60,4% of the OS companies consider that 6D.BIM highly impacts their activities.

#### **7D.BIM Concerns Evaluation**

The seventh BIM dimension of the objects is used by managers in operating and maintaining facilities throughout their life cycle, enabling participants to manage and track relevant asset data such as component status, specifications, maintenance, operation manuals, and warranty dates, among others (Kocziszky & Somosi, 2016). The use of 7D.BIM may result in more accessible and faster parts replacement, fulfilling and optimizing a streamlined asset lifecycle management over time (Gillen, 2013). Through monitoring and managing the built environment in real-time, the 7D.BIM makes it possible to manage assets, specify maintenance models and, create preventive and curative maintenance procedures (Sune & Gibb, 2013), negotiate guarantee conditions, among other operations, relating to each element (object) incorporated in the design from the beginning of the project to the demolition of the building. By integrating BIM.SC implies providing low-maintenance products on a global scale. Moreover, through BIM libraries, competitors may access the maintenance level of the rivals' products.

In this regard, from the answers to the questions related to the operation and maintenance of the elements, it was found that 71.7% of the respondents evaluate "very high" or "high" concerns related to the low-maintenance products, and 64.5% of the concerns

related to the response time to customers (Table 6). Moreover, from the analysis of the concerns, it was found that 66,0% of the OS companies consider that 7D.BIM highly impacts their activities.

#### **8D.BIM Concerns Evaluation**

The eighth BIM dimension represents the information on safety aspects from construction to demolition. Once incorporated into BIM, this information lets designers quickly develop actions, documentation, safety plans, simulations, and situation analyse before construction starts. That is, integrating BIM.SC requires providing products that positively impact people's health and safety. In this regard, from the answers to the questions related to emergency plans and preventing security issues, it was found that 66.6% of the respondents evaluated as "very high" or "high," the concerns related to zero security issues and 64.2% the concerns related to healthy products. Moreover, the concerns analysis found that 65.4% of OS companies consider that 8D.BIM highly impacts their activities.

#### **Conclusion**

Given the challenges posed to businesses by the global drift to BIM.SC in AEC, the digital transformation of the OS companies seems to be impactful for their activities:

1. From the survey it was found an average convergence of 65.5% in concerns related to the market: (1) real-time BIM-collaboration capability (65,4%); (2) in-time delivery according to the market demands; (3) zero tolerance to defects (quality) (63,7%); (4) green label products (59,0%); (5) products recyclability; (6) energy-efficient processes & products (61,8%); (7) low maintenance products (71,7%); (8) response time to customers (64,5%); and (9) services incorporation (61,4%). This concerns in convergence to market may result from the direct connection between BIM users and providers. This connection publicly exposes providers to a global market rating, from which it may be inferred that in BIM.SC, a fundamental challenge for OS companies, is ensuring high-level customer satisfaction ratings (Table 1).

Table 1: Convergence towards concerns on Customers' Experience

real-time BIM-collaboration	65,4%	3D.BIM
zero tolerance to defects	63,7%	5D.BIM
energy efficient products	61,8%	6D.BIM
easy maintenance of products	71,7%	7D.BIM
response time to customers	64,5%	7D.BIM
zero security issues	66,6%	8D.BIM
healthy products	64,2%	8D.BIM
Convergence towards Customers' Experience	65,4%	

2. From the survey carried out, it was found an average convergence of 66.8% on concerns related to efficiency: (1) competitive prices at a global level (68,2%); (2) quick time to deliver products (53,6%); and (3) low carbon footprint in processes & products (63,1%); (4) healthy products (64,2%); and (5) no security issues (66,6%). This convergence to efficiency may be explained since, through efficiency, the manufacturing companies can offer price-wise products. That is, through efficiency, the manufacturing companies ensure competitive conditions to integrate the AEC supply chain successfully. Therefore, it may be inferred that in BIM.SC, a fundamental challenge for OS companies, is to ensure high efficiency (Table 2).

Table 2: Convergence towards concerns on Efficiency

in-time delivery and according to the market demands	69,2%	4D.BIM
competitive prices at a global level	68,2%	5D.BIM
low carbon footprint in processes & products	63,1%	6D.BIM
Convergence towards Efficiency	66,8%	

**Acknowledgments:** The authors express appreciation to the Inovmineral4.0 Project, for the support they received on their work

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3. From the survey carried out, it was found an average convergence of 65.2% on concerns related to their investment capacity to acquire production scale and digital infrastructures: (1) digital infrastructures (47,7%); (2) advanced shop floor technologies acquisition (59,6%); (3) skills admission (56,5%); and (2) scale to provide projects regardless the dimension (70,8%). Therefore, it may be inferred that in BIM.SC, a fundamental challenge for OS companies, is to ensure financial solutions.

Table 3: Convergence towards concerns on Investment

digital shop-floor technologies acquisition	59,6%	3D.BIM
capacity for projects regardless its dimension (scale)	70,8%	4D.BIM
Convergence towards Investment	65,2%	

From the above, it may be concluded that in BIM-oriented supply chains, the OS company's challenges were ensuring efficiency, scale, and customer satisfaction. Moreover, shifting the operations to digital requires ensuring connectivity among actors. That is, to catch BIM.SC the companies must adequate their capabilities in infrastructures, shopfloor means, and human resources, among other capabilities which require investment capacity. However, bank debt is usually the only way to finance investments for stone companies, mainly SMEs.

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# Decarbonization challenges in natural stone quarries

J. L. Frazão<sup>1\*</sup>, I. P. Frazão<sup>2</sup>, J.P. Frazão<sup>3</sup>

- (1) Project Manager, Fravizel S.A., \*Jorge.Fraza@Fravizel.com
- (2) Marketing Manager, Fravizel S.A.
- (3) Human Resource Manager, Fravizel S.A.

**Summary:** *Mineral resources extraction (mining or quarrying) and construction are key sectors in today's economies. Natural stone extraction requires the use of specific dynamic capabilities and technology throughout several stages until the dimensional block is obtained. Also in natural stone quarrying, the decarbonization must take place to meet the GHG emission targets established by the European Union, i.e., carbon neutrality in 2050. In this study, we resume possible paths for the decarbonization of natural stone quarrying stages with Fravizel Technology. In our study, we found that some stages will be easier to decarbonize than others. Electrification will be the most important path, furthermore, increasing productivity and hybridization with diesel/electric will be a solution in the short-medium term to lower GHG emissions per extracted volume.*

**Keywords:** *Natural stone, Quarrying, Technology, Dynamic Capabilities, Decarbonization, GHG emissions.*

## Introduction

Mining and quarrying are essential industries that provide the raw materials needed for many aspects of modern life but it should be aligned with the European *Critical Raw materials Act*, that sets clear actions to ensure access to a sustainable supply of strategic raw material.

However, like other industries, these industries also have a significant impact on greenhouse gas (GHG) emissions, contributing to the global climate crisis. In Europe, there is a growing commitment to reduce emissions and achieve carbon neutrality by 2050 (European Committee of the Regions, 2022), with many countries setting ambitious targets for decarbonization.

According to the European Environment Agency, in 2019, GHG emissions from the mining and quarrying sector in the European Union were around 196 million tonnes of CO<sub>2</sub> equivalent, representing approximately 3% of total EU emissions. This highlights the urgent need for the sector to reduce its emissions if Europe is to achieve its decarbonization goals (Förster et al., 2021)(EuroStat, n.d.).

To address this challenge, there is a growing focus on developing and implementing decarbonization strategies in the mining and quarrying sector, including exploring new technologies and practices that can help to reduce emissions while maintaining productivity and competitiveness. By doing so, the sector can play a crucial role in supporting Europe's transition to a low-

carbon economy and helping to mitigate the impacts of climate change(EU, 2018)(IEA, 2021)(Un.org, n.d.). Nevertheless, mining and quarrying have an important role in local, countries and worlds economy. In Portugal the annual production of natural stone from 2009 to 2019 increased 33% to 3 574 349 ton with a commercial value of 170 377M€ (ASSIMAGRA, 2021).

With increasing demand for resources, quarrying is one of the few remaining viable options for most global economies((Gazi et al., 2012)). Nevertheless, it's possible to innovate and increase productivity without breaking with established practices and available technologies ((Gazi et al., 2012)).

The impact on local socio-economic plays a key role in the development this industry ((Prno, 2013)). Energy wise, the location of mining and quarrying sites is important to have electric connection.

The decarbonization of the mining and quarrying sector is expected to have significant impacts on the human resources within the industry. As the sector moves towards a low-carbon future, it will require new skills, technologies, and ways of working, which will require investment in upskilling and reskilling the workforce. This constant adaptation means that companies must develop new capabilities to remain competitive, i.e., dynamic capabilities.

Dynamic capabilities can be divided into capabilities (1) to sense and shape opportunities and threats, (2) seize opportunities and (3) remain competitive by combining, protecting and, when necessary,

reconfiguring the company's intangible and tangible assets. Companies need to thrive their development, internationalization and networking capabilities, remembering that motivations are intended to respond to market challenges (David J. Teece, 2007).

The type of stone and quarry site determine the type of machinery used, size of stone mass extracted and final size of dimensional stone block. Nevertheless, new methods and techniques can bring productivity to the sector.

The quarry exploration has several phases to obtain the final dimensional stone with commercial measurements ready for transport. These phases depend on the stone hardness but can be divided into four phases: Drilling, cutting, tilting and squaring (Frazão & Frazão, 2013). Inside each phase different equipment and methods can be used.

### **Phase 1. Drilling phase**

Drilling phase has the purpose of completing triangulation holes so that the diamond wire machine can cut all the sides of one bench and obtain the paralepipedic shape stone mass. The main advantage is the longer drilled holes and consequent larger stone masses. Alternatively in softer stones, chainsaw machines can be used to replace phase 1 and 2, but the cut length is limited to the length of the chain with the added probability of the blade getting stuck under the bench in a horizontal cut, especially in discontinued stones.

The drilling machines used in this stage are mostly pneumatic machines with Down the Hole Hammers (DTH). Pneumatic DTH systems have some limitations in energy efficiency, GHG emissions, hole deviation, dust control, in longer drilling depths and with unconsolidated rock.

It is not uncommon to see diesel powered air compressors near machines in quarry sites. The burn of fossil diesel highly contributes to not only GHG emissions during the work of DTH rigs, but also at idle, when DTH machines are being prepared, adjusted, or moved. Another issue with energy efficiency is leakage in air lines.

From an environmental point of view, the CO<sub>2</sub> emission factor in KgCO<sub>2eq</sub>/kW.h varies depending on the European country, being the EU average 229t CO<sub>2eq</sub>/kW.h in 2020 and Portugal 185t CO<sub>2eq</sub>/kW.h in that same year (European Environmental Agency, 2022).

Quarry operations are very demanding on equipment and accessories and without the proper maintenance air leaks can reach up to 40% of total air produced increasing even more the energy consumption and emissions. Typical quarry DTH operates from 6 to 10 m<sup>3</sup>/min at 5-7 bar, depending on the air pressure available where the machine is, that normally is far from the compressor.

DTH machines for quarrying applications are mostly column type, tensioned to the ground with chains for support. This type of setup is labour intensive because operators need another machinery, i.e., wheel loader or excavator for transport and setup. All this causes increased overall CO<sub>2</sub> emissions due to fuel consumption.

Fravizel technologies have several capabilities to provide energy efficiency solutions in this process such as electrification and automation, hydraulic drilling, electro-hydraulics, and own mobility. Being remote controlled it increases safety and four times more productivity.

This phase efficiency can improve with electrification and better mobility, lower setup time, lower machine running costs and CO<sub>2</sub> emissions. Also, hydraulics can bring greater power density improving productivity with much higher drilling rates.

### **Phase 2. Cutting**

In this phase diamond wire machines are used to separate the stone mass from the wall using previous triangulation holes made by DTH machines. These machines depend on a wheel loader for its mobility and the time used to move the regular diamond wire machines and CO<sub>2</sub> emissions caused by the wheel loader, can be reduced by adding technology such as mobility.

Fravizel technologies has several capabilities like high production performance and its own mobility.

### **Phase 3. Tilting**

In the Tilting phase, the objective is to tilt the stone mass away from the wall so it can be further processed into a dimensional block. The faster it's done, the faster quarry front is free for another section being cut. For larger mass stones more than one excavator or wheel loader are used, two or three are normally used contributing all to a significant increase in GHG emissions.

But with a high-power stone mass displacement system, attached to a carrier machine, such as a hydraulic tracked excavator, it's capable to generate approximately 260t of force, and to displace up to 2000t of stone mass in less time. This can be done with just one medium sized excavator, therefore significantly reducing the GHG emissions on this phase. It's also expected to have less wear on the carrier machine (tracked excavator or wheel loader), less time to perform this phase, compared with substitutes, less operation cost, less machines and human resources mobilized on this phase (Frazão & Frazão, 2013).

#### **Phase 4. Squaring**

After the stone mass has been pushed (tilted) to the ground, it's necessary to cut it into transportable blocks with stone chainsaws, diamond wire, and/or with drilling machines. The removal of this stone mass from quarry front is imperative to continue the exploration further. To increase production and achieve cost reduction, this requires larger stone masses to be processed and depending on the stone characteristics, it might be necessary to use more than one process to have the final dimensional block.

Some of the technologies used in the squaring phase rely on diesel engines, don't have own mobility increasing the lack of efficiency and the GHG emissions.

Fravizel has technologies that has several capabilities like mobility and maneuverability to every quarry angle driven by a hybrid energy source.

A mobile drilling machine and a mobile diamond wire machine are a key asset in quarrying management. The added autonomous mobility with high and long reach, and wide cutting length enables fast deployment and cutting in all the surface area of the bench.

Overall, one key element for squaring phase is to clear quarry front as soon as possible and this requires machines capable of processing larges sections of stone and have mobility for faster deployment. Like in any industrial process electrification is a key method to achieve high efficiency and low operational costs.

Another important aspect is the waste produced during squaring phase. Transforming the stone mass into transportable blocks means that it needs to be sectioned into smaller sections and the waste produced is proportional to the cutting width as also the energy required.

If we consider the advantage of having less waste - more volume to sell – the advantages are well seen environmentally and commercially.

#### **Discussion**

Phase 1 can be more efficient by diverting away from

compressed air stationary machines. Compressed air is expensive and without proper leak control leads to a very inefficient process in quarrying. Also using mobile machinery increases productivity by not depending on human force or other machinery for its mobility on quarry front. Fravizel technologies allows the reduction of 81% in energy costs and 87% in CO<sub>2</sub> emissions, contributing to the decarbonization of the sector. (Source: Fravizel)

Phase 2 can be more efficient with the use of mobile machinery and less resources involved to start the operation.

Phase 3 can be a safer with lower emissions of GHG using less resources. Fravizel technologies allows an added productivity of 83%. (Source: Fravizel).

Phase 4 is the one that has more choices, with different ways and technologies better adapted to each quarry and stone.

Fravizel technologies enables the reduction up to 82% in energy costs and less 85% in CO<sub>2</sub> emissions contributing to the decarbonization of the sector (Source: Fravizel).

In diamond wire cutting, shifting the machine from stationary to mobile electric machinery can reduce the GHG mainly because they don't need other machines, i.e., wheel loader to be moved around the quarry.

Also, digitalization plays a key role in quarrying and mining operations due to its potential to improve productivity, safety, and sustainability. By using digital technologies, such as automation and the Internet of Things (IoT), companies can optimize their operations, reduce costs, and increase efficiency. Digitalization can help to improve safety by reducing the need for human intervention in hazardous areas, and by detecting and preventing equipment failures before they occur. Finally, digitalization can support sustainability by reducing energy consumption and emissions, and by enabling more efficient use of resources.

#### **Conclusion**

The overall efficiency of a natural stone quarry depends on several factors. It's a fact that natural stone quarrying has a high percentage of waste which represents not only the wasted material but also wasted energy and cumulative and unnecessary GHG emissions.

One key impact of decarbonization is likely to be a shift towards more renewable energy sources and electrification of quarrying operations, which will require new technical expertise in areas such as battery

storage, renewable energy technologies, and electric powertrains and machines. This could potentially create new job opportunities in areas such as engineering, operations, and maintenance, while also requiring existing workers to acquire new skills and knowledge.

Another potential impact of decarbonization on human resources in mining and quarrying could be a shift towards more sustainable and efficient practices. This could involve reducing waste and improving the circularity of the industry, as well as developing more environmentally friendly quarrying techniques and optimizing workflows and reorganizing the way to explore inside the quarry. Such practices could require different types of expertise and could potentially create new job opportunities.

However, it's also important to note that the transition

to a low-carbon mining and quarrying sector will continue to depend on the availability of human resources and this transition to digitalization and carbon neutrality is a matter of corporate survival.

Overall, the decarbonization of mining and quarrying is a must have in this industry, it's an opportunity to have an even more environmentally friendly product because, being a natural product, it should be the perfect example of sustainability, not only environmentally, but also economically and socially. Dynamic capabilities are organizational and strategic routines through which managers change their resources to generate value creation strategies (David J. Teece, 2007). That's the great opportunity.

**Acknowledgments:** The authors can express their appreciation or acknowledge to support and help their received on their work etc.

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# RISK ASSESSMENT AND MANAGEMENT IN MACHINE MANUFACTURING FOR THE STONE INDUSTRY

A. Pata<sup>1</sup>, A. Silva<sup>2,3\*</sup>

<sup>1</sup> D. Dinis Higher Institute (ISDOM), Marinha Grande, Portugal

<sup>2</sup> CEFAGE-UE, Center for Advanced Studies in Management and Economics, Évora, Portugal

<sup>3</sup> MARE, Marine and Environmental Sciences Centre, Polytechnic Inst. Leiria, Portugal

\*a.silva@zipor.com

**Summary:** *Accordance with safety standards and legislation are some responsibilities assigned to those who develop and build machines. Risk assessment and risk management applied to the design, conception and operation of a machine can contribute to compliance with a range of requirements that help to significantly reduce, or even eliminate, the risks of a machine failing in terms of safety. Thus, safety functions and risk assessment methods can be applied to ensure that limits are met without endangering people's health and safety. To standardize the risk assessment of machines in the phases that are the responsibility of the manufacturer, a preliminary risk analysis is suggested in the design phase and the application of the HRN method in production. The proposal has been tested and validated on a CNC stone processing machine, StoneCUT<sup>®</sup>mill and the risks associated with this machine in the design phase are found to be 5.59 and, in the construction phase, acceptable risks with HRN = 0,0006.*

**Keywords:** *assessment, risks, machinery, management, stone.*

## 1. Introduction

The construction of a machine arises from the need to automate a manual process according to the customer's specifications. The starting point depends on what phase the study of the manufacturing process is at. There is still a need for knowledge in the industrial world about resolving high risks through risk assessments and safety functions, and it is sometimes unclear in an industrial environment. Therefore, it is of utmost importance to know the diversity of Risk Assessment methods that exist and understand their applicability and safety functions. Since they are of vital importance in the production of machinery because of the high risks if they fail, there may be severe unwanted occurrences such as deaths and amputations, or there may be highly relevant hazards that entail civil and criminal liability. However, scientific research can provide new opportunities for machine manufacturers, contributing to the construction of safe machines according to techniques, methods, tools, standards, legislation, and advanced technology. Gaining a better understanding of risk assessment methods to ensure compliance with these requirements enables machine builders, without great expense, to apply methodologies to provide customers with compliant products that meet all applicable standards and legislation and remain competitive in an increasingly demanding and uncertain marketplace. On the other hand, knowing how to apply evaluation methods that are not part of the manufacturer's responsibilities, that is, in the operational phase of the machine, allows them to provide customers with some guidelines.

## 2. Methodologies for risk assessment

The literature has several methods for assessing machine risks: quantitative, semi-quantitative and qualitative. These methods can be applied in three phases: design, construction, adequacy, and operational.

- In the design, we can use the following methods:

(1) qualitative – (PRA) *Preliminary Risk Analysis*: which is generally applied to the initial phases of a new design. Its application can be critical to reducing costs and unnecessary concerns and even avoiding severe accidents. It is an initial analysis critical in investigating new and little-known systems when there is no or little experience with risks in their operation. It makes it possible to determine the risks and preventive measures before the operational phase. It should be complemented by more demanding and more specific techniques (Barreto & Meiriño, 2016; Barros, 2013).

(2) quantitative – *HAZOP (Hazard and Operability Study)* deals with the study of possible failures, errors, or deviations. The method serves to identify risks to people, equipment, environment, or organizational objectives. Keywords are used that question the design intent or operating conditions. It can address all forms of design deviation due to design deficiencies. Over a series of meetings, a multidisciplinary team evaluates the design phase if changes are deemed feasible. The evaluation may be performed during operation. However, the cost-benefit ratio may not justify the required changes. Current information about the

system, process, or procedures is analyzed. Inputs to consider may be drawings, flowcharts, layouts, and procedures (e.g., operation, maintenance, emergency response). New designs should be completed with other techniques (Barros, 2013). The risk assessment must also be done during its development and completion.

An engineering technique used to define, identify, and eliminate known and potential failures, problems and errors in a system, design, process and service before it reaches the customer. (3) semi-quantitative (intuitive) – *FMEA (Failure Mode and Effect Analysis)*: allows for the analysis of each component of a system systematically, the various failure modes that may occur, their causes and their effects on the operation and safety of the system. It helps to identify the potential failure modes of a product (e.g., machine, equipment) or a process, the effects of the failures, and assesses the criticality of these effects on product functionality, also providing essential information on reliability prediction and product or process designs (Barros, 2013; Lipol & Haq, 2011).

- In the manufacturing /adaptation of a machine: (4) quantitative – *HRN (Hazard Rating Number)* is used to estimate the risks on machines (Fig.1). According to a sequence of steps, the limits of the machine are determined, identifying the hazard, estimating the risk, assessing the risk, devising countermeasures, and establishing an acceptable safety standard for the work. It is considered one of the main tools for risk assessment on machinery. While adapting a machine (Safety of Machinery and Equipment), there is the risk assessment phase, where this method can be applied (Santos & Rodrigues, 2020; Steel, 1990).

Rating	LO	FE	DPH	NP	HRN*
0.003	Almost impossible, possible only under extreme circumstances				0 to 10 Very Low
0.1			Scratch/bruise		10 to 50 Low (though relevant)
0.5		Annually	Laceration/mild ill health effect		
1.0	Highly unlikely, though conceivable	Monthly	Break - minor bone or minor illness (temporary)	1-2 persons	50 to 500 High
1.5	Unlikely, but could occur	Weekly	Break - major bone or major illness (temporary)	3-7 persons	
2.0	Possible, but unusual		Loss of 1 limb/eye or serious illness (temporary)	8-15 persons	500 to 13,500 Unacceptable
2.5		Daily	Loss of 2 limbs/eyes or serious illness (permanent)	16-50 persons	
4.0		Hourly	The real engineering	More than 50 persons	
5.0	Even chance, could happen	Constantly	Fatality		
8.0	Probable, not surprised				
10.0	Likely, only to be expected				
12.0					
15.0	Certain, no doubt				

\*HRN = LO x FE x DPH x NP

Fig. 1. Default values.

– *OiRA* risk assessment that helps to quantify and prevent risks, which contains two main parts: a list of the risks in work activity and an action plan to prevent them. It is a tool used to assess health and safety risks in the workplace. It helps identify and assess a particular activity's working conditions, addresses the most frequent hazards, and explains how to avoid them. Addresses the risks to workers, including working with the machine and machinery used in each occupational

sector. It provides a step-by-step solution for risk assessment, starting with identifying workplace risks, then guiding the user through implementing preventive actions and ending with risk monitoring and reporting (Eu-Osha, 2023).

(5) semi-quantitative (intuitive) – *Simplified Method*: serves to quantify the amplitude of the risks and the hierarchy in the intervention. The starting point is the detection of non-conformities. Using the simplified risk assessment system, it identifies hazards, evaluates, prioritizes, and controls risks associated with activities and processes to determine those that can be tolerated and not tolerated. Checklist and Risk Assessment (e.g., MARAT, NTP330) are used. – *Integrated Method*: Identifies hazards, values, evaluates, ranks, and controls risks. Estimates probability, exposure and consequences. Calculates residual risk. Justifies the construction of the machine economically (Almeida et al., 2018; Melo & Carvalho, 2011).

(6) quantitative/qualitative – *Event Tree Analysis*: helps to identify which events may follow an initiating event; studies emergency control systems; Starts with the failure of a system component. Identifies a possible data sequence; it can enter numerical values and evaluate quantitatively. It must be known the process well (Barros, 2013). – *Fault Tree Analysis*: It starts with the damage and continues with the causes that may have caused it, calculates the probability of occurrence of primary or intermediate events, and allows the analysis of systems. Allows revealing critical failures and a more complete understanding of the system; determines the most critical sequence; finds the combination(s) that need to be prevented; can be used to assess many failures; does not need to reach a quantitative analysis. Requires knowledge of Boole's algebra and an experienced team; may need to perform a preliminary study (Barros, 2013).

(7) qualitative / semi-quantitative (intuitive): Targets the unsafe act; Identifies hazards through task analysis (Santos, 2020).

- A customer, in the operational phase of the machine, can perform risk assessment using different methods, depending on the application: (A) *quantitative method - Risk Assessment and Occupational Accidents - MARAT* - allows the level of existing risks to be determined and consequently coherently order them to establish priorities for intervention on them (risks). The simplified guiding method in which a comparison between the probability of a detected failure and the estimated probability level can be established based on accident records and statistical methods. No absolute values are used. Scales are used with levels of risk, probability of event and consequence, which makes it essential to choose the number of levels to be used to make it easier

to differentiate situations and locate appropriate levels. (B) *Qualitative method – DOUA (Direct Observation of Unsafe Acts)*: enables the Identification of unsafe acts committed by workers while performing their tasks; Use of checklists; Analysis of positions. (C) *Semi-quantitative (intuitive) William T. Fine*: enables the Identification of hazards and the ranking and control of risks. Estimates probability, exposure, and consequences and justifies actions economically.

### 3. Case Study

#### 3.1. Problem approach and objectives

Knowing a machine's hazards and risks is fundamental to identifying the laws to be respected and the standards to be used. Thus, it is essential to identify the risks of a machine before it is marketed to prevent the health and safety of people. The most crucial thing in industrial safety is to make a rigorous risk assessment. How to do it still generates distinct ideas and some questions in an industrial environment. Despite national and international methodologies and standards (e.g., EN 12100:2010) to guide risk assessment and help manufacturers solve design, solve and report identified risks intrinsically.

So, the overall objective of this case study is to present practices to improve the application of safety functions in industrial manufacturing machine production. The secondary objectives are: (1) To understand the legislation to be applied to the constituents of a machine; (2) To know the hazards that machines can provide; (3) To understand the dangers that operators may be exposed to; (4) To study the methods that allow minimizing risks and dangers; (5) Elect skills in the application of standards and laws; (6) Build tools to assist in the correct application of the safety component; (7) Develop simple and effective solutions to minimize risks and dangers; (8) Present an acceptable solution to use by the industrial machine builders.

#### 3.2. Risk assessment in the machine design phase

The managers of a design are in close and permanent contact with the customers throughout the lifetime of the design and the machine. Engineering supports from idea to production. When designing, manufacturing, and marketing machines, it is necessary to demonstrate that they meet all the requirements and demands of the country where they will be used. A series of laws, Europe-wide laws, called directives, must be complied with. In the design phase, it is proposed to evaluate management, technology, customers, suppliers, and partners, as well as external factors such as cultural and economic issues. This assessment allows us to quantify the risk (low, medium, high) and its consequences

(negligible, design carried out with deviations, design not carried out). This results in the determination of the level of risk. According to a proposal presented, the stone processing machine was assessed to validate the assessment in the design phase (Fig.2).

Project Risk Assessment											M.43.02
											Version 1
Project Name:		Risk			Consequence						
		0.25	0.5	0.75	0.25	1	1.5				
Group	Risk Description	Relative Weight	Low	Medium	High	Unavoidable	Project carried out with deviations	Project not carried out	Risk Level		
Management	Managing Director Leave the Company								0		
	Project Leader Leave the Company								0		
	Market potential								0		
	Activity that limits the continuity of the project								0		
Technology	Project Value (the value of the risk is proportional to the value of the project)								0		
	Level of adequacy of existing technologies to those needed in the Project								0		
	Level of appropriateness of technologies mastered by the Company								0		
	Scale-time								0		
Customer / Suppliers /	Changing the project's objectives by the Client/User Partner (History)								0		
	Delay of the Technological Partner in the development of activities								0		
	Delay of the Client/User Partner in the development of activities								0		
	Delay of the Specifying Partner in the development of activities								0		
Business Context (external)	Client/User Partner Stability considering the partner's investment								0		
	Accessibility of Technology Partners								0		
	Customer Awareness of Partner/Supplier								0		
	Supplier's delay in delivering the product/service								0		
Business Context (internal)	Sub-TOTAL								0		
	Complexity in the implementation applicable to the product								0		
	Complexity in transport								0		
	Ability of the user sector								0		
TOTAL	Geographic proximity of the Partner								0		
	Level of innovation culture and predisposition to risk of the Client/User Partner								0		
	Sub-TOTAL								0		
	TOTAL								0,00		
Scale:		Date of Assessment:		Risk Weighted Level							
Minimum 0,63		Evaluation carried out by:									
Maximum 11,25											

Fig. 2. Proposal to use at the design phase.

Generically, the scale ranges from 0.63, since not all risks are possible to eliminate but rather minimize, to a maximum of 11.25. These values were adapted based on the Katz scale created in 1963.

#### 3.3. Application of the HRN method: Construction phase

A risk assessment on a machine under construction involves identifying all possible risks associated with using the machine. This includes considering possible mechanical, electrical, physical, and environmental hazards. After assessing the risks, it is important to evaluate the probability of that risk occurring and the severity of its damage. Finally, measures need to be taken to eliminate these risks wherever possible.

The application of the HRN risk assessment method encompasses several processes, including risk assessment, determining machine limits, hazard identification, risk estimation, risk assessment, verification that the machine is safe, and, if not, recommending improvements (e.g., design modifications, protective measures, signage, PPE, or safe procedures). To facilitate future risk assessments, a flowchart was built (Fig.3) with all the processes for applying the HRN assessment method. It was chosen to keep the colours in the HRN values in the flowchart to visually help understand the state of the risk assessment, where in the worst-case scenario (black -  $500 < x < 1000$ ), one must redo the machine and in the best-case scenario. The SISTEMA software (Safety Integrity Software Tool for the Evaluation of Machine Applications) is used to design, calculate, and demonstrate that the safety functions are correct. A free tool created by the ISO 13849-1/2 standard authors. The

German Institute of Labour, it is an international harmonized standard.

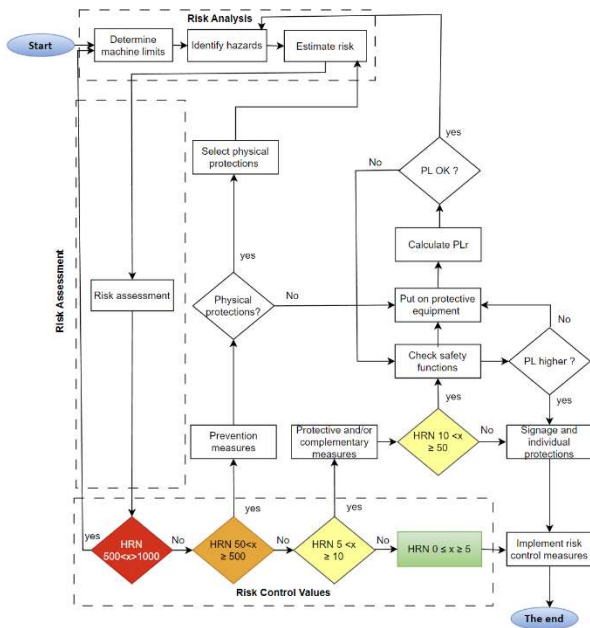


Fig. 3. Proposal to use the HRN method.

This tool can be used in constructing any machine during the development phase of the 3D model when all functional zones and the whole operating process is being developed and during the final assembly phase, the testing and validation of the equipment. For a correct risk assessment of the machine, it is necessary to record the information for the risk assessment. Training for a few hours is needed to understand how to interpret the alarm messages. A practical advantage for the user is that every parameter change is immediately reflected in the user interface, impacting the entire system. After completion of the risk estimation, risk assessment was performed to determine whether risk reduction is necessary. If risk reduction is necessary, prevention measures must be selected and applied. The applicability of the risk reduction should be determined after that of the risk reduction measures.

During this interactive process, the risk assessment team also checked whether additional hazards were created, or other risks increased when new prevention measures were introduced. If additional hazards do occur, they should be added to the list of identified hazards, and the prevention measures that address them should be applied. Meeting the risk reduction objectives and achieving a favourable risk comparison result demonstrates that the risk has been adequately reduced (ISO12100, 2010).

Risk reduction can be achieved by eliminating hazards separately or simultaneously reducing the elements that determine the associated risk. These risk reduction

measures should be applied following a sequence known as the three-step method (ISO12100, 2010).

Step 1: Intrinsic preventive measures - eliminate hazards or reduce associated risks through appropriate choice of machine design principles and interaction between exposed persons and the machine.

Step 2: Protective and complementary preventive measures - considering the intended use and foreseeable misuse, protective and complementary preventive measures can be taken to reduce the risk when it is not possible to eliminate a hazard or sufficiently reduce the associated risk by using intrinsic preventive measures.

Step 3: Information for use - where risks remain after intrinsic prevention measures, protective measures and complementary prevention measures have been applied, the residual risks should be identified in the information for use.

It is expected that this proposal, presented in the flowchart, will facilitate the risk assessment done by industrial machinery manufacturers. Following the flowchart, three steps are then proposed that serve as a guide for performing this type of risk assessment on a machine:

• *First step:*

Risk Assessment				
Review:				
Project:	Machine:	Client:	Date:	/ / 20
<b>Dangerous Areas</b>				
Zone	Name	Description	Localization	
1				
2				
3				
4				
5				
6				

Fig. 4. First step: identifying the hazardous zones of a machine.

So, the first part is to fill out a document (Fig.4). The paper has the data necessary for the Identification of the risk analysis recorded, and we will identify, if possible, all accident-prone areas, risk areas, for machine operators, maintenance technicians and other operators who may be in the surrounding area.

This first part is always common to all risk assessments done on the machine during its design, unless changing or redoing some functional area may create a new hazard.

• *Second step:*

The assessment begins (Fig.5).




Risk Assessment									
Project: Machine: Client:								 Review: _____ Date: / / 20	
HRN (Hazard Rating Number)									
Assessment data									
Zone	Danger	Description	Risk	LO	FE	DPH	NP	HRN	Improvement Actions
1									
2									
3									
4									
5									
6									

Fig. 5. Second step: HRN Method.

In this step, it is crucial to correctly identify the type of hazard, how it is constituted, and its associated risk. This data will influence the assessment values when the HRN.

• **Third step:**

In the last part of the machine evaluation (Fig.6), we can see the risk assessment summary, which describes the risk assessment phase carried out after the functional and structural development in the 3D model of the equipment.

Risk Assessment Results		Made by:	Verified by:	Approved by:
Signature: _____		_____	_____	_____
Date: / / 20		/ / 20	/ / 20	/ / 20
Project: Machine: Client:				
HRN (Hazard Rating Number)				
CAD 3D				
Evaluation phase				
Operation phase				
Nature of dangers				
Hazard impact				
Observations				
Risk estimation		Actions to minimize risk		Norms
LO				
FE				
DPH				
NP				
HRN				

Fig. 6. Third step: results.

Thus, the description of the risk assessment is performed in the 3D structural and functional development phase of the machine to complete the development by contemplating the safety elements for its physical construction. The other parameters, Operating Phase, Activity, Nature of Hazards, and Impact of Hazards are also important in summary but are only changed during the development of the machine if there is a drastic change in the design. Thus, in the second and third parts of a risk assessment, the information records will always differ due to the appropriate interventions during machine development. The last part of the proposed document contains the evaluation method values (Fig.1) to make the selection of values for each parameter of the HRN method formula more intuitive.

**3.3. Stone processing machine**

**3.3.1. StoneCUT®mill**

The StoneCUT®mill is a (CNC) Computer Numerically Controlled machine (Fig.7). The stone processing

machine was used to test and validate the risk assessment proposal.



Fig. 7. StoneCUT® mill machine.

The CNC StoneCUT®mill is an innovative and ecological system, industry 4.0 designed to provide the following key benefits:

- (1) Full capacity operating as cutting machine:
  - Tilting system for slab loading;
  - Vision system to scan slab defects and quality zones;
  - Automatic head tilting 0-90°;
  - Five interpolated axes to make profiles;
  - Automatic cutting software
- (2) FULL operating as a milling machine:
  - Automatic tool exchange;
  - Automatic head tilting 0-90°;
  - Five interpolated axes;
  - Milling 3D software;
  - Vacuum cups positioning assistance software.
- (3) Industry4.0 functionalities:
  - Web server to operate in "Internet of Things" mode;
  - Direct connection to office/smartphone/tablet;
  - Online technical support.

**3.3.2. Limits of machine**

The limits of use include various aspects, such as intended use and reasonably foreseeable misuse. Since there is only one mode of operation on this machine, the initial startup and shutdown limit the procedures to only two. All others required for proper operation must be performed by qualified persons (e.g., manufacturers, service technicians with advanced knowledge). It only involves risks for operators from other sectors with knowledge of its operation and its surroundings during its operation. The machine's dimensions can be adjusted to the area available for implementation. The range of

movements of the operators and maintenance operations in certain areas can limit the number of operators to a maximum of one. During the machine's lifetime, there is an imposition on the limit of hours of motorization operation. For these to work correctly, the manufacturer must make a technical intervention, leaving it to the customer's responsibility. The stone causes a significant deposition of waste leading to the requirement of daily cleaning of all machines so that it has a correct performance.

### 3.3.3. Identification of hazardous zones

Each area is assigned a code when identifying accident-prone areas (e.g., machine operators, maintenance technicians, and other operators who may be in the area surrounding the equipment). In the table created to perform the risk assessment, along with the codification, a brief description of each zone is presented (Fig.4) so that it is easy to identify it in the equipment and consult the information that identifies the type of danger. In this way, the filling out the evaluation tables is quicker and more enlightening.

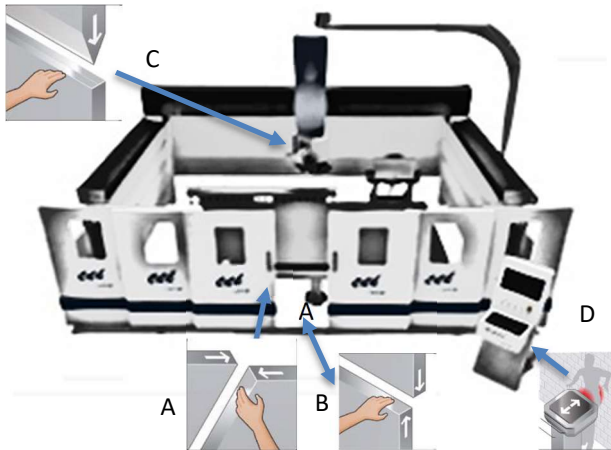


Fig. 8. Codification at CAD 3D StoneCUT® mill machine.

Five danger zones are identified precisely: (A) Open and close de door; (B) Feeding and Parts exit; (C) Cutting motion; and (D) Work-related musculoskeletal injuries. In identifying hazards, one must consider unintentional operator behaviour or reasonably foreseeable misuse of the machine, human interactions throughout the life cycle of the machine and the interactions identified in the standard (ISO12100, 2010), such as:

- adjustment
- testing
- learning/programming
- starting
- feeding the machine

- product removal from the machine
- stopping the machine
- stopping the machine in an emergency
- restarting after jamming or blocking
- restart after an unplanned stop
- troubleshooting/troubleshooting
- cleaning and tidying up
- preventive and corrective maintenance.

Considering the possible interactions and operation, it is concluded that the hazards identified in the various zones are mechanical, with different consequences depending on the zone.

### 3.3.4. Risk assessments

With the results obtained, depending on the risk obtained, together with the technicians involved in the design, the measures to be taken to reduce the risks are. In this phase, it is fundamental to register the actions to eliminate the hazard and to present a summary in the Hazard Identification table, which exposes the descriptive: when the machine was developed, in which mode of operation accidents can occur, description of the operator's activity, what type of hazards are associated, the impacts of the hazards on the operators and finally a brief description of how the risk assessment was done. The maximum risk of the machine is identified by the zone in which the HRN value is highest; these values are presented here.

The risks of this machine are musculoskeletal injuries and entrapment of the hand.

The risks of this machine are musculoskeletal injuries and entrapment of the hand precisely when:

- Loading the slab into the machine (Fig.9).



Fig. 9. Slab loading.

- In processing operations if the operator tampers with the safety functions of the machine (Fig.10).



Fig. 10. Stone cut.

- When changing tools because the machine (Fig.11) door opens for the robot to transfer the tools, and some person approaches when it is running.



Fig. 11. Tool Exchange.

- And when and when the cut stone comes out (Fig.12).



Fig. 12. Parts unloading

Finally, the proposals for risk reduction are registered. To support the use of the proposals and so that the full development of the machine results in a safe machine, the standards to be used in the construction must be registered too.

### 3.3.5. Results

In the Design phase, the machine has a weighted risk of 5.59 (Fig.13).

Project Risk Assessment										M.43.02	
										Version 1	
										2019/July	
Project Name:		StoneCUT® MILL		Risk			Consequence				
				0,25	0,5	0,75	1,0	1,25	1,5		
Group	Risk Description	Score Weight	Low	Medium	High	Intelligent	Project can not be carried out with Deviations	Project can not be carried out	Risk Level		
Management	Managing Director Leave the Company	1	x						1,125		
	Project Leader Leave the Company	1		x					1,25		
	Market potential	1					x		1,25		
	Activity that limits the continuity of the project	1						x	0		
Project Value (the value of the risk is proportional to the value of the project)		1							0,625		
SUB-TOTAL		5	GROUP WEIGHT			1		500%	0,625	2,00	
Technology	Level of adequacy of existing technologies to those needed in the Project	1	x						0,375		
	Level of appropriateness of technologies mastered by the Company	1		x					0,375		
	Know-how	1			x				0,375		
SUB-TOTAL		3	GROUP WEIGHT			1		500%	0,375	1,25	
Customer's Competence / Motivation	Changing the project's objectives by the Client/User Partner (history)	1	x						0,25		
	Delay of the Technological Partner in the development of activities	0		x					0		
	Delay of the Client/User Partner in the development of activities	1			x				0,375		
	Delay of the Specifying Partner in the development of activities	0				x			0		
	Client/User Partner's skills by considering the partner's investment	1					x		0,625		
	Accessibility of Technology Partners	1		x					0,375		
	Existence/Awareness of Partner/Supplier	1			x				0,625		
Supplier's delay in delivering the product/service		4	GROUP WEIGHT			3			1,875	0,94	
External Groups (contractual)	Amendment to the legislation applicable to the product	1		x					0,75		
	Complexity in transport	1			x				0,75		
	Stability of the user sector	1		x					0,375		
	Geographic proximity of the Partner	1			x				0,25		
	Level of innovative culture and predisposition to risk of the Client/User Partner	1				x			0,25		
SUB-TOTAL		5	GROUP WEIGHT			1		100%	2,125	0,25	
Scale:								TOTAL		Risk Weighted Level 5,59	
Minimum 0,63										Date of Assessment: jul/19	
Maximum 11,25										Evaluation carried out by: Agostinho de Silva	

Fig. 13. Design phase.

Then, we performed risk quantification in the construction phase using the HRN method. The values (Fig.14) were adapted to CNC machines.

LO – Likelihood of Occurrence	NP- Number of Persons	FE – Frequency of Exposures
0,033 Almost impossible Can occur under extreme circumstances	1,000 1-2	0,001 rarely
1,000 Highly unlikely But can occur	2,000 3-7	0,500 Annually
1,500 Impossible Although inconceivable	4,000 8-15	1,000 monthly
2,000 Possible But not usual	8,000 16-50	1,500 semi-annually
5,000 Any Chance It can happen	12,000 > 50	2,500 daily
8,000 Likely No surprise		4,000 hours
10,000 very likely Expected		5,000 constant
15,000 Certainty Without a doubt		

HRN	Result	Risk	Assessment
0-1	Acceptable	Very Low	Consider possible actions Maintain protective measures.
1-3	Low	Low	Ensure that protective measures are effective. Improve with complementary actions.
5-10	Significant	High	Actions should be taken to reduce or eliminate the risk.
10-50	Very High	Very High	Ensure that protective guards or safety devices are implemented.
50-100	Extreme	Extreme	Reduce the equipment. Interrupt the activity until the risk is eliminated or reduced.
100-500	Unacceptable	Unacceptable	
500-1000			
> 1000			

Fig. 14. Quantifying risk using the HRN method.

To conclude, this evaluation was used at the customer's facility in the machine installation's final testing and validation phase. The machine used to test and validate the proposal presented (Fig.15) to serve as a model for other manufacturers, according to the HRN evaluation, presents an acceptable risk (HRN  $0 \leq x \leq 1$ ).

Risk Assessment Results		Made by:	Verified by:	Approved by:
Signature: _____		_____	_____	_____
Date: ___/___/20__		___/___/20__	___/___/20__	___/___/20__
Project: XYZ Machine: StoneCUT® Mill Client: XPTO				
HRN (Hazard Rating Number)				
Evaluation phase	CAD 3D			Assembly
Operation phase	[Diagram]			Normal automatic operation
Nature of dangers	[Diagram]			Cleaning and maintenance with / perifer
Hazard impact	[Diagram]			Mechanical hazards
Observations	[Diagram]			Physical hazards that may cause injury to operators or others who approach the machine during normal operation. This evaluation was used at the customer's facility in the final testing and validation phase of the machine installation.
Risk estimation	Actions to minimize risk			Norms
LD	0,003			EN 349
FE	0,1			EN 415 4
DPH	2			EN ISO 12100
NP	1			EN ISO 13849-1
HRN	0,0006			EN ISO 13853
	It was advisable to apply signage and mark the floor. Provide the client with a list of safe procedures for cleaning operations or maintenance interventions to be performed without accidents			EN ISO 14120

Fig. 15. Construction phase.

The physical risks that could cause harm to operators or others approaching the machine during regular operation were analyzed. HRN = 0.0006, an acceptable value, is advisable to apply signage and mark the floor and provide the client with a list of safe procedures for

cleaning operations or maintenance interventions to be performed without accidents.

#### 4. Conclusions

Ensuring the correct operation of a machine at the time of its final assembly is essential to ensure that all elements are correctly designed. Failure to confirm the interconnection between all moving parts and that there is no interference between the various designed parts can compromise the operation and safety of machines. Thus, all parts are developed to fulfil the function for which the machine was designed. Pay attention to all this importance. The most important thing is to ensure the machine's safety of the operator and the environment where the machine will operate.

Structural safety requires calculating the forces and moments of inertia that the machine structure is subjected to so that all the cinematics do not come loose and hit an operator. Mathematical calculations indicate that what is being applied is resistant. This makes it legal to use, proves that the machine is within safe

parameters, and does not create a new risk. Sometimes these calculations are made by an external company which provides the service.

Creating safe procedures is one of the procedures that most often fails when building machines. It is generally taken for granted that manufacturers have the experience and capacity to guarantee the safety of the equipment—fundamental skills in their construction and use. In developing a new machine design, it is essential to consider safe procedures that apply to both manufacturers (construction phase) and users (operational phase of the machine).

For a correct risk assessment, there must be a team rather than just one person doing the risk assessment. A risk assessment has to be done by at least four people, who play different roles, one to perform the function of the machine user, another for the maintenance function, who knows the technical part, another for the process engineering function because he is the person who knows the layout of the production process, and the Superior Technician of Safety at Work, an expert in the prevention of occupational risks.

**Acknowledgments:** The authors appreciate the Inovmineral4.0 Project - *programas mobilizadores clusters de competitividade e outras dinâmicas coletivas* - Portugal 2020 14/SI/2019, for the support on their work.

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# PROMOTION OF ORNAMENTAL STONES QUARRIED IN THE NORTH AND SOUTH AEGEAN REGIONS OF GREECE USED IN LOCAL ARCHITECTURE

Konstantinos Laskaridis<sup>1\*</sup>, Angeliki Arapakou<sup>1</sup>, Michael Patronis<sup>1</sup>, Christos Papatrechas<sup>1</sup> and Ioannis Kouseris<sup>1</sup>

(60) Hellenic Survey of Geology and Mineral Exploration (HSGME), Lithos Laboratory, Acharnae, Athens, Greece, \*[laskaridis@igme.gr](mailto:laskaridis@igme.gr)

**Summary:** *In the Aegean Islands of Greece there have been rich natural stones' deposits of internationally recognized high quality since ancient times, which have played a key role in the islands' economic growth. The use of local stones in regional construction projects have contributed substantially to the sustainable architecture of the islands. The scope of the paper is to overview the importance of these stones and link traditions and local use to their characteristics and availability presented in an Atlas. This Atlas provides scientific information about the relevant geological setting, petrography, photographic record depicting applications of each extracted stone, and corresponding results derived from the laboratory tests for determining their physical-mechanical properties and their mineralogical and petrographic characteristics, which have great impact on the architectural design.*

**Key words:** *Atlas, ornamental stones, Aegean Islands, local architecture*

## 1. Introduction

In the Aegean Islands of Greece there have been significant natural stones' deposits of internationally recognized high quality since ancient times. During the Neolithic period (6500 BC), stone quarrying took place in many Aegean Islands, such as, Limnos, Lesvos, Milos, etc., for the construction of "millstones" (Atlas, 2022). "Millstones" (Fig. 1) were used for cereal milling, nut and olive processing, ore crushing etc. and they have played a primary role in the economic growth and human well-being since the Prehistoric Ages.



Fig. 1. "Millstones" in Lesvos.

Then, marble trade developed in the entire Mediterranean. In addition, the Cycladic civilization has numerous archaeological finds, which prove the

extensive use of the art of "dry stone walling", as shown in Fig. 2. the term "art of drystone walling" indicates the construction of stone structures without any binding material (dry). Drystone structures can either be highly visible, as in the case of the impressive clusters of terraced cultivations, or discrete and humble, as in the case of farming structures. They are always aesthetically adapted to the environment. The art of drystone walling was inscribed on the National Inventory of Intangible Cultural Heritage on 2014 (website: Intangible Cultural Heritage of Greece) and on the Representative List of Intangible Cultural Heritage of Humanity (website: UNESCO, Intangible Cultural Heritage).

The oldest quarries in ancient Greece operated around the 7th century BC in the Cyclades, and especially on the islands of Naxos and Paros. A little later, extraction and exploitation of white marble began in Tinos, Andros and Anafi. The marble of Naxos was widely used in monuments and sculptures of the sacred sites of Delos and Delphi. Naxos' Temple of Apollo – Portara (Fig. 3), which is the island's emblem and main landmark, is a huge marble gate and the single remaining part of an unfinished temple of Apollo of 530 BC (Website: Naxos and Small Cyclades). Naxos marble (Naxos Crystallina) is used nowadays for various interior and exterior decorations, such as building façades, flooring, stair construction, as well as for the restoration of archaeological monuments, such as the Tholos in the Sanctuary of Asklepios Epidaurus, the Ancient Sanctuary of Apollo and Demeter at Gyroulas in Sagri. Fig. 4 depicts pictures taken from Naxos quarries.

The marble of Tinos has been detected in Roman Antiquities and in Middle Ages remains, while the marble of Lesvos has been used for monuments in Rome, Pompeii, Smyrna and Pergamum.



Fig. 2. The art of drystone walling in Andros and Kythnos.



Fig. 3. Naxos' Temple of Apollo - Portara.

A great part of the Aegean islands' growth from the past until today is due to the rich marble deposits. The use of local natural stones in local construction projects has contributed substantially to the sustainable architecture of the Aegean islands. The slate roofs in Ikaria (Fig. 5), the various constructions (houses, churches, etc.) built with the red stone of Chios (Fig. 6), the paving roads and residences constructed with the well-known reddish ignimbrite of Lesvos, the use of the light-grey

trachydacite of Limnos in many old and new buildings of the island, the applications of Sifnos gneiss-schist in terraces and other constructions, the stone houses, stone masonry and stone pavements in Kea and in Kythnos are some notable examples of Aegean architecture (Fig. 7, Fig. 8 and Fig. 9).



Fig. 4. Naxos' quarries.



Fig. 5. Slate roof in Ikaria.

Terraces built with stone walls by using local materials are common in most Aegean islands such as in Sifnos and Andros, as shown in Fig. 10. It is also worth mentioning that in Tinos there are a lot of pigeon houses (more than 600) which are estimated to appear during the Venetian period (1207-1715) (website: [tinosecret.gr](https://tinosecret.gr)). Pigeons, offering meat and fertilizer, were the main export product of Tinos when trade began flourishing. With the advent of the Venetians on the island the first pigeon houses were built, and the systematic breeding of pigeons began. Tinian craftsmen used the slate as a building material, and the pigeon house as a canvas to imprint excellent examples of folk art and their own architectural point of view (Fig. 11). (Source: <https://tinosecret.gr/en/pigeon-houses/>).

Most of the ornamental stones extracted in the Greek islands are suitable for interior and exterior applications, for flooring and paving, for masonry, for the construction of terraces, while some of them have been used from the past years in sculptures, furniture, decorative pieces or even jewellery.

In addition, restorations by using natural stones have offered great opportunities for contributing to a sustainable architecture, as it can be seen in the Greek islands. The unique landscape of each island, close to nature, has been achieved by the construction of stone houses and other stone design projects using local materials.

The above highlight the diachronic importance of the Greek natural stones and underline the current need for a Greek natural stone Atlas.

## 2. Atlas of Greek natural stones

The scope of the paper is to overview the importance of the Greek natural stones and link traditions and local use to their characteristics and availability presented in the corresponding Atlas.

Kythnos, Lesvos, Limnos, Naxos, Paros, Patmos, Samos, Sifnos, Tinos and Chios.

The Atlas provides scientific information for each ornamental stone, concerning name, colour and lithology, the relevant geological setting, the petrographic examination, and corresponding results derived from the laboratory tests for determining physical-mechanical properties. It contains also pictures taken from licensed quarries as well as photographs depicting applications of each extracted stone in local architectural projects.



Fig. 6. Constructions with Mesta stone and Thymiana stone of Chios.

Highlighting the qualitative and technical characteristics of the ornamental stones quarried in the South and North Aegean islands, which play a key role in the sustainable architecture of each island, is the main purpose for the creation of an Atlas of 27 different types of stones extracted in the islands of Andros, Ikaria, Kea,



Fig. 7. Applications of ornamental stones in the North Aegean islands (Limnos–Kaspakas stone, Lesbos–Styphis stone and Mystegna stone, Patmos).



Fig. 8. Applications of ornamental stones in the South Aegean islands (Paros – Gneiss schist, Sifnos – Kontou stone, Naxos – Gneiss schist, Kythnos – Andrianou stone)

The laboratory tests performed for determining physical-mechanical properties of the stones, include apparent density, open porosity, water absorption, flexural strength, uniaxial compressive strength, abrasion resistance, rupture energy, frost resistance, breaking load at dowel hole. These features have great impact on the architectural design. The ornamental stones have numerous applications considering their technical characteristics. Some of them are weather – resistant making them suitable in extreme climates, others are ideal for facades, wall cladding, due to their high breaking load at dowel hole, others are durable and highly resistant for specific construction projects.







Fig. 9. Applications of ornamental stones in the South Aegean islands (Kea – Petrousa stone, Kea – Ag. Symeon stone, Tinos – Marble of Pyrgos, Tinos – Marble of Vathi).

All laboratory tests were carried out according to the European standards (EN Standards). Specimens from the 27 types of stones were appropriately prepared in order to perform the aforementioned tests at the ornamental stones quality control laboratory of the Hellenic Survey of Geology and Mineral Exploration (HSGME), named “Lithos” Laboratory. These tests are described below:



Fig. 10. Terraces built with stone walls in Sifnos and Andros.

**Apparent density and open porosity:** The test procedures were conducted on 6 specimens from each stone according to EN 1936:2006. Apparent density, expressed as  $\text{kg/m}^3$ , is the ratio between the mass of the dry specimen and its apparent volume. Open porosity is the ratio (as a percentage) between the volume of the open pores and the apparent volume of the specimen.

**Water absorption at atmospheric pressure:** It is expressed as a percentage, by the ratio of the mass of the saturated specimen to the mass of the dry specimen, as described in EN 13755:2008., performed for 6 specimens of each stone.

**Flexural strength under concentrated load:** A 10-specimen set from each stone was prepared to undergo the test whose experimental procedure is described in EN 12372:2006. The average value of each set was calculated, expressed in MPa. This test is very important, especially for applications in places exposed to different kinds of load.

**Uniaxial compressive strength:** It is expressed by the ratio of the failure load of the specimen and its cross-sectional area before testing (in MPa). The procedure was carried out for 10 specimens of each stone according to EN 1926:2006.

**Abrasion resistance:** The test was performed according to EN 14157:2017 – Method B – Böhme abrasion test. The abrasive wear was determined as the loss in

specimen volume, expressed in  $\text{cm}^3/50\text{cm}^2$ , for 3 specimens of each stone.

**Rupture energy:** It was determined for 6 specimens (in joules), by the dropping of a spherical steel ball from given increasing heights until specimens broke, as described in EN 14158:2004.

**Frost resistance:** 10-specimen sets from all stones start their freeze–thaw cycling tests in a saturated condition according to EN 12371:2010. Each cycle consisted of a 6h freezing period in air from  $+17\text{ }^\circ\text{C}$  to  $-12\text{ }^\circ\text{C}$ , followed by a 6h thawing period in water from  $-12\text{ }^\circ\text{C}$  to  $+17\text{ }^\circ\text{C}$ . Specimens were subjected to 48 cycles. After completion of the cycles, visual inspection was carried out to score their behaviour and then flexural or uniaxial compressive strength was determined in compliance with EN 12372:2006 or EN 1926:2006.



Fig. 11. Pigeon house in Tinos built with Tinos stone of Aghia Kalloni.

**Breaking load at dowel hole:** This test, conducted for 10 specimens of each stone, consists of applying a force in a direction perpendicular to the face of a specimen through a dowel previously placed in a hole drilled in one of its sides and measuring the breaking load of the specimen (expressed in N), as defined in EN 13364:2002. This test should be performed in cases where natural stones will be used for cladding or lining in buildings.

The calculated average values of the results from the above physical-mechanical tests are given in Table I. The average values of the flexural strength under concentrated load (flex.) or the uniaxial compressive strength (un. comp.) after exposing the specimens to 48 frost resistance cycles are shown in the last column.

Petrographic examination was carried out according to EN 12407:2019, in thin sections prepared for samples of the 27 stones. Macroscopic description included the general colour, the rock structure, the grain size, macroscopic cracks, pores or cavities, evidence of weathering or alteration, etc. Microscopic description depicted the constituents of the stones, such as

percentage of minerals and grains, groundmass, discontinuities. Fig. 12 illustrates a microphotograph of a thin section of Andros stone, as it is presented in Atlas.

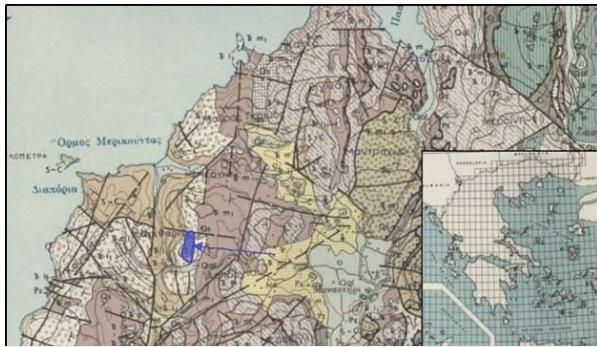


Fig. 12. Microphotograph of a thin section of Andros stone.

Andros stone is a greenish chlorite-epidote-actinolite gneiss-schist. Chlorite and epidote are responsible for its greenish colour. According to the petrographic examination the rock texture is granoblastic to lepidoblastic, its fabric is schistose to folded, characterized by slaty cleavage (schistosity). It consists of chlorite (56%), feldspars (25% - albite, orthoclase), quartz (6%), muscovite (5%), epidote (4%), actinolite (4%), and of accessory minerals: zircon and apatite. Chemical analysis demonstrated percentages of the main elements composing Andros stone, such as: 49% of  $\text{SiO}_2$ , 22% of  $\text{Al}_2\text{O}_3$ , lower percentages of  $\text{Fe}_2\text{O}_3$ , MgO, CaO, LOI,  $\text{Na}_2\text{O}$ ,  $\text{TiO}_2$ ,  $\text{K}_2\text{O}$ , MnO, and of the trace elements (i.e., V, Mn, Cr, Co, Ni, Cu, Zn etc.) expressed in mg/kg.

The name of each natural stone was defined according to EN 12440:2017, which corresponds to a particular type of rock with a specific place of origin while its type (lithology of the stone) was described based on EN 12407:2019.

The geological setting contained in Atlas provides the relevant geological map and information about the geology of the area where the ornamental stone is quarried referring also to the geological age and unit, based on the geological maps of Greece, created by the formerly Institute of Geology and Mineral Exploration of Greece (IGME - the new HSGME). An example of the geological setting presented for Mesta stone of Chios island is illustrated in Fig. 13.



**Geology:** Basal limestones: well-bedded, medium to dark-grey and occasionally red. At the base marly. Sandy transition to the basal sandstones and basal conglomerates. Syngenetic breccias and pseudoitic limestones abundant. Locally intercalations of thick dolomite beds. Thickness: variable, maximum 120m. Fossils: lamelibranches, gastropods, conodonts.

(Source: Geological Map of Greece 1:50000, CHIOS SHEET – SOUTHERN PART)

**Geological age:** Skithian – Lower Anissian

**Geological unit:** Basal limestones

Fig. 13. Geological setting as presented in Atlas for Mesta stone of Chios.

The quality control of the natural stones achieved through study of their properties, enables the prediction of their behaviour over time. The study of their mineralogical and petrographic characteristics determined by macroscopic and microscopic examination, describes their aesthetic image. All these scientific information is proved to be crucial for a stone type in order to specify its potential applications.

### Conclusions:

The paper overviewed the diachronic role of Greek natural stones in the Aegean islands highlighting their importance for local construction. The continuation of traditional methods for construction of new Buildings and renovation of existing ones makes necessary the creation of an Atlas compiling all characteristics of these natural stones. The paper summarized the contents of the “Aegean natural stone Atlas” that can aid designers, constructors, and architects in choosing the correct stone for any application.

Table I: Average values of the physical – mechanical properties of each ornamental stone determined in “Lithos”.

Stone name	Physical – mechanical properties								
	Appar. density (kg/m <sup>3</sup> )	Open poros. (% vol)	Water absorp. (% wt)	Uniaxial Comp. Strength (MPa)	Flexural Strength (MPa)	Break. load at dowel (N)	Abrasion. Resistance (cm <sup>3</sup> /50cm <sup>2</sup> )	Rupt. ener. (J)	Frost resist. after 48 cycles, Flex. or uniax. comp. strength (MPa)
Andros Stone	2840	1.9	0.5	47	18.5		18	8	19.9 (flex.)
Kyparissi Stone of Ikaria Island	2700	0.4	0.2	73			19	4	55 (un. comp.)
Arethousa Slate of Ikaria Island	2680	3.0	1.1		23.1		10	11	29.4 (flex.)
Aghios Symeon Stone of Kea Island	2690	1.0	0.3	66	33.7		31	9	25.3 (flex.)
Petrousa Stone of Kea Island	2680	1.5	0.5	31	24.0		9	13	25.0 (flex.)
Aghioi Anargyroi Stone of Kea Island	2700	1.0	0.3	39	20.9		13	8	22.6 (flex.)
Dryopida Stone of Kythnos Island	2620	3.2	0.8	66	25.1		12	12	24.4 (flex.)
Andrianou Stone of Kythnos Island	2700	0.7	0.2	140					158 (un. comp.)
Stypsis Stone of Lesbos Island	2180	15.2	4.6	62	10.3		9	5	89 (un. comp.)
Mystegna Stone of Lesbos Island	2190	13.2	4.4	96	18.3		6	4	20.1 (flex.)

Kaspaka Stone of Limnos Island	2300	10.9	4.4	83	19.6		13	5	20.2 (flex.)
Vigla Stone of Limnos Island	2380	11.2	3.6	81	11.0		13	7	10.1 (flex.)
Naxos gneiss-schist	2700	1.3	0.4	66	17.7		7	8	18.8 (flex.)
Naxos Crystallina	2710	0.3	0.1	55	9.4	1500	23	4	8.3 (flex.)
Paros Stone	2610	2.2	0.8	125	21.9		9	7	23.1 (flex.)
Paros Semi-white	2700	0.3	0.1	63	13.4	2100	16	4	12.2 (flex.)
Stone of Patmos Island	2420	4.4	1.5	138	19.0		9	4	17.9 (flex.)
Ash-Gray Banded marble of Samos Island	2710	0.4	0.1	128	20.6		16	4	17.7 (flex.)
“Kontou” Stone of Sifnos Island	2720	1.1	0.6	81	23.2		7	9	21.5 (flex.)
Tinos Stone of Ysternia	2680	1.1	0.3	66	15.9		26	8	12.5 (flex.)
Grey marble of Pyrgos Tinos	2710	0.5	0.2	131	15.8	1850	16	4	13.9 (flex.)
Red-white marble of Vathi Tinos Island	2710	0.5	0.2	89	10.3	1700	21	5	9.6 (flex.)
Tinos Stone of Aghia Kalloni	2720	1.7	0.4	65	18.6		9	14	18.2 (flex.)
Marlas Stone of Tinos Island	2930	1.2	0.4	97	29.9	2600	10	7	28.1 (flex.)
Mesta Stone of Chios Island	2700	0.5	0.1	51			14	6	66 (un. comp.)
Reddish-Orange Thymiana Stone of Chios Island	2230	18.9	7.0	56	13.2		27	4	16.8 (flex.)
Reddish Thymiana Stone of Chios Island	2280	17.6	6.4	55	14.6	1500	22	4	17.1 (flex.)

**Acknowledgments:** The authors gratefully acknowledge financial support from the Greek “OPERATIONAL PROGRAMME: COMPETITIVENESS, ENTREPRENEURSHIP AND INNOVATION 2014-2020 (EPAnEK)”, PROJECT OPYGEK, SUBPROJECT 3: Actions for the exploitation of Primary and Secondary Raw Materials (DYOPY).

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# SUSTAINABILITY AND DURABILITY OF URBAN STONE PAVING: A PROPOSAL FOR AN INNOVATIVE APPROACH FOR DESIGN, CONSERVATION AND REDEVELOPMENT WORKS

G. Signori\*

(61) Mapei SpA, \*[g.signori@mapei.it](mailto:g.signori@mapei.it)

**Summary:** *Durability and sustainability are the main final requirements of stone paving.*

*The first answer to sustainability issues is to ensure a long 'life' for the paving, i.e. durability. Then, the key aspects (stresses and performance characteristics of the stratigraphic package) shall be identified and taken into account at the design stage.*

*According to current and/or mandatory regulations, the characteristics of each element of the stratigraphic package are determined by means of rupture tests, which investigate the limit state of the individual layer, but unfortunately do not provide any information on fatigue resistance or on the behaviour of the package as a system of interacting layers undergoing continuous stresses during life-service.*

*Thanks to a specific software, an innovative approach has been developed for carrying out numerical analyses aimed at defining the performance level of different paving packages*

**Key words:** *stone paving, durability, sustainability, fatigue, stratigraphic package*

Urban squares and streets in the heart of historic city centers serve multiple functions: crossroads of people, they have often guided the development of the city, and with the colors, materials and textures of the paving contribute strongly to the deep identity of a city.

In many cases, moreover, stone paving lends beauty and value to the urban landscape (Fig. 1).



Fig. 1. St Peter's Basilica in Rome, with the square paved with sanpietrini, the technique used for the most important roads in Rome since the end of the 16th century.

Furthermore, in addition to the aesthetic and identity value related to the historical tradition of places, stone pavements must also ensure maximum social sustainability, i.e., be usable for all categories of citizens, including those with reduced mobility.



Fig. 1. Slab and cobblestone paving in the historic center of Bergamo Alta. In addition to the aesthetic and identity value related to the historical tradition of the places, stone pavements must also be socially sustainable, that is, inclusive, and allow their use by all categories of citizens, including those with reduced mobility with wheelchairs and strollers

That is why it is so important to make, preserve and safely maintain stone paving.

Today, stone vehicular paving must meet very demanding and burdensome requirements due not only to weathering, but also to continuously increasing needs and stresses related to traffic, wear and tear, and mechanized maintenance operations.

The UNI 11714-1 Stone floor, wall and ceiling coverings – Part 1: Instructions for the design, installation and maintenance [1], published in 2018, introduces a technical approach to the choice of the stratigraphic package according to both the use (i.e., load class) and the type of stone paving to be laid, and accordingly indicates durability, sustainability, maintainability,

cleanability and safety of the work as the final decisive requirements.

#### **Durability of natural stone coverings according to UNI 11714-1.**

Section 5.3 of UNI 11714-1 states durability as the final requirement for cladding, as follows:

*Durability is mentioned among the general requirements of stone coverings, and corresponds to an important expectation of the user. This is significantly conditioned by the intended environment and the operating conditions, as well as the nature, thickness and finish of the stone material, the support and the laying. Given the variability of the intrinsic characteristics of different stone materials, their properties are to be assessed with attention, according to the intended use and chosen finish, in order to ensure the expected durability.*

*Durability shall be ensured during the design phase, following clause 7 of this standard, the choice and specification of the stone materials based on a comparison between the mechanical, chemical and thermal-hygrometric stresses associated with the intended environment and the foreseeable operating conditions (7.2.1 and annex B) with the corresponding properties of the materials, according to the specific product standards and indications given in clause 6. The durability of a covering is also influenced by the laying quality and correctness, according to the instructions given in clause 8, as well as the quality and correctness of maintenance (9 and annex E).*

#### **Sustainability of natural stone coverings according to UNI 11714-1.**

Section 5.6 of UNI 11714-1 states durability as the final requirement for cladding, as follows:

*Increasing sensitivity to issues of environmental protection, climate change, the limits of using non-renewable resources and energy demands is oriented not only to building design and construction but also the installation of stone coverings, starting from the choice of products and materials which guarantee measured and declared performances in line with key parameters of environmental sustainability and environmental quality.*

*For example, the following indicators may be mentioned:*

- *recovery and recycling of natural minerals;*
- *use of regional resources;*
- *reduction of carbon dioxide (CO<sub>2</sub>) emissions into the air;*

- *integrated pollution reduction (emissions into the atmosphere, water bodies, the*

*soil); as well as reductions in water consumption;*

- *reduced use of substances that are harmful to health and the environment.*

- *quality of indoor air (environmental cleaning of chemical and biological pollutants);*

- *improved energy efficiency;*

- *end-of-life recycling of products and materials.*

*In relation to the above-listed indicators, natural stone, like other constituent products of stone coverings, are qualified for their excellent positioning in terms of sustainability: this positioning is partly associated to the intrinsic properties of natural materials, and partly determined by the commitment towards and developments in increasingly sustainable processes and products over the past few years, above all in Italian industry.*

*For the choice of covering materials and products in the design phase (natural stones, adhesives, grouts, etc.) preference shall be given to those with clear and measurable environmental sustainability indicators declared by the manufacturer.*

#### **Sustainability and durability of stone paving.**

The binomial sustainability+durability expressed by UNI 11714-1 is one of the key points also for the CAM regulation, i.e. the minimum environmental requirements defined by the Ministry of the Environment on the basis of GPP (Green Public Procurement), aimed at guiding public administrations towards a rationalisation of consumption and purchasing, providing indications to identify the best design solution, product or service from an environmental point of view, throughout the life cycle, thus with a view to conservation and maintenance, according to market availability.

Ensuring the "longevity" of the paving is undoubtedly the first form of saving in terms of sustainability: in fact, it means using public funds more frequently, reducing the consumption of environmental resources and limiting the social impact of maintenance, also in the interest of the citizens, who will be able to enjoy the asset without interruption for ongoing work.

The key point is therefore durability, with the identification and management of the key aspects that contribute to this already in the project.

In fact, in the design phase, which is a complex of material, stratigraphic, functional and construction

choices, it is appropriate to dimension the pavement in relation to the conditions with which it will have to coexist during life service.

### **The destination classes of external paving**

The classification of the various types of external paving depends on the accesses, uses and destinations of the paving, which are defined in point 6 of UNI 11714-1.

The classes for external paving, P4, P5, P6, P7, P8 and P9, are defined in accordance with appendix A of UNI EN 1341 and apply to the following intended uses:

- *Class P4* – exclusively pedestrian, residential external paving, e.g.: balcony, terrace, patio, courtyard, residential paths, exclusively pedestrian pavements
- *Class P5* – public/commercial external pedestrian paving and cycle paths, e.g.: external terraces of restaurants, bars, exclusively pedestrian public footpaths and cycle paths, etc.
- *Class P6* – residential/public paving with occasional vehicle traffic, e.g.: pavements used for parking, driveways, Car park or ramp/drive for garages; courtyards and areas connecting buildings and car parks
- *Class P7* – pedestrian paving, markets used occasionally for deliveries and emergencies, e.g.: Squares occasionally used by light and heavy vehicles at reduced speed (sacristies, cemeteries, etc.), Market squares and loading/unloading areas, squares used for events, festivals, etc.
- *Class P8* – pedestrian paving often used by heavy vehicles, e.g.: 30 km/h areas, urban streets and squares with restricted traffic, public car parks, public access ramps
- *Class P9* – road and street paving, e.g.: urban streets, road with preferential lanes for public transport or obligatory routes, highly trafficked roads, roundabouts, humps and speed ramps.

The UNI 11714-1 standard prescribes to identify the expected stresses and the maximum driveability, taking into account that class P4 is a 'pure pedestrian', i.e. it does not in any case contemplate the transit of vehicles with wheels such as bicycles, motorbikes and, of course, rescue and cleaning vehicles.

In design, therefore, it is essential to provide for the heaviest possible degree of transits on the paving once it is in operation: for example, flush pavements, where there is no physical barrier preventing vehicles from passing over them, must inevitably be designed according to the same transits as the road, or time-based LTZs, which must be sized according to the actual amount of transit with the greatest impact, regardless of temporary restrictions on opening to traffic.

### **Stone paving stresses**

In order to better qualify the carriageway classes, the UNI 11714-1 standard includes in Appendix B the Classification and analysis of the operating conditions, in particular in tables:

- B.1 Main stresses for internal and external coverings
- B.2 Pavings: analysis of stresses and requirements for different intended environments
- B.5 Main variables to consider in relation to the intended use of pavings
- Main variables to be considered with respect to intended use for horizontal coverings.

### **The definition in the design phase of the operating conditions of stone paving: normative indications and an innovative approach for numerical analysis**

Usually, this activity is concentrated in the verification of the expected stresses (environmental conditions, magnitude of the loads, emergencies and constraints on the stratigraphy due to pre-existences, etc.) to be cross-referenced with the performance characteristics of the elements making up the stratigraphic package, such as the geotechnical properties of the ground, those of the subgrade, the bedding, the stone elements and the joints.

According to current and/or mandatory standards, the characteristics of each element of the stratigraphic package are determined by means of fracture tests, which investigate the limit state of the individual layer, but unfortunately do not provide indications regarding two decisive aspects

1. the fatigue strength, which is most likely the stress with the greatest impact on the durability of the system.
2. the behaviour of the package seen as a system of collaborating layers continuously subjected to stress throughout the life of the paving.

In the absence of test methods dedicated to investigating these two aspects, Ciri Edilizia e Costruzioni - Centro Interdipartimentale di Ricerca Industriale dell'Università Di Bologna, has developed numerical finite element models for performing numerical analyses aimed at defining the performance level of various paving packages. In particular, three-dimensional solid models with about 350,000 finite elements have been realised, while the type of load adopted is the concentrated load scheme no. 3 referred to in Ministerial Decree 17/01/2018 for road loads (NTC2018).

The numerical analyses carried out were aimed at defining the performance level of two different types of stratigraphic package, both for slabs (40x80x4 cm) and



for cubes (10x10x10 cm): the first type adopts a pre-blended cementitious mortar screed with high mechanical strength and high resistance to environmental factors (MAPESTONE TFB60 by MAPEI S.p.A., 7 cm thick for slabs, 5 cm thick for cubes), bonded with grouted joints using pre-blended cementitious mortar with high mechanical strength and high resistance to environmental factors (MAPESTONE PFS, 7 cm thick for slabs, 5 cm thick for cubes), 7 cm thick for the slabs, 5 cm thick for the cubes), combined with joints grouted with pre-blended cementitious mortar with high mechanical strength and high resistance to environmental factors (MAPESTONE PFS, joint width 1 cm); the second type uses a classic sand and cement screed as the bedding layer (7 cm for the slabs, 5 cm for the cubes) combined with water-cement grout for grouting the joints (1 cm).

The main performance properties used in the analyses are summarised in Table I.

The numerical analyses were conducted on three-dimensional portions of pavements with approximate dimensions of 5x5 m<sup>2</sup>, founded on a thickness of approximately 4.5 m of soil. The different materials were modelled using three-dimensional 'brick' type elements, hexahedral at 8 nodes for the pavement package and tetrahedral at 4 nodes for the soil.

The load of 150 kN, concentrated on a load area of 40x40 cm<sup>2</sup>, by means of a non-linear incremental static analysis, was applied to the model considering a load multiplier  $\lambda$  varying from 0.1 to 2.0. Therefore for  $\lambda=2.0$  the vertical load applied to the model is 300 kN (twice the normative indications). The numerical models and subsequent finite element analysis were developed using MidasFEA 2016 v1.1 software and Midas FEA NX 2020 v1.1 software.

Through simulation, it was possible to compare the behaviour of the different packages considered. In particular, the effect of the increase in load intensity on vertical displacements, horizontal, vertical and tangential stresses, horizontal and vertical deformations, and above all, the overall crack pattern was considered.

The results of the first simulations showed the following information:

- a. the great difference in the behaviour of packages based on traditional mortars, which show significant deformations and cracks at an early stage, compared with packages based on pre-mixed mortars (MAPESTONE System), which, given the same external stresses, have lower stress levels.
- b. From the point of view of cyclic loading (fatigue), the lower tension variation under load of premixed mortar-based floors highlights their capacity

to resist a greater number of cycles before manifesting mechanisms such as cracking or detachment. This emerges with a view to making use of S-N fatigue curves such as Wohler's.

c. The critical points from which the greatest positive deformations and package cracks arise are the interface between the stone element's rib and the grout filling (Fig. 2). Based on this information, a specific product was formulated in 2020 for grouting the joints of architectural stone pavings, capable of improving the adhesion of the joint filler with the stone. The product is a pre-mixed cement mortar with high mechanical and environmental resistance that is characterised by a low elastic modulus combined with a tear strength three times greater than the other products in the line and normal grouting products. The first 40 months of experimentation using this product (MAPESTONE PFS2 FLEX, approximately 80,000 m<sup>2</sup> of paving) confirm that the path indicated by the simulations corresponds to the real contexts.

d. - the contribution, whether positive or negative, of the geotechnical characteristics of the load-bearing soil, which is combined with the packages: in the case of soils with poor geotechnical properties, these "command" the durability of the package regardless of the composition of the paving package, it being understood that deformations and cracking occur in a short time in packages based on traditional mortars and in a longer time in packages based on premixed mortars.

At the moment, the results are to be interpreted in qualitative and semi-quantitative form, pending the conducting of a capillary experimental campaign on full-scale samples aimed at the direct verification of what emerged from the theoretical and "limited" study of the packages under investigation.



*Fig. 2. The typical alteration of stone paving, with the filling of the joints deteriorating and crumbling: this is how the disruption of the entire paving package is triggered.*

## Conclusions

The simulation makes it possible to compare different packages by investigating vertical displacements, horizontal, vertical and tangential stresses, horizontal

and vertical deformations, and above all, the crack pattern.

This approach, innovative in its kind, is configured as a preliminary design support to ensure durability and limit the maintenance needs of architectural stone floors.

These initial numerical results obtained from the calculation reports will be followed by an experimental phase on full-scale samples aimed at empirically

verifying the interesting ideas that have emerged, some of which have already been applied through the formulation of specific products consisting of pre-mixed cementitious mortars with a low elastic modulus and high tear resistance (3 times higher than comparative pre-mixed cementitious mortars) for grouting the joints of architectural stone paving.

Layer element	Elastic modulus	Compressive strength	Flexural strength	Tensile stiffness
	E (GPa)	fcm (MPa)	ftm (MPa)	Gf, tensile (N/mm)
Granite slab	42	200	16	-
Porphyry cube	57.9	180	12	-
Bedding: Mapestone TFB60	28	55	8	0.2
Grout filling: Mapestone PFS2	28	55	8	0.2
Fibre-reinforced concrete slab (20 cm)	29	30	4	0.1
Bedding: Sand-cement	10	15	0.3	0.02
Grout filling: Cement grout	10	5	0.1	0.02

Table I. Summary table of the main performance properties used in the modelling of the various elements of the stratigraphic package.

**Acknowledgments:** I'm very grateful to prof. Claudio Mazzotti from Ciri Edilizia e Costruzioni - Centro Interdipartimentale di Ricerca Industriale dell'Università di Bologna for his collaboration, support and help.

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**POSTER SESSION  
CONFERENCE PAPERS**

## DIGITAL TRANSFORMATION OF THE NATURAL STONE SECTOR

Ana Sofia Amaral /Marta Ferreira

### ABSTRACT

The CTCV - Technological Centre of Ceramic and Glass, with Assimagra - Portuguese Association of Marbles, Granites and Related Branches developed a project called “The Stone4.0 Age - a Digital Transformation of the Stone Sector”, whose objective was to support SME of the natural stone sector in the incorporation of industry 4.0 concepts and practices, empowering companies and providing digital tools adjustable to the reality of each organization.

There is a perception in the sector of the advantages of digitizing tools, namely:

- Reduction of time associated with data management and processing;
- Creating flow and increasing speed of response in providing the service or product;
- Guidance for the implementation of continuous improvement practices focused on adding value;
- Greater control and specificity of industrial costing (maintenance, production and human resources domains);

The project began with a benchmarking study on the use of digital tools among companies in the sector, analyzing their applicability and scalability, covering extractive, processing, trading companies or the combination of these activities. The benchmarking was supported by a questionnaire to diagnose the state of maturity of companies in the natural stone sector towards the challenges of industry 4.0 and digitalization and, in this way, to better understand their reality and needs.

The topics covered were:

- General Characterization of Companies
- Legal and Regulatory Compliance
- Digitization of the Production Control System or Quality Management System
- Technological Resources/ Digital in the Company
- Qualitative Cost Structure
- KPI (management, production, maintenance)

The companies in the sector, which may already have production control systems in place, have some gaps, namely in the requirements associated with quality management and organizational support processes, requirements that are highly valued by their customers. These gaps need to be minimized, so that the variability of the products available to the market is reduced and so that the quality requirements can meet the increasingly demanding requests of customers. It is necessary to strengthen the production control systems with improvement tools in order to make the performance more agile and quick, so that the process is as efficient as possible.

Customizable digital tools were developed, in the form of guides, with individualized links for each tool. The i4.0 Digital Tool Guides for Production Control and Quality Management Systems intended to respond to the requirements of continuous improvement of the global efficiency of processes to create value, optimize their potential and drive companies towards the digital use of documentation, with the ease of access, communication to the team and action, which they provide in a format that facilitates their analysis.

The digitization of documents and records allows access to a high volume of data in real time, its knowledge is an ally of integrated management and contributes to faster and more informed decision-making.

# SELECTIVE AND SUSTAINABLE EVALUATION OF ORNAMENTAL ROCK RESOURCES BY OBTAINING A THREE-DIMENSIONAL DEPTH MODEL BASED ON THE APPLICATION OF SEISMIC METHODS AND DATA MINING TECHNIQUES

A. Espín<sup>1\*</sup>, M. Reyes<sup>1</sup>, A. Gil<sup>1</sup>

<sup>1</sup>Marble, Stone and Materials Technological Centre, road of Murcia. Cehegín (Murcia) Spain. \*[antonio.espin@ctmarmol.es](mailto:antonio.espin@ctmarmol.es)

**Summary:** *In the Monte Coto exploitation of the company Mármoles Coto S.L. There are important unknowns about the deep structure of the minerable layer, so the main objective is to extend the current knowledge of the limestone structure to an approximate depth of 300-400 m from the surface. To achieve the proposed objectives, the development of a deep seismic campaign is proposed using two seismic profiles of refraction and wide-angle reflection. The refractive seismic profiles will be complemented by tomography measures that will generate models of propagation velocity distribution of P waves (VP) in the subsurface from the analysis of the first arrivals (P waves).*

*The results obtained confirm the presence of the limestone layer in depth up to a depth of 360 m, ensuring important reserves of material, so the following phases of this project are very necessary once the layer in depth is secured. A decrease in the dip of the layer has been observed, important when evaluating the exploitable reserves in the future.*

*Exploitable material reserves have been demonstrated below 357 m level, it's necessary to demonstrate the quality of the materials, both physical-mechanical as commercial. To take advantage of the existence of old boreholes, were recognized by means of optical televiewer and spectral gamma probes. As the result of the obtained data, a log was made where the different materials have been described and characterized, including with identification of the microfossils they contain.*

*From different laboratory tests as well as from drill hole data that have been incorporated into the 3DEC program, information has been obtained for the analysis of the stability ratio for exploitation-depth of exploitation. With that information, a stable model has been developed that will not collapse, thus ensuring the operation for the next 21 years, detecting small instabilities that will be corrected during the work. These results allow make block models of the quarry and the different exploitable layers, measure the volume to extract per layer and selecting the new exploitation method.*

**Key words:** *geophysical, seismic, model 3D, probes, 3DEC.*

## Introduction and objectives

The Monte Coto quarries has a long history about ornamental rock exploitation and has many importance due to the characteristics of the deposit. It's a structurally complicated area due to the influence of the movement of the Mula-Fortuna-Crevillente fault, there are outcrops linked by thrusts and faults, being from different palaeogeographical domains, materials from the External Subbetic and the Palaeogene limestones from the lower-middle Eocene belonging to the Internal Prebetic.

The quarry, owned by Mármoles Coto SL, has unknowns about the structure at deep of the mineable layer, so the main objective is to extend the current knowledge of the limestone structure to a depth of approximately 300-400 metres from the surface.

The overall objectives of this work are i) obtaining an accurate 3D geological model down to 400 m depth; ii) Evaluation of existing stocks; iii) Optimisation of

deposit resources and increase the tailings valorisation; iv) Development of a three-dimensional economic model of the reservoir in terms of quality and location of reserves; v) Methodology development for the in-depth study of sites.

To achieve all the objectives of the project, the investigation has been approached in different phases, aimed at obtaining the necessary information for reservoir design and exploitation. Specifically, the following workflows have been applied: deep refraction/reflection wide-angle seismic; deep down-hole seismic; laboratory analysis for the collected samples; reservoir modelling using borehole test probes data and stability analysis of the operation.

## Study area

The studied area is located to the SE of Pinoso, on sheet No. 870 (Pinoso) of the national topographic map, scale

1:50,000 of the I.G.N. in the Municipal District of Pinoso in the "Monte Coto" area (Fig. 1).



Fig.1.- Situation of the study area.

Present materials in this area belong to the External Zones of the Betic Mountain range. During the Mesozoic, these materials formed part of the sedimentary cover of the southern continental margin of the Iberian plate, where two main domains can be distinguished: the Prebetic Zone and the Subbetic Zone. These areas are subdivided into Inner Prebetic and External Prebetic for the first zone, and Inner Subbetic, Middle Subbetic and Outer Subbetic for the second. (Gallego *et al*, 1981).

As a general description of our study area, there are an anticline structure with NE-SW direction and 8 km<sup>2</sup> long formed by massive Paleogene fossiliferous limestone corresponding to the central inner Prebetic subdomain, from the Lower-Middle Eocene. It's composed by a package of between 50 m and 200 m in depth and with a dip of 45-70°, of cream limestones with remains of microfauna of nummulites, milliolids, gypsinids, discocyclines, echinids, red algae, coralline algae and alveolines. On the top, these materials have a marlier character.

The quarry extracts a marble limestone (medium and light background limestone) of high ornamental value, commercially known as "Crema marfil Coto". It is a compact, cream-coloured nummulitic limestone of carbonate sedimentary origin, petrographically described as biosparite, composed of foraminifera and irregularly distributed calcite veins. (Herrera Herbert, 2018).

According to the geological mapping, the cream limestone formation has a series of green marl limestones on the bottom and a series of marl limestones and limestones of no commercial value at the top. The interest layer of limestone is approximately 140 m thick and has a dip of 70 degrees at the current mining face.

## Methodology

Different seismic methods and techniques were applied in the area, as detailed below:

### -Deep refraction/reflection wide-angle seismic

The main objective of applying this technique is to extend the current knowledge of the limestone structure (direction, dip, presence of discontinuities...) to a depth around 400 metres from the surface. The refraction seismic profiles will be complemented by tomographic models that generate models of P-wave propagation velocity distribution (VP) in the subsurface from the analysis of the first arrivals (P-waves).

The equipment used for the acquisition of these seismic profiles were:

- Texan seismic stations (US company REFTEK): thirty-five stations were installed and were connected to geophones with a nominal frequency of 4.5 Hz and protected by plastic bags (Fig. 2).



Fig. 2.- TEXAN station with vertical component geophones.



Fig. 3.- Taurus stations used in the study.

- Taurus seismic stations (American company Nanometrics): ten stations were installed and were equipped with GPS signal for accurate time corrections. They were connected to three-component and low frequency geophones of 1.0Hz nominal frequency. The stations were housed in a thermal enclosure (Fig. 3) to protect them from the elements.
- Guralp stations (American company Nanometrics): five stations were installed and were connected to three-component and low-frequency geophones with 1.0 Hz of nominal, model LE-3Dlite of the Lennartz brand (Fig. 4). The stations were encased

in a thermal enclosure to protect them from the weather.



Fig. 4.-Three-component Lennartz LE-3DLite geophone.

– Trimble 5800 Centimetre Accuracy GPS Total Station: all reading points and blasts were stationed using a high precision centimetric GPS with a base at a high point in the quarry. The points have been obtained from the ERVA network of the Valencian cartographic institute in UTM projection ETRS89 ZONE 30 and geoid EGM2008 REDNAP of the National Geographic Institute in absolute coordinates.

Six blasting (B1 – B6) points were programmed for each seismic profile acquired and their position was based on the known geometry of the limestone layer of interest and the limitations using explosives within the quarry enclosure.

For each blast, 10 m long holes were drilled, each hole was lined with PVC to ensure its integrity and filled with 3.57 kg of explosive gel which was handled by qualified quarry technicians.

The data processing was made with OCTAVE and MATLAB softwares (The MathWorks Inc., 2022), and the interpretation of the results was carried out by technicians of Marmoles Coto S.L. and the Centro Tecnológico del Mármol, Piedra y Materiales (Marble, Stone, and Materials Technological Centre).

#### **-Deep down-hole seismic**

In this phase, the objective is to determine the dynamic elastic modules from the analysis of the interval velocities and the average velocity, measured from the P-wave and S-wave arrival times. These waves propagate from the surface and go downwards to seismic sensors installed vertically within the borehole. Down-Hole seismic is also done on two old boreholes in the quarry.

The components of the equipment used were:

- Sledgehammer: the seismic source used was a 6.5 kg sledgehammer impacting on a Teflon plate for the vertical strikes and on the edge of a reinforced timber beam (Fig. 5) for the horizontal strikes.
- Borehole geophone: a multi-component seismic receiver placed inside a stainless-steel tube was

used. This seismic receiver has 4 horizontal sensors and one vertical sensor.

– Seismograph: a Summit Compact seismograph was used to collect the digital signal from the sensors, combine it and transmit it to a portable field computer.

– Disparador: the sledgehammer is equipped with a digital trigger to accurately determine the impact moment and initiate seismic recording.



Fig. 5.- Metal plate and sledgehammer used for the generation of seismic waves.

Down-Hole assays were acquired by lowering the borehole geophone every 5.0 m depth along both boreholes. At each station, the borehole geophone was placed by means of a rubber plug that fixed the sensor to the formation walls. To fix the geophone was used a constant pressure of 1bar measured by a manometer from the surface. In the borehole 1 (Fig. 6), data were acquired at 34 reading points, and in borehole 2, were recorded at 27 points, distributed every 5 metres depth, from the surface.

The multi-component geophone inside the borehole was connected to a field computer for the data acquisition software and the borehole geophone was attached to the surface by a hollow kevlar tube which housed the data transmission line.

The data processing was made with programmes developed in OCTAVE (Eaton, 2002) and PYTHON (van Rossum, 1995).



Fig. 6.- Location and measurements in borehole 1.

### -Stability and reservoir modelling analysis

Starting with the geophysical survey where different probes was used, logging some physical parameters and 360° digital and oriented pictures of the borehole wall in the whole drillhole continuously. This information collected is processed with specific software to obtain structural data of reservoir.

The probes were used are: QL40 OBI 2G optical televiewer which allows to obtain important lithological and structural data, identifying the different discontinuities *in situ*, and Gamma Ray QL40-GRA gives information about the lithology overall on the argillaceous levels and the clay concentration in the materials.

For the stability analysis and due to the complex geometries and the potential instabilities, the better tool is 3D numerical modelling in discontinuous environments to simulate intra and inter blocks mechanism.

The used methodology is detailed below:

- Obtention of the elasto-plastic parameters of the geotechnical unities through the available boreholes data.
- Analysis of data from geomechanical stations, borehole joint descriptions and laboratory joint strength tests to estimate the distribution and shear strength of discontinuities and their scale effect.
- 3D numerical modelling and calculation of the factor of safety using the shear strength reduction technique for both the current situation and the planned design.
- Analysis of the results and reinterpretation of the shear strengths of the discontinuities, 3D numerical modelling and calculation of the factor of safety of the current situation and the planned design, according to the new assumptions adopted.
- Reinterpretation of the present discontinuities and redesign of the final slope, 3D numerical modelling and calculation of the factor of safety of the present situation and the planned design according to the new hypotheses and proposed designs.
- Inclusion of the seismic acceleration of the area in the calculations.

First, a precise topography is carried out by drone flight and the geology of the study area is projected on the orthophotomap. Then the stability study of the initial state and the newly designed final shaft is developed. In addition, the initial data, the calculation criteria adopted, the design model and calculation assumptions are described. The analyses have been implemented in the 3DEC finite difference zones method (Itasca, 2020).

### Results and discussion

The main results of the project are detailed below.

#### -Deep refraction/reflection wide-angle seismic

Two seismic profiles were obtained, they have 3.400 m long, with direction 143°N and 60 m separated (Menke, 1989). One of which is from refraction seismic, and the other is from wide-angles seismic that goes parallels across the quarry. In Figure 7, shows one ray tracing for blasting.

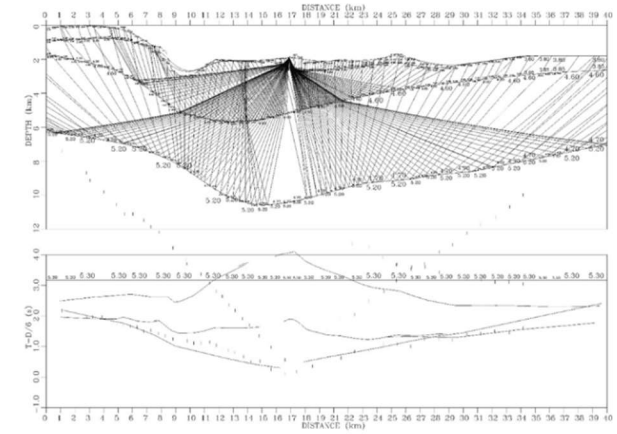


Fig. 7.-Ray tracing for blasting B6; (below) calculated running times (continue lines) and observed (vertical bars).

As of the interpretation of the ray tracing and the adjusted measured and observed running times the following P waves velocity model (Fig. 8). Based on this model, three discontinuities have been detected through the identification of the reflexion families. Remarkable the discontinuities with a maximum depth cote of -220 m with P waves velocities between 5.2 and 5.3 km/s. The other remarkable discontinuity with a maximum depth cote of -500 m and with P waves velocities arranged 5.5 and 6.5 km/s.

With the tomographic inversion process, a P waves seismic velocity model (Fig. 93) has been obtained, using the firsts observed arriving times (reflected waves) (Socco, 2008; Telford, 1991).

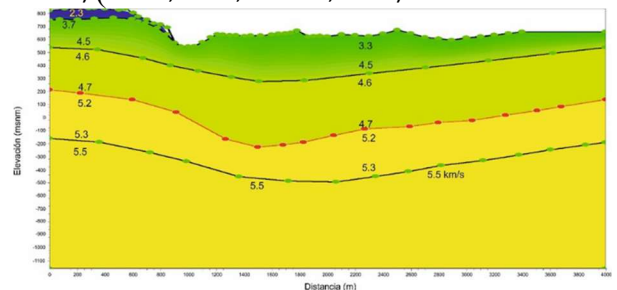


Fig. 8.- P waves velocity model expressed in km/s.



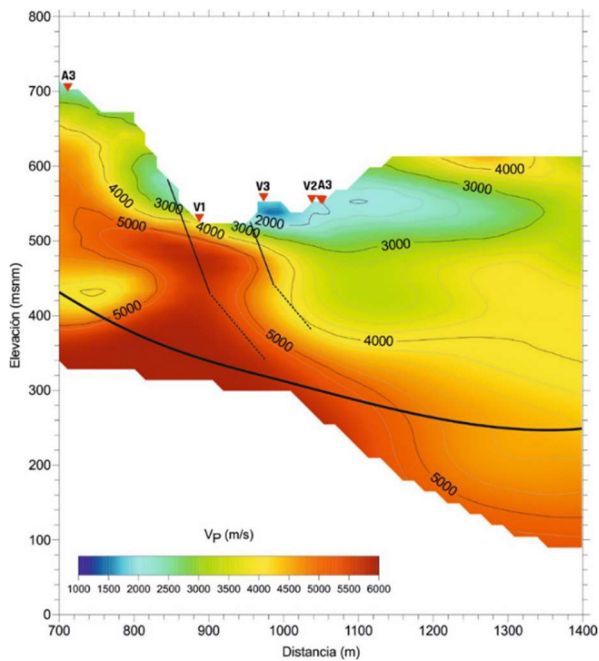


Fig. 9.- Detail of P waves seismic velocity model.

This model presents P waves seismic velocities, between 1000 and 6000 m/s. The velocities distribution allows determine top the strong dip of the geological structures that are present in the quarry area.

In the model, can identify low velocities (<1800 m/s) related with the fills areas of the quarry.

The studied limestones, appears as a high velocity anomaly (4000 - 5000 m/s). The material located at the top and bottom (loams and loamy limestones) has velocities between 1800 and 3000 m/s. That high velocity anomaly shows a decreasing in the dip of the limestones at depth.

#### -Deep down-hole seismic

The main results obtained by the two Down-hole essays are described below. The borehole 1 essay reached a depth of 165 m, obtaining measured points each 5 m and generate 34 arrivals both from the P and S waves. The Figure 10 shows the interval velocities for the P and S waves and indicates a soft increasing tendence for both waves.

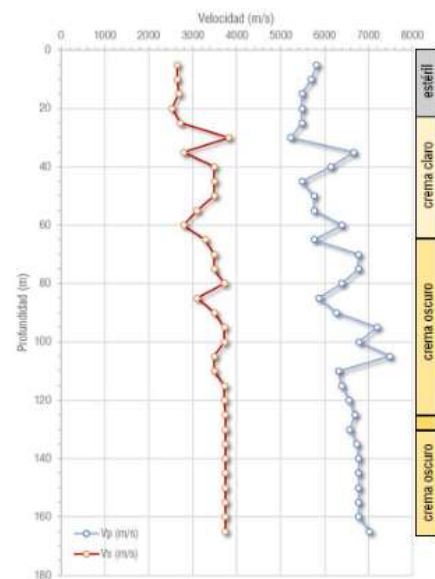


Fig. 10.- Interval velocities for the P (blue) and S (red) waves.

Interval velocities are very vulnerable to small errors in the arriving times determinations. By this, the layer velocities are also calculated by lineal regression the observed arriving times. The table 1 shows the calculated lineal regression of the arriving times and its depth, according to the described lithologies observed in the boreholes and the quarry. Using the layer velocities of table 1 and assuming a material density of 2.670 Km/m<sup>3</sup>, the dynamic-elastic module values are obtained and shown in table 2.

Lithology	Depth (m)	V <sub>P</sub> (m/s)	V <sub>S</sub> (m/s)
Top mine tailings	0.0-23.7	5.575	2.630
Cream limestone	23.7-65.5	5.883	3.237
Dark-cream limestone	65.5-126.7	6.600	3.563
Dark-cream limestone	126.7-166.8	6.790	3.740

Table 1.- Seismic velocities obtained by lineal regression in the borehole 1.

Lithology	Depth (m)	$\sigma$	G (GPa)	E (GPa)	K (GPa)
Top mine tailings	0.0-23.7	0.36	18.5	50.1	58.4
Cream limestone	23.7-65.5	0.28	28.0	71.8	55.1
Dark-cream limestone	65.5-126.7	0.29	33.9	87.7	71.1
Dark-cream limestone	126.7-166.8	0.28	37.3	95.8	73.3

Table 2.- Dynamic-elastic modules of the main lithologies identified in the borehole 1.

The borehole 2 essay reached 125 m, obtaining measured points each 5 m generating 27 waves P and S arrivals. The figure 11 shows the seismic assembly of the Down-hole essay in this borehole.

The figure 12 shows the interval velocities for P and S waves. The obtained velocities are like borehole 1, and to the present lithology.

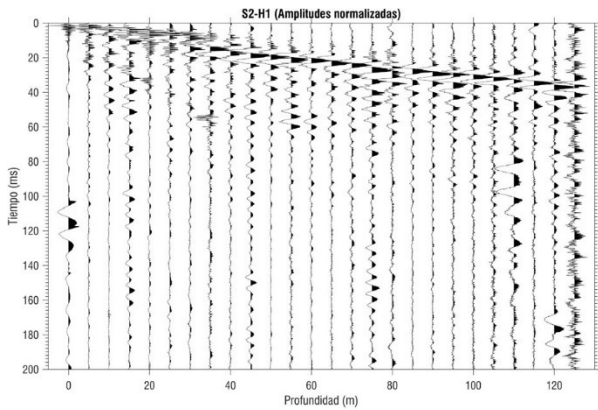


Fig. 11.- Seismic assembly of the Down-hole essay in this borehole.

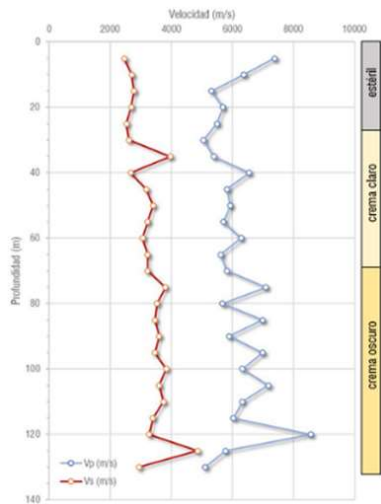


Fig. 12.- Interval velocities for the P (blue) and S (red) waves.

Table 3 shows the calculated linear regression seismic velocities as in the borehole 2. And like in the borehole 1, assuming a material density of 2.670 kg/m<sup>3</sup>, dynamic-elastic modules are obtained and shown in the table 4. (Caterpillar, 2000).

Lithology	Depth (m)	V <sub>p</sub> (m/s)	V <sub>s</sub> (m/s)
Top mine tailings	0.0-30.5	5.722	2.641
Cream limestone	30.5-71.0	5.901	3.180
Dark-cream limestone	71.0-123.0	6.478	3.590

Table 3.- Seismic velocities obtained by lineal regression in the borehole 2.

Lithology	Depth (m)	$\sigma$	G (GPa)	E (GPa)	K (GPa)
Top mine tailings	0.0-30.5	0.36	18.6	50.9	62.6
Cream limestone	30.5-71.0	0.30	27.0	70.0	57.0
Dark-cream limestone	71.0-123.0	0.28	34.4	88.0	66.2

Table 4.- Dynamic-elastic modules of the main lithologies identified in the borehole 2.

### - Stability and reservoir modelling analysis

Due to the velocity is distributed in a range of values, the maximum and minimum values were calculated. The minimum represents high alteration grades and the maximums low alteration grades.

Lithology	V <sub>p</sub> (m/s)	V <sub>s</sub> (m/s)	$\rho$ (kg/m <sup>3</sup> )	$\sigma$	G (GPa)	E (GPa)
Marl and marlaceous lime (high alteration)	1600	700	2200	0.38	1.08	2.98
Marl and marlaceous lime (low alteration)	2600	1200	2500	0.36	3.6	9.83
Clear limestones (high alteration)	3000	1700	2600	0.26	7.51	18.99
Clear limestones (low alteration)	4500	2500	2700	0.28	16.88	43.09

Table 5.- Dynamic-elastic modules of the materials present in the quarry.

P waves values are obtained from the seismic velocity model (Fig 10), and the S waves velocities form horizontal components of the three-components stations. That values are shown in table 5 and used to simulate future possible scenarios of the quarry.

The obtained image by Televiewer probe is processed by WellCAD™ software (Advanced Logic Technology). Figures 13 and 14 shows an example of the results in boreholes 1 and 2. The provided results shows the discontinuities relations, relevant to use for modelling and predict the stabilities of the rock massif.

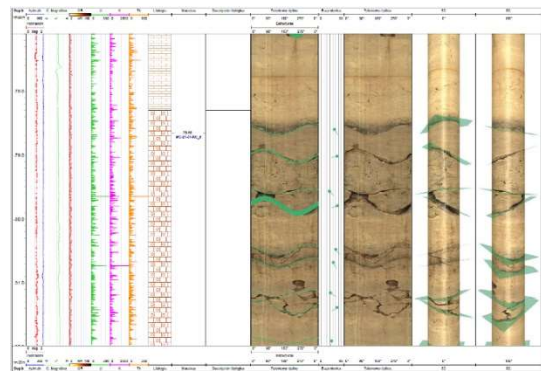


Fig. 13.- Example of processed section in borehole 1.

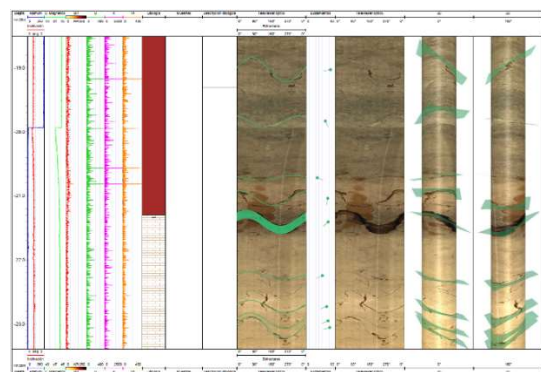


Fig. 14.- Example of processed section in borehole 2.

The layers and discontinuities mapped in the quarry are analysed, which is shown in the figure 15.

After the statistical analysis the discontinuities are grouped in two main families and five subfamilies, taking account its orientation and typology. Table 6 shows the average values for each discontinuity's families.

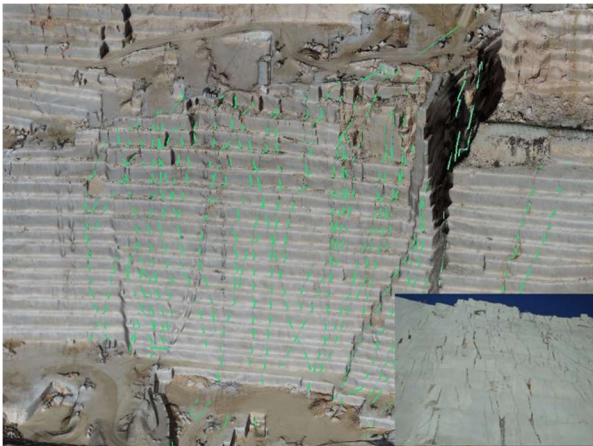


Fig. 15.- Present discontinuities in the main quarry slope, west zone. Slope detail (low-right corner).

Family	Subfamily	Orientation	Dip	Sense
J	J1	N159E	81	SW
J	J2	N152E	75	NE
S	J3	N48E	62	NW
S	S <sub>0</sub>	N47E	63	SE
S	J4	N40E	90	vertical

Table 6.- Obtained average values for the discontinuity's families.

Remarkable, the subfamily S<sub>0</sub> with dip to southwest and subparallel to the quarry front slopes can generate gravity displacements due to wedges. By this, the relation of S<sub>0</sub> and the other discontinuities subfamilies are analysed.

Bands of discontinuities J have been detected concentrated in certain areas of the quarry, crossing it from top to bottom. In each band, one of the J1 or J2 subfamilies predominates, orientating the maximum slope lines of the J planes to the north-east and others to the south-west. The S planes are laterally compartmentalized and staggered by the J family, and vertically by the J<sub>3</sub> and J<sub>4</sub> subfamilies.

With all the information, two numerical models are finally built, in 3DEC, representing the current geometry of the operation and the proposed final design. These models' dimensions are 1500 m parallel to the operation, 1500 m perpendicular to the slopes, and the lower contour has been placed 150 m below the bottom of the open pit. The calculated factor of safety (FoS) value is above 1.5 for most of the slope.

Figure 16 shows a final view of the 3D geometric model, according to the geology considered in the calculation (Varona, *et al* 1998).

Finally, a design of the final hole (Fig 17) has been carried out, which main objective is to eliminate the potentially unstable sectors detected in the last calculation hypothesis, deleting instabilities in some cases, or minimizing them by tilling in others.

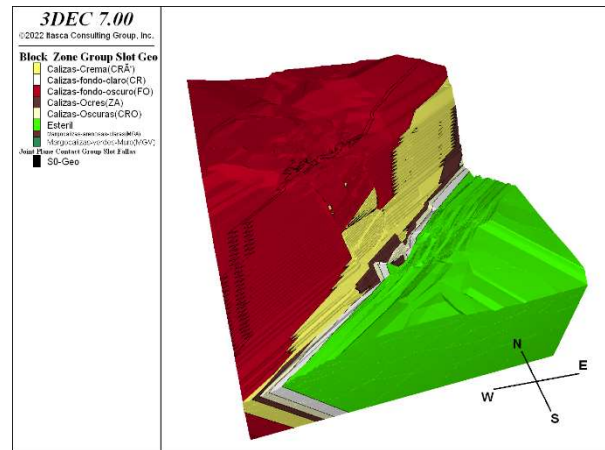


Fig.16.- View of the final design with the considered geology.

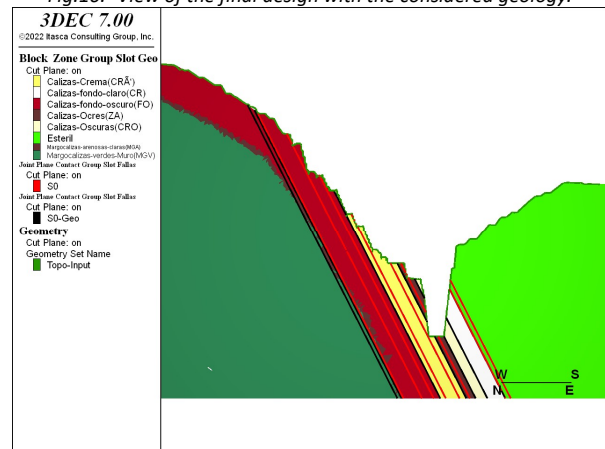


Fig.17.- Final hole design and geology with its structures.

Based on these new models and calculation assumptions, can determine that:

- No potential instabilities with FoS<1.6 are obtained for the current design model, both for the static and the seismic situation.

- In static situation, doesn't exist relevant potential instabilities with a FoS<1.6. Only some very local potential instability is observed with FoS=1.3. Whereas in a seismic situation, potential instabilities appear in the J1 family with a FoS=1.2.

After this study, the planning of slopes stability must consider the geometric relationships between the discontinuity planes of the rock massif, removing the block displacement risk by gravity. To optimize mining performance, the orientation of the benches should follow the structural directions of the rock mass, matching with the J and S families. And on the frontal slopes, the possible wedges generated by the intersection with the S-planes must be monitored and the slopes must be parallel to the direction of S<sub>0</sub>, to minimize the FoS.

On the lateral slopes, the possible wedges generated by the intersection of the J1 (western) and J2 (eastern) planes must be monitored. The intersection of these

slopes with the S0 planes must also be monitored because they can also generate instability processes. And the wedge generated in the final slope by the intersection with the J plane shall be eliminated. The ideal state is a final side slope with no intersections with J-planes (or they have been eliminated) and the blocks bordering on their lower part with an S<sub>0</sub> plane intersected by the slope, resting on the lower bank.

### Conclusions

The seismic velocity model has identified several layers that could be associated with the contacts between materials identified in the superficial geology. And it confirms the presence of a limestone exploitable layer at depths of up to 360 m, the layer shows a decrease in dip, which is important for the evaluation of exploitable reserves in the future.

The velocity distribution of the P waves seismic velocity model coincides with the observed surface dips.

The analysis of old boreholes by the probes has provided valuable information on the different families of discontinuities present and their characteristics in depth.

The calculation models implemented in 3DEC represent with high accuracy both the current design and the final design of the mining slopes, as well as the distribution of the geotechnical units present in the area.

A stable model that doesn't collapse has been achieved, so that the operation has been assured for the next 21 years, with small instabilities detected that will be rectified as the exploitation is being developed.

It's recommended that a yearly inspection of the quarry stability of the be carried out to update the geometry of the different discontinuities and layers, recalculating the safety factors.

**Acknowledgments:** This project has been possible thanks to the collaboration and equipment used by the company Mármoles Coto S.L., and financing by CDTI "Proyecto de INVESTIGACIÓN Y DESARROLLO (IDI-20201210)".

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# How can paper be produced from mineral sources instead of cellulose?

Anabela P. Massano<sup>1\*</sup>, G. Martins<sup>2</sup>, J. Fernandes<sup>3</sup>, P. Brito<sup>2</sup>, A. Mateus<sup>1</sup>

(1) Centre for Rapid and Sustainable Product Development, Polytechnic Institute of Leiria, Marinha Grande, Portugal.

\*[anabela.p.massano@ipleiria.pt](mailto:anabela.p.massano@ipleiria.pt)

(2) Valoriza, Polytechnic Institute of Portalegre, Portalegre, Portugal.

(3) MVC Mármore de Alcobaça Lda, Alcobaça, Portugal

## Summary:

Paper is a well-known material created in Asia and which production has evolved through many centuries. Traditionally, paper is obtained from processing treatments of vegetable fibers. Rapid growth trees, such as eucalyptus, are among the usual sources of fibres for papermaking.

In last decades, a new type of paper has been invented. This new material is based on calcium carbonate and polyethylene and a small proportion of other components.

The greatest difference between the conventional paper and this one is that while the former is based on cellulose, the latter is a composite based on mineral particles and a polymer as a binder. Also, this mineral-based material does not involve water consumption nor cutting trees.

PAPEPO project aims to develop an experimental strategy involving diverse characterization techniques and processing methods. It is expected to provide results that will enable finding a formulation to produce the new generation of paper.

**Key words:** Mineral paper, calcium carbonate, residues, polymers, polyethylene.

## Introduction

Paper production technology was first discovered in China around two thousand years ago.

The term "paper" that we know so far, corresponds to a thin material where most of its components is cellulose fibers, either in the form of virgin fibers obtained from wood or non-wood plants, or secondary fibers from recycled waste papers. It may also be obtained from recycling textiles made of cotton or linen, for instance.

The conventional paper making process typically starts with the defibrillation of trees through intensive mechanical and chemical processes. A complex sequence of operations is needed to separate and cook the vegetable fibres until obtaining, in a first phase, a cellulose paste. This requires the use of multiple tools and technologies in successive steps. The usual workflow involves pulp making, pulp dewatering and complex finishing phases. The whole process uses multiple chemical additives and large amounts of water. The energy consumption is very high, namely during the drying phases of the paste. In fact, sheet formation of paper is carried out on a

machine whose working principle is the process of removing water from the paper stock. It starts with the drainage and vacuum of water in a wire section. After passing through wire part of paper machine, about 80-75% of water still remain in the wet sheet before it is transferred to the press part. Herein, more water will be pressed out from the wet sheet resulting more compact and stronger sheet. Finally, with the moisture content of about 20-25%, the water in the wet sheet is evaporated using sets of dryer cylinders heated by steam.

An alternative to the conventional paper has emerged gradually around 30 years ago in Asia and today, many commercial brands are available worldwide. The new concept of paper is a material that has no trees or other natural fibres in its composition. Instead, it may be classified as a composite material based on calcium carbonate minerals and a polymeric phase that acts as a binder between the solid particles. The polymeric component constitutes, typically, only 20-40 %wt of the material. Some thermoplastic polymers, as high density polyethylene (HDPE), are usually selected because these are easily recyclable. But the major component, that can reach 60-80% wt of the material

is normally calcium carbonate. This is a very common mineral in nature and may be found at quarries of limestone or marble. The mineral based paper usually also contains a small percentage of additives that are required for the processing technologies of the production. While some additives may be necessary to obtain a convenient viscosity during processing of the material, others are related to the mechanical properties of the sheet or even to the ability of receiving ink or other writing or printing pigments. These additives may be classified as coupling agents, lubricants, plasticizers, dispersing agents and antistatic compounds, to mention the most important ones. The additives are needed so that the mineral paper resembles the conventional version as much as possible. Despite the high mineral content, that increases the stiffness of the blend, this new type of paper must be flexible and tear resistant. The polymeric components contribute to these properties, being also responsible for the water proof feature of the surface. The hydrophobicity is pointed out as advantageous regarding the spending of pigments or inks in the printing of the mineral based paper.

In addition to the advantages on the material properties, the manufacturing processes are also considered as more sustainable than the conventional ones. Mineral based paper may be produced by melt blending of the components and the obtained composite is subsequently shaped into a film, usually through blow extrusion technology. The temperature conditions, air pressure, air volume, traction speed and many other parameters must be thoroughly controlled. The thickness of the film must be appropriate for the final applications of this material. Surface coating is also mentioned as step in the processing line as a solution for printing or writing problems.

Along these last years, the chemical composition of mineral based paper has been proposed with many variations both in the mineral and in the organic phases. Some advances in processing technology are also expected to occur, as described in some patents. Anyway, even considering the inclusion of minerals as silica or magnesium and biopolymers or other biodegradable polymers in the organic phase, the fact is that the basis of the invention constitutes an important means of having a much more sustainable materials than conventional cellulosic paper.

In the present study, a commercial sample of mineral paper was analyzed for its qualitative and quantitative chemical composition. Calcium carbonate and HDPE were used to compare the FTIR vibrational spectrum of mineral paper with these pure reagents. A

thermogravimetric study was conducted to quantify the mineral and organic components of the commercial sample.

Moreover, a preliminary study of blends made of limestone residues and diverse polymers was undertaken. Different proportions were tested and the obtained materials were hot pressed into thin sheets to evaluate some of their properties. The technical details about the components of the mixtures cannot be described because of the intention of arriving to a patent at the end of the project Papepo that is at the basis of the present study.

## Materials and Methods

A commercial sample of mineral paper from a Emanagreen notebook was analyzed.

Calcium carbonate from Sigma Aldrich and HDPE from Repsol were used as references for FTIR analyses. HDPE and other polymers were used in the preparation of blends.

Residues from a limestone quarry were dried and sieved until obtaining an adequate size range for the proposed application. Particles inferior to 0.1 mm were selected to blend with the polymers.

FTIR analysis - The vibrational spectra were obtained using a Nicolet Magna IR-750 spectrometer (Nicolet, Madison, WI, USA) in the attenuated total reflection (ATR) mode. The spectra were registered in the 500–4000  $\text{cm}^{-1}$  region using 64 scans and a spectral resolution of 4  $\text{cm}^{-1}$ .

DSC-TGA Simultaneous Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) - was used to determine the content of minerals and polymers since degradation occurs at different temperatures. These tests were performed using a SDT Q600 equipment (TA Instruments, New Castle, DE, USA), under nitrogen flow at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  from 25  $^{\circ}\text{C}$  to 600  $^{\circ}\text{C}$ . Samples weighted around 6 mg.

Torque rheometry - This technique, which provides an indirect measure of the viscosity of a material, was performed using a Plastograph Brabender equipped with a mixing chamber of 55  $\text{cm}^3$ . The rotation speed was set to 60 rpm and the temperature to 150-190  $^{\circ}\text{C}$ , depending on the selected polymers.

Hot press- this equipment was used to press fragments of the materials obtained in the plastograph mixer. The materials were contained inside metallic frames of 1-2 mm thickness.

## Results

A sheet of mineral paper from a notebook of a commercial brand was analyzed. As depicted in Figure 1, it has an appearance similar to conventional paper. However, its surface is brighter and smoother. It enables writing with a pen and with a pencil.



Figure 1 – Commercial sample of mineral paper.

The vibrational spectrum of the mineral paper sample was obtained, being most of its bands readily identified by comparison with reference materials (Figure 2). The typical bands from carbonate were observed at 700, 855 and 1400  $\text{cm}^{-1}$ . On the other hand, the bands from polyethylene were observed at 718, 1485  $\text{cm}^{-1}$  and also at 2846 and 2915  $\text{cm}^{-1}$ . In the spectrum of the mineral paper, the band from the organic component at 1485  $\text{cm}^{-1}$  is overlapped by the band centered around 1400  $\text{cm}^{-1}$  from carbonate. However, another band at 1000  $\text{cm}^{-1}$  is also observed. This does not correspond to any of the two expected components, being due to an unknown reagent. This shows that the mineral based paper contains other chemicals. These can be part of the coating, in case it exists.

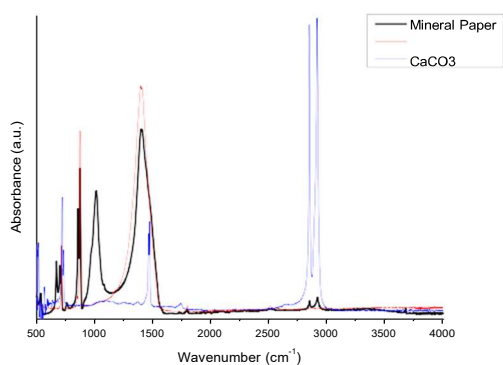


Figure 2 – Vibrational spectra of the commercial sample of mineral paper, calcium carbonate and HDPE.

FTIR was also used to compare the calcium carbonate from the residues with that from laboratory origin. The spectra were similar, despite the residues were not as white as the sample supplied by Sigma Aldrich.

To determine the amount of minerals in the commercial sample, the results from DSC-TGA (Figure 3) were analyzed. In the 25-600  $^{\circ}\text{C}$  range, the events correspond to the polymer melting at around 130  $^{\circ}\text{C}$ , followed by its thermal degradation at 400-500  $^{\circ}\text{C}$ . The remaining weight proportion was around 75% wt, which corresponds to the mineral content of the sample.

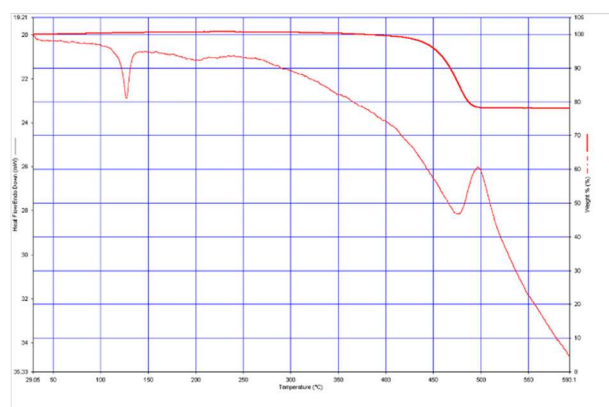


Figure 3 – DSC-TGA from the commercial sample of mineral paper.

Having these results in mind, the study to prepare a blend of minerals and polymers started with a selection of raw materials that included calcium carbonate obtained from quarry residues and also some polymers.

Several combinations were prepared through the variation of components and respective proportions. One of the objectives was to maximize the concentration of mineral residues. The mixtures were prepared under controlled conditions of temperature, volume and speed of rotation of the equipment. During the blending process, measurements of torque were obtained, being a means to monitor the viscosity and ascertain if the solids concentration was under the critical value for each tested composition. The appearance of one such blend is exemplified in Figure 4. It is notorious that the melted material has some elasticity.



Figure 4 – Preparation of mixtures in a Plastograph mixer.

After cooling the obtained materials, these were reduced to smaller fragments and these were subsequently hot pressed using square frames (Figure 5).



Figure 5 – Hot pressed mixture under thin square shape.

### Acknowledgments

This work is supported by the FCT (Fundação para a Ciência e a Tecnologia) support of CDRSP-IPLeia (Strategic Projects UIDB/04044/2020 and UIDP/04044/2020; and Associate Laboratory ARISE LA/P/0112/2020); and ANI, through the project PAPEPO – Papel Pedra Portugal; nº 69882; and finally, IPLeia (Institute Polytechnic of Leiria) and IPP (Institute Polytechnic of Portalegre).

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Patent CN101851365A - Stone paper and manufacturing method thereof

Patent CN103131145B - Completely biodegradable stone paper material and preparation method thereof

The square samples were, in general, darker than the initial mixture. It was latter observed that using a longer period of mixing at above melting temperature of the polymers, the materials became darker, too. The square samples with different thickness values showed interesting properties, mostly with some of the selected polymers at certain mineral concentrations. With others, the squares could break easily, and those combinations of materials were discarded.

### Conclusions

The present study makes a small review of the state of the art regarding mineral paper, mostly to compare it with the conventional cellulosic material. The advantages are highlighted. Regarding the experimental part, the study shows that mineral based paper is real and the information from the commercial supplier whose sample was analysed is close to their claims. Some experimental routes that were taken to develop an innovative formulation of mineral based paper are described. However, it was not possible to enter any details on this subject because of the intention to obtain a patent in the context of the project that sponsored this research.



# 3D PRINTING OF COMPOSITES BASED ON RESIDUES OF CALCIUM CARBONATE FROM QUARRIES

Anabela P. Massano<sup>1\*</sup>, G. Martins<sup>2</sup>, M. Franco<sup>1</sup>, D. Sousa<sup>3</sup>, J. Fernandes<sup>4</sup>, A. Mateus<sup>1</sup>

- (1) Centre for Rapid and Sustainable Product Development, Polytechnic Institute of Leiria, Marinha Grande, Portugal.  
[\\*anabela.p.massano@ip.leiria.pt](mailto:anabela.p.massano@ip.leiria.pt)
- (2) Valoriza, Polytechnic Institute of Portalegre, Portalegre, Portugal.
- (3) Mechanical Engineering Institute IDMEC-IST, Lisboa, Portugal
- (4) MVC Mármore de Alcobaça Lda, Alcobaça, Portugal

## Summary:

Calcium carbonate is one of the most abundant minerals on Earth and accounts for about 4% of the Earth's crust mainly as limestone and marble. Industrial extraction from quarries and transformation of these stones generates large amounts of residues that can be used in new applications. The development of innovative products and instrumental means of production may be determinant on lowering the environmental impact caused by these residues. In particular, the use of calcium carbonate particles combined with a polymeric matrix may be used in the synthesis of composite materials with properties adequate for 3D printing through a digitally controlled process. The global procedures, from stone extraction to the final products, may be optimized into a circular economy process with maximum energy efficiency and minimum waste production. This study shows some fundamental steps of this innovative production process, from the composite synthesis by extrusion to the 3D printed products.

**Key words:** Calcium carbonate, polymer, composite, 3D print, sustainability.

## Introduction

At a time when natural resources are being spent at an alarming rate, mostly in developed countries, scientists are interested in contributing to find alternatives of materials and processing technologies that may be more sustainable. The alliance between science and industry is of high importance and many different approaches may be found to meet circular economy solutions. In this context, some industries are adopting strategies as using residues as raw materials and processing techniques that originate the least possible wastes. That is the case of 3D printing technologies that enable rapid prototyping with freedom of design, customization, and capacity to develop complex shapes and systems.

3D printing has been invented in 1986. It consists of a technology for creating shapes and intricate patterns using three-dimensional modelling. The process involves the deposition of layers of material, one in top

of another. The initial invention was called stereolithography and since then, other techniques have emerged such as fused deposition modelling, powder bed fusion and inkjet printing, among other advancements. The introduction of computer aided design (CAD) systems has been most valuable for these technologies of additive manufacturing. The versatility of the techniques extends to the diversity of materials that can be used, as well as to the numerous possible applications. Over the last decades, 3D printing has progressed using diverse procedures, equipment, and materials. Nowadays, it is being used in areas such as construction, biomechanics and prototype manufacturing, enabling the production of limited series of products at lower costs relative to conventional technologies.

All classes of materials are being used in a wide range of 3D printing machinery that must be appropriate for each of the multiple and diverse materials and respective applications. Conventional polymers and

biopolymers, metals and alloys, ceramics and cements, as well as composite materials have been applied in the manufacture of products such as complex parts of instruments, lightweight aerospace structures, electronic components, architectural structures, biological tissues, bone scaffolds, and many more examples at diverse scales of work. Depending on the final application, the selection of raw materials may also lie on residues, being a way to implement reusing and recycling strategies in the industry. The choice for recyclable end-products or even biodegradable ones, whenever possible, depends on the initial selection of raw materials.

In this context, solutions that integrate the use of thermoplastic polymers are important since these are recyclable. Polyethylene (PE) and polypropylene (PP) are probably the most used thermoplastics in the world. These are both semi-crystalline polymers. PE may be produced under diverse chemical structures, hence having different densities and different related properties. Low-density polyethylene (LDPE), linear low density polyethylene (LLDPE), medium density polyethylene (MDPE), and high-density polyethylene (HDPE) present different degrees of crystallinity, being LDPE typically that with the lowest value. This property is reflected on lower temperatures for melting and for crystallization compared to its analogues. Moreover, the lower structural density of LDPE may be useful on the preparation of blends or composites with high content of solid dispersed fillers such as fibres or particles. In addition to the variable density, the molecular weight of polymers has also influence on that capacity. In a melt blending process, polymers with different values of melt flow index, which is directly dependent of molecular weight, may show viscosity problems in blends with different concentrations of the dispersed filler. Thus, finding an adequate composition for a certain processing technology must take these factors into account.

Lately, there is a growing interest in combining thermoplastic polymers with fillers of mineral additives, such as limestone powders, to build composite materials with improved characteristics, obtaining an environmentally and economically

## Materials and methods

The polymers selected for this study were Low Density Polyethylene (LDPE) and Polyethylene (PP) that were acquired to Exxon Mobil and SK GeoCentric, respectively. The reference for LDPE is Lupolen 1800S, with MFI of 20 g/10 min (190 °C; 2.16 Kg). For PP, the reference is Yuplene PP copolymer BX3920, with MFI of 100 g/10 min (190 °C; 2.16 Kg).

Zinc Stearate M 305 was supplied by SAPEC Química SA.

efficient production of parts. Limestone powders that constitute residues from the extraction and transformation industry of stones may be used in the synthesis of these composites, enabling the industry to diversify its offer while taking actions for using new technologies as 3D printing to obtain added-value products of low environmental impact.

Limestone is composed of calcium carbonate. It is considered as an essential feedstock material with application in several sectors, from construction to agriculture and pharmacy. Although it may be used in bulk, the powder form finds more and more applications each day. Many of these applications involve the preparation of blends with polymers for the production of green parts, being the polymeric component removed afterwards through heat treatments or other ways. Then, the obtained piece may be sintered to obtain the desired configuration, including controlled porosity, and resistance. This methodology may be simplified through the use of additive manufacturing. Different 3D printing technologies have been proposed to produce calcium carbonate containing items as diverse as artificial rocks or bone scaffolds. For each product, different technologies were tested so that the best one could be selected for each product. This alerts for the need to explore the technical capacities of each technique, each instrument and also each material to process.

This study is focused on developing composite material formulations based on polymeric matrices of LDPE and of PP, mixed with calcium carbonate powders from quarry residues. These materials must be adequate for 3D printing in two different prototype equipments, one for larger products than the other. However, it should be mentioned that the material compositions must first meet the requirements for processing through extrusion using also different machines for the two working scales. The particle size and the concentrations of the minerals that were blended with both polymers were previously studied in order to maximize the calcium carbonate for each 3D printer.

Calcium carbonate was supplied by MVC Portuguese Limestones. The mineral was obtained from the industrial activities of extraction and transformation of natural stones, constituting powder residues in loosely compacted form. These residues were dried at 55 °C until constant weight and were subsequently sieved to two sets of size ranges. One set between 0.3 and 0.5 mm and another set inferior to 0.1 mm. The larger particles were used with LDPE and with PP in the production of composite granules and the smaller ones, with PP and Zinc Stearate in the production of composite filament.

The synthesis of the composite materials was made by extrusion to obtain the following compositions expressed in mass percentages:

Material A – 55% LDPE + 45% Calcium Carbonate (particle size range 0.5-0.5 mm)

Material B – 66% PP + 33% Calcium Carbonate (particle size range 0.5-0.5 mm)

Material C – 55% PP+33% Calcium Carbonate (particle size inferior to 0.1 mm) + 12% Zn Stearate

The extrusion of the composite with LDPE polymeric matrix was processed in the range 150-170 °C, whereas those of PP matrices were processed in the range 160-180 °C. The extrusion of materials A and B was performed using a double screw extruder at semi-industrial scale and the obtained composite were reduced to granules at the end of the process. Material C was processed using a smaller extruder with a single screw and a nozzle of 1,75 mm diameter to enable the production of a thin filament.

The thermal analysis by DSC-TG of the composites was made using a STA 6000 Perkin Elmer equipment. Samples of 7 mg, approximately, were placed in an alumina pan. All samples were heated from 25 °C to 200 °C at a heating rate of 20 °C/min and held isothermally at that temperature for 10 min. Afterwards, the samples were cooled down back to 25 °C at the same 20 °C/min rate. An inert atmosphere was used, being the flow rate of nitrogen 20 mL/min.

The granules from materials A and B were used in 3D printing using a prototype extruder that has a robotic arm attached to the machine. The printed object was obtained by deposition of successive layers of melted material into a square shape of sides around 20 cm length and with walls making a 45° angle relative to the deposition surface. The processing temperatures ranged 160-185 °C.

The filament from material C was used in a different prototype extruder that works like a plotter. The filament enters the machine, and the deposition was tested using a simple flat shape measuring around 5 cm width. The processing temperatures ranged 160-185 °C.

## Results and discussion

The limestone residues, after being dried and sieved, had the appearance depicted on Figure 1 that shows some of the different size ranges obtained in the preliminary treatment of the minerals. The selected size ranges were used in the preparation of the blends through extrusion. The processing of Material A and Material B occurred in the double screw extruder

depicted in Figure 2 and the granules obtained at the end of the process are exemplified in Figure 3. The extrusion proceeded without any problems, even in the case of Material A that had a higher concentration of calcium carbonate. The melted blend was homogeneous, and the filament did not break despite that amount. The fact that LDPE was used as the polymeric matrix of this composite enabled the use of such concentration. To obtain an equivalent behavior in Material B with PP, that had higher melt flow index, the concentration of minerals had to be lower than in Material B.



Fig. 1. Calcium carbonate residues after being dried and sieved to different size ranges.



Fig. 2. Calcium carbonate powders are mixed with polymeric components through a double screw extruder. The melted filaments will solidify after passing a water bath. Processing method used to produce Materials A and B.



Fig. 3. Granules obtained after fragmentation of the solidified extruded filaments.

After drying the granules of both materials, these were tested in the 3D printer to obtain the shapes presented in Figures 4 and 5. In both cases, the materials presented an adequate flow for the continuous deposition, but the solidification was probably too slow, causing the deformation of the products. The adhesion between each layer was strong, anyway.

The processing temperature of Material A should be lower since it is based on LDPE that has the lower melting temperature. However, the equipment operated only from 160 °C. This should not be a problem using Material B, based on PP, but the obtained products also presented significant deformations. Although it is usually found that PP solidifies faster than LDPE, this was not observed in both blends with calcium carbonate. The change in some parameters of the printing process, such as the speed and the geometry of the product may be important to overcome the deformations as the solidification occurs.

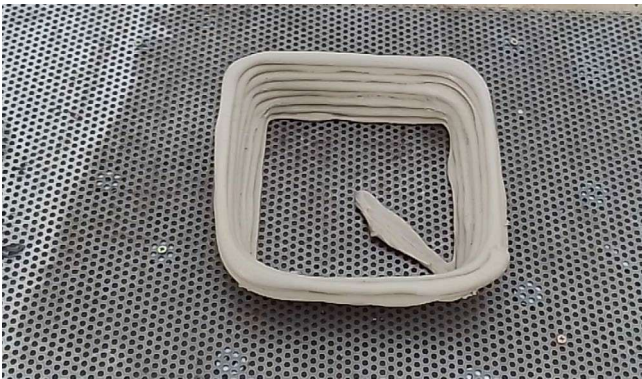


Fig. 4. Tri-dimensional shape made using Material A.

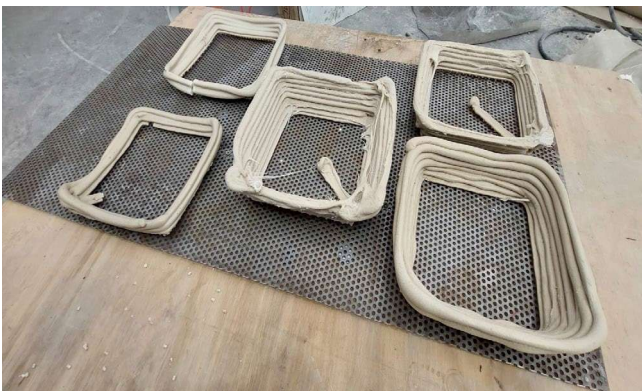


Fig. 5. Tri-dimensional shapes made using Material B.

As for Material C, it has the same percentage of minerals as Material B, but the polymeric matrix contains zinc stearate and PP. Zinc stearate was used to decrease the viscosity of the blend of the minerals and PP. Together with the use of smaller particle sizes, that combination enabled the successful production of a thin filament of composite that is showed in Figure 6,

at the top of the 3D printer for small products. The printed shapes are showed in Figure 7. In this case, the tridimensional shapes had the intended characteristics, almost without deformation of the products.

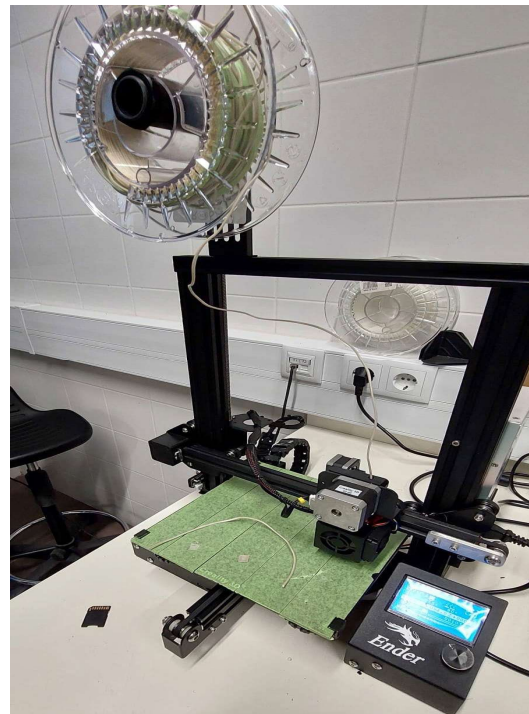


Fig. 6. 3D printer using a filament of Material C.

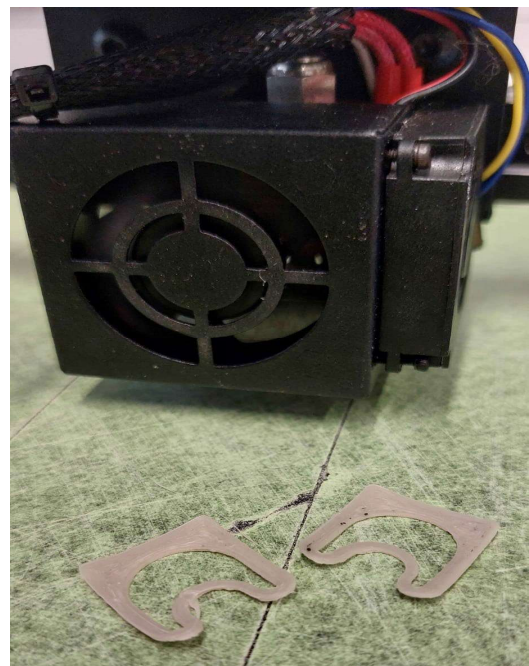


Fig. 7. Products obtained with Material C.

The thermal behaviour of the studied materials may be explained using the DSC results presented in Figures 8, 9 and 10. The melting temperature of Material A is 113 °C, as expected for its LDPE based composition and the crystallization occurs at around 91 °C involving the

energy of 11 mW approximately. Material B and Material C melt around 163 °C and 164 °C, solidifying at 120 °C and 113 °C, respectively. The energy of crystallization is approximately 15 mW and 13 mW. These results point to the fact that Material A needs a higher difference in temperature to solidify completely. This may be because of the higher content in mineral particles that are known to have high heat capacity. All the materials were extruded at above 160 °C and the deformation did not occur in Material B probably because of the architecture of the printed products that had much lower thickness.

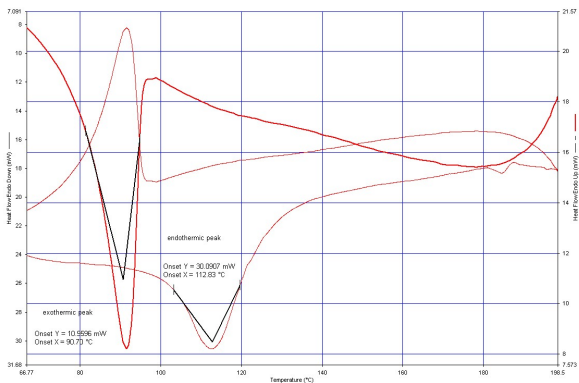


Fig. 8. DSC results from Material A.

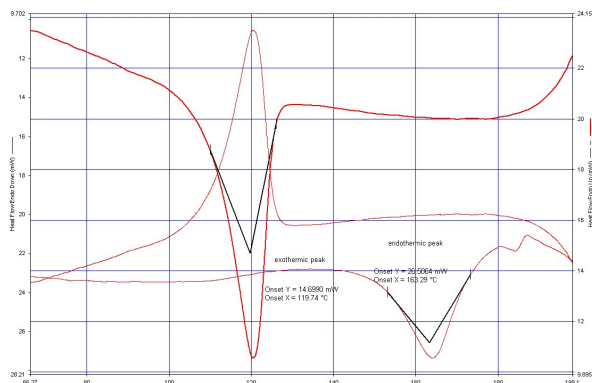


Fig. 9. DSC results from Material B.

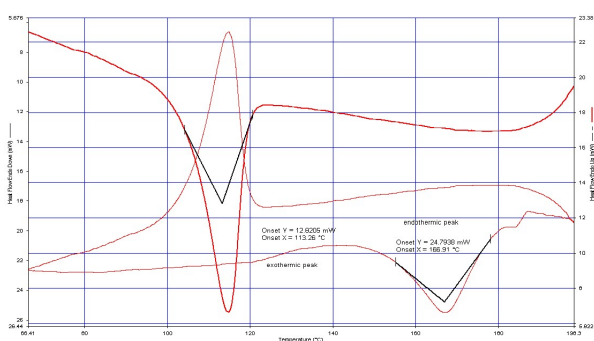


Fig. 10. DSC results from Material C.

## Conclusions

The results obtained in this study have been important to establish some experimental conditions for using calcium carbonate residues as fillers of thermoplastic composites to be produced using 3D printer

technology. Variables as polymer composition and structure, melt flow index and temperatures of physical transitions, as well as particle size range of the mineral fillers, were studied to obtain material that could be synthesized through two different extruders and then transformed into products using two different 3D printers. The obtained products showed that some processing parameters may have to be adjusted to obtain the intended quality of the end-products. For the larger printer, the limitations may be overcome by designing products with larger dimensions, or by lowering the speed of the process of printing to enable a slower solidification. The process associated with the smaller printer originated end products without deformations, as intended.

## Acknowledgments

This work is supported by the FCT (Fundação para a Ciência e a Tecnologia) support of CDRSP-IPLeiria (Strategic Projects UIDB/04044/2020 and UIDP/04044/2020; and Associate Laboratory ARISE LA/P/0112/2020); and ANI, through the project INOVMINERAL 4.0 – Tecnologias avançadas e software para os Recursos Minerais; nº POCI-01-0247-FEDER – 046083; and finally, IPLeiria (Institute Polytechnic of Leiria)

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## Main deterioration patterns found in Lioz Limestone at Rio de Janeiro, Brazil

Mozer, A.G.S.<sup>1</sup>, Castro, N.F.<sup>1,2</sup>, Mansur, K.L.<sup>1</sup>, Ribeiro, R.C.C.<sup>2</sup>

<sup>1</sup> Universidade Federal do Rio de Janeiro—Geosciences Institute, Post-graduate program: Av. Athos da Silveira Ramos 274, Rio de Janeiro, RJ 21941-916, Brazil; <sup>2</sup> Centre for Mineral Technology, Av. Pedro Calmon 900, Rio de Janeiro, RJ 21941-908, Brazil

Lioz was widely used in Portugal's and Brazil's historical buildings and in other countries that Portugal colonised during its maritime expansion. This limestone presents different colours and receives different names depending on its aspect and the extraction area. The most common are Lioz (the beige one), Amarelo Negrais (the yellow one exploited at Negrais), Encarnadão (the red one) and *Chainnette* (the cross-cut beige variety that looks like a chaine). In Rio de Janeiro this stone can be seen in facades, ornaments, walls, floors and other uses in churches, buildings and monuments from the 16th century onwards. Some examples are the Rio de Janeiro's landmark built in Lioz and brought to Brazil in 1565, the Estácio de Sá Tombstone (1583) and the Santo Antônio Convent and Church (1620). Lioz received the IUGS - Heritage Stone designation in 2018 a recognition granted by the International Union of Geological Sciences (IUGS) to natural stones used in heritage monuments and edifications significant for human culture.

The city of Rio de Janeiro has a polluted and saline environment, which combined with the tropical weather and frequent vandalism, can accelerate the degradation of this stone. Despite the compactness of Lioz and its outstanding characteristics as a building material, its fossils and stylolites can be weakening zones. The presence of clay minerals and iron oxides in Amarelo Negrais and Encarnadão also makes them more susceptible to alterations, those varieties have a slight increase in porosity. They show intense discolouration and loss of components, especially when used on floors and external applications. Because of that, nowadays this stone is mainly used inside the constructions.

When applied outside, in ornaments and facades, is observed the presence of black crusts, deposits and soiling due to the polluted environment, microkarst in ornaments and fonts, pitting and biological colonization. Efflorescence and blistering can be seen in buildings, both related to salt precipitation. Fractures and abrasion are frequent on floors and stair steps. When used on floors, Lioz shows discolouration and loss of components, these two are also observed in altars, baptismal fonts and other ornaments. A glossy aspect is present in locals where people tend to touch, as in an 18th century font located in the vestry room at the Santo Antônio Church. Graffiti is a typical deterioration pattern found at Rio's monuments and the attempt to clean it can cause bleaching to the stone's surface. Missing parts usually occur at corners and parapets. The fossils control the differential erosion since they are more resistant to weathering than the matrix. These are more frequent on floors.

Knowing these deterioration patterns and their causes is helpful for future conservation strategies.

# Technology Paths towards Coopetition Ecosystems | The Portuguese Stone Sector

A. Silva<sup>1\*</sup>, A.J. Cardoso<sup>2</sup>

(1) CISE - Covilhã, Portugal; CEFAGE-UE, Évora, Portugal; MARE, Leiria, Portugal, \*a.silva@zipor.com

(2) CISE - University of Beira Interior, Covilhã, Portugal

**Summary:** *Architecture, Engineering, and Construction (AEC) has embraced the Building Information Modelling (BIM) technology. It is possible to cocreate digital twins, shaping procurement and reducing costs, time, and waste, representing a serious threat to the stone sector in Portugal. Mainly medium enterprises (SMEs) it is a relevant sector in the Portuguese economy, for which the recourse to debt is usually the only way to get investment financing. In this context, literature in Management and EU officials point out collaboration as a solution. However, a work carried out by a group of researchers from the BCG Henderson Institute and published by the MIT Sloan Management Review (2019) highlights that only 15% of collaborative cases reach the long term. For underlying potential solutions to this question, along with this research, a survey on a sample representing 67% of the population was carried out to evaluate the improvement in collaboration from technology-enabled "secrecy" environments in the Portuguese ornamental stone (OS) sector. This survey found that in the level of collaboration under the current state-of-the-art environment, only 2,2% of the high-level digital companies accept collaboration with rivals. Controversially, in a technology-enabled "secrecy" environment, 90,1% of these companies should accept to collaborate with rivals. From these findings, a technology-enabled secure environment should improve the collaboration among companies to catch the opportunities. However, this improvement only happens in companies with a relatively high digital experience.*

**Keywords:** *Coopetition, BIM, ornamental stone, collaboration, Information systems*

## 1. Introduction

Designing environmentally friendly buildings that provide both high performance and cost savings is of increasing interest in developing sustainable cities. Architecture, engineering, and construction (AEC) have embraced the Building Information Modelling (BIM) technology in this context. It is possible to cocreate digital twins, shape procurement, and reduce costs, time, and waste. This BIM generalization tends to reshape the AEC supply chain, pushing providers to operate in a global marketplace. In this regard, the AEC providers, like stone companies, must shift their operations towards a digital marketplace shaped by the potential mandatory use of BIM (Vilas-Boas et al., 2019).

Mainly for small and medium enterprises (SMEs), the Ornamental Stone sector (OS) is relevant to the Portuguese economy. Directly employing more than 16,600 people, this sector is one of the foremost private job generators in inland regions, registering sustained export growth, placing Portugal as the eighth country in the International Trade of OS and the second country in the world international trade per capita.

## 2. Problematic

The BIM technology transforms the AEC into a digital and global supply chain, where information

interoperability between all actors is one of the pillars. It requires the actors, whether they are customers, suppliers, or any other, to interact through technology accessible from any personal or professional system, enabling customers' direct access to the most competitive suppliers.

The BIM dimensions are complementary elements of information such as cost, accuracy, sustainability, and compatibility, among other information (Sacks et al., 2010). By creating and using these dimensions, one can better understand the planning of the work to the management of the undertaking sustainably (Corry et al., 2015).

The first three BIM dimensions represent the comprehensive model of general construction plans. This dimension represents the model and projects related to architecture, structures, and installations (Wang et al., 2015). Through these three dimensions, it is possible to detect divergences, inconsistencies, and failures before proceeding to execution, making this dimension indispensable to avoid errors that could compromise the schedule and the budget (Wong & Zhou, 2015). The fourth BIM dimension represents the planning process to link the construction activities in time schedules with 3D models to develop a real-time graphical simulation of construction progress against time (Smith, 2014). This correlation makes it possible for the manager to follow the physical progress of the

construction and, with the simple movement of the computer cursor over the schedule, to see the work being calmly and smoothly built as if in a film (Cheung et al., 2012). The fifth BIM dimension represents the cost dimension associated with the model, where each element (solid bloc) is linked to the cost database (Marinho, 2014). In this regard, the masonry shown in the walls, for example, becomes associated with the budget and the respective production and execution costs, whether these are labor equipment, among other costs (Matthews et al., 2015). The sixth BIM dimension represents sustainability since it can no longer be absent in any project (Vargo & Lusch, 2017). It is not a market differential but a requirement. In this way, this dimension of BIM allows several analyses to be carried out focusing on sustainability (Wang et al., 2015). The seventh BIM dimension represents the "as built" information with operation approach and maintenance (Smith, 2014). The seventh BIM dimension allows extracting specific information on materials, operating manuals, and warranty dates. Through this dimension, one can also carry out a detailed control of equipment warranty, maintenance plans, manufacturers' and suppliers' data, operating costs, and even photos not only of materials and equipment but of everything inside the walls, not visible at a later stage of the work operation (Wagner et al., 2017). The eighth BIM dimension represents the embedded emergency plans and prevents security issues regarding safety and prevention of workplace accidents (Krishnan et al., 2012). Through the eighth BIM dimension, it is possible to determine risks in the model, encourage the creation of protection suggestions for high-risk profiles and recommend control of threats in the work area (Smith, 2014), helping professionals avoid accidents and improving the team's performance.

The functionalities resulting from these eight dimensions seem like marketplace enablers (Dallasega et al., 2018). Through the BIM generalization, the supply chain becomes collaborative and global, leading to global-scale procurement in AEC, where the provider's survival will depend on their competitiveness at a global level, requiring a critical dimension of investment capacity.

### **Research problem**

Referred to as a pillar in the developed regions, for the SMEs to achieve the digital supply chain requirements, the management, business, and supply chain literature converge to solutions based on long-term collaborative alliances among companies (Friend et al., 2020). At the same time, the European Commission points out the establishment of urgent industrial alliances as a key to

making the European manufacturing SMEs successful in the digital supply chain context (European Commission, 2021). Moreover, recent literature on ecosystems shows substantial growth in co-competition research, described as simultaneous competition and cooperation among business rivals (Bicen et al., 2021).

As in other European countries, the OS sector in Portugal consists of SMEs, for which the recourse to debt is usually the only way to get investment financing. Because excessive indebtedness makes SMEs more vulnerable to fluctuations in demand, both the literature (Geissbauer et al., 2018) and European Union officials point out collaboration between companies as an alternative to debt (European Commission, 2021). Through collaboration, companies may (1) acquire scale to supply projects of any size, (2) increase competitiveness by concentrating their own resources on the products and services in which they are most efficient and effective, (3) guarantee market deliverables according to commitments, regardless of the size of its projects.

However, a work carried out by a group of researchers from the BCG Henderson Institute and published by the MIT Sloan Management Review (2019) highlights, to some extent, worrying signs about collaborative actions among companies. According to this research, only 15% of the collaborative cases reach the long-term, and most of the survivors are large companies like Microsoft and Amazon, among other giants (Reeves et al., 2019).

From the literature in social sciences (Crick, 2019; Ho et al., 2020; Uribe et al., 2020), the end of collaboration among rivals is related to negative experiences of past access by competitors to critical information or other differentiating and fundamental assets of companies, from which we can pose a question: May a technology-enabled "secrecy" environment improve the collaboration among competitors?

For underlying potential solutions to this question, along with this research, a survey will be used and applied to a representative sample of Portuguese stone companies. A survey will evaluate the improvements in collaboration from the current state-of-the-art to Technology-enabled "secrecy" environments.

### **3. Methodology**

Despite its small size, Portugal has a diversified and significant reserve of stones suitable for ornamental use. Therefore, more than ever, the ability to add value through the digital cocreation of products with clients and architects at a global level, still in the conception (design) phase of the work, should be one of the



fundamental focuses of Portuguese suppliers of stone products.

### **Digital steps**

In the "Prospective Strategic Study," presented by CEVALOR and ASSIMAGRA (2004), the need to develop a digital-lean strategic path for Ornamental Stone was called the "leanstone booklet" (Silva, 2018). Embracing innovative technologies and techniques along successive steps in the Ornamental Stone sector has acted as a lever for the full innovative development and competitive potential.

The first leanstone step (LS#1) was the technological output of a 2004-2004 partnership including three technological companies, three adopter companies, and two research centers. Using the LS#1 technologies, the OS companies can flexibly transform raw materials, obtain expressive results in reducing raw materials waste, converting them into productivity gains, and responsiveness to small orders in the domestic and European markets (Almeida & Silva, 2022).

The second leanstone step (LS#2) was the technological output of a 2010-2013 partnership, including four technological companies, twelve adopters, and three research centers. Using LS#2 technologies, OS companies significantly increase productivity and efficiency today (Silva & Gil, 2020).

The third leanstone step (LS#3) was the technological output of a 2016-2018 partnership, including two technological companies, two company adopters, and three research centers. Regardless of the order dimension, these technologies enable OS companies to respond to customized projects, significantly reducing materials waste, energy, and ecological footprint and ensuring a significant increase in productivity. By using these technologies, companies become ready to shift their operations to Industry 4.0.

The fourth leanstone step (LS#4) was the technological output of a 2017-2020 partnership includes two technological companies and two company adopters, and three research centers. Using LS#4 technologies, OS companies can take a significant step towards collaborative and intelligent manufacturing. Oriented towards the cocreation of value in marketplaces, the Inovstone4.0 technologies will allow OS manufacturers to collaborate with customers in a growing, personalized, and optimized co-creational approach along the opportunities, regardless of the scale.

Mainly for medium enterprises (SMEs), the OS sector is relevant to the Portuguese economy. Despite the improvements from these leanstone steps and skills

admission over the last decade, this sector, beyond a lack of investment capacity, keeps traditional family-based management, of which it is part, a lack of collaborative mindset.

### **Artifact requirements to improve collaboration**

Digitization converts analogue information (e.g., text, images, sound, and physical attributes) to a digital format (Ng & Wakenshaw, 2017). Digitization and networks have altered the concept of place, where a task is performed, and where resources are delivered (Lusch et al., 2010). Human service activities are taking on physical tools and forms to create greater efficiency for faster and more consistently replicable services (Zhong et al., 2017). Cyber layers become more entangled and inseparable from material things and humans (Pohlmann & Kaartemo, 2017).

Digital-based Artifact - Connecting customers directly to providers (Haleem et al., 2018) and globally rated by customers' satisfaction (Medberg & Grönroos, 2020), the manufacturing SMEs' access to the digital supply chains requires a high level of manufacturing efficiency (Bayne et al., 2017). In this regard, business literature highlights manufacturing efficiency (Faller & Feldmüller, 2015) and customers' satisfaction-oriented mindset (Raut et al., 2019) as the main conditions for accessing and remaining in the supply chain. As digital technologies are now increasingly capable of adjusting to their environment as operant resources (Akaka & Vargo, 2014), from the information systems literature (Howard & Björk, 2008), the "Digital-based" can be stated as the first Artifact's requirement to improve the collaboration among companies.

Interoperability is a central issue in technology-based ecosystems (Ruokolainen & Kutvonen, 2009). Interoperability is the ability to exchange and share resources (e.g., information and devices) and use them meaningfully (Succar & Kassem, 2015). In case of fault, cooperation will be interrupted, while interoperability problems can range from simple technological incompatibilities to conflicts between business strategies (Leal et al., 2019). Moreover, interoperability in service ecosystems has technical, semantic, and pragmatic concerns (Ruokolainen & Kutvonen, 2009).

Interoperable Artifact - understanding interoperability as a requirement for ensuring an effective collaboration within a network (Leal et al., 2019) in such a way that either their mutual goals become fulfilled, or their cooperation is dissolved in a controllable manner in case of a fault (Ruokolainen & Kutvonen, 2009), arguably, the Secure technology to improve the company's collaboration must comply in terms of technical

interoperability, semantic interoperability, and pragmatic interoperability issues (Leal et al., 2019). Technological facilities must be compatible such that communication paths can be established. All actors in the network must access and give access to the shareable information with other actors in the coopetition network. Semantic interoperability - exchanged information and message exchange patterns must be ensured by all actors. Pragmatic interoperability – expectations, business rules, and organizational policies of collaborating parties' compatibility. Therefore, from the information systems literature, "Interoperability" can be stated as the second Artifact's requirement to improve collaboration among companies.

Usable and accessible Artifact - to reach a sufficiently secure system, the Artifact, to improve the company's collaboration, must ensure safe accessibility and usability to actors' resources to leverage the value cocreation interactions most effectively in the ecosystem. The Artifact must ensure the simultaneous collaboration among competitors along the business opportunities (from the lead to opportunity win or lose).

Systemic assemblage's enabler Artifact - Systemic assemblages refer to objects or devices working together (Doganova & Eyquem-Renault, 2009) and with the ability to do things that none of these objects could perform on their own (Ng et al., 2015) (Ng & Wakenshaw, 2017). In this regard, Artifact must become a systemic enabler of assemblages to improve the company's collaboration.

Transactions enabler Artifact - through the Artifact to improve the company's collaboration, companies can modularize their practices as tasks for designing thin crossing points that allow new Internet-connected constituents to be brought in and for new transactions to occur (Ng et al., 2018). In this way, the Artifact must represent a schema that views design processes as acts of decomposition and aggregation to achieve architectures that preserve and enhance a hierarchy of loosely-coupled parts (Storbacka et al., 2016) (Henfridsson et al., 2014).

### Population and sample

The Portuguese OS sector has around two thousand and five hundred active companies (Silva & Pata, 2022) operating upstream, downstream, or both. However, from this gross population, around a thousand and five hundred companies operate micro-companies or craft workshops with at least three workers in a local market. Therefore, for this research, the net population of one

thousand OS companies spread over eighteen Portuguese Districts.

From this population, the interviews were conducted with a sample of six-hundred and seventy companies, representing 67.0% of the population. Moreover, this sample represents a total turnover of 89.3% and exports 95.9% of the population.

### Digital Enterprise Levels

The literature in management, digital technologies, and the building industry points out that the BIM benefits as depending on the level of connectivity of the parties involved in the AEC value chain (Müller et al., 2018). Because some requirements, such as investment capacity in production technologies and skills, to get efficiency, flexibility, scale, among others, are essential to be successful, the sample was divided into stages of digitization of its operations.

The Digital Level thus determines the level at which a given company is in terms of the use of digital technologies. This index was determined based on the face-to-face survey and on-site visit, assessing the company's digital production equipment, the means of management and integration with production, and the collaborative means that are part of the digital marketplace.

For this research purpose, are considered as "no digital level" (ND#0) those companies not using any computerized system in production and, therefore, have not started the digitalization path. As the "first digital level" (ND#1), those companies have used computerized production systems for over a year. As the "second digital level" (ND#2), those companies already depend on digital equipment in production, with at least two computerized machines on the shop floor. As the "third digital level" (ND#3), those companies integrate the information generated by computerized machines on the shop floor with the management information. As the "fourth digital level" (ND#4), those companies integrate collaborative data generated by computerized shop floor technologies with the management. They use this real-time information in the marketplaces. The following questionnaire was put available to the respondents:

1. Introductory concepts: *BIM and CAD meaning.*
2. Introduction: *The construction projects will cease to be carried out in CAD and will be developed in BIM, changing the model of prescription and purchase of the ornamental stone. To respond to these market changes to get orders, it will become crucial that ornamental stone suppliers interact digitally with the architect in BIM during the design phase, can provide orders of any size, and strictly meet delivery*

deadlines. Additionally, any provider will tend to be digitally evaluated by all actors involved in the business.

3. First question: *In this context of market change, and considering your experience, do you consider that in the future, you may participate in projects in collaboration with other competitors?* Possible answer: *yes; no; maybe.*
4. Introduction: *Suppose, hypothetically, that someone developed a technology to search for and supply works together with other suppliers of the type of online banking systems:*
  - a. *It would allow them to carry out market research together with other competitors.*
  - b. *To add to their commercial proposals, specialized productive resources of these partners without their knowledge.*
  - c. *To guarantee commercial confidentiality until the buyer's decision.*
  - d. *After the buyer's decision, to automatically reserve the specialized resources to be acquired by their project partners.*
5. Second question: *Under these new "commercial secrecy" conditions, would you consider collaborating on projects with other competitors?* Possible answer: *yes; no; maybe.*

All companies were interviewed according to this questionnaire, face-to-face from February 2022 to February 2023. The results were saved in an Excel file.

#### 4. Results

Following the research objective, we found that despite the threat to OS companies from the BIM-shaped procurement, under the current state-of-the-art collaborative environment, they seem far to accept collaborating with rivals. Especially the most digitally advanced companies, from their negative collaborative experiences, refuse to collaborate with their rivals.

From the data, we found on the low level in digital experience respondents, 28,1% of the companies without a computerized production system (ND#0) and 25,8% of the companies using only one computerized system (ND#1) accept to collaborate under the current state-of-the-art environment. However, for the high-level digital respondent companies, 14,1% of the companies already integrating the information generated by computerized machines on the shopfloor together with the management information (ND#3), and only 2,2% of the companies integrating collaborative data generated by computerized shop floor technologies together with the management and market, accept to collaborate under the current state-of-the-art environment (Table 1).

Table 1: Collaboration level under state-of-the-art environment

State-of-the-art	yes	no	maybe
ND#4 group	2,2%	89,8%	8,0%
ND#3 group	14,1%	77,1%	8,8%
ND#2 group	22,3%	56,8%	20,9%
ND#1 group	25,8%	37,5%	36,7%
ND#0 group	28,1%	33,2%	38,7%

Controversially, by using a trustable technology, the answers are quite different. Ensuring "commercial secrecy" conditions, (1) carrying out market opportunities together with other competitors, (2) allowing companies to add to commercial proposals competitors specialized resources without their knowledge, (3) guaranteeing commercial confidentiality until the buyer's decision, and (4) once happens the buyer decides to automatically reserve the specialized resources to be acquired to their project partners.

1. For the low-level digital experience respondent companies, under a "commercial secrecy" environment, 22,2% of the ND#0 companies and 28,9 of the ND#0 companies accept to collaborate under the current conditions. The probability of collaborating with rivals in an information-secure environment for these low-level digital companies remains the same.
2. for the high-level digital respondent companies, 65,9% of the ND#3 companies, and 90,1% of the companies integrating collaboratively the data generated by computerized shop floor technologies and the management in marketplaces, accept collaborating under a "commercial secrecy" environment (Table 2).

Table 2: Collaboration level under Technology-enabled environment

Technology-enabled	yes	no	maybe
ND#4 group	90,1%	1,8%	8,1%
ND#3 group	65,9%	15,6%	18,5%
ND#2 group	46,4%	22,1%	31,5%
ND#1 group	28,9%	33,2%	37,9%
ND#0 group	22,4%	32,6%	45,0%

#### Conclusions and final considerations

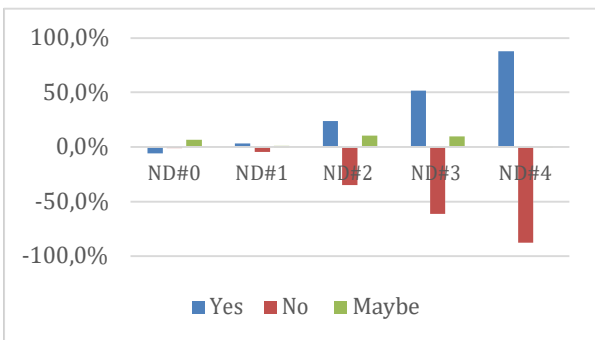
To evaluate the collaboration improvement in the Portuguese ornamental stone (OS) sector, a sample representing 67% of the population has been interviewed. Moreover, this sample has been split into five groups according to their digital level of maturity.

From the results, enabling the "commercial secrecy" by through a technological artifact seems to improve the collaboration trend among the high-level digital stone companies significantly. However, there are no significant improvements in the low-level digital stone groups of companies (Table 3).

Table 3: Table 4: Collaboration improvement from the current state-of-the-art to Technology-enabled "secrecy" environments

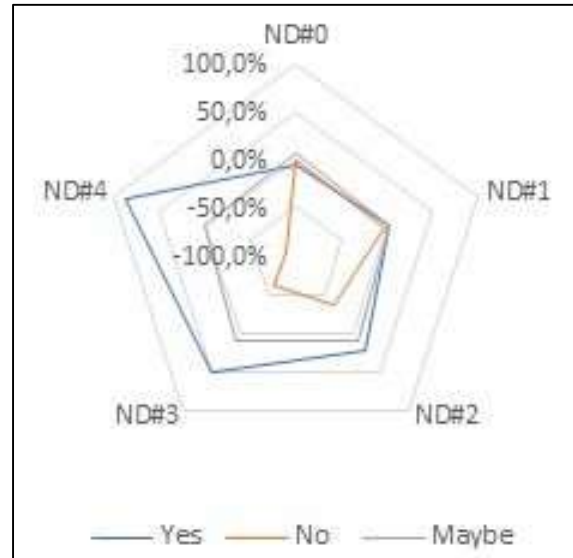
Improvement on Collaboration	ND#0	ND#1	ND#2	ND#3	ND#4
Yes	-5,7%	3,1%	24,1%	51,8%	87,9%
No	-0,6%	-4,3%	-34,7%	-61,5%	-88,0%
Maybe	6,3%	1,2%	10,6%	9,7%	0,1%

As companies evolve at their digital level, they rely more on digitally supported environments. They are, therefore, more sensitive to the potential impacts of these technologies on their business (Graphic 1).



Graphic 1: Digital level vs. embracing digitally supported collaboration

Companies with a low digital level, perhaps because they have less experience in international markets, will be less sensitive to the future impacts of the procurement change in BIM. As a result, the increase in the willingness to collaborate when there is security in the preservation of differentiating assets is very high in digitally evolved companies and relatively low in companies with little digital experience. The companies that advance in the digital path are probably the most export-oriented. They tend to be aware of BIM procurement's impact on their activity. Therefore, they look at collaboration to mitigate the BIM procurement threats to their activities (Graphic 2).



Graphic 2: Collaboration trend to mitigate the BIM procurement threats.

A hypothetical technology-enabled secure environment in BIM procurement should increase trust among rivals, improving their collaboration to catch the opportunities. Moreover, this improvement happens only in companies with experience in digital operations. Moreover, in a trusting environment, the higher the digital level of the companies, the more likely they are to collaborate, thus avoiding the over-indebtedness consequences.

From these conclusions, we encourage researchers to carry out in-depth on the specifications of a future artifact that must be designed, demonstrated, and evaluated regarding its usability and reliability, ensuring a collaborative environment among SMEs.

**Acknowledgments:** The authors appreciate the Inovmineral4.0 Project - *programas mobilizadores clusters de competitividade e outras dinâmicas coletivas* - Portugal 2020 14/SI/2019, for the support on their work.

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## Abstract

**Name: Catarina Baleia**

**Title: Nanostructured Functional Stone Coatings**

Inglês: Stone can undergo deterioration from natural and/or anthropogenic causes. Specifically, water is one of the main factors that contribute to the complex degradation mechanisms. The goal was to develop nanostructured functional coatings to impart resistance to water. Additionally, the surface treatment should maintain the appearance of natural stone. Hence, the coating was based on the use of Silica Nanoparticles (SiNPs) to create a surface roughness and, consequently, promote hydrophobic properties onto the stone. To optimize the hydrophobic character and adhesion to the stone surface the approach was to develop a surface treatment based on alkoxy silanes silica precursors. The hydrophilic SiNPs with 65 and 400 nm size were surface modified with hydrophobic propyl groups. In a first stage, different SiNPs dispersions were formulated to study the effect of alkoxy silane mixture, nanoparticle size, nanoparticle type (non-functionalized and functionalized), and nanoparticle concentration on the wettability of glass substrates. The proof of concept for the hydrophobic coating based on 3 w/v% of functionalized 65 nm SiNPs dispersed in 90 % TEOS + 10% PTES was the Cinzento Vimieiro granite (Contact Angle  $\sim 45^\circ$ ). The treated stone showed an average Contact Angle (CA) of  $123^\circ$  and the maximum measured value was  $139^\circ$ , which was close to the demanding threshold of superhydrophobicity (CA  $> 150^\circ$ ). Hence, the goal of the thesis was successfully achieved and so, the coatings have the potential to help the industry increase the value of natural stone materials. In particular, hydrophobic properties provide a competitive advantage to the stone industry.

## Monitoring of a Natural Stone Cutting CNC Machine: Early Insights on Internet of Things applications for Improved Efficiency and Sustainability in the Stone Industry

C.D. Henriques<sup>1\*</sup>, D.R. Siva<sup>2</sup>, P.A. Amaral<sup>3</sup>, C.A. Silva<sup>3</sup>

(62) MIT Portugal Sustainable Energy Systems, Instituto Superior Técnico, Universidade Técnica de Lisboa, IN+ Center for Innovation, Technology and Policy Research, 1049-001 Lisbon, Portugal, \*[Carlos.diogo@tecnico.ulisboa.pt](mailto:Carlos.diogo@tecnico.ulisboa.pt)

(63) Department of Mechanical Engineering, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1, 1049-001, Lisbon, Portugal

(64) Instituto Superior Técnico, Universidade Técnica de Lisboa, IN+ Center for Innovation, Technology and Policy Research, 1049-001 Lisbon

**Summary:** *The natural stone industry is a vital sector for the construction industry, but it also has significant environmental and social impacts that require attention. Energy consumption is a key issue, as it affects both economic and environmental sustainability. In this study, we instrumented a natural stone cutting computerized numerical control (CNC) machine with Internet of things (IOT) devices to gain insights into its energy consumption and operation.*

*Over several months, we collected data that allowed us to observe the operation hours of the machine during work and off-work times and estimate its moves in the x, y, z directions, as well as the table. We also added vibration and distance sensors to the machine head and plan to analyse those data. The analysis of the energy data, including power factor, voltage, reactive energy, and active energy, can provide insights into maintenance considerations for improved efficiency and sustainability in the stone industry.*

*The instrumentation of old machinery offers many advantages, including improving the efficiency and sustainability of the stone industry, reducing its environmental impact, and promoting a safer work environment. The analysis of the energy data can provide insights into maintenance needs and potential improvements, which can increase machine lifespan and decrease downtime, leading to increased productivity and reduced costs. This study highlights the importance of energy monitoring and instrumentation for the stone industry and its potential to improve efficiency and sustainability.*

**Key words:** *Natural Stone, Energy Efficiency, CNC, Maintenance, IOT*



## Introduction

The natural stone industry is a significant contributor to the global economy, with the demand for natural stone products increasing in recent years [1]. The industry is known for its unique qualities of durability and aesthetic appeal that make it a preferred choice for various architectural and decorative applications. However, the industry's production processes are also associated with high energy consumption and environmental impact [2].

To address these issues, researchers and industry practitioners are increasingly exploring ways to optimize existing machinery and adopt energy-efficient technologies [3]. As a result, the use of energy monitoring devices has become more prevalent in manufacturing industries, including the natural stone industry [4-5]. These devices can provide real-time data on machine performance and energy consumption, which can then be used to identify areas for improvement.

In this paper, we present our work on the instrumentation of a natural stone cutting CNC machine with energy monitoring devices. Over the course of several months, we collected data on the operation hours of the machine during work times and off work, as well as estimates of the machine's movements in the x, y, z, and table directions using sensors. The data collected from the sensors was used to identify patterns and correlations between machine performance, energy consumption, and other factors.

To further our analysis, we also added vibration sensors and distance sensors to the machine head, with plans to analyse the data from these sensors in the future. Through our observations and analysis of the energy data, we gained insights into the maintenance and prevention of machine issues, as well as safety concerns. By identifying areas for improvement, we aim to contribute to the overall goal of making the natural stone industry more sustainable.

Our work adds to the growing body of research on energy-efficient technologies in the natural stone industry. Instrumenting older machinery with energy monitoring devices can lead to significant improvements in machine performance, reduced energy consumption, and cost savings [6-11]. In addition, it can also help to mitigate environmental impact and improve the industry's overall sustainability [12,13]. Therefore, our study provides valuable insights for practitioners and researchers looking to improve the efficiency and sustainability of natural stone production.

## Methodology

The natural stone cutting CNC machine used in this study was a model. This machine was owned and operated by a leading manufacturer in the stone cutting Portuguese industry but is representative of the industry as whole, since it is a fairly common equipment and the configuration is also a standard in the industry. The machine was instrumented with non intrusive energy monitoring devices, measuring, power, current, voltage energy both active and reactive, as well as power factor. The energy monitoring devices were expertly installed in the triphasic main circuit supply of the CNC cutting machine to provide accurate readings of its energy consumption (Fig. 1).



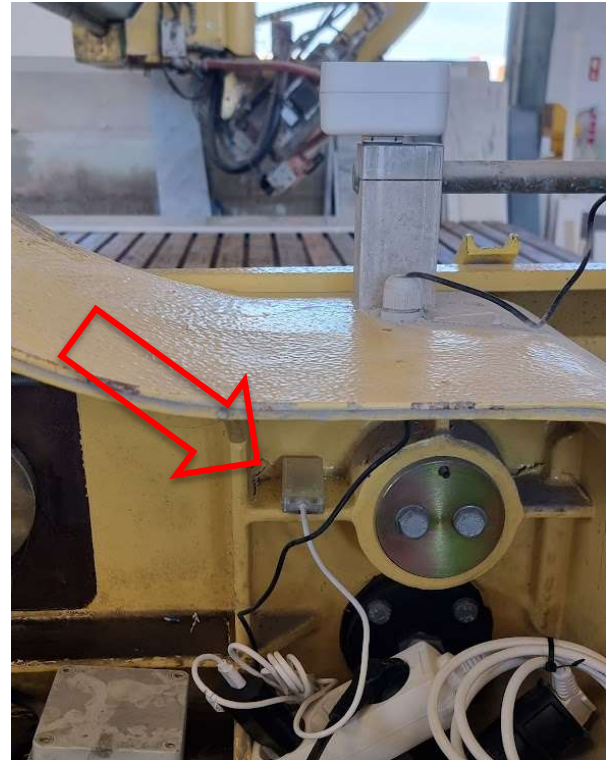
*Fig. 1. External switchboard with energy monitoring device.*

The device used for energy monitoring utilizes Modbus communication protocol to transmit the data to a GSM gateway for processing and sending data to a cloud server for storage. In addition, the machine was equipped with a light sensor glued to the CNC working light (Fig. 2), distance sensors (Fig. 3) and vibration sensors (Fig. 4), which were attached to the machine to capture data on the machine's movements during cutting.

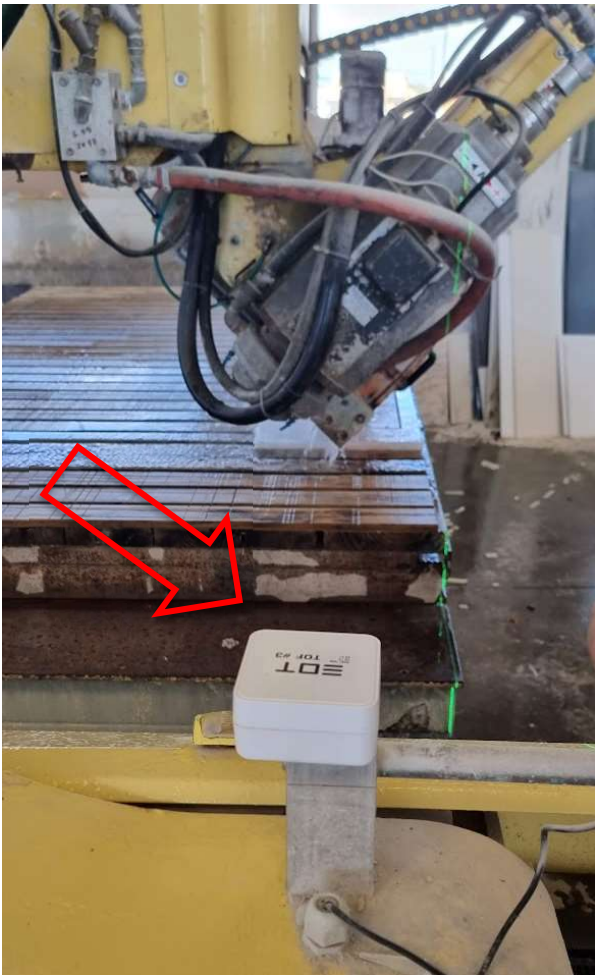
The vibration sensors were used to measure the machine's vibration levels, while the distance sensors were used to estimate the machine's movements in the x, y, and z directions. For the communication process of the devices other than energy, we employed low-power long-range radio frequency (RF) transceivers based on the LoRaWAN (Long Range Wide Area Network) protocol. This protocol enabled the devices to communicate their data over long distances and through obstacles found in factories with low power consumption, making them ideal for our purposes.



*Fig. 2. Light Sensor collecting information on the stop/working state of the CNC machine.*



*Fig. 4 vibration sensor collecting data on the machine operation*



*Fig. 3 distance sensor collecting data on the machine movement*

The data collected from the energy monitoring devices, vibration sensors, and distance sensors were stored in a central database for analysis. The analysis involved identifying patterns in the data and correlations between the energy consumption and the machine's movements.

The data analysis also involved identifying anomalies in the data, such as spikes in energy consumption or vibration levels. These anomalies were investigated further to determine their cause and potential impact on the machine's performance and maintenance requirements. A customized dashboard was developed to display the energy data in a user-friendly way, enabling the operator to monitor and analyze the energy consumption of the machine over time.



Fig. 5 Energy monitoring dashboard

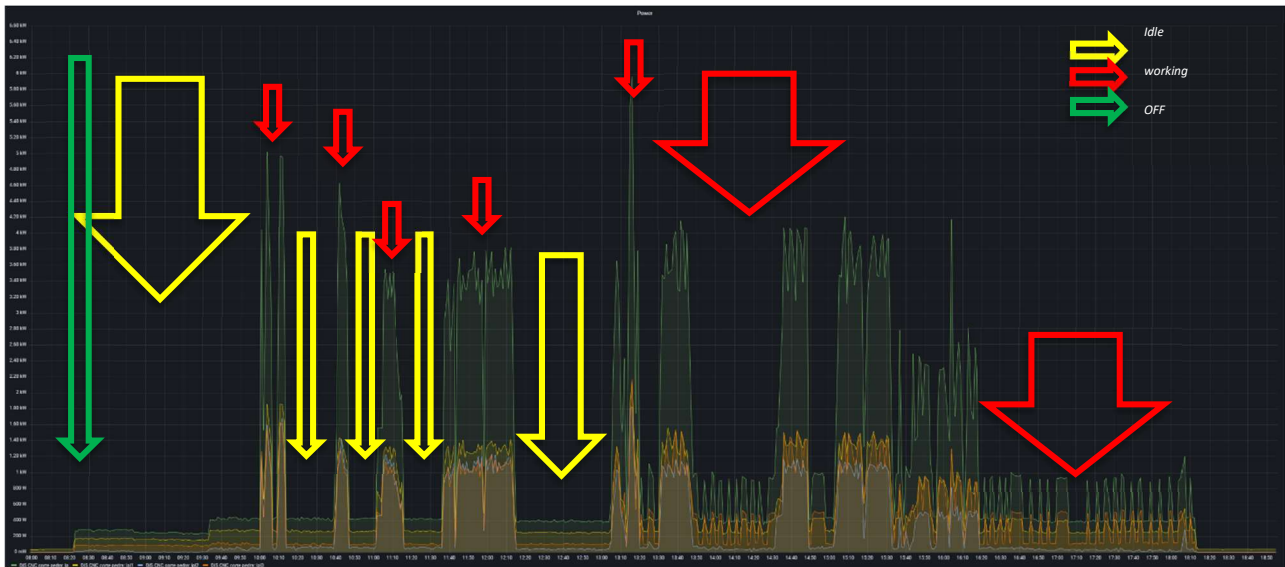


Fig. 6 Example of a workday power consumption of the CNC machine

This study builds on previous research in the field of energy-efficient technologies in the natural stone industry [14-16] by employing a comprehensive approach to instrumenting and monitoring the natural stone cutting CNC machine. The use of multiple sensors and devices from different manufacturers enabled a more comprehensive analysis of the machine's energy consumption and performance, which can inform future efforts to optimize machinery and reduce energy consumption in the industry.

## Results

Over the course of several months, we collected data on the operation hours of the machine during work times and off work. The dashboard (Fig. 5) allowed us to find that the machine was operated for an average of 8.5 hours per day during work times, and for an average of 1.5 hours per day during off-work times.

The data collected on the energy consumption of the machine, including active energy, reactive energy, voltage, and power factor. Our analysis of this data showed that the machine consumed an average of 0.6kWh of active energy and 0.2kVAr of reactive with a power factor average is around 0.67.

The dips in voltage correlated with machine operation are not substantial showing that the energy supply is well within conformance parameters. Also, we could access the average energy consumption of the machine (Fig. 6) and the different values obtained during different types of operation, such as the average consumption in Idle is around 400Wh, cutting the average is around 800Wh in simple operations up to an average of 3kWh during heavy load operations.

Using sensors attached to the machine, we estimated the movements of the machine in the x, y, z, and table directions. We found that the machine performed an average of 30 thousand movements per day.

The vibration sensors collected the machine natural frequencies and while we are yet to fully analyse and validate this data we anticipate that they will provide valuable insights into the performance and maintenance of the machine.

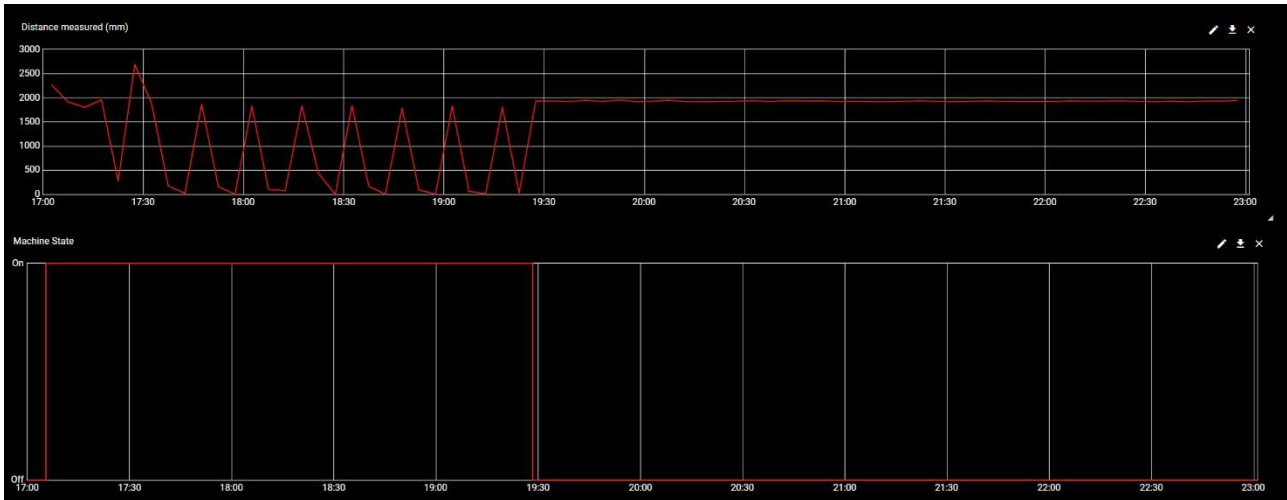
The distance sensor and light sensors correlated directly and themselves with the energy monitoring giving us insight on the redundancy of these equipment (Fig. 7)

## Discussion

Our results demonstrate the effectiveness of energy monitoring devices in optimizing natural stone cutting CNC machines. By collecting and analysing data on machine operation and energy consumption, we can

be a cost-effective solution for smaller companies or those with limited resources for new equipment.

Limitations of our study include the relatively short duration of data collection and the small sample size. Future research could explore the long-term



identify areas for improvement in both machine performance and energy efficiency. Energy quality was successfully evaluated, and power factor correction was recommended for both energy efficiency and financial

effectiveness of internet of things and energy monitoring devices and their impact on energy savings and machine maintenance in larger natural stone cutting operations.

*Fig. 7 Correlation between light and distance sensors*

savings.

One of the key findings of our study was the CNC operation time management evaluation. There is a significant amount of savings and efficiency to be obtained from a better utilization of the machine. In average the machine is in Idle 22% of the workday. It is typical for the machine to be turned on early in the morning without working for the next hour and to be let in idle during the lunch time. Considering this amount of time is not productive for the company and that the idle energy consumption is significant, this highlights a potential area for optimization through scheduling adjustments or automatic shut-off mechanisms to reduce energy consumption during non-working periods.

Another important insight we gained was the relationship between different sensors such as machine movements and energy consumption between light sensor and machine movements. Further analysis of this data could lead to improved maintenance and safety measures. Also, it shows that some of these sensors are redundant enough in terms of the information provided giving more and better options to different types of equipment and factories where there might constraints to the installations of some of these sensors.

Our study adds to this body of research by demonstrating the feasibility and effectiveness of retrofitting older machinery with energy monitoring devices and other internet of thing's devices, which can

## Conclusion

The natural stone industry is one of the oldest and most important industries in the world, providing materials for a wide range of construction and architectural projects. However, it is also known for its high energy consumption, which has negative environmental and economic impacts. To address this issue, our study focused on the instrumentation of a natural stone cutting CNC machine with energy monitoring devices, vibration sensors, and distance sensors.

Our data collection methods enabled us to gain insights into the machine's operation, energy consumption, and movements in different directions. We found that the machine's energy consumption was not optimized and there were opportunities for improvement in different areas such as workday operation, energy quality, and energy efficiency. By analysing the data collected from the sensors, we identified potential solutions that could lead to more sustainable and efficient natural stone industry practices.

Our work highlights the importance of energy-efficient technologies in the natural stone industry and provides practical insights for companies looking to improve their machine performance and workflow. By using sensors and energy monitoring devices to optimize the use of existing machinery, companies can reduce their energy consumption, lower costs, and contribute to a more sustainable industry.

The correlation between the distance and light sensors with energy monitoring devices also indicates the redundancy of certain instruments, which can help factories make better decisions on the equipment they need. Additionally, we found that the machine consumed energy even when it was not in operation, which suggests that there is room for improvement in the machine's energy efficiency. These findings have important implications for energy management and machine maintenance, as well as for the overall sustainability of the natural stone industry.

Future work could include further analysis of data collected from the vibration and distance sensors, which could provide more detailed information on machine

performance and maintenance. Additionally, the implementation of additional energy-efficient technologies in the natural stone industry could lead to significant improvements in energy efficiency and sustainability.

In conclusion, our study demonstrates the potential benefits of instrumentation and monitoring in the natural stone industry. By implementing energy-efficient technologies and using sensors to optimize machine performance, companies can reduce their environmental impact and improve their bottom line. Our work provides valuable insights for future research and practical applications in the natural stone industry.

**Acknowledgments:** This paper is supported by “Sustainable Stone by Portugal” – A project part of the Portuguese recovery and resilience plan with the objective of adding value to the Natural Stone industry supporting a digital, sustainable, and qualified future.

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# Structural use of high-strength granites: mechanical behaviour and applications

C. Pinto<sup>1\*</sup>, J. Fonseca<sup>2</sup>

(65) Centre of Materials and Building Technologies (C-MADE), University of Beira Interior, cmp@ubi.pt

(66) University of Beira Interior

**Summary:** *Some types of granites, with compressive strength higher than 120 MPa, can have a great potential for structural use, in which the mechanical properties of the material permit lightweight structures. Some possibilities of long-span arch bridges and other special structures can be referred to as potential cases of new stone structures. Those solutions correspond to different structural applications of high-strength granites when compared with traditional stone structures, like classical arch bridges. The proposed applications imply the combination of stones with modern techniques like prestress, in which the high mechanical strength of the material is useful. However, the structural use of high-strength rocks implies knowledge about their mechanical behaviour, like the internal destruction under compression loadings and the failure modes. As described in the present paper, the mechanics of spalling and its consequences seem to be a central question for the structural use of high-strength granites, on which research should be focused.*

**Keywords:** *granite, strength, structures, spalling.*

## 1. Introduction

Some types of granites have a compressive strength higher than 120 MPa, which seems to be a reason to analyse the potential use in structural applications. The use of granite as structural material comes from immemorial times, as exemplified by some Cathedrals. However, in these types of structures, the equilibrium is a question of proportions and shape (Heyman, 1997), with a small dependence on the material's compressive strength.

More recently, a stone bridge in China (Danhe Bridge - HighestBridges.com) (Chen, 2009), with an arch of 146 m span, established a world record concerning stone bridges. However, being a massive structure, it seems possible to consider that the material is not used efficiently, concerning compressive strength, particularly for high-strength rocks.

The examples referred to above, being important uses of natural stones, do not reflect the potential application of natural stones, in which good mechanical properties are used in an assumed way. For example, this type of application is suggested by (Leonhardt, 1979), for long-span and slender prestressed beams.

The structural behaviour of classical stone structures is strongly conditioned by the absence of tensile strength, particularly in the joints. That conditioning was not a limitation for the master builders due to a design based on the relation between forms and forces (Billington, 1979). Currently, seems not to be viable a type of design like classical constructions. However, there is an opportunity to use classical materials in relevant

structural applications, in association with modern solutions, and apply adequately the principles used by the past master builders.

The present paper aims to assume the structural use of high-strength granite in the current scenario, focusing on the material mechanical behaviour, and some potential applications, like a prestressed granite pillar (Pinto, 2008).

## 2. Mechanical behaviour of high strength granite

### 2.1. Compressive strength and failure modes

The granite used in the research described in this paper was obtained in quarries in Pinhel, district of Guarda, Portugal, with the designation as "Pinhel gray granite" (Figure 17).

Compressive strength was evaluated through compression tests with different variants (Pinto, 2008), with a minimum value near 110 MPa and a maximum value of 156 MPa.

In tests with specimens with dimensions 100x100x200 mm<sup>3</sup>, a mean compressive strength of 150 MPa was obtained. The failure occurred in a very explosive way, with global fracture and loss of material (Figure 18).

Figure 19 contains the axial stress-axial strain diagram obtained in a test until a maximum applied stress of 70 MPa. From that diagram, a value of 47 GPa for the elasticity modulus was obtained.



Figure 17: Pinhel gray granite specimen.



Figure 18: Specimen with dimensions 100x100x200 mm<sup>3</sup> after failure.

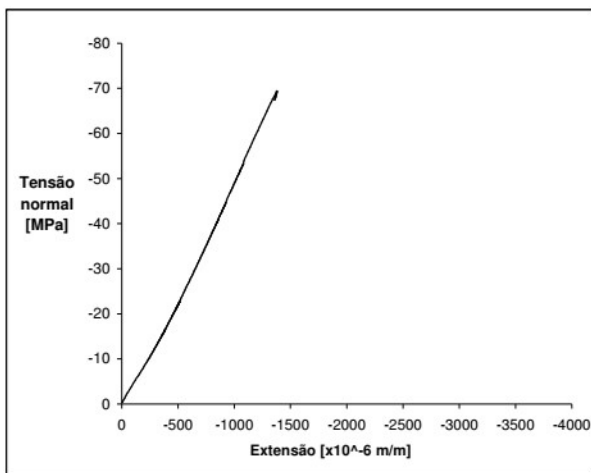


Figure 19: Axial compression stress vs.. axial strain until a maximum compression test of 70 MPa.

In a variant test, the effects of surface smoothness in the faces in contact with the compression test machines were evaluated, with specimen dimensions 50x50x200 mm<sup>3</sup>. It is considered a smooth surface when cutting by a diamond disk is adopted.

Values of Table II show that the roughness of the surface reduces the compressive strength of the specimens, due to the stress concentration associated with the irregular contact between the specimen ends and the testing machine steel plates. Figure 21 and Figure 20 show the failure modes for the two variants of Table II. The roughness of the end surface (Figure 20) implies the internal destruction processes near that zone, with the formation of a wedge element that creates an axial fracture. Contrarily, with two smooth surfaces failure occurs by loss of corner materials and an axial fracture (Figure 21). The observed differences between the two tests show the importance of smooth surfaces of specimens, to avoid decreasing compression strength.



Figure 20: Failure of specimens with a smooth end and the other rough

Table II: Compressive strength with different ends in contact with machine end plates.

Variant	Compressive strength [MPa]
Two smooth ends	146
One smooth end and one rough end	109





Figure 21: Failure of 50x50x200 mm<sup>3</sup> with both smooth ends.

A particular test was made using sandboxes in the ends, in an attempt to minimize the effects of the steel end plates. Two types of sand were used in the tests, with different compressive strengths and grain sizes. Values of compressive strength near 50 MPa and 100 MPa were obtained for the two types of sand. After the test, crushing of the sand was observed (Figure 23), which can be simultaneously associated with the failure of specimens. The higher value of compressive strength was obtained using the finest and strongest sand.



Figure 22: Specimen with end sandboxes.



Figure 23: Crushed sand after the compression test.

The failure modes permit to understand the effect of the crushed sand. Failure was characterized by the successive formation of mini-pillars between axial fractures. The specimen was successively subdivided into different parts (Figure 24), starting from the first division into four parts (Figure 25). The division process occurred under constant compression loading.

Tests with sandboxes simulate the effect of mortars in compressed joints between high-strength granite elements. The compressive strength of mortars will determine the compressive strength of the elements, independently of the high compressive strength of the granite. The crushed sand, like a deformable mortar, will create a transversal effect of tensile stresses (Figure 26) that results in the observed axial fractures. The brittle behaviour of high-strength granite facilitates the formation of axial fractures.



Figure 24: Failure with the division of the specimen into mini-pillars between axial cracks.



Figure 25: First division into four parts when maximum loading was attained.

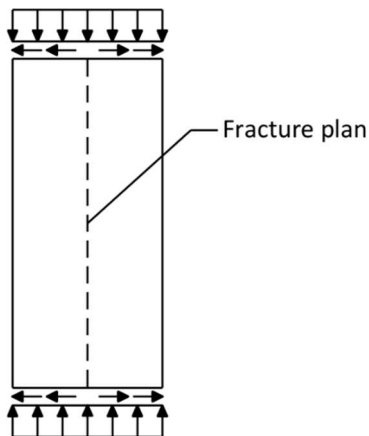


Figure 26: Transversal effect of crushed sand in compression tests with sandboxes.

## 2.2. Bending strength

The tensile strength of Pinhel gray granite was determined through three points bending tests. The material was tested for three types of specimens concerning three different directions of their cutting. Those directions were defined according to the information from the quarry about the strong and weak directions of exploitation.

Table III: Bending strength for three directions.

Bending plane	Tensile strength [MPa]
Normal to the strong direction	17,9
Normal to the weak direction	9,2
Normal to the intermediate direction	13,0

## 2.3. Models for failure

Internal destruction of rocks in compression is associated with the formation of cracks parallel to the principal compression stresses (Gramberg, 1988). Thus, in non-confined compression tests, cracks will appear in the axial direction and not in inclined directions, as proposed by Mohr-Coulomb failure theory, associated with shear failure. Different studies about the mechanical behaviour of rocks show the limitations of shear failure theories to describe the real behaviour of rocks, especially for high-strength rocks (Gramberg, 1988), (Martin & Chandler, 1994), (Diederichs, 1999). Thus, aiming for relevant structural use of natural stones, as high-strength granite (Pinto, 2015), implies research about coherent models for the description and analysis of mechanical behaviour.

The proposed model for the internal destruction of rocks, like granite, corresponds to understanding the compressed material as a structural system formed by micro or mini-pillars between cracks (Figure 27 and Figure 28), (Pinto & Fonseca, 2013). Inclined planes of failure are the result of the system of micro-pillars, when the progression of cracks is limited (Martin & Chandler, 1994).

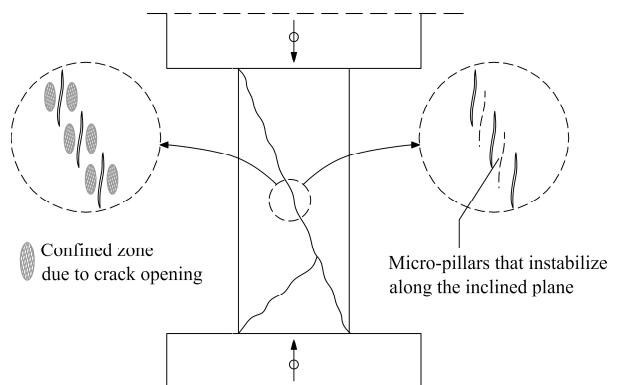


Figure 27: Failure by global deformation and simultaneous instability of micro-pillars forming inclined "sliding" planes.

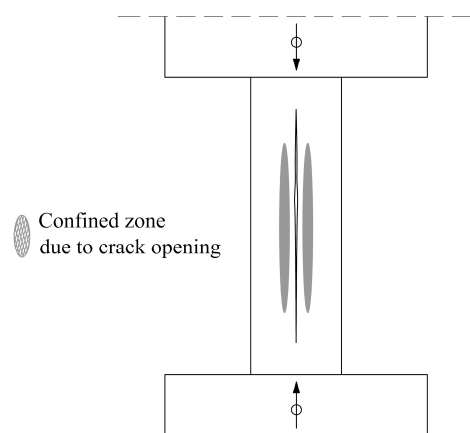


Figure 28: Progression of a long crack inhibiting adjacent cracks.

### 3. A structural application

#### 3.1. Description

An ashlar structure cannot generate internal tensile forces, to equilibrate the applied loadings. That limitation can be solved through an adequate choice of the relation between form and forces or using prestress. Instead of “natural” pre-compression generated in arch or vault structures by dead load, it is possible to apply compression forces using the prestressing technique (Chilton, 2000).

There are some examples of prestressed stone structures involved in beams (Sebastian & Webb, 2021b) (Ye et al., 2014b). However, the research at the University of Beira Interior aimed to develop solutions for long-span beams or slender elements subjected to high compression forces. These types of elements can be used in arch bridges, a new type of bridge construction (Fonseca & Pinto, 2019).

Figure 29 and Figure 30 describe a solution of a prestressed granite column (Pinto, 2008) with two external cables and lateral struts. Transversal stiffness created by the cables is mainly influenced by the cross-sectional area and the lateral opening (length of the lateral struts). Beyond the effect of pre-compression, the prestressing cables can create transversal stiffness to construct long-span beams or slender columns.

Friction between the smooth granite surfaces, and the prestress permits to have dry joints with the capacity to transfer transversal forces.

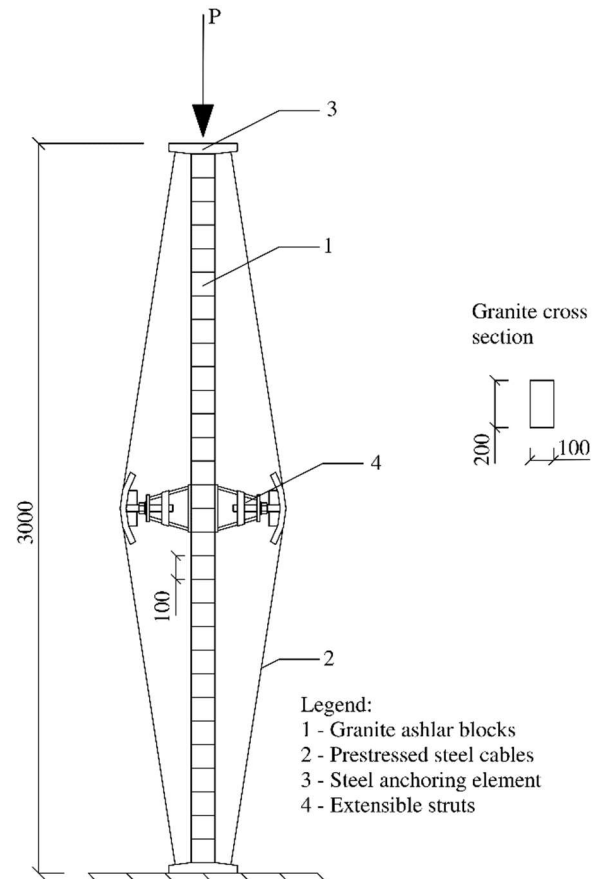


Figure 29: Frontal view of a prestressed granite column.



Figure 30: Image of a prestressed granite column.

#### Compression test

An axial compression test of a prototype confirmed the viability of the solution. The test was made with previous characterization of the defects and imperfections, with influence in the loading capacity and failure mode.

The compression test was made in five loading steps with increasing maximum loadings: 200 kN, 400 kN, 700 kN, and 1000 kN.

Figure 31 and Figure 32 contain the deformation curves in transversal and axial directions until the ultimate loading is equal to 1000 kN.

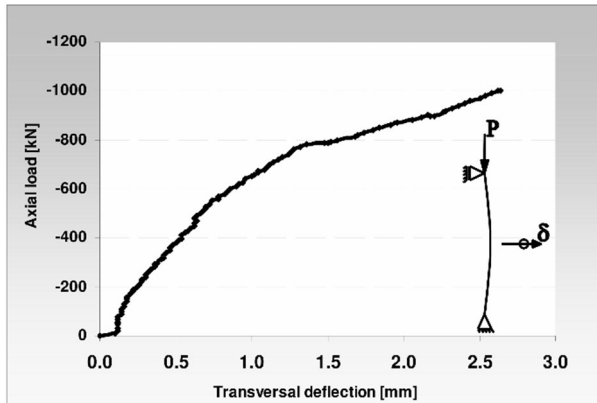


Figure 31: Axial loading versus transversal displacement curve of the tested prototype.

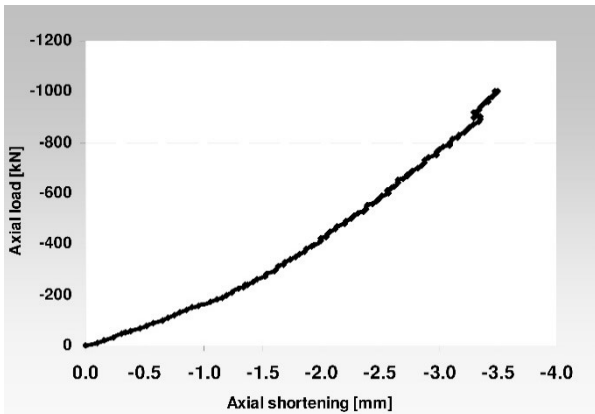


Figure 32: Axial loading versus axial shortening of the tested prototype.

Before the compression test, some imperfections and defects were observed. After the application of prestress some cracks were seen, possibly related to some initial damage to the granite prisms. The progression of those initial cracks was limited or inexistent, even for the maximum loading of 1000 kN.

The most relevant defect was related to a non-plane face of the steel plate where the cables were anchored, and the external compression loading was applied (Figure 17).

The non-planar steel face implied a “knife” loading that induced transversal tensile loadings in the stone prisms. The resultant fracture had a significant progression until a maximum length of 1.9 m (Figure 34). The long crack created a slender sub-pillar whose buckling would cause the global failure of the column. However, the progression of the fracture and buckling of the sub-column was prevented by transversal reinforcement (Figure 35).

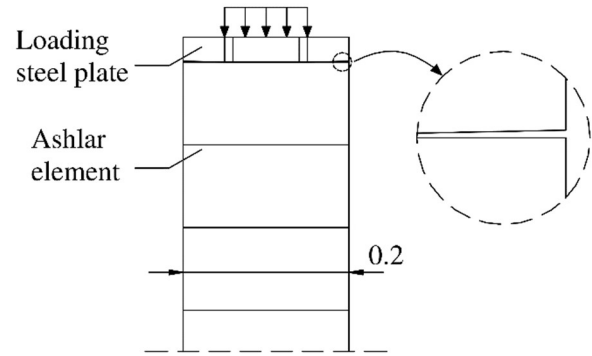


Figure 33: Scheme of the defect in the end of the pillar in which the compression loads are transferred only through the central part.

shows the fracture pattern in the reconstruction of the pillar after failure. The pillar failure occurred after the rupture of reinforcement r2, due to fracture c5, formed during the last loading phase.

Fractures c1 and c2 were observed during the pillar construction and could have resulted from initial imperfections of the granite prisms. It is important to note that these initial fractures did not progress during compressive loading, because global conditions were favourable and prevented its opening.



Figure 34: Longitudinal fracture with a maximum length of 1.9 m.



Figure 35: Transversal reinforcement applied to prevent progression of long axial crack and buckling of the sup-pillar.

Internal destruction of rocks like high-strength granite is a result of cracks formed by direct or indirect tensile stresses. Thus, understanding the failure of that type of material corresponds to identifying the source of those tensile forces and analysing the behaviour of the resultant system from a structural point of view.

The proposed structural use corresponds to slender columns using prestressing cables to produce bending stiffness and increase the critical loadings. The proposed solutions can be used for elements of arch bridges or beams.

The brittle nature of high-strength granite implies higher relevance of imperfections, like the non-planarity of the surfaces. The use of mortar can produce a reduction of compressive strength, as a function of the mortar strength. A smooth surface resultant from cutting with a diamond disc is adequate to avoid compressive strength reduction.

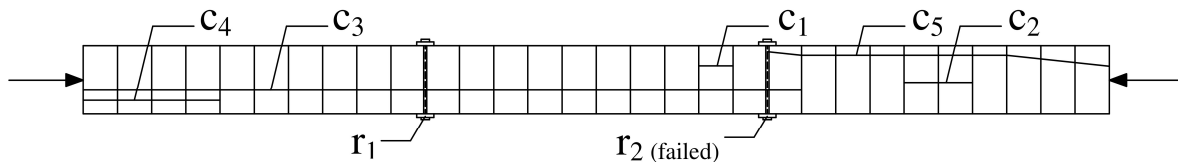


Figure 36: Scheme of the cracks c1 to c5 in the reconstitution of the pillar after failure.

The cracks c3, c4, and c5 had a significant and fast progression under the action of significant compression load.

The performed test shows the viability of the proposed solution to build slender columns or beams using high-strength granite. The tested pillar had different defects which conditioned the loading capacity. Thus, the obtained failure load of 1000 kN is a lower value in comparison to the possible maximum loadings obtained in pillars with better construction quality, concerning the planarity of the joints and adequate detailing of the end zones, where stones will interact with other materials, like steel.

The proposed system can be developed for special structures like slender arch bridges or footbridges. In all cases the association with other structural systems can be advantageous, being possible to build slender and lightweight structures.

## 4. Conclusions

The present paper shows the good mechanical properties of Pinhel gray granites, with high compressive and tensile strengths that can be explored. Those mechanical properties justify the evaluation of the use of the material in relevant structural applications.

**Acknowledgments:** This work has financial support from FCT/MCTES through national funds (PIDDAC), under the project C-MADE-UIDB/04082/2020.

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## **Sustainability in the Extractive Industry of Ornamental Stone in Portugal: Planning and Management of the Territory**

Catarina Santos, Célia Marques, Nelson Cristo

ASSIMAGRA – Portuguese Association of the Mineral Resources Industry, csantos@assimagra.pt

ASSIMAGRA - Portuguese Association of the Mineral Resources Industry, cmarques@assimagra.pt

ASSIMAGRA – Portuguese Association of the Mineral Resources Industry, ncristo@assimagra.pt

The activity of natural stone quarrying in Portugal, experienced a significant increase from the mid-1980s when several favorable factors were combined, such as a period of economic expansion, new markets access conditions, technological advances that allow the extraction of stone more optimally and the introduction of new financing systems adapted to the expansion of extractive activity.

However, in recent years, the extractive industry has encountered great difficulties, namely the exhaustion of licensed areas, together with the lack of alternative areas included in territory management instruments, which prospect the bottleneck of this activity and the absence of planning for many years, showing the disorganized way in which, the quarries are present in the territory.

The need to order the quarrying industry and rationalize the exploitation of resources, led the sector to rethink its way of acting in the territory. An example of this is the case of quarries in the “Maciço Calcário Estremenho”(MCE), which are currently organized according to Integrated Projects (IP), which aim to make extractive activity compatible with the conditions of territorial planning, with the purpose of planning the quarrying areas, the definition of methodologies and rules of exploration and landscape recovery, considering the occurrence of the geological resource and environmental imperatives.

As a way of materializing the IP on the field, the companies constituted together with the territory's management entities, Management Commissions of Extractive Clusters, which ensure: (i) support and management of horizontal measures to the quarries cluster to comply with EIDs; (ii) Direct relation and connection with entities to solve the measures to be implemented; (iii) support for the EID measures implementation and monitoring; (iv) support for the compatibility of IPRA's and IP's elements with individual quarry licenses.

The results of this model are monitored annually in order to evaluate the measures that are being implemented, with constant control of Air Quality, Ambient Noise, Groundwater Quality, Cultural Occurrences, Fauna and Flora. At the level of habitats, industry compensation measures are also carried out, such as the reforestation of degraded areas and the recovery and requalification of habitats.

Currently, the sector is working on a new territorial planning solution to face the constraints of the extractive clusters of the “Anticlinal de Estremoz” (Portuguese Marble Zone), based on the same organizational form tested in the MCE.

Keywords: resource, quarrying, territory, environment

# INDUSTRY 4.0 CONCEPT IN THE PORTUGUESE NATURAL STONE CLUSTER

C.E. Cremonini<sup>1</sup>, J.C. Vasco<sup>1,2\*</sup>, C. Capela<sup>1,3</sup>, A. Silva<sup>1,4,5,6</sup>, M.C. Gaspar<sup>1,7</sup>

- (1) School of Technology and Management, Polytechnic Institute of Leiria, Leiria, Portugal, \*[joel.vasco@ipleiria.pt](mailto:joel.vasco@ipleiria.pt)
- (2) Institute for Polymers and Composites (IPC), University of Minho, Guimarães, Portugal
- (3) Centre for Mechanical Engineering, Materials and Processes (CEMMPRE), University of Coimbra, Coimbra, Portugal
- (4) CEFAGE-UE, Center for Advanced Studies in Management and Economics, Évora, Portugal
- (5) MARE, Marine and Environmental Sciences Centre, Polytechnic Institute of Leiria, Leiria, Portugal
- (6) CIIC, Computer Science and Communication Research Centre, Polytechnic Institute of Leiria, Leiria, Portugal
- (7) ADAI-LAETA - Industrial Aerodynamics Development Association, Coimbra University, Coimbra, Portugal

**Summary:** *This research provides an overview of Industry 4.0 systems and technologies applied to the ornamental stone industry, focusing both on the effects on mineral resources and on the energy use in daily operation. Additionally, research and development initiatives that enable the ornamental stone industries to be more efficient and sustainable by addressing important issues such as economic growth, environmental impact, and social well-being are key to attain such objectives. Integral to these developments are the increasing digitalization of manufacturing systems and their integration with discrete and numerical models to replicate shop-floor operations. In addition, these technologies are essential for the efficient utilization of materials, be they raw materials or manufacturing instruments. The unique situation of the Portuguese ornamental stone industry is highlighted, and the ensuing benefits are described and thoroughly analysed.*

**Key words:** *Industry 4.0, Manufacturing systems integration, Mineral resources, Sustainability*

## 1. Introduction

With the current industry's ongoing digital transformation, the resulting increase in productivity and efficiency show to be the primary drivers of innovation and change across all sectors of our economy (Cheng et al., 2023; Du & Jiang, 2022).

Considering this impact, the European Union refers to the Industry 4.0 (I4.0) paradigm as the organization of production processes based on technology and devices autonomously communicating with each other along the value chain: a smart factory model of the future, using computer-driven systems to monitor physical processes, creating a virtual copy of the physical world, and making decentralized decisions based on self-organizing mechanisms (Ghobakhloo et al., 2021; Soori et al., 2023).

Such increasing digitalization of manufacturing industries in which physical objects are seamlessly incorporated into the information network, leads to production systems that are vertically networked with business processes within factories and enterprises and horizontally networked with spatially dispersed value networks that can be managed from the time an order is submitted through outbound logistics (Benitez et al., 2023; Z. Liu et al., 2022; Schubert et al., 2023).

## 2. The Industry 4.0 Paradigm

As a result of these developments, the distinction between industry and services becomes less significant, as digital technologies transform industrial products and services into hybrid products that are neither exclusively goods nor services. Indeed, both the Internet of Things and the Internet of Services are regarded as Industry 4.0 components (Malik et al., 2021; Peter et al., 2023; Pivoto et al., 2021).

Consequently, the key characteristics of Industry 4.0 paradigms (Nakagawa et al., 2021) can be summarized as follows:

- interoperability: the cyber-physical systems, such as work-piece carriers, assembly stations, and products, allow humans and smart factories to communicate with each other and keep connected;
- virtualization: by connecting sensor data to simulation models and virtual plant models, this creates a virtual copy of the smart factory;
- decentralization: the capacity of cyber-physical systems to make autonomous decisions and produce locally using technologies like 3D printing;



- real-time capabilities: these abilities to acquire and analyze data enable immediate delivery of the derived insights;
- service orientation: the increased data and knowledge of the system allow for and increase focus on robust real-time services to be performed and provided;
- modularity: the individual module replacement or expansion enables smart factories' adaptability to variable demands.

In addition, quality and documentation requirements are increasing. Industry 4.0 is a new development in an industry that has revolutionized and digitized its various sectors. This new paradigm introduces a new industry concept, the smartification and digitization of factories, to create intelligent factories using connected devices, data analysis, and artificial intelligence technologies to automate processes (Ching et al., 2022; Stock & Seliger, 2016).

Industry 4.0, also known as the fourth industrial revolution, addresses these challenges by facilitating end-to-end communication between all production-relevant IT assets and production-relevant physical assets. According to this implementation strategy, the primary aims of Industry 4.0 (Antonino et al., 2019) may be highlighted as:

- IT systems vertical integration in production and automation engineering;
- horizontal integration of diverse IT systems across the value-chain;
- engineering consistency throughout the entire lifecycle;
- product customization by means of small quantities or even as a single lot size;
- new social infrastructures for work.

This approach of the Fourth Industrial Revolution and the current trend of automation technologies in the manufacturing sector is primarily related to significant recent enablers such as the cyber-physical systems (CPS), the Internet of Things (IoT), and cloud computing.

In the Industry 4.0 paradigm, embedded systems, semantic machine-to-machine communication, IoT, and CPS technologies are bridging the gap between the virtual and real worlds. In addition, a new generation of industrial systems related to smart reconfigurable machines and factories is emerging to manage the complexity of cyber-physical environment production systems (Morgan et al., 2021).

### 3. Recent Trends of the Ornamental Stone Sector

According to the United Nations, industrial development is essential for both income generation and the enhancement of living conditions (Navarrete et al., 2020). In turn, appropriate infrastructures provide facilities for industry and society, while innovation boosts technological capacities and leads to the development of new skills (De Andrade Régio et al., 2017; Régio et al., 2016). These factors are key for the Ornamental Stone Industry as it allows passing from a resource exploitation paradigm to a sustainable value-chain of natural resources to be efficiently integrated into modern practices and solutions.

#### 3.1. Environmental sustainability vs. Economic growth

Integrating environmental sustainability with economic development and well-being is one of the greatest global challenges to reduce environmental degradation caused by economic growth and increase productivity (do more with less). Decoupling of resources and impacts is required to promote sustainable consumption and alter production paradigms, thereby facilitating the transition to a circular economy (Neves et al., 2020).

This integration is reinforced by two aspects: on the one hand, they are built slowly as the relationships between individuals share norms and mutual trust; on the other hand, they are also integrated with individuals from different spheres, but with common experiences. Such cooperation process includes a generalized incorporation of cross-fertilization of haves and have-nots, specifically technologies in the production and transformation processes, as well as the creation of suitable strategies for the production and promotion of Portuguese stone products, pre- and post-sale, support services, and internationalization (Carvalho et al., 2018a).

Much of this growth is related to the development model of the companies, *i.e.*, maturity is supported on a growth whose time assumes significant relevance. Particularly in Portugal, between the natural stone sector and the footwear industry a so-called cross-fertilization has recently taken place, *i.e.*, an exchange of knowledge between dedicated technologies of both sectors lead to mutual benefits and improvements. In this sense, the dynamics of cooperation between companies has been verified with significantly positive results, and its similarity with the evolution of the footwear sector should be highlighted (da Silva et al., 2020).

#### 3.2. Mineral resources

It is acknowledged that for some nations with natural mineral resources, these assets are essential for

promoting their sustainable development and, consequently, contributing to the growth of their economies (Dubiniński, 2013). Furthermore, these mineral resources are regarded as nonrenewable natural resources, and they can be metallic (including iron, copper, tin, and lithium) or nonmetallic (quartz, limestone, marble, granite, ardosia, gypsum, sand, and coal). As for Portugal, many of the available nonmetallic minerals are mineral aggregates, and Portugal is abundant in terms of quantity, quality, and variety (Amorim Dinis & Sousa, 2003).

Materials used in applications of Industry 4.0 must be able to withstand the rigorous requirements. Novel applications require advanced composites and responsive (smart) materials that are compatible with emerging manufacturing technologies such as 3D printing and CNC machining. As a result of the significant progress in fields such as nanotechnology, these materials must have properties such as light weight and improved mechanical properties, as well as a sustainable production and general life cycle. In the context of circular economy, products made from these materials must be designed and manufactured with these factors in mind (Nascimento et al., 2019).

The potential of a nation's geological resources can be viewed as an economic development factor with increasing strategic significance and a strategic opportunity that should be leveraged (Ketels & Memedovic, 2008). Thus, representing an economic/political advantage for a country's economic strategic mosaic, local mineral resources should allow for its global revenue and value creation (Andersson, 2020).

### **3.3. Energy usage and storage**

The recent advancements in the energy sector, most notably on what concerns energy storage, smart infrastructure, and renewable energy, have had a significant impact on the fourth industrial revolution. A device (such as an automaton, drone, or wearable) that can only operate for a few minutes has limited utility. The advancements in energy storage technologies, such as batteries, enable industry 4.0 devices to operate long enough to be beneficial in their operating environment. On the other hand, the new information and communication technologies (ICT) introduced as part of Industry 4.0 have produced new energy distribution and management technologies (Tsaramirsis et al., 2022).

In the future decades, few other chemical elements will be as significant from an energy standpoint as lithium. Recently, lithium has become an essential component of battery electrolytes and electrodes, requiring a purity level of greater than 95.5%. Due to its low atomic mass,

its charge and power-to-weight ratio is considerable. Lithium-ion batteries have the highest voltage and energy density of all rechargeable batteries and are favored for use in portable devices where high energy capability, low weight, and compact volume are essential (Heredia et al., 2020; Y. Liu et al., 2023).

## **4. 14.0 and the Portuguese Natural Stone Sector**

### **4.1. Impact of the Portuguese Natural Stone Cluster**

The growing need to access new markets and invest significantly in product, service, and process innovation leads to the formation of strategic alliances that can be crucial for a company's international operations. This can only be accomplished by exporting goods and/or services, or by physically entering the market in which it intends to operate.

Current companies should adopt networked business models, as there is a growing recognition that the peripheral technologies of one company are frequently the core activities of others. For cooperation to be effective, the parties involved must share the same values and pursue the same ends. The latter must be explicit, coherent, and motivating in addition to being realistic, consistent, hierarchical, and quantifiable.

In this context, specialized clusters (Ketels, 2011) consist of geographically-located groups of entities (companies and institutions) whose interconnections develop alongside relevant technological support and are founded on how the players manage to benefit from stakeholder relationships, sustain and strengthen competitive advantage, which is primarily due to three factors: (1) the interrelationships between the various participants; (2) the speed and ease of information and knowledge circulation; and (3) its accessibility.

Turning potential competitors into allies and suppliers of complementary products and services contributes to the development of new businesses, thus allowing for the neutralization of prominent rivals as threats and the creation of network economies by businesses with complementary assets.

These relationships are organized and focused on a thematic cluster, thus enhancing the performance of the companies, enhancing the competitive advantage of the members, fostering innovation, and attracting knowledge based on internal investment. Innovation occurs when those involved are in proximity, fostering the flow of information and the exchange of ideas. To such an end, the creation of a Mineral Resources cluster should be strongly committed to a partnership and network dynamics.

In 2009, the Portuguese Natural Stone Cluster was established to increase the national and international visibility of the country's Ornamental Rocks Sector and as a positioning tool for a group of companies involved in extraction, processing, and production of machinery and equipment under the VALORPEDRA project. Later, in February of 2017, the Cluster Portugal Mineral Resources was formally recognized as the new name.

The role of national universities has been fundamental in organizing the ideas that result in innovative and attractive projects in the Portuguese Mineral Resources cluster foster the attraction of companies to become more competitive and efficient. In this sense, the creation of an alliance with the purpose of overcoming the difficulties experienced in the Portuguese Stone Sector, implies that there is mutual trust and great commitment of all the stakeholders.

In this context, the main objectives of the Portuguese Mineral Resources cluster are:

- produce knowledge and induce innovation;
- enhance value creation and internationalization;
- promote efficiency in the use of resources;
- empower the stakeholders of the cluster;
- strengthen synergies between complementary activity sectors.

#### **4.2. Portuguese Research and Development Projects and the Portuguese Natural Stone Sector**

In this section, some of the most relevant Portuguese mobilizing research and development projects will be addressed.

##### **4.2.1. The JetStone Project**

The JetStone Project was created based on the collaboration between ten entities in the business, science and technology system. This project took place between 2005 and 2008 with the aim of increasing production flexibility and reducing the response time to market demands. According to the Technological Diagnosis of the Natural Stone Sector and Intervention Areas made by CEVALOR, in 2010 (Cabral De Melo et al., 2017), with the implementation of this mobilizing project, it was possible to:

- develop new and improved solutions for automatic cutting;
- differentiate and improve the product quality by using laser technology, which allowed the creation of non-slip marble and granite floors;
- develop an automatic raw material stock control system integrating a virtual simulation of the

capacities of the company's existing cutting machines, which consequently guaranteed the optimization of raw materials and final product quality.

The design and creation of nine lean prototypes and six test and quality control devices, all validated in a production environment and currently used by several dozen manufacturers, was the project's most noticeable outcome (Antunes Da Silva, 2014).

With the aid of the technologies created as part of the Jetstone Mobilizer Project, ornamental stone businesses can now transform raw materials in a flexible manner and achieve noticeable reductions in raw material waste, which leads to productivity gains and increased responsiveness to microorders in the domestic and/or European market (Silva et al., 2020).

##### **4.2.2. The InovStone Project**

The Inovstone Consortium was responsible for the second wave of digital technology advancements for Portuguese Ornamental Rocks. Nineteen businesses and organizations from the scientific and technological system participated in a more extensive mobilizing collaboration that presented the Inovstone Mobilizing Project (Antunes Da Silva, 2014).

This research and development initiative, which was created to carry on the leanstone ideology's implementation after the foundation of Cluster VALORPEDRA, has a useful life between 2010 and 2013. It featured a collaboration of 15 entities, including natural stone enterprises, equipment firms, associations, and entities of the Scientific and Technological System, and produced eight solutions (PPS) and fourteen prototypes.

The goal of this initiative was to develop PPS-level innovations—Product, Process, and Service—to give the Natural Stone cluster's member companies the tools they need to innovate in these three areas. The following principles served as the project's foundation:

- increased involvement of a larger group of the natural stone cluster's companies and entities;
- attract critical mass to the sector, thus increasing the value of the entire chain;
- seek the development of novel, contemporary, and distinctive manufacturing techniques that might add value and improve the standard and competitiveness of businesses in the industry;
- encourage the expansion of the cluster's exports either through product improvements in terms of pricing and technology or through a shift in mindset and attitude based on the businesses that have achieved notable levels of success by implementing the aforementioned approach.

The execution of such driving Research and Development (R&D) programs, like the JetStone and InovStone projects, has significantly influenced the growth of the competitiveness of the stone industry in Portugal (Frazão, 2016).

The use of the technologies developed under the InovStone project enables one to respond to medium-sized commercial opportunities in a flexible and competitive manner on global markets, as well as to significantly reduce raw material and energy waste, according to a number of recent studies conducted by authors from various Universities. The same research suggest that ornamental stone enterprises have been given a substantial push by the InovStone technology to transform their operations into Industry 4.0 (Silva et al., 2020).

#### 4.2.3. The FlexStone Project

The FlexStone Research and Development Project, which ran from 2010 to 2013, produced the third generation of digital technology advancements for the natural stone technology. This FlexStone Project, supported on the leanstone concept, produced two innovative, cutting-edge prototypes. The following short-term goals were attained through the execution of this project (Cabral De Melo et al., 2017).

- ensure continuity in established markets with room for expansion;
- introduction new markets;
- increase product innovation and differentiation to better serve markets that are more demanding;
- continue the companies' technological development and integration;
- promote the Portuguese Natural Stone - Stone.PT brand and the sector's and companies' communication strategies;
- meet the demands of demanding markets (the primary markets for Portuguese natural stone), invest in product certification and long-term guarantees of the same.

The advancements made as part of this consortium project have led to technologies that allow businesses in the ornamental stone sector to respond to customized projects of any scale optimally and in an integrated manner, with a further reduction in waste of raw materials, energy, and ecological footprint, as well as a guarantee of a significant increase in productivity. This project has produced technology advancements that permit ornamental stone enterprises to bridge the gap between their operations and Industry 4.0 (Silva et al., 2020).

#### 4.2.4. InovStone 4.0 and BIM technologies

The fourth wave of technological developments in Industry 4.0 for ornamental stone was portrayed by the Inovstone4.0 Mobilizer Project (InovStone 4.0 Project - Advanced Technologies and Software for Natural Stone, 2022). Seven institutions from the Research and Innovation Systems (universities and others) and 17 companies have been involved in the development and implementation of the InovStone 4.0 Project. They provide a crucial role in ensuring the application of science, fostering interactions between universities and businesses, and, most importantly, ensuring the sector's ongoing evolution. In the framework of BIM procurement in the construction industry, it seeks to promote InovStone 4.0 Collaborative Production (Antunes Da Silva et al., 2016).

BIM is a type of software that enables the representation of a building's physical and inherent characteristics as a guided model using objects connected to a database. This innovative approach to building design is altering the construction sector and will shift procurement, or how we buy, toward standardized goods. Portugal's ornamental stone industry places a strong emphasis on product personalization, which gives it a competitive advantage. This highlights the significance of the InovStone 4.0 mobilizer project, which will establish the necessary conditions through its collaborative network for the ornamental stones to give competitive advantages with the BIM implementation mandate (da Silva, 2018).

This creative dynamism in the decorative stone cluster is best demonstrated by the InovStone 4.0 mobilizer project. The approach that economically stimulates knowledge, turns concepts into products, fosters innovation, and increases industry and company competitiveness is the promotion of technical progress. Portugal needs such technological progress that stimulates economic expansion to compete in markets like Germany, France, and England, we must develop new products' technologies, manufacture them, and promote them through strong brands.

It is already clear from the industrial environment tests being conducted in these businesses that the prototypes will perform better than anticipated and that ornamental stone businesses can advance towards collaborative manufacturing and smart manufacturing by using these ground-breaking technological solutions. InovStone 4.0 technologies are designed to enable customers and suppliers to collaborate in a growing, personalized, and optimized co-creational approach to commercial opportunities of any scale. This is especially true with Building Information Modelling (BIM) users (Silva et al., 2020).

### 4.3. Benefits and sustainability in the stone industry

Companies today are moving beyond traditional industry silos and fusing into network ecosystems, creating new potential for innovation for many existing organizations as well as new problems. This one of the key motivations that mobilize the Portuguese ornamental stone industry to reduce waste and increase flexibility began with the first wave of technological development projects back in 2004.

In this context, the following research question may arise: What is the impact of R&D Mobilizing Projects on the sustainability, efficiency and image of Portuguese ornamental stone businesses? In response, it should be noted that ornamental stones are naturally available, non-renewable resources that must be used and handled in an efficient manner in order to reduce waste output and increase profitability. Considering the global average for the extraction of ornamental stones, only a third of the raw stone material that was harvested makes it to the market as a finished good. The other two thirds are waste. For any plan of competitive economic growth in nations aiming for sustainable development, the proper valuation of ornamental stones is key.

Due to this and the fact that ornamental stones are non-renewable natural resources, they must be extracted and handled properly for both economic and environmental reasons (Carvalho et al., 2018b).

Portugal's stone industry depends on its companies' capacity to provide value in the global marketplace. Portugal, while being a relatively small country, has a vast variety and quantity of stone to sell to the global market. Stone is an easily exportable product (Frazão, 2016).

The Portuguese stone industry has chosen a framework that aims to improve sustainable consumption and production patterns through sustainable management and efficient use of natural resources. This program, which takes the shape of R&D mobilization initiatives in business consortiums, sought goals and challenges to go forward in all directions and produce powerful firms that are more environmentally, socially, and economically sustainable (da Silva & Almeida, 2020)

The extraction and optimized transformation of ornamental stones using new machine tools have a clear impact on competitiveness, with improved energy efficiency, less waste, less water consumption, and faster operation times. As a result, new and improved products and processes can be developed, taking better advantage of the resource's natural attributes, and introducing modern and sustainable technologies (Carvalho et al., 2018b).

It is accepted that Portugal has a history with stone and a considerable variety of rocks that can be used as ornaments. The country's history is preserved in stone and by stone and can be found by simply traveling through the area that is now known as Portugal (Antunes Da Silva, 2014).

Portugal possesses a sizable and diverse stock of stones that are appropriate for aesthetic purposes, despite its modest area (Siegesmund & Snethlage, 2011).

There are two categories of marketable products: stone sold raw, and stone sold processed. The industrial process is typically divided into four phases: extraction, transportation, transformation, and marketing. According to the established commercial language, there are four different types of rock that have been discovered in Portugal and used for decorative purposes: marbles, limestones, granites, and schists (Antunes Da Silva, 2014). Once extracted, the various stone types provide a diversified range of goods depending on the various transformation processes, which are then distributed to the widest range of markets (Galetakis & Soutana, 2016).

One of the world's top manufacturers of ornamental stones, Portugal has had an average yearly increase in tons of 4% over the past 50 years (Carvalho et al., 2018b). According to information made available by the National Association of Extractive and Manufacturing Industry (ASSIMAGRA - Associação Portuguesa dos Industriais de Mármore, 2021), Portugal's sector is distinguished by the extreme importance it presents in its economy, giving rise to 2,112 businesses that employ 13,380 people nationwide and had a turnover of around 872 million euros in 2018.

The most prevalent technologies in this industry are some of the most cutting-edge in the world, used by Portuguese processing companies : (A) Stone Jet, an advancement in technology tested in the tanning industry, which enables the plate classification and defect detection through appropriate equipment, software, and an operator who makes the arrest and digitalization of the raw material's flaws prior to the cutting process; (b) Stone Cut, which utilizes the same technological features but in a more straightforward manner, permits an increase in production and straight cuts; (c) Computer Numerical Control (CNC), which enables digitalized numerical control of five axes, permits the modeling of parts in multiple forms, enabling the creation and implementation in 3D projects; Last but not least, (d), the Stone Cut Mille, is a device that combines various features, including scanning, sorting, finishing, cutting, and modeling. All the above mentioned are key technologies that successfully integrate the current Industry 4.0 concept.

## 5. Summary and Conclusion

This investigation into the Portuguese ornamental stone market serves as an excellent case study on the significance of industry digitalization. To turn the ornamental stone business from a rigid, traditional industry into a flexible, collaborative, and digitalized industry for the future of society, a strategic set of research and development initiatives were conducted in the Portuguese ornamental stone sector and are now being conducted at an industrial level.

These R&D initiatives cover the whole stone industry value chain, providing a comprehensive understanding of all processes, including marketing, extraction, design, preparation, planning, production, finishing, and dry layout of stone products.

Such digital transformation is crucial because it directly addresses the Triple Bottom Line principle and affects all environmental, social, and economic elements. As a result, the health and wellbeing of the stone industry's stakeholders is effectively maintained. Also, as shown, ornamental stone natural resources are used more effectively. Finally, improving business outputs has positive consequences on the economy and on people's well-being.

This shows that the Industry 4.0 concept not only addresses technological improvements and industry principles, but also approaches holistically dedicated sectors, such as the ornamental stone cluster to improve the efficiency and outputs through all of its value-chain.

**Acknowledgments:** The authors acknowledge the Inovmineral 4.0 Project - *Programas mobilizadores clusters de competitividade e outras dinâmicas coletivas* - 14/SI/2019 COMPETE2020 (POCI-01-0247-FEDER-006375), for the support on their work.

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# DIGITAL TWINS AND THE ORNAMENTAL STONE INDUSTRY: KEY FACTORS

C.E. Cremonini<sup>1</sup>, J.C. Vasco<sup>1,2\*</sup>, C. Capela, A. Silva<sup>1,4,5,6</sup>, M.C. Gaspar<sup>1,7</sup>

- (1) School of Technology and Management, Polytechnic Institute of Leiria, Leiria, Portugal, \*[joel.vasco@ipleiria.pt](mailto:joel.vasco@ipleiria.pt)
- (2) Institute for Polymers and Composites (IPC), University of Minho, Guimarães, Portugal
- (3) Centre for Mechanical Engineering, Materials and Processes (CEMMPRE), University of Coimbra, Coimbra, Portugal
- (4) CEFAGE-UE, Center for Advanced Studies in Management and Economics, Évora, Portugal
- (5) MARE, Marine and Environmental Sciences Centre, Polytechnic Institute of Leiria, Leiria, Portugal
- (6) CIIC, Computer Science and Communication Research Centre, Polytechnic Institute of Leiria, Leiria, Portugal
- (7) ADAI-LAETA - Industrial Aerodynamics Development Association, Coimbra University, Coimbra, Portugal

**Summary:** *This paper discusses the application of Digital Twins (DTs), which are virtual replicas of physical systems, to a real-world industrial application of an ornamental stone manufacturing company. Implementing DTs can save time and money during prototype design and provide continuous diagnostics and optimization throughout a machine's production run. In addition, DTs are beneficial for cost savings because they consider physical processes holistically. In addition, the article emphasizes that the implementation of a DT must be done with care and that there are still numerous problematic issues to be resolved. The article also discusses the incorporation of additional software tools to enhance DTs and the significance of comparing the virtual model's results to the actual ones. The article concludes that Digital Twins technology will have a significant impact on business value and that it is beneficial to use this technology despite the need for caution during the design phase.*

**Key words:** *Digital Twins, Industry 4.0, Simulation, Applications, Ornamental Stone Sector.*

## 1. INTRODUCTION

Digitalization and automation have become increasingly essential to the market as the demand for industrial production efficiency has risen. This has required the development of more realistic and reliable simulation tools. In comparison to hardware models, digital models offer greater flexibility regarding use cases and lower resource requirements (Rosen et al., 2015; Tao et al., 2019). To standardize the creation of digital twins and reduce production costs, the Association of German Engineers (VDI) and the Association for Electrical, Electronic and Information Technologies (VDE) have published a guideline on virtual commissioning (Janda et al., 2019).

Digital Twin (DT) technology is one of the most prevalent areas of Industry 4.0, with the goal of achieving total virtualization through digitization. DT involves incorporating data in both directions between a physical and virtual machine. A DT is a virtual model of a real-world physical system that simulates its behaviour and monitors operations using real-time data sent by sensors on that system (Tao et al., 2019). When applying DTs to prototyping, engineers can validate the efficacy of industrial concepts without the need for prototypes by simulating the whole device. Early detection and

correction of design flaws significantly reduces the cost of rectifying them during operation (Rosen et al., 2015).

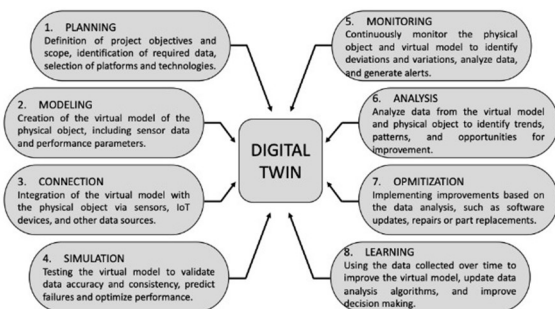
In complex industrial processes such as manufacturing and production, simulations are essential to optimize processes. Hence, DT technology can increase manufacturing process efficiency and generate cost-effective consumer products. DT technology is also essential in engineering education to maintain curricula and education content and to provide students, instructors, and businesses with dedicated and efficient DT tools (Schuster et al., 2016; Tao et al., 2019).

CAX software has grown to be an integral component of any design office that applies complex simulations. They can be used for calculations (CAE), design (CAD), and simulation of manufacturing (CAM). These programs include Inventor, Solid Edge, Solid Works, and NX. Through the Mechatronics Concept Design (MCD) application, the NX program provides tools for interactively simulating the complex motion of a mechatronic system (Fait et al., 2022). The MCD application consists of several subcomponents, including system engineering, mechanical concept, simulation, mechanical, electrical, automation, and design collaboration (Holzer et al., 2022).

The use of DTs in the industry allows companies to improve production efficiency, reduce costs, and increase product quality. With accurate, real-time simulation, flaws can be detected and corrected even before production, thus avoiding waste, and lost time. In addition, DT technology can be used to maintain optimized production operations and increase the efficiency of the manufacturing process (Tao et al., 2019; Yang et al., 2017).

In conclusion, DT technology represents a significant step forward for industry and engineering education. With increasingly accurate and realistic simulation tools, valuable results are generated that can be efficiently used in real-world applications (Fait et al., 2022; Janda et al., 2019). Notably, in addition to its benefits for production efficiency, DT technology can also bring about significant sustainability advancements. By simulating production processes in their entirety, it is possible to identify areas for enhancement to reduce the waste of energy, materials, and natural resources and to minimize the production's environmental impact. This can assist businesses in meeting rising demands for more sustainable and accountable production (Liao et al., 2017a; Tao et al., 2019).

When broken down into well-defined stages, implementing a DT in a dedicated industry can be carried out efficiently and effectively. Figure 1 illustrates the process of developing and deploying a DT in the Ornamental Stone Sector, from data acquisition to results analysis. Understanding these stages is crucial for the effective deployment of a DT to such a real-world application.



## 2. LITERATURE REVIEW

Industry 4.0 is one of the driving forces behind the development of DT technology. The implementation of the DT methodology is regarded as a crucial element for the transformation of manufacturing and, consequently, the industry's future. DTs allow for the optimization of the product development process, the reduction of

costs, the acceleration of time to market, and even the prediction and avoidance of potential production problems (Janda et al., 2019; Tao et al., 2019).

One of the best-known tools for creating DTs is Mechatronics Concept Designer (MCD), a subsystem of Siemens' PLM software system and a platform on NX developed to address mechatronic product design and automatic motion simulation issues. It comprises of two systems: the physical system and a virtual system that contains all the information about the physical system. (Tao et al., 2019), (Tao et al., 2022).

Maslow's pyramid is a psychological theory that depicts five levels of human needs, from physiological to self-actualization (Maslow, n.d.). This pyramid is used in Industry 4.0 to represent the hierarchy of cyber-physical systems, from sensors to management software, that facilitate automation of production processes and data-driven decision making (Ryan & Deci, 1985). Figure 2 depicts the application of Maslow's hierarchy of needs to the ornamental stone industry, specifically a CNC milling machine for the production of stone slabs.

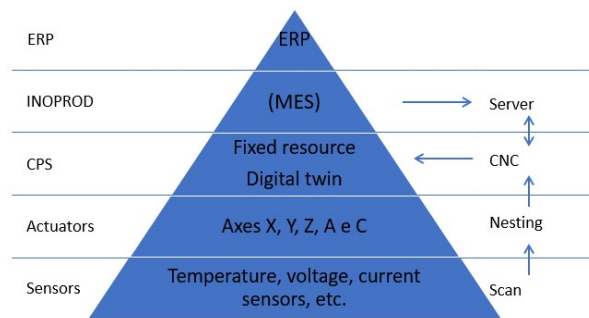


Figure 2: Maslow's pyramid applied to Industry 4.0: hierarchy of cyber-physical systems and the role of management software.

The sensors at the base of the pyramid collect data from the physical environment, such as temperature, pressure, and vibration, and send it to the cyber-physical system. The second level of the pyramid, the actuators, receives this data and then acts on the physical environment, conducting tasks such as moving machinery and activating control systems (Lee et al., 2015; Liao et al., 2017a).

The cyber-physical system occupies the third level of the pyramid and is responsible for processing and analyzing the data received from the sensors and actuators and making decisions based on this information. This layer consists of a network of devices, including computers, sensors, and actuators, that collaborate to automate production processes (Liao et al., 2017a).

In the context of Industry 4.0, the Manufacturing Execution System (MES) is an instrument belonging to the fourth level of Maslow's hierarchy of needs. This tool is essential for the automation of production, as it

permits the accumulation and analysis of production process data in real time, resulting in enhanced efficiency, productivity, and product quality. The use of MES is directly related to the DT concept, which involves the creation of a digital replica of a product, process, or system to enable simulations and virtual optimizations prior to actual implementation (Rosen et al., 2015; Tao et al., 2019).

At the apex of the pyramid is the ERP (Enterprise Resource Planning) management software, which utilizes the collected data to optimize production processes and make strategic decisions. This software is responsible for integrating the various company departments, including production, sales, finance, and logistics, and provides the management team with an integrated view of the company's production processes and performance indicators (Liao et al., 2017a; Rosen et al., 2015).

In this way, Maslow's pyramid is a useful analogy for conceptualizing the hierarchy of cyber-physical systems in Industry 4.0 and how the integration of these systems can lead to increased production efficiency, higher quality, and reduced costs (Liao et al., 2017a).

### 2.1. Digital Twin Technology Applications

DT technology has numerous applications across numerous industries. In the manufacturing industry, it can be used for product design, simulation, and optimization, making it one of its primary applications. DTs can be used to create a virtual model of a product or process, which can be used to simulate various scenarios and optimize the design and production processes. This can result in substantial cost reductions and a shorter time to market (Liao et al., 2017a; Tao et al., 2019).

This technology also has applications in the field of maintenance and repair. By using DTs to monitor equipment performance and forecast potential failures, maintenance can be performed proactively, thereby reducing downtime and boosting productivity. It has been demonstrated that predictive maintenance is more effective and less expensive than traditional reactive maintenance (Liao et al., 2017a; Tsai et al., 2020).

DT technology has extensive and diverse applications, and its implementation has the potential to revolutionize numerous industries. Future applications of digital siblings are likely to become even more inventive as technology continues to advance (Tao et al., 2019).

## 3. METHODOLOGY

This paper explores the capabilities of automation in solving real-world problems by employing digital tools and DT technology. Using a virtual model created with Siemens NX Mechatronic Concept Designer (MCD) and Simit, the objective is to provide practical and comprehensive applied examples.

This strategy intends not only to improve problem comprehension, but also to increase efficiency and accountability in their resolution.

The kinematic model is derived from simplified 3D models for simulation purposes. Individual rigid bodies are defined and then connected via joints, allowing the virtual machine to replicate the movements of a physical machine. This is crucial because it ensures that the simulation is as accurate as feasible.

In this case-study, the MCD software serves as a tool for the mechatronic part of the machine, allowing the definition of the 3D model and all of its movements, in addition to sensors, physical characteristics, and input/output signals. This allows for extremely precise control over the virtual machine's behaviour.

The first step in creating a DT is to define the physical and mechanical characteristics of the machine's various components. The Mechatronic Concept Designer enables you to simulate the dynamic behaviour and interactions of the various parts by allowing you to add rigid body and collision body properties, as well as designate the movement possibilities of their connections (sliding joint, ball joint, etc.). This enables the simulation to be extremely accurate and realistic.

MCD is a commercial application within Siemens NX software that facilitates physics-based simulation and is utilized in virtual commissioning and the creation of digital twins. The advantage is the close proximity to the Siemens NX CAD tool, which makes the software simple to use. The reuse library enables validation of the designed product by allowing the user to rapidly add data to the functional model, such as joints, motion, sensors, crash behaviour, and other kinematic and dynamic properties for each component (Fait et al., 2022).

In addition to enabling the definition of signals and signal adapters, the MCD software allows for the development of event-based machine behaviour governed by virtual sensors. Position function control is used to define the speed, acceleration, force, etc., at which the part can move within its defined position based on the connections. This allows the simulation to be extremely accurate and effective in solving real-world issues (Holzer et al., 2022).

#### 4. DISCUSSION

Prior to contemplating the use of a dedicated DT in a production system of the Ornamental Stone Sector, several steps and factors must be taken into account beforehand.

The main goal of this applied DT is to reflect reality as accurately as possible of the corresponding manufacturing system of the ornamental stone industry. Due to the complex dynamics involved, however, some features are extremely difficult to replicate. Future optimization relies heavily on the successful design of a digital counterpart. Real-world mechanical and electronic issues can be studied and verified in a safe, regulated environment. These research findings can be used to predict and prevent prospective system problems before they occur.

In this ornamental stone device, as for other industrial applications, older mechanical applications are frequently replaced by servo drives and intelligent motion control, thereby enhancing the system's dynamic and power characteristics. Additionally, mechatronic systems tend to be more flexible and endure less wear and strain.

The MCD does not simulate sensors in reality; rather, it replicates their functionality. It does not address their physical characteristics. Real sensors can get unstable in certain environments (due to magnetism, rapid motion, etc). As also observed by Jaya (Institute of Electrical and Electronics Engineers Malaysia Section et al., n.d.), this can result in unanticipated errors during prototype development, highlighting the need to test and optimize a system prior to implementation.

When considering the use of STEP files, these can be imported into the NX mechatronics concept designer, but they are converted into "Siemens part files," an internal Siemens data format, which imposes import restrictions. In addition, MCD permits mesh-by-mesh collision, with the limitation that each mesh may contain no more than 1024 vertices. MCD can therefore only exhibit original meshes with a maximum of 1024 vertices. In addition, the vertices are always distributed uniformly throughout the body and cannot be locally concentrated to obtain a more accurate representation of collision-heavy regions. Future projects must consider software and hardware constraints and adjust simulation models accordingly.

In our DT applied to the specialized ornamental stone system, the MCD enables OPC signal mapping with the PLC and other applications. This mapping enables the OPC server and simulation object to communicate. This means that simulation objects can be controlled by a

(virtual) PLC and simulation processes can function as control process parameters. Future concepts include attempting to communicate with a real PLC and programming a rudimentary control using the JOINT coordinate system. It is imperative to remember that the simulation environment must be meticulously calibrated and tested in order to produce accurate results.

In addition, incorporating this DT into our system can result in improved decision-making, cost and labour savings, and an increase in system efficiency. The use of this type of DT can also lead to a reduction in environmental impact, as it permits the early identification and resolution of problems that may result in the waste of natural resources.

Nevertheless, it is key to remember, that a digital counterpart can never be an exact representation of the actual system. As mentioned by *Liao et al.* (Liao et al., 2017b) there are actual limitations related to the available hardware and software, as well as the accuracy of the employed models that affect the sensing operating environment of the actual system which may differ significantly from the simulation environment, thus leading to some degree of inaccuracy in the measured results.

Therefore, it must be considered that such applied DTs must be meticulously designed and validated to ensure their reliability and accuracy. Also, it is key to enhance that our DTs must be continuously updated and improved to reflect system changes.

In a nutshell, it can be highlighted that the implementation of a dedicated DT to an applied system of the ornamental stone industry allows for improving the design, testing, and operation of existing solutions, resulting, therefore, in more efficient, safer, and sustainable systems.

#### 5. CONCLUSION

Digital twin technology could seem to be an overly complex simulation, but if applied properly, it can save a significant amount of time and money when designing prototypes. Continuous diagnostics and optimization throughout a machine's entire production time will be a game-changing factor.

The successful design of a digital twin will be crucial for future optimization, as the complexity, quality, and adaptability of the complete production or individual products will continue to increase. Therefore, digital twins should be structured with as much design and production flexibility as possible.

DTs can provide real-time data on the condition and status of physical assets through continuous learning. Management will be able to monitor systems, develop more precise plans, and forecast, for instance, the time required to service critical components.

DTs are particularly useful for saving costs because they allow for a holistic view of physical processes, which will be particularly true in situations where large amounts of data will be required. However, it will be necessary to approach the design phase very carefully because there will still be many problematic aspects to consider beforehand to the simulation of the system.

When considering the implementation of a dedicated DT to an industrial-level application due to the need to analyse and characterize complex real-world interactions in a simplified approach. However, and as referred by Tao *et al.* (Janda *et al.*, 2019; Tao *et al.*, 2019), even though it may be very difficult to predict and simulate real-world complexity, it will still be worthwhile to use this technology.

For the case of MCD, there is a great potential for DT improvement and functionality enhancement. The integration of other software packages will enable to include hydraulics, heat transfer and other effects to the digital model. These increased functionalities may belong to a second age of DT and its validation will require to compare the digital results to the results obtained on the ornamental stone system.

For communication between different programs and devices, the OPC server will be a good solution. Also, the use of the mechatronics module will make it possible to use the robot to perform the simulation using a pre-

programmed motion sequence (in PLC program). The XML data exchange method will be applied between MCD and other PLM subsystems, which will help integrate the data of MCD and other subsystems, to enhance the advantages of information exchange, provide the implementation method of continuous connection in relevant systems, and enhance the core competitiveness of products. Hence, as mentioned by Lysek *et al.* (Konstantinov *et al.*, 2017; Lysek *et al.*, 2019) it will be the basis of collaborative design in MCD, which will not only be suitable for Siemens PLM systems, but also for other similar software systems or Internet development engineering.

In general, DT technologies offer significant opportunities for increasing the efficacy and precision of design and production processes. Although there are still some additional obstacles to overcome, such as the need to incorporate additional numerical models and the complexity of the physical world's interactions. In addition, the integration of other software tools and communication between programs and devices can contribute to the enhancement of the efficacy of DTs.

The DT technology will likely play an ever-increasing role in the future of the industry, and businesses that adopt and implement this technology will enjoy a substantial competitive advantage. Adoption of this technology has a high potential for cost reduction and enhancement of product's commercial value, making it a prospective strategy for companies in a variety of industrial sectors, particularly the Ornamental Stone Industry.

**Acknowledgments:** The authors acknowledge the Inovmineral 4.0 Project - *Programas mobilizadores clusters de competitividade e outras dinâmicas coletivas - 14/SI/2019 COMPETE 2020 (POCI-01-0247-FEDER-006375)*, for the support on their work.

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# GEOHERITAGE ELEMENTS BETWEEN EXPLOITATION AND CONSERVATION. CASE STUDIES FROM ROMANIA

D.C. Papp<sup>1\*</sup>, V. Cetean<sup>1</sup>

(1) Geological Institute of Romania, email of the 1<sup>st</sup> author: [mail.deliapapp@gmail.com](mailto:mail.deliapapp@gmail.com)

## Abstract:

A geological heritage element is typically defined as a site or an area of variable extent that possess special scientific or landscape value and that can provide essential information about a unique geological event or phenomenon. If the landscape value of a heritage element is easily recognized, the scientific value is often disregarded without the constant involvement of the scientific community. The current work aims to inventory and promote some rock occurrences from Romania that are of particular importance for the history of geology or for the explanation of some geological phenomena. The inventory list includes type-locality for petrographic types (as dacite and ditroite), concretions (trovants) occurrences with high aesthetical value, type-locality for sedimentary formations, and some fossiliferous sites. If some of them are in restricted areas and benefits from protection, others are currently exploited as construction material or are subject of decay.

One of the most relevant examples is the Poieni quarry in the Apuseni Mountains that represents the type-locality of dacite. Dacite is a volcanic rock formed by rapid solidification of silica-rich lava that is intermediate in composition between rhyolite and andesite. The term dacite was introduced in the scientific literature by Guido Stache in 1863 and now is generally assumed in the petrographic literature. Etymologically, the word dacite derives from Dacia, the ancient name of the present Romanian territory. Currently, the old quarries for crushed stones are merged into a single large modern quarry. Andesites, dacites and microgranodiorites are extracted for road and railway construction. In the absence of any warning regarding the importance of the dacite occurrence and without any protection, the type-locality for this petrographic type is at risk of being lost.

Another relevant example is the overuse of trovants as ornaments for buildings and gardens. Trovants are sandstones concretions with a harder structure than the stratigraphic layer in which they develop. They occur as nodular, spheroidal, ellipsoidal, discoidal, or dendritic mineral aggregates with a massive, concentric, or stratified texture, ranging in size from a few millimetres to a few meters. Their scientific and educational importance was recognized by the establishment of the open-air museum in Costești (Vâlcea county). In contrast, other occurrences (e.g. Feleac concretions) are completely unprotected and the trovants can be extracted and utilized without any regulation.

Using specific criteria, the degradation risk was assessed for all the inventoried sites. Some of them have high degradation risk requiring specific geoconservation plans. Degradation risk was found to be low for the sites located within National or Natural Parks, which provides the framework for legal protection. A policy for the balance between conservation and exploitation is to be found and integrated in the national/local legislation, carrying the ideals of protection, education, and sustainable development.



## Stone filament for hybrid systems of additive manufacturing of products in composite materials

Dora Sousa<sup>1</sup>, M. Leite, M. Garcia, Pedro Amaral

<sup>1</sup>dsousa@tecnico.ulisboa.pt, IDMEC, Instituto Superior Técnico, Portugal

The extractive industry sector in Portugal is historically very dependent on the construction market, therefore, exposed to the volatility that is characteristic of it.

This work is an important step towards a more efficient and sustainable use of natural resources, which is based on their growing appreciation, continuous optimization of production processes and reduction of waste. The project is expected to contribute to opening the door to the use of endogenous resources, mainly from the central region, namely the limestone exploited there, in advanced and innovative technologies such as additive manufacturing.

This line of research aims to develop materials, innovative compounds, from the transformation of extracted natural limestone, for application in advanced technologies of additive manufacturing by extrusion of thermoplastic matrix compounds.

The limestone powder is dried and sifted, mixed with up to 50% PLA or another thermoplastic through melt blending methodology. A 3devo filament maker was used to spool the filament (1.75 mm). A 3devo granulator was previously used to shred the recycled polymers prior to use in the filament maker.

In Italy (Esposito Corcione et al., 2018) demonstrated the suitability of Lecce Stone waste as a filler for high loaded bio-composite polymers (PLA) for the 3D printing of ornamental industrial products.

Scanning electron microscopic investigations of PLA/stone composite filaments were performed on the fractures surfaces to evaluate defects, such as air inclusions, the sample morphology and analyze the dispersion of stone particles in the PLA matrix.

Esposito Corcione, C., Palumbo, E., Masciullo, A., Montagna, F., & Torricelli, M. C. (2018). Fused Deposition Modeling (FDM): An innovative technique aimed at reusing Lecce stone waste for industrial design and building applications. *Construction and Building Materials*, 158, 276–284. <https://doi.org/10.1016/j.conbuildmat.2017.10.011>

# FIRST INVENTORY OF HISTORICAL QUARRIES ASSOCIATED TO THE ARCHITECTURAL HERITAGE OF ANDALUSIA (SPAIN)

Álvarez Areces, E<sup>1</sup>; Fernández Suárez, J.<sup>1</sup>; Martínez Martínez, J.<sup>1</sup>; Orche Amaré, P.<sup>2</sup>; Baltuille Martín, J.M.<sup>1</sup>

(1) CN IGME-CSIC – Instituto Geológico y Minero de España, [e.alvarez@igme.es](mailto:e.alvarez@igme.es)

(2) Servicio de Minas. Secretaria General de Industria y Minas. Junta de Andalucía.

**Summary:** *The historical quarries associated with the construction of the historical architectural heritage have a great importance, since it provides historical, geological, and technological information, and establishes the relationships between the building and the physical environment in which it was built. This set of information is essential for understand the building, its production and construction from the quarry to the construction site. In this work are presented the results obtained in the Autonomous Community of Andalusia, in southern Spain, with the study of 675 monuments, and the location of 798 quarries associated with its construction. Emblematic buildings such as the Alhambra in Granada, the Cathedral of Sevilla, or the Mosque of Córdoba, among others, have been included in this research. These works will continue in the future to have comprehensive information on the historic quarries associated with the construction of the numerous and valuable Spanish architectural heritage.*

**Key words:** *quarry, architecture, geology, rocks construction, Andalusia (Spain)*

## Introduction

The different civilizations throughout history have left a legacy in stone through their constructions and architecture. All this architectural heritage represents the common past and there are numerous scientific and technological initiatives for its study, conservation and dissemination. In this sense, historical quarries have achieved a certain prominence in recent decades, with research groups, at national level (Spain) and at European level, dedicated to the study of these issues and with a high scientific production. Since 2014, the Geological and Mining Institute of Spain (CN IGME-CSIC) has been developing the National Inventory of Historic Quarries associated with architectural heritage (INCHaPA project), with the main objective of locating quarries associated with the construction of important heritage. Spanish architecture, becoming an excellent tool to deepen the knowledge of architecture, its production and its relations with the physical environment.

This paper presents the results in relation to the Autonomous Community of Andalusia, located in the south of Spain, with an extension of 87.600 km<sup>2</sup> representing 17% of the total territory of the country. To these results must be added those obtained in the previous project in the Autonomous Community of Extremadura, both complete approximately 25% of the

entire territory of Spain. In the future, new territories are completed in order to have a complete inventory of the country in the medium term. This inventory will provide unpublished and interesting information in different fields of knowledge, geological, historical, architectural, technological, socio-economic, etc.

## Methodology

This work develops a systematic methodology for the location of historical extraction areas (Fort, 1996; Álvarez Areces, 2015; Baltuille Martín et al., 2017; Fernández Suárez et al., 2020). This systematic methodology, developed by IGME, presents the following aspects (Fig.1).

### *Query of historical documentation*

Information on the situation of historical quarries can be obtained by consulting the following documentation: historical archives, construction factory books, memories of intervention projects in the building, old maps, historical aerial photography, institutional databases, maps of industrial and geological rocks and minerals, etc.

### *Sampling in the architectural-artistic heritage*

As a general rule, the samples will be taken in areas where they do not affect the visual value of the work, trying to do it on the interior faces of the blocks and in areas with less aesthetic value. The quantity of sample

taken must be adjusted to the analyses and tests that will be necessary for the characterization of the materials.

#### *Petrological characterization of materials*

To locate the quarries, it is essential to have a thorough knowledge of the materials used in the construction of the monument. Generally, the studies to be carried out are petrographic, mineralogical, paleontological content, geochemical characterization, petrophysical characterization.

#### *Regional geological documentation*

In this phase, the cartographic work of the Geological Survey of Spain, with its geoscientific information, is particularly important, especially The Geological Map of Spain at a scale of 1:50.000 (2<sup>nd</sup> Series) and the GEODE (scale 1:50.000 continuous digital geological mapping). Other cartography like, Industrial Rocks Map (1:200.000) or Database of Industrial Rocks and Minerals (BDMIN), are interesting.

#### *Outcrop selection*

Once located on the map the formations that may have similar characteristics to those used in the construction of the Historical-Artistic Heritage, the outcrops should be selected on the basis of: geographical characteristics, topographic characteristics, and aerial photo recognition.

#### *Recognition of outcrops*

In this phase, it is necessary to carry out a classical methodology for the study of geological materials, with specific geological mapping and sampling.

#### *Specific geological mapping*

The detailed geological mapping is of great importance for the location of quarries, because it allows the delimitation of the different types of materials. Once the area of extraction of the stone materials has been identified, several vestiges of the extractive work can generally be found: Traces or imprints that, broadly speaking, can be included within the following categories: quarry face, bench of exploitation, ashlar fragments, marks on the rock massif and marks on ashlar.

#### *Database and INCHaPA geographic information system*

The data set is included in a database that stores patrimonial, geological and technological information, as well as a geographical information system where

extraction marks, exploitation areas and quarries are georeferenced.

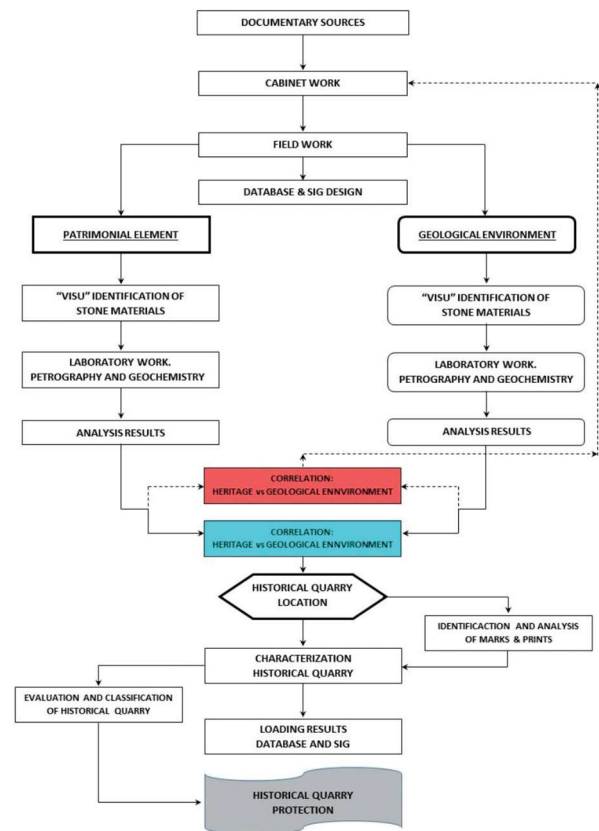


Fig. 1. Methodological scheme developed by the Natural Stone and Monumental Heritage Unit of the Geological Survey of Spain used during the development of the INCHaPA Project.

### **Objectives**

The main objectives set out in this Research are:

- Identification and characterization of the rocks used in the construction of the architectural heritage of Andalusia.
- Location of historical quarries associated with architectural heritage.
- Characterization of historical quarries, georeferencing signs and extractive marks.
- Petrological characterization of quarry rocks.

The partial objectives include:

- Incorporation of the data obtained in a National Database of Historical Quarries associated with Architectural Heritage (BDINCHaPA).

-Carry out a technological, geographical and temporal study from a historical perspective, with the establishment of conclusions.

-Determine possible archaeological sites associated with the quarries.

-Promote protection figures, drawing up maps with the historic quarry sites, for their conservation, future exploitation, tourist or educational use.

### Research results

The location of historic quarries is of great importance from an architectural point of view, since they allow information on the production of the architectural complex, work in the quarry, rock accumulation for construction and construction planning.

The study of architectural heritage and stone construction materials has a significant visibility in recent decades, and there are numerous research groups working in this line of research, in Spain and also in Europe.

The joint study of the building and its quarries provides new information, and in some cases resolves questions that traditionally had no answer. Likewise, the location of the quarries associated with the architectural heritage is of great importance from a historical and cultural point of view, since it provides information on communication routes, the technology available in certain historical periods and the historical and geographical evolution of the populations. Another important aspect is its scientific interest, since with the location of the quarry, rock of a sufficient size is available to be able to carry out petrological and durability tests, allowing to know the physical-mechanical characteristics of the construction rocks and their future behavior.

In the case of Andalusia, the proposed work methodology has allowed us to have an overall vision of the architectural heritage in the region and its relationship with the physical environment, with geology and rocks. The results of this research may be the starting point for new studies in the historical, archaeological, geographical field, etc., with further advance knowledge about the use of construction materials in heritage in the south of Spain, movements and transport of materials from their extractive areas (quarries) to the place where they are used (buildings), and finally the existence of stone production centers and workshops with an influence on the territory and on architecture in different historical periods and architectural styles.

In the context of the research, a total of 675 monuments belonging to the eight provinces that are part of the Autonomous Community of Andalusia have been studied, in Almería (60 monuments), Cádiz (60), Córdoba (102), Granada (98), Huelva (56), Jaén (126), Málaga (56) and Seville (117). These monuments correspond to different typologies, the most numerous being those corresponding to the religious architecture (278 monuments) including cathedrals, churches, monasteries, hermitages, etc. Defensive architecture (246 monuments) is also widely represented, including castles, towers, fortified churches, walls, etc. Regarding civil architecture, a total of 116 monuments corresponding to palaces, noble houses, theaters, etc. were studied. Finally, architecture was studied in relation to public works (25 monuments) among which bridges, fountains, roads, etc. stand out, and finally megalithic architecture (4), historical-monumental ensembles (3) and funerary art (3) (Fig. 2), (Fig. 4).

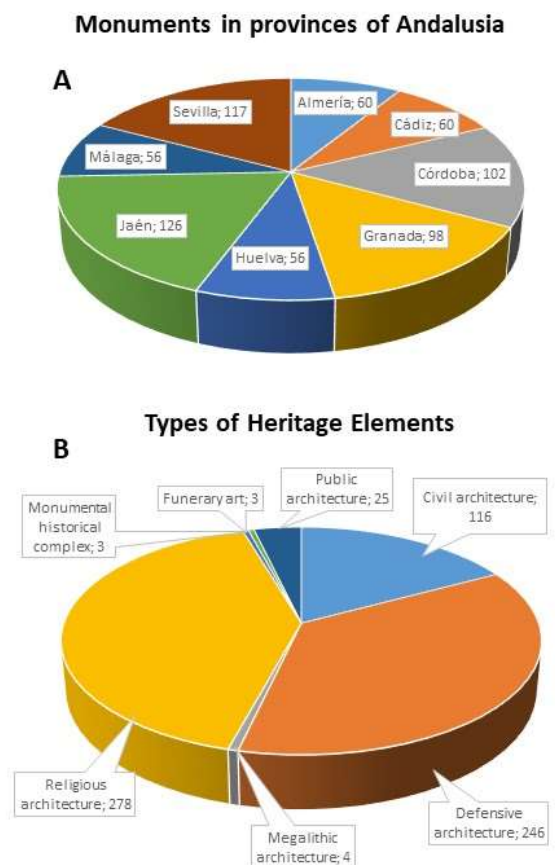


Fig. 2. A. Monuments in provinces of Andalusia, B. Types of Heritage Elements (monuments).

In relation to these architectural elements and after applying the methodology explained in this work, 798

associated quarries have been located. In these quarries various types of igneous, sedimentary and metamorphic rocks are exploited, the most numerous being limestone quarries, followed by calcarenite, sandstone, granite and marble.

The types of quarries located are divided into open-air quarries, underground quarries, and mixed quarries, and in them, masonry, ashlar masonry, brick, lime, as well as mill stones or other elements of daily use are obtained as a product for construction.

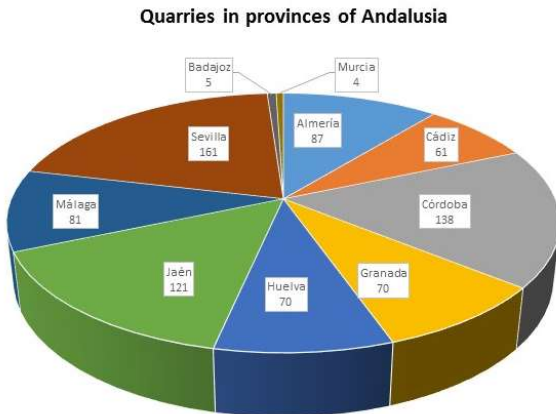


Fig. 3. Quarries associated to architectural heritage in provinces of Andalusia and others (Badajoz, Murcia)

The distribution of quarries in the different provinces of Andalusia is: Sevilla, Córdoba and Jaén are the provinces in which the most quarries associated with architectural heritage have been located, with a total of 161, 138 and 121 quarries, respectively. The provinces of Almería and Málaga, 87 and 81 quarries respectively, and finally the provinces of Huelva and Granada with 70 quarries located in each of them, and 61 quarries in the province of Cádiz (Fig. 3) (Fig. 5).



Fig. 4. Architectural heritage in Andalusia. A. Mosque in the city of Córdoba (Córdoba), B. Jabalquinto Renaissance Palace in Baeza (Jaén), C. Walls in the city of Ronda (Málaga), D. Cathedral of Seville (Seville), E. Alcazaba of Almería, F. Jerez de la Frontera Cathedral (Cádiz).

The location of large-scale quarries also allows them to be classified according to their periods of activity, contextualizing them in different historical periods. This aspect allows to determine exploitation techniques, to know the planning of the work in the

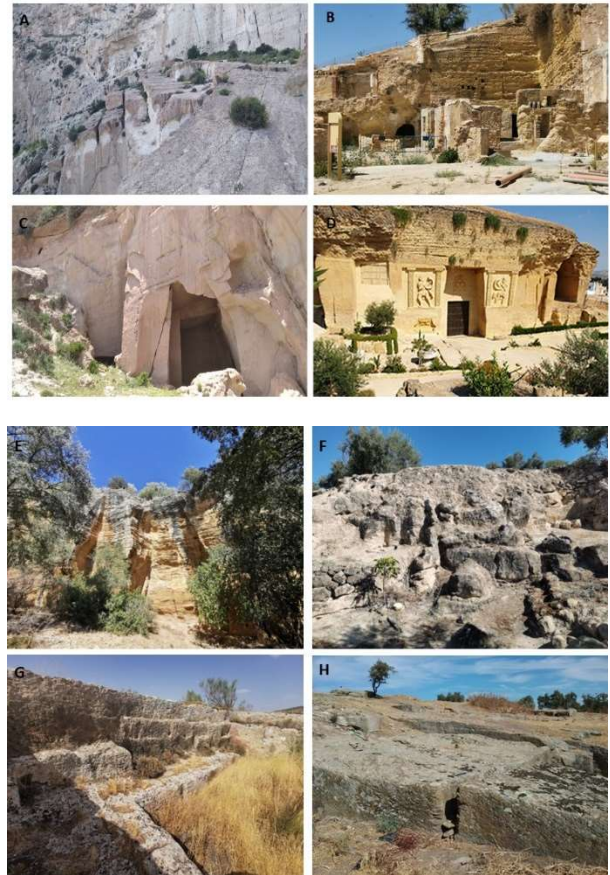


Fig. 5. Quarries associated to architectural heritage. A. Open pit quarry in Puerto and Bayana (Almería), B. Open pit quarry in San Pitar, Vélez Málaga (Málaga), C. Underground quarry in El Tesoro and Cuatro Cuevas (Almería), D. Osuna Mixed Quarry (Seville), E. La Albaida open pit quarry (Córdoba), F. Atalayón Baeza extractive area (Jaén), G. Vadolosyesos open pit quarry (Málaga), H. San Nicolás del Puerto quarry (Seville).



*Fig. 6 Historical-extractive marks associated with the different exploitation methods. A. Wedges in the Atalayón extractive area, Baeza (Jaén), B. Hole marks in the Vélez Blanco quarries (Almería), C. Quarries of San Pitar in Vélez -Málaga (Málaga), D. Peak marks in Lora del Río quarry (Sevilla).*

quarry and its strategies. The conservation, in many of them, of historical-extractive marks provides us with interesting technological information in relation to the different historical periods that in the future will deserve a classification and analysis (Fig. 6).

### Protection and recovery of historic quarries

The study of historical quarries is today an essential element for a better knowledge of architectural and monumental heritage works. Its interest, from the constructive point of view (facilitate rehabilitation with the original material), technological (extraction tools and methodologies), ethnological and socio-economic (quarries as historical poles of social-regional development), are some examples.



*Fig. 7. Conservation of historic quarries. A. Quarries of San Cristobal, accumulation of waste. Puerto de Santa María (Cádiz), B. Quarry of San Juan de Terreros, electrical installations in the quarry (Almería), C. Quarry in Castril, affected by the construction of a house (Granada), D. Quarry of San Pitar, Vélez-Málaga, musealization initiative (Málaga).*

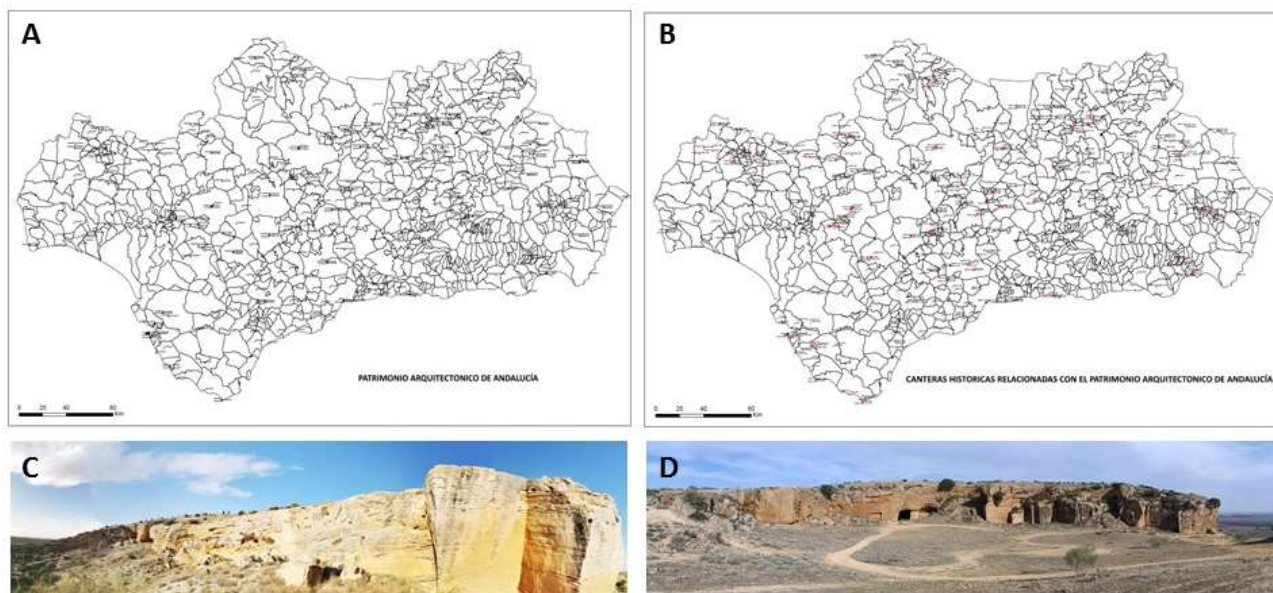
The conservation of many quarries has been affected by intense anthropization, urban developments that have not protected them in the respective urban plans, as well as their use as landfills or waste spaces (Fig. 7).

In order to preserve these extractive areas, the IGME proposes to the competent administrations the need to create a specific protection figure for them. So that when there is a need to intervene on the heritage asset and use the original rock, this rock is located and can be extracted from the historic quarry. This double character of protection and use for heritage is what should mark future lines of action and conservation initiatives.

### Conclusions

The results of the research project in Andalusia present very interesting results from a scientific point of view, with a total of 675 studied monuments and 798 quarries associated with their construction. All these data allow us to have an overall vision, which represents an advance in the knowledge of the architectural heritage in Andalusia, and the knowledge of the production of historic buildings from the quarry to the construction site (Fig. 8).

Finally, with this study it will be possible to develop future initiatives for the protection, recovery and definition of routes that integrate heritage architecture, geology and quarries, with the aim of disseminating these spaces that are of great scientific interest, technological, cultural, etc. and that society in general learn to value and respect them.



*Fig. 8. A. Distribution map of the patrimonial elements studied in Andalucía, B. Location of the historical quarries in the INCHaPA project Andalucía, C. Los Canterones. Estepa (Seville), D. Quarry Cueva de la Batida, Carmona (Seville).*

**Acknowledgments:** The authors express their gratitude for the support and help received on their work by the Mines Service, General Secretary for Industry and Mines, Junta de Andalucía.

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# STRATEGIES AND EXPLOITATION OF THE PARAMOS MIOCENE LIMESTONES IN THE CONSTRUCTION OF EARLY MEDIEVAL HISPANIC BUILDINGS

Álvarez Areces, E.<sup>1</sup>; Utrero Agudo, M<sup>a</sup>.Á.<sup>2</sup>

(1) CN IGME-CSIC – Instituto Geológico y Minero de España, [e.alvarez@igme.es](mailto:e.alvarez@igme.es)

(2) EEA/EEHAR-CSIC – Escuela de Estudios Árabes/Escuela Española de Historia y Arqueología en Roma

**Summary:** *The Paramos limestones of lacustrine origin are contextualized in one of the largest Spanish Cenozoic Basins, specifically the Duero Basin, in the northern region of the Iberian Peninsula. They are massive limestones with fossil remains of gastropods, operculums, caraceae and ostracods, which usually appear in the form of molds (moldic porosity), and outcrop on the tops of the hills, ending the Miocene series.*

*The study of the architecture dated to early medieval architecture (8th-10th centuries) in this context has allowed us to know in detail the construction sequence of two important buildings from this period, the churches of Santa María de Wamba and San Cebrián de Mazote, both in the province of Valladolid in the Autonomous Community of Castilla y León (Spain). Both buildings were analyzed in a multidisciplinary way by means of archeological and geological methodologies, establishing their different construction phases and identifying and characterizing the stone materials used in order to approach their origin and extractive areas. With this information, we are able now to better understand the production of these buildings and their links with the physical environment over the Early Middle Ages.*

**Key words:** *Paramos Miocene limestone, early medieval age, archaeology of architecture, geology, quarry.*

## Introduction

The study of early medieval ecclesiastical constructions, dated to between the 8th and 10th centuries, by applying archaeological and geological methodologies has allowed us to deepen our knowledge of their construction process and technology, along with the productive context to which they belong. These results are the consequence of more than a decade of collaboration in numerous research projects between the authors, studying the buildings dating to this historical period from a multidisciplinary perspective.

The results obtained so far allow us to link the quarry with the building, to understand the construction technique used, and to relate these buildings to the physical environment, in search of the ideal material for the construction, including both stones as structural elements (ashlars and masonry) and as part of the decorative programs.

Coordination between the work in the quarry and on the construction site had to be essential, ensuring the suitable materials for certain uses, guaranteeing the continuity of the architectural project by collecting

material, and finally placing the stones in their precise place in the work.

In this work, the results are presented for the churches of Santa María de Wamba and San Cebrián de Mazote, in which the exploitation strategies to obtain construction resources, as we will see, present certain analogies.

## Geographical and geological context

The studied architectural ensembles are geographically located in the Duero Basin, in the north-central region of Spain, in the province of Valladolid. Their geological context is the Basin Cenozoic Duero.

Duero Basin is one of the large intracontinental tertiary basin's characteristics of the interior of the Iberian Peninsula, and the buildings studied are located specifically in the northern sub-plateau. Stratigraphically, it is characterized by the presence of neogene sediments, on one hand, and tertiary sediments corresponding to the filling of a foreland depression, on the other, the latter traditionally assigned to the quaternary and associated with the process of erosion and emptying of the Basin (Fig. 1).



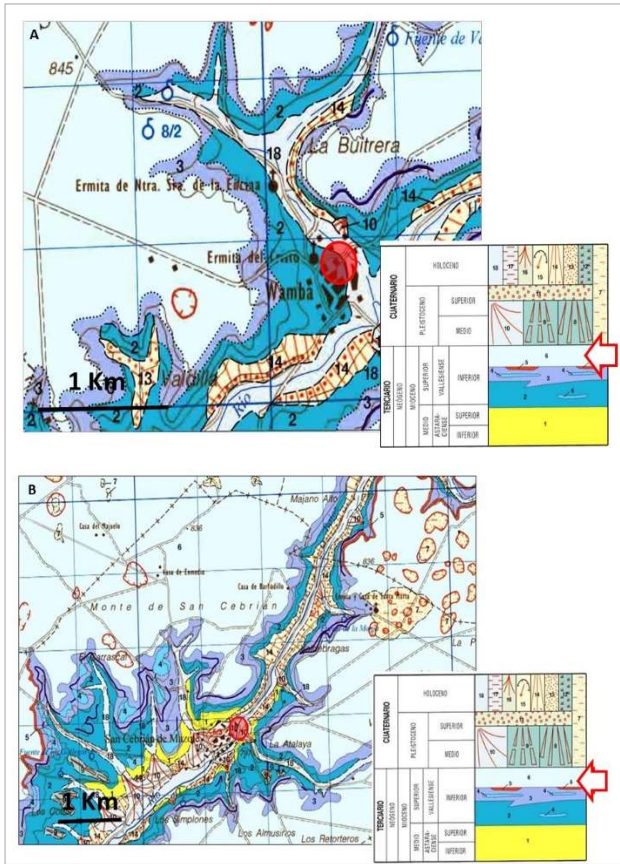


Fig. 1. A. Geological context of Santa María de Wamba, B. Geological context of San Cebrián de Mazote, highlighting the paramos limestone levels. Source: IGME (2007).

As mentioned before, the churches of Santa María de Wamba and San Cebrián de Mazote are located in the province of Valladolid, where tertiary outcrops are extensive, consisting of three lithological sections. The massive limestones with fossil remain of gastropods, operculums, caraceae and ostracods, usually appear in the form of molds (moldic porosity), and geological outcrops are on the tops of the hills, ending the Miocene series.

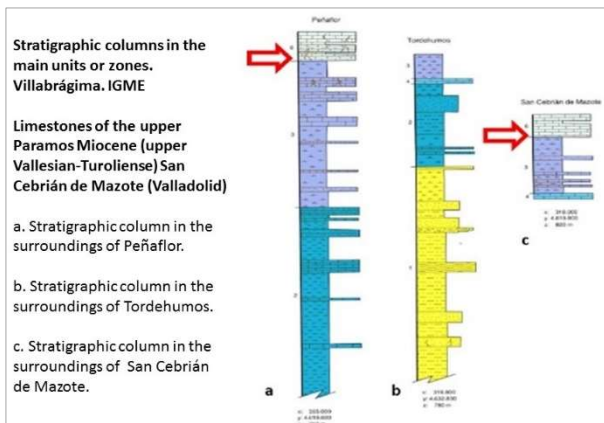


Fig. 2. Stratigraphic columns corresponding to the paramos limestones, in the surroundings of San Cebrián de Mazote, highlighting the paramos limestone levels. Source: IGME (2007)

This series is made up of three lithological sections: *tierra de campos facies* (Astaracian), of sand and silt; *cuestas facies* (Vallesian), of limestone and marl; and culminating the paramos, limestones or *paramos facies* (upper Vallesian-Turoliense), all these made up of massive and homogeneous limestone (Fig. 2).

### Early Medieval Hispanic Buildings

Early medieval ecclesiastical constructions and their quarries have been studied. Among them, we can mention the churches of San Miguel de Escalada, Santiago de Peñalba and San Isidoro, all of them in León; Las Mesas de Vilalberde in Málaga; Santa María de Melque and San Pedro de La Mata, both in Toledo; and Santa María de Wamba and San Cebrián de Mazote, both in Valladolid.

Santa María de Wamba and San Cebrián de Mazote, objects of this investigation, present constructive analogies regarding the use of structural materials for the construction of their walls. They were built with miocene limestone from the paramos. In both buildings, similar strategies are used to take advantage of the resources available in their environment. The materials used to cut their decorative elements are not the object of this work (Fig. 3).



Fig. 3. A. Church of Santa Maria de Wamba, B. Use of miocene paramos limestone in the construction of its walls, C. Church of San Cebrian de Mazote, D. Detail of paramos limestone in its walls.

The methodology of the archeology of architecture was applied to the analysis of both buildings with the aim of knowing their historical-constructive sequence, represented graphically with the so called Harris matrix, and of defining the original early medieval building (Harris, 1979; Utrero Agudo, 2017). On the other hand,

the methodology for the location of historic quarries, with the analysis and sampling of the building and the correlation of samples with those selected at the geological outcrops, allowed to locate the related historic quarries or extractive areas (Fort, 1996; Álvarez Areces, 2015; Álvarez Areces, Baltuille Martín, 2017; Baltuille Martín et al., 2017; Fernández Suárez et al., 2020).

### Petrological characteristics of the paramos miocene limestones.

In the construction of the churches of Santa María de Wamba and San Cebrián de Mazote, it was used the *paramos limestones facies* and the limestone associated with *cuestas facies*, the latter with great lateral development. These limestones correspond to a rock classified as compact biomicrite limestone (Folk, 1962) or a bioclastic packstone limestone (Durham, 1962). Macroscopically, it is a fine-grained, homogeneous limestone with an absence of anisotropy, presenting good technical conditions for carving the ashlar stones of the walls and arches. Polarization light microscope studies indicate their high content of allochemicals, predominantly ostracods, with gastropods and characea. Bioclasts make up the structural framework of these rocks, which present a grain-supported texture with a micritic matrix (Fig. 4).

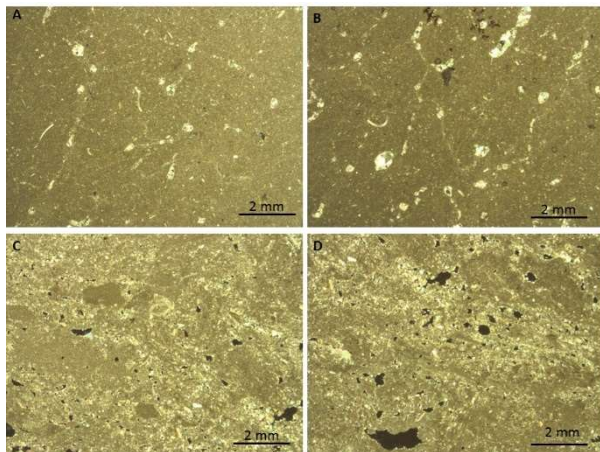


Fig. 4. A. Photomicrograph of paramos limestone sample, textural aspect, crossed nicols (LPA) Objective 2.5x/0.06. Selected sample in Santa María de Wamba, B. Texture of a sample selected in San Cebrián de Mazote, crossed nicols (LPA), C. Textural aspect of sample selected in a quarry associated to Santa María de Wamba, crossed nicols (LPA), D. Texture, crossed nicols (LPA), sample selected in a quarry associated to San Cebrián de Mazote.

The mineralogical composition of this rock, determined by X-ray diffraction, is calcite (97%) and, as minor elements, presents quartz.

### Technological characteristics of the paramos miocene limestones

The results of the technological characterization tests have been provided by the laboratories of the Geological and Mining Institute of Spain (IGME). They correspond to the results for the analysis of the physical-mechanical behavior of the paramos limestone, within the framework of the project *Characterization of the rocks used in the construction of the cultural heritage of the St' James Way* (Álvarez Areces, 2015) (Table I).

Paramos limestone physical-mechanical characteristics	
Apparent density (Kg/m <sup>3</sup> )	2025
Open porosity (%)	24,7
Compressive strength (Mpa)	27
Flexural strength (Mpa)	4
Water absorption (%)	9

Table I. Results for the analysis of the physical-mechanical behavior of the paramos limestone. Source: IGME (2015).

### From the quarry to the building

The prospecting work focused on the search for the quarries associated with the construction of the two churches of Santa Maria de Wamba and San Cebrián de Mazote.

From the selected samples in the buildings and their macro and microscopic petrological characterization, the geological prospecting paid specially attention to the recognition of nearby outcrops corresponding to the Miocene series (*cuesta facies* and *paramos facies*).

The rocks used in the construction present a series of textural characters, fossil content, composition and sedimentary features of the depositional environments associated with these facies.

In this way, more than ten possible points of exploitation possibly associated with the construction of Wamba were identified, within a radius of 3 km, with the exception of an exploitation area located 6 km to the east, in the proximity of Villanubla village. In the case of San Cebrián de Mazote, there are seven exploitation points associated with its construction, located in an area of 2 km radius distance from the church (Fig. 5).

In both cases, for the construction of their walls, the paramos limestones were exploited, materials that culminated the Miocene series and the upper levels of

limestones corresponding to the *cuestas facies*. Therefore, the stone materials used are of a local nature, with the exception of the decorative materials in its interiors that combine new pieces (capitals, friezes and reused (bases, shafts, capitals).

In both cases, the exploitation strategy corresponds to a perimeter exploitation of the resource, optimizing to the maximum the volume of rock extracted and the operations or labors for its benefit. This perimeter exploitation leaves, as it has been proven, numerous exploitation points along the geological outcrop.

The presence of marly levels on the lower part of the paramos limestone layers, or the existence of centimeter-thick limestone layers, in the set of marl corresponding to the *cuestas facies*, greatly affects the method of exploitation used.

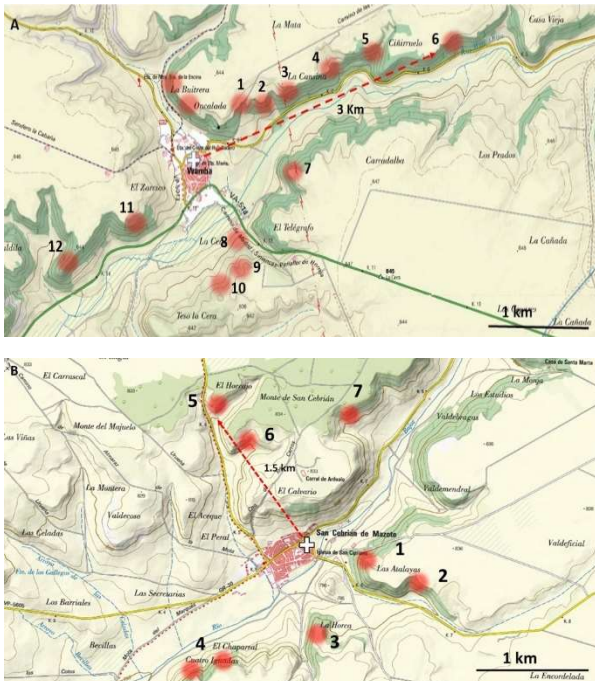


Fig. 5. Extractive areas in which the limestones of the miocene series are exploited (*facies cuestas* and *paramos*). A. Main extractive areas related to the construction of Santa María de Wamba, B. Extractive areas related to the construction of the San Cebrián de Mazote.

In the case of the *cuestas facies*, the layers with centimetric thicknesses are used to obtain masonry in the buildings. They present thicknesses between 10 and 40 cm, and there is a total correspondence between the thickness of the geological layer and the thickness of the masonry used in the church wall (Fig. 6).

With respect to the *paramos facies*, the thickness of the layers presents sizes from 50 cm to 1,50 m. The method of exploitation is based on the great lateral continuity of the layers and the possibility of carrying out cross-sections that optimize to a great extent the extractive

efforts. The lateral continuity and the existence of two pre-cut surfaces on the ceiling and wall (top-bottom) make it possible to simplify the extraction tasks by making transversal cuts to obtain the blocks (ashlars).

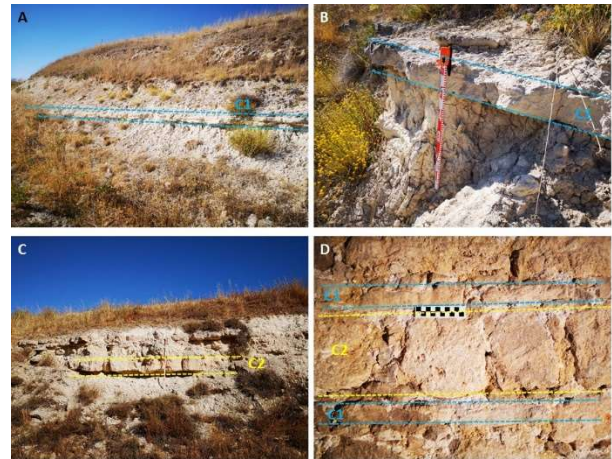


Fig. 6. Extractive area associated with the construction of Santa María de Wamba, for obtaining masonry of the carbonate levels, *cuestas facies*. A. Limestone layer outcrop, C1 (*cuestas facies*), B. Detail view, of layer C1 limestone *cuestas facies*, C. Limestone layer outcrop, C2 (*cuestas facies*), D. Interior wall of Santa María de Wamba, identification of the limestones corresponding to the geological outcrops of *facies cuestas* (Miocene). Layers C1 (10 cm) and C2 (40 cm), same thicknesses are observed.

In this case, the layers with metric thicknesses preserve historical-extractive marks such as wedges, ashlars, worked blocks, etc. Likewise, there is a correspondence between the thickness of the layers corresponding to the *paramos* limestones outcrops and the dimensions of the ashlars used in the construction (Fig. 7).

In both cases, the extractive process is favored by the morphology of the outcrops, the presence of marly levels and natural pre-cut surfaces in the contacts between geological materials, which allow mining operations to be optimized by making cross cuts to obtain ashlars or masonry depending on the thickness of the layer.



Fig. 7. Extractive area associated with the construction of San Cebrián de Mazote, for obtaining ashlars of the carbonate levels, *paramos facies*. A. Extractive marks (wedges), B. Historical-extractive marks on the *paramos* limestones, C. wedges marks, D. Blocks and ashlars accumulated on the slope.

## Conclusions

The study of the early medieval churches of Santa María de Wamba and San Cebrián de Mazote and of their stone materials is not only an effective mean for locating the origin of the materials used and determining their technological characteristics, is also an opportunity to obtain further information on the capabilities, resources, distribution and use of building resources.

The relationship of architecture with the physical environment are demonstrated in this study, in which the Miocene limestone is used for the construction of buildings. A direct relationship is

clearly observed between the geological outcrops and the construction of the walls.

Limestones are used as a resource and construction material (ashlar, masonry), and as a consequence, a large number of exploitation areas are identified in which historical marks of this extractive activity are preserved.

All this information should contribute to the knowledge of the stone material in the construction process, to the extractive activity in the quarry and exploitation strategies, and finally to the relationship between both spaces, approaching the production of these buildings of the Early Middle Ages.

**Acknowledgments:** The authors express their gratitude to the Cultural Heritage Service of the Junta de Castilla y León for the permission to take samples in buildings and the facilities for their study of the buildings. Research tasks were developed and funded by the projects entitled *Arqueología de las iglesias hispánicas del siglo X: la circulación de modelos arquitectónicos y decorativos*, references HAR2017-84927-P and PID2020-116931GB-I00, granted by the Spanish Ministry of Science (MCIN) and AEI. Stone materials of the decorative elements of San Cebrián de Mazote are currently being analysed thanks to the funding provided by Fundación Palarq.

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# BOÑAR STONE (LEÓN, SPAIN) AS A CONSTRUCTION RESOURCE: HISTORICAL HERITAGE AND PROSPECTS

Álvarez Areces, E.<sup>1</sup>; Utrero Agudo, M<sup>a</sup>.Á.<sup>2</sup>; Alonso-Jiménez, A.<sup>1</sup>; Santos, R.C.P.<sup>3</sup>; Aldeiturriaga Rozas, M<sup>a</sup>.A.<sup>4</sup>

- (1) CN IGME-CSIC – Instituto Geológico y Minero de España, [e.alvarez@igme.es](mailto:e.alvarez@igme.es)
- (2) EEA/EEHAR-CSIC – Escuela de Estudios Árabes/Escuela Española de Historia y Arqueología en Roma
- (3) UFOP – Universidade Federal de Ouro Preto (Brazil)
- (4) Mármoles OASA, León (Spain)

**Summary:** Boñar stone is an Upper Cretaceous (Coniacian-Campanian) dolomitized limestone that crops out near the village of Boñar, in the province of León, northern Spain. Geologically, it has been traditionally called the Boñar Formation, an eminently carbonate succession with sandy and clayey intercalations, which in some sectors is over 300 m thick. In the region, there are numerous small indications in which Boñar stone was historically exploited, although currently only the Sierra Redonda quarry remains active.

Its use in the architectural historical heritage is extensive in the province and in the city of León, with outstanding examples such as the Cathedral of Santa María de la Regla, the Hospital of San Marcos and the Royal Collegiate Church of San Isidoro of León, among many others.

It is currently used as a construction material, with different finishing, being used in different parts of the Spanish geography and exported abroad.

**Key words:** Dolomitized limestone, quarry, architectural heritage.

## Introduction

Boñar stone is an Upper Cretaceous (Coniacian-Campanian) dolomitized limestone, used mainly in the construction of the architectural heritage of the city of León, but also with outstanding examples of its use in the province of León, located in northwestern Spain (Fig. 1).

This stone material is part of the rich architectural heritage of the historic city of León, where there are important examples of architectural styles from the Romanesque, Gothic and Renaissance periods.

## Geological context

Geologically, it has been traditionally called the Boñar Formation (Evers, 1967), an eminently carbonate succession with sandy and clayey intercalations, which in some sectors is over 300 m thick (Fig. 2).

The most significant structures in the area are the Las Bodas syncline and the Losilla anticline, whose axial traces present an approximate NW-SE orientation. Between them and subparallel to the León fault, the Sabero-Gordón fault is available, whose late settling seems to be the origin of said folds (Heredia, 1998).

The geological context of the outcrops is the southern edge of the Cantabrian Zone (ZC). In the study of the geological outcrops, it has been possible to divide four members: i. calcarenitic member; ii. Clay-sandy member; iii. Member of alternations of marls,

limestones and calcarenites; and iv. dolomitic member. The deposit of these materials has been related to the generalized rise in sea level during the Late Cretaceous that flooded the depositional environments of the Voznuevo Formation on which it overlaps.

In the case of the last dolomitic member, it corresponds with the Boñar stone, which is still exploited at the Sierra Redonda quarry.

The calcarenitic and alternating members are exploited to obtain masonry stone, corresponding to sparitic limestone. The dolomitic member was used in the construction of the historical architectural heritage and is also still used now for new constructions.

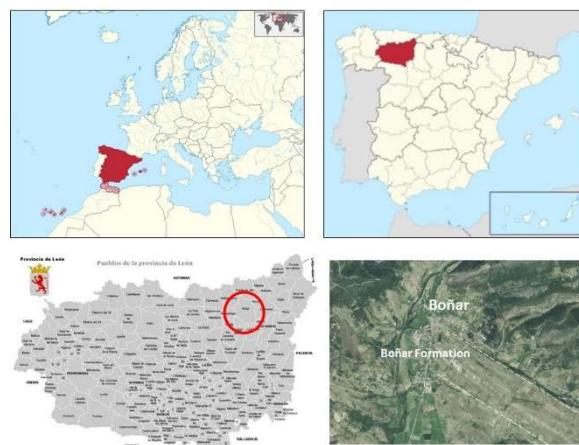


Fig. 1 Situation of the Boñar stone in the Spanish geographical context

## Main petrological characteristics

Boñar stone is a rock that is not too abrasive, yellowish in color and in some cases pinkish, compact, finely crystalline, massive in appearance and sometimes nodular or banded. It presents iron oxides, as well as small geodes with calcitic recrystallizations. Laminations, bioturbations and tracks stand out as sedimentary structures, which correspond to lagoon-type sedimentation (García de los Ríos, Báez Mezquita, 2001).

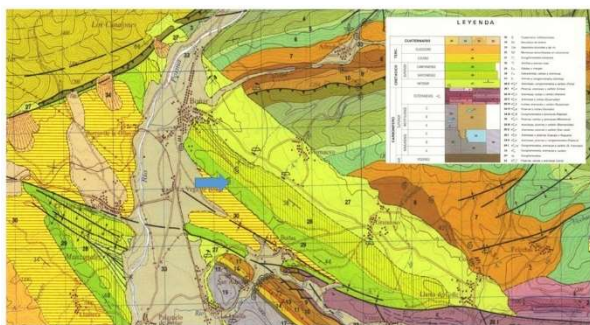


Fig. 2. Geological map around the village of Boñar (León), E: 1:50,000, (IGME, 1984). The location of the Sierra Redonda quarry, where the Boñar stone is currently exploited, has been indicated.

Microscopically, it is a rock formed essentially by dolomite, with sizes between 20-100  $\mu\text{m}$ , and larger calcite crystals can also be observed, although they show variations. It presents a crystalline texture of better or worse preserved fossil grains. The relict fossil remains correspond to oolites, foraminifera, shell fragments, etc. They present terrigenous components in a small proportion, being the most frequent clayey minerals and quartz. Iron oxide staining is common on the edges of fossil grains and in intercrystalline void spaces (Fig. 3).

The mineralogical composition of this rock, determined by X-ray diffraction, is dolomite (77%), calcite (23%) and, as minor elements, phyllosilicates and alteration minerals.

Regarding its porosity, notable variations are observed. In some samples, practically no pores are detected and, in others, there are a large number of pores with a good distribution. They also present moldic pores with an average size of 0.5 mm.

There are other varieties within the Boñar stone, in relation to the arrangement in the quarry banks. These varieties present small differences on a micro and macroscopic scale, in terms of color, texture and compaction.

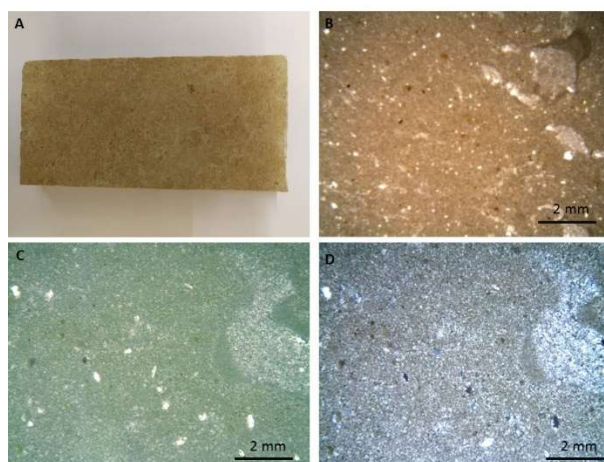


Fig. 3. A. Boñar stone appearance in visu. B. Photomicrograph of Boñar stone sample, textural aspect, parallel nicoles (LPNA) Objective 2.5x/0.06. Selected sample in architectural heritage. C. Textural aspect with parallel nicoles (LPNA) of a sample selected in the Boñar stone quarry. D. Textural aspect with crossed nicoles (LPA) of the sample selected in a quarry.

## Technological characteristics

The results of the technological characterization tests have been provided by the company Mármoles OASA, S.L. They correspond to the results of the analysis of the physical-mechanical behavior of the Boñar stone and its durability against the alteration agents of the constructions (Table I), mainly the heladicity, very frequent in the north of Spain, as well as the thermal shock.

## Resource exploitation and quarries

In the region, there are numerous small indications in which Boñar stone was historically exploited. All these historic quarries have been abandoned and correspond to small-sized open pit quarries. The Boñar stone was historically mined, especially its member i. calcarenitic, and iii. alternations of marls, limestones and calcarenites. Historical exploitation marks are identified, such as wedges and worked blocks, among others (Fig. 4).

Since 1452, there are historical references to the exploitation of the Boñar stone, in the chapter acts of the Archive of the Cabildo of the Cathedral of León.

Boñar stone physical-mechanical characteristics	
Apparent density (Kg/m <sup>3</sup> )	2510
Open porosity (%)	10,5
Compressive strength (Mpa)	117
Flexural strength (Mpa)	16
Abrasion resistance (mm)	28
Slip resistance (USRV)	73
Anchorage resistance (N)	1950
Thermal shock (%)	0,06
Heladacity (%)	10

Table 1. results for the analysis of the physical-mechanical behavior of the Boñar stone and its durability

In these documents, the names of a group of places appear, all of them relatively close, located to the northwest of the province, approximately 40 km from the city of León, which supplied stone to the works. All of them are located in the surroundings of the village of Boñar.

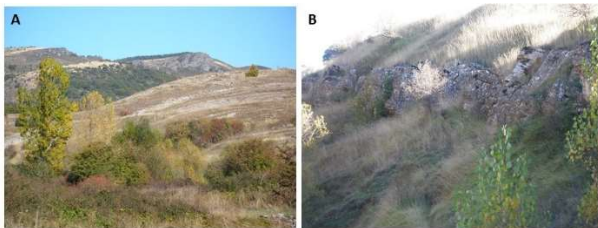


Fig. 4. A. Geological outcrops of the Boñar formation (limestone and dolomites) Cretaceous age, in the proximity of the village of Boñar, B. Detail of a stratum of the Boñar formation, in the proximity of the quarry.

Places such as Valdepiélagos, La Vecilla, Robres (present-day, Robles or Robledo de la Valcueva), Prado (possibly, Prado de la Guzpeña or the place of the Arroyo del Prado), Santa María, etc., are mentioned. In these places, the Boñar formation outcrops are limestones and dolomites, and are exploited for the construction of buildings (Marcos Fierro, 1992).

Based on the observations, the most favorable areas for the historical exploitation of this resource are considered to be the northern flank of Las Bodas syncline, currently in exploitation, and the southern flank of the Losilla syncline, from the vicinity of the town Barrillos de las Arrimadas. In both cases, they present a great lateral continuity, a considerable thickness, sufficient textural homogeneity and, in general, low fracturing and high rock/sterile ratios.

The rocks of the Boñar formation have been exploited since ancient times, especially different banks of recrystallized and partially dolomitized limestone. In historical references of these exploitations are

described as it follows: "They have three banks, the finest is titled the King; he carves out very easily; its color is light orange; the other two, although of outstanding clarity, are more inferior and whitish in color" (Madoz, 1847). This text already points out the differences between the many varieties within the Boñar stone, which affected not only its aesthetic appearance but also its mechanical properties and, therefore, its uses in construction.

Currently, the Boñar stone is exploited only in the Sierra Redonda quarry, the only exploitation that remains active. Other exploitations were closed more than a decade ago. Located 1 km southeast of the village of Boñar, it has been working since 1945, with the third family generation running it. The exploitation front presents dimensions of 50 m in length, seven layers corresponding to the member iv. Dolomitic. This is formed by compact beige and beige-pink dolomites from 0.5 to 1.05 m thick and the reaching maximum exploitation is 1500 m<sup>3</sup>/year (Fig. 5).

In the quarry, the extraction of the blocks is carried out by benchers aided by wedges that are later cut with a disc saw to obtain pieces of different dimensions. It is marketed as blocks, boards, slabs, etc.

#### Architectural heritage and uses

Boñas stone is used to build architectural historical heritage in the province of León and especially in the main city, where outstanding examples from the medieval to the contemporary ages, such as the Cathedral of Santa María de la Regla, the Hospital of San Marcos, the Royal Collegiate Church of San Isidoro and the Town Hall Building, among many others, are still preserved.

Its use has been confirmed since the 10th century in the construction of the interior arches of the basilica of the monastery of San Miguel de Escalada (Gradefes; Utrero Agudo, Álvarez Areces, 2022), located close to León. This is the first historical verification of its use in architecture. The local low-quality materials (calcretes and Miocene limestones) in the surroundings of the monastery are only used to build the walls (ashlars and rough masonry), but Boñar stone is employed to execute works that demand high quality materials, such as for carving decorative elements and building the arches of the arcades.

Subsequently, the Boñar Stone was widely used at the Collegiate Church of San Isidoro, an example of pre-Romanesque and Romanesque construction (Utrero et al., 2017), and later on at the Cathedral, an outstanding example of Gothic period, and many other religious buildings in the city of León. Specifically, Boñar stone is used for the construction of the Cathedral from the 15th

century, replacing tertiary caliches (calclform soils), locally called as red and yellow “piedra del país”. These materials showed however a bad behavior against alteration over the time and tertiary limestone had to be substituted, not only at the cathedral, but at many other constructions. In all these buildings, it can be observed the versatility of the Boñar stone used in the construction of walls as ashlars, and in the preparation of their decorative programs, allowing the carving of high-quality decorative elements.

Its use is also identified in the civil architectural heritage of the city, with examples such as the Palace of the Guzmanes, the Hospital of pilgrims of San Marcos or the Old Town Hall, among many others (Fig. 6).

In addition to its use in León and its province, Boñar stone has been employed at other places in Spain, such as the construction of a bank building, houses or hotels in the city of Santander. Likewise, it was exported for the construction of the New Town Hall building in Malabo (Equatorial Guinea), for its building use on the Caribbean Island of Aruba and also for the restoration of the Fontainebleau castle in France.



Fig. 5. Sierra Redonda quarry. A. Location of the quarry on an orthophoto, southeast of the village of Boñar (León). Source: Iberpix viewer (IGN), B. Outcrops in the proximity of the quarry in which old exploitation marks (worked blocks, wedges, etc.) can be identified, C. Sierra Redonda quarry and its advance from the west, D. Exploitation benches in front of the quarry, E. An image of the quarry taken during the cold winter in León, F. Stockpiling blocks.



Fig. 6. Architectural heritage in León built with Boñar stone. A. Royal Collegiate Church of San Isidoro, B. Hospital of San Marcos, C. Cathedral of León, D. Palace of the Guzmanes, E. Interior arches of the Monastery of San Miguel de Escalada. F. Decorative elements in the Hospital of San Marcos, use of stone from Boñar, G. Old Town Hall, H. León Town Hall or Poridad Palace, I. Boñar stone monument to the Kings of León, J. Use of Boñar stone in a bank building in Santander (Cantabria). Source: sedesocialsantander.

## Conclusions

The Boñar stone constitutes the most prominent stone element in the rich architectural heritage of the city of León. Its use is confirmed from the 10th century until today in an outstanding group of buildings of civil and religious typology, among which the Collegiate Church of San Isidoro (12th century) and the Cathedral (15th century) stand out.

It is a cretaceous dolomitized limestone that can be used on site as a structural element and also, due to its characteristics, to carve decorative programs. The use of this material exploited 30-40 km north of the city of León, in the proximity of Boñar, gives its own personality to the important architectural heritage of León. Currently, only the Sierra Redonda quarry remains active and there are evidences of historical exploitation in the cretaceous outcrops (limestone and dolomites) in the area of Boñar.



The public administrations should encourage its use in rehabilitation works of the historical heritage, as well as

make the society aware of its importance in the historical architectural legacy of León.

**Acknowledgments:** The authors express their gratitude for the support and help received in their work by Mármoles OASA, S.L. (Boñar, León) who have contributed for this publication the results of the characterization tests of the Boñar stone, information on the quarry, as well as photographic documentation used in this work.

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# AGRONOMIC POTENTIAL OF USING ORNAMENTAL STONES WASTE AS A SOURCE OF AGRICULTURAL FERTILIZATION

G.R. Camara<sup>1\*</sup>, E.S. Santos<sup>1,2</sup>, A.J.Z. Gualandi<sup>1,2</sup>, L.L.L. Silveira<sup>1</sup>, M.A. Neves<sup>2</sup>, C. Chiodi Filho<sup>3</sup>, N.F. Castro<sup>1</sup>

- (1) Center for Mineral Technology (CETEM), \*[camara.gdr@gmail.com](mailto:camara.gdr@gmail.com).
- (2) Federal University of Espírito Santo (UFES).
- (3) Brazilian Association of the Ornamental Stones Industry (ABIROCHAS).

**Summary:** *The volume of waste (by-products) generated in the production process of ornamental stones is the main environmental problem of the sector, while the import limitations and increase in fertilizer prices highlight the vulnerability of the Brazilian agricultural production system and put the world food supply at risk. Alternatives for the use of mining by-products and agricultural fertilization are essential for the sustainability of these activities, and this is the objective with this research. The by-products used in this study include three syenites (CB, CI, and MG), one alkali-granite (OI), and one varvite (AR), evaluated for their agronomic potential to improve soil quality. In the analysis of the results, the by-products meet the requirements of the legislation, and are considered potential soil remineralizers. After 150 days of soil incubation with the different by-products under study, RA showed the best agronomic potential, followed by CB, OI, CI and MG.*

**Key words:** *agrominerals, soil remineralizer, rock powder, sustainable development.*

## Introdução

The fourth largest food producer in the world, representing one-fifth of the Gross Domestic Product (GDP) and 48% of exports (IBGE, 2020; CNA, 2021), Brazil is an agribusiness powerhouse.

Considering the world trend of growing food consumption at about 6% per year, for agricultural production to keep up with demand, it will be necessary to increase the production and productivity of crops, which are directly related to the fertility of the soil-plant system. However, in the conventional cultivation system, it becomes necessary to import soluble fertilizers, which today correspond to approximately 80% of the national annual requirement, resulting in high production costs, loss of competitiveness, and vulnerability to other countries (SANTOS; GLASS, 2018; GLOBALFERT, 2021).

The dependence on the national agricultural production system can be exemplified by the limitations on the shipment of fertilizers from Russia to Brazil, as an indirect consequence of the diplomatic conflicts established between Russia and Ukraine in the year 2022. According to the Brazilian Ministry of Economy (COMEXSTAT, 2022), about 23% of chemical fertilizers imported by Brazil in 2021 came from Russia. If added to

the dependence on fertilizers coming from Belarus, the seventh largest exporter of fertilizers to Brazil and an ally of Russia, the degree of national dependence rises to 26%. Thus, the search for alternative sources acquires great importance for the future of Brazilian agricultural production (BRITO et al., 2019).

As Brazil is the fifth largest producer and exporter of ornamental stones in the world (ABIROCHAS, 2021a;b), the use of waste generated in this production chain, estimated nationally at 18.0 million tons in 2019 (ABIROCHAS, 2020), meets the Circular Economy and can, about product innovation, provide more sustainable and competitive economic development for industries in the mining sector concomitant to the supply of alternative sources of agricultural fertilization (CAMARA et al., 2021).

Given this scenario, studies that recommend the use of different waste products from the production chain of ornamental stones together with the search for alternatives that accelerate the solubilization of nutrients present in the rocks, become necessary so that the production chain of ornamental stones can become more sustainable, as well as the Brazilian agribusiness less dependent on external inputs.

Considering the hypotheses that the materials under study have: (i) potential for use as soil remineralizers, meeting the requirements set out in the NI; (ii) ability to release potassium to the soil; and (iii) greater solubilization of nutrients when inoculated with different microorganisms, this study aimed to analyze the potential for agronomic use of coarse waste generated by the processing of different types of ornamental stones as soil remineralizers.

After the characterization of the materials, the results of the incubation process are presented, according to the agronomic protocol provided in the Brazilian Normative Instruction (IN) No. 05, of March 10, 2016, of the Ministry of Agriculture, Livestock and Supply (MAPA) (BRASIL, 2016), which will be referred to as either Normative Instruction or NI throughout this material.

### Material and methods

The rocks selected for this study include three syenites (CB, CI, and MG), one alkali granite (OI), and one varvite (AR), collected from coarse residues (by-products) generated in quarries or sawmills (non-standard blocks, rock fragments, hulls, drill cores, etc.). The characterization of each rock is described in the results and discussion topic.

The sequence of the methodology presented below complies with the guidelines provided by Normative Instruction, which recommends that studies conducted to evaluate the potential use of certain rocks as soil remineralizers include the requirement of geochemical and mineralogical analyses that prove the suitability and efficiency of the material. Thus, they are presented in the following order: comminution of by-products, physical characterization, determination of the chemical composition by X-Ray Fluorescence (XRF), loss on ignition (LOI), analysis of the hydrogen potential (pH), determination of the mineralogical composition by X-Ray Diffraction (XRD), quantification of the free silica content by Rietveld Analysis, analysis of potentially toxic elements by solubilization and incubation test.

For the comminution of by-products, the samples were sent to the Center for Mineral Technology - Regional Center of Espírito Santo (CETEM - NRES) and fragmented with the aid of a sledgehammer. Subsequently, the fragments were comminuted in a jaw crusher and sieved in a vibrating sieve with a 28 mesh (0.06 mm) screen. The material retained on the sieve was taken to a roller mill for grinding, again sieved on a 28 mesh screen and the material retained on the sieve was ground in a disc mill and mixed with the material passing the sieve for further homogenization and quartering, following methodology adapted from Góes et al. (2010).

The physical characterization of the by-products was determined from the particle size distribution. For this, a laser granulometer model Mastersize Hydro 2000SM from Malvern Instruments was used. 500 mL of water were added to the device's container, keeping it under agitation of 1700 rpm for 30 minutes until it reached the necessary obscuration to perform the measurement. Then, the sample was gradually added until it reached the ideal obscuration index for reading.

The determination of the chemical composition of the samples was performed at CETEM - NRES using an X-ray Fluorescence Spectrometer (XFR), Bruker S2 Ranger model. We used 10 grams of each by-product powder sample, with particles smaller than 0.105 mm, manually comminuted in agate mortar and pressed at 20 tons for 15 min in a manual press (Mauthe Maschinenbau, PE-011 model). The loss on ignition (LOI) of each sample was also determined by heating at a temperature of 1000 °C for 2 hours in a muffle furnace, INTI, model FL1300/20, with a heating ramp of 5 °C per minute.

For analysis of hydrogen potential (pH), an aliquot of each ground sample was mixed in distilled water at a ratio of 1:1 by weight, according to the methodology proposed by MAPA (BRASIL, 2017), the mixture was homogenized in a Quimis magnetic stirrer (model Q5261) and then the pH was measured in a pH meter Marte (model MB 100).

The determination of the mineralogical composition of the samples was performed via X-ray diffraction (XRD) using a Bruker-AXS Model D8 Advance Eco diffractometer. The following operating conditions were adopted: Cu K $\alpha$  radiation (40 kV/25 mA), with 0.01° 2 $\theta$  step, 92 seconds counting time per step with a state-of-the-art silicon drift type position sensitive linear detector (with energy discrimination) LynxEye XE, collected from 5 to 80° 2 $\theta$ . Qualitative spectrum interpretation was performed by comparison with standards contained in the PDF 4+ relational database (ICDD, 2020) in Bruker Diffrac.EVA software.

Aiming to quantify the free silica content (silicon dioxide - SiO<sub>2</sub>) in the samples submitted to XRD analysis, the Rietveld method (Rietveld, 1969) was applied. The analysis and interpretation of the XRD results and the Rietveld analysis were performed at the Technological Characterization Sector (TCS) of the Mineral Analysis Coordination (COAMI) of the Mineral Technology Center in Rio de Janeiro, Brazil (CETEM - RJ).

To assess the solubilization of the metals Arsenic (As), Cadmium (Cd), Mercury (Hg), and Lead (Pb), a waste solubilization test was conducted using the methodology described in the ABNT NBR 10006:2004 standard (ABNT, 2004). The extracts were quantified

using an inductively coupled plasma optical emission spectrometer (ICP-OES).

For the incubation test, soil samples were collected from the experimental area of the Center of Agricultural Sciences and Engineering (CCAEE) at the Federal University of Espírito Santo (UFES), Alegre Campus, at a depth of 0 to 0.30 m. The soil has a clayey texture (46% clay + 36% coarse sand + 10% fine sand + 8% silt).

To accelerate the solubilization of nutrients in the studied by-products, two solutions of microorganisms known as plant growth-promoting inoculants were used. The first solution, identified by the acronym BF, was composed of a combination of *Pseudomonas fluorescens* and *Azospirillum brasiliense* at a concentration of  $1 \times 10^{11}$  colony-forming units per liter (CFU/L). The second solution, identified by the acronym BT, was composed of a combination of *Bacillus subtilis*, *Bacillus amyloliquefaciens*, and *Bacillus pumilus* at a concentration of  $1 \times 10^{11}$  CFU/L. Inoculation was performed using a graduated pipette, and the volume of inoculum followed the manufacturer's recommendation.

A  $5 \times 3 \times 2 + 3$  factorial experiment was used for the incubation test. Specifically, the experiment included 5 by-product samples (MG, CB, CI, OI and AR), 3 different doses of each by-product (0%, 40% and 60%), 2 different microbial solutions (BF and BT), and 3 additional control treatments, with 3 replicates for each sample unit. Each unit consisted of 300 grams of sieved soil homogenized with the respective treatments. The samples were packed in transparent plastic bags, placed on metal shelves in a controlled environment with an average temperature of 25 °C and indirect sunlight, and evaluated at different incubation periods (0, 30, 60, 90, 120 and 150 days) for a total of 33 treatments in 594 sampling units. The soil moisture of each sample unit was maintained at 80% of field capacity, and all sampling units had a small opening for gas exchange with the medium. The experiment was repeated 6 times with different sets of soil samples.

Every 30 days, for a period of 150 days, the experimental units were analyzed for the following parameters: routine analysis, including available Calcium (Ca), Magnesium (Mg), exchangeable Aluminum (Al), Phosphorus (P), Potassium (K), total acidity (H + Al), pH, base saturation (V), Aluminum saturation (m), exchangeable base sum (SB), and cation exchange capacity at pH 7.0 (T); analysis of micronutrients Iron (Fe), Copper (Cu), Zinc (Zn), and Manganese (Mn); and analysis of Remaining Phosphorus (Prem). The soil analyses were performed by the Soil Laboratory of UFES, which is accredited and certified by Embrapa Solos (Brazilian Agricultural Research Corporation).

The obtained results were subjected to variance analysis. Whenever the variables evaluated were significant, the Scott-Knott test was conducted with the aid of R software, version 4.1.2 (R CORE TEAM, 2021), at a 5% probability of error.

## Results

To evaluate the potential of the different waste rocks studied in this work, to act as remineralizers for soils intended for agriculture, the results obtained were analyzed and compared with the rules on the definition, classification, specifications, guarantees, and tolerances provided for in Normative Instruction.

### *By-product characterization*

The by-products of ornamental stones, collected in the form of rock fragments, were prepared for the incubation tests by means of grinding. After this procedure, the samples became a particulate material whose granulometry is described in Table I. In the by-products MG, CB, CI, and OI, the predominant fractions are fine sand and coarse sand, unlike the RA, where the silt fraction predominates.

All materials studied are composed mostly of Si, followed by Al, which are normally present in the crystal structure of silicate minerals (Table II). In third place come Fe, Ca, and K, followed by Mg and Na, also very common constituents of silicate minerals present in rocks. The other elements present can be considered 'trace' or of little significance. The sum of bases, involving CaO, MgO, and K<sub>2</sub>O contents, should be greater than or equal to 9%, and K<sub>2</sub>O content greater than or equal to 1%, according to Normative Instruction. The by-products have a similar alkaline nature, with pH values ranging between 7.57 and 8.48, with the AR by-product having the highest value.

The silicon (Si) contents presented in Table 2 do not refer to the so-called "free silica" but to the total silica, including that present in all minerals of the silicate group. The quantification of free silica (quartz - SiO<sub>2</sub>), necessary to meet the proposed Normative Instruction, was done using Rietveld analysis with the data obtained by X-ray diffractometry (XRD) (Table III).

The minerals present in higher concentrations are quartz, albite, microcline, oligoclase, muscovite, orthoclase, and augite, with smaller fractions of others. For the free silica content, all by-products showed values equal to or less than 25%, which is the maximum value allowed by Normative Instruction, for a material to be considered a soil remineralizer. Thus, all the samples of the residues studied fit the free silica content set by the NI.

**Table I.** Granulometric distribution of the studied residues, after grinding.

	<b>Distribuição Granulométrica (%)*</b>				
	<b>MG</b>	<b>CB</b>	<b>CI</b>	<b>OI</b>	<b>AR</b>
Silte	1.03	0.46	0.99	1.20	98.26
Areia Fina	54.74	32.74	44.26	48.72	1.74
Areia Grossa	44.23	66.80	54.75	50.08	0.00

\*The silt fraction comprises particles with diameters between 0.002 mm and 0.06 mm and the sand fraction has a grain diameter ranging from 0.06 mm to 2.0 mm, with 0.06 mm to 0.20 mm corresponding to the fraction of thin sand; 0.20 to 0.60 mm of medium sand and 0.60 to 2.0 mm of coarse sand (ABNT NBR 6502:1995).

**Table II.** Chemical composition and pH of the studied residues.

<b>By-product</b>	<b>Chemical Composition (%)</b>												<b>pH</b>
	<b>SiO<sub>2</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>CaO</b>	<b>K<sub>2</sub>O</b>	<b>MgO</b>	<b>Na<sub>2</sub>O</b>	<b>TiO<sub>2</sub></b>	<b>P<sub>2</sub>O<sub>5</sub></b>	<b>Outros</b>	<b>LOI<sup>(1)</sup></b>	<b>SB<sup>(2)</sup></b>	
MG	60.9	14.1	4.27	3.43	7.97	2.98	3.73	0.56	0.72	0.93	0.41	14.38	7.57
CB	58.1	17.6	6.08	3.06	5.16	1.09	6.02	1.21	0.53	0.99	0.16	9.31	7.75
CI	56.8	14.0	5.10	5.40	8.15	3.48	2.45	1.24	1.45	1.64	0.29	17.03	7.98
OI	59.6	18.0	4.73	4.01	6.65	1.96	2.36	1.01	-	1.44	0.24	12.62	8.06
AR	60.4	17.7	5.89	1.34	3.47	4.64	2.12	0.88	-	0.64	2.92	9.45	8.48

\*1 LOI - loss on ignition; \*2 SB - Sum of Bases (sum of CaO, MgO and K<sub>2</sub>O contents).

**Table III.** Mineralogical composition of residues and percentage of minerals calculated by the Rietveld method.

<b>MINERAL</b>	<b>% RIETVELD</b>				
	<b>MG</b>	<b>CI</b>	<b>CB</b>	<b>OI</b>	<b>AR</b>
Quartz	4.46	1.96	-	3.51	29.10
Albite	17.86	9.38	19.19	-	24.48
Microcline	31.28	22.61	8.38	-	13.66
Actinolite	9.39	2.57	5.09	4.59	-
Oligoclase	7.58	15.26	29.45	45.52	-
Muscovite	2.18	5.85	2.49	3.74	19.11
Fluorapatite	1.24	2.50	1.42	1.44	-
Dolomite	-	0.18	0.21	-	1.57
Kaolinite	0.27	0.12	0.15	0.16	2.61
Orthoclase	18.05	18.31	18.71	36.23	-
Nepheline	-	0.55	5.42	-	-
Sodalite	-	0.21	1.08	-	-
Augite	6.82	17.47	5.83	3.60	-
Cancrinite	-	1.28	0.97	-	-
Titanite	0.88	1.74	1.61	1.22	-
Chlorite	-	-	-	-	9.47

In Brazil, the solubility of elements and substances from a solid waste (by-product) is predicted using the solubilization test according to the Brazilian Association of Technical Standards - ABNT (ABNT, 2004). The results obtained from the application of this test to the by-products studied (Table IV) allow us to consider them

inert for the elements As, Cd, Hg, and Pb, as provided in the NI.

**Table IV.** Element contents in the obtained solubilized extracts.

Content in the solubilized extract (mg L <sup>-1</sup> )	By-products					
	MG	CB	CI	OI	AR	T-IW <sup>(*)</sup>
As	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01
Cd	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	0.005
Hg	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.001
Pb	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01

T-IW<sup>(\*)</sup>: threshold for inert wastes (ABNT, 2004).

#### Analysis of the use of by-products as potential soil remineralizers by the incubation method

Significant differences were observed among the treatments studied, regardless of the inoculation of microorganisms BF and BT, for the variables pH, P, K, Na, Ca, Mg, Fe, Cu, Zn, Mn, H+Al, SB, T, V, and Prem.

Due to the large amount of statistical information to be presented, a table of notes was created to show the average variation of each of the analyzed variables in comparison to the control, at 30, 60, 90, 120, and 150 days after the application of treatments (DAA). In other words, the variation observed in the treatments, which were soil + by-products (T2 to T11), soil + microorganisms (T12 and T13), and soil + by-products + microorganisms (T14 to T33), was calculated by discounting the naturally occurring variation in the

control (T1). This was done to ensure a clear understanding of the effect of using the by-products on the chemical and physical aspects of the soil.

To construct the table of scores, initially, the statistical groupings defined after variance analysis and the Scott-Knott test at 5% probability were analyzed for the mean variation of each variable and within each evaluation period. Zero scores were assigned for the results referring to the control group (T1) and, from this, other scores were established according to the groupings. For example score '3' for group 'a', '2' for group 'b', '1' for group 'c', '0' for group 'd' (T1 - witness), and '-1' for group 'e'. In the end, we quantified the scores given for each variable at 30, 60, 90, 120, and 150 days, allowing us to see, in a single table, which were the best treatments within each of the variables analyzed (Table V).

**Table V.** Final grades attributed to the average variation of the soil variables analyzed in comparison with the control, after 150 days of treatment application.

TREATMENT	DESCRIPTION	ANALYZED VARIABLE														FINAL GRADE	
		pH	P	K	Na	Ca	Mg	Fe	Cu	Zn	Mn	H+Al	SB	T	V		Prem
T1	Control	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
T2	MG 40%	8	10	0	0	-3	-4	0	1	-4	0	11	-1	-6	9	2	23
T3	MG 60%	9	13	0	0	-3	-5	0	3	-4	0	11	-1	-7	11	4	31
T4	CB 40%	8	4	10	6	-4	-2	0	0	9	-4	11	9	6	11	3	67
T5	CB 60%	9	6	17	11	-4	-3	0	0	10	-4	11	11	9	11	3	87
T6	CI 40%	7	17	0	0	-4	-2	0	9	-2	0	8	-1	-6	8	1	35
T7	CI 60%	9	23	0	0	-4	-4	0	10	-4	0	11	-1	-7	11	4	48
T8	OI 40%	9	4	0	0	-1	-3	6	1	3	4	11	0	-6	10	2	40
T9	OI 60%	11	9	0	0	-1	-7	8	2	3	5	11	-1	-6	11	3	48
T10	AR 40%	13	4	1	0	4	6	11	17	6	13	11	3	-1	11	-4	95
T11	AR 60%	14	7	1	0	5	7	18	22	6	21	11	3	-1	11	-3	122
T12	BF Microorganism	1	0	1	0	-1	-1	0	2	0	0	1	0	0	1	0	4
T13	BT Microorganism	1	0	1	0	-2	0	0	1	-2	0	1	-1	0	0	1	0
T14	MG 40% + BF	6	9	0	0	-3	-4	0	4	-4	0	6	-1	-4	4	3	16
T15	MG 60% + BF	8	13	0	0	-4	-5	0	7	-4	0	10	-3	-8	9	3	26
T16	MG 40% + BT	7	10	0	0	-2	-2	0	6	-4	0	8	-1	-5	6	3	26
T17	MG 60% + BT	8	13	0	0	-2	-4	0	6	-4	0	11	0	-5	9	3	35
T18	CB 40% + BF	8	6	12	7	-3	-1	0	0	8	-4	10	15	13	10	3	84
T19	CB 60% + BF	9	10	13	15	-2	-4	1	-3	13	-4	11	15	14	11	2	101
T20	CB 40% + BT	9	3	9	10	-3	-4	0	-3	8	-4	10	12	10	11	1	69
T21	CB 60% + BT	10	7	12	13	-3	-4	0	-4	13	-4	11	13	10	11	2	87
T22	CI 40% + BF	9	20	0	0	-1	-3	0	5	-3	1	11	-1	-6	11	0	43
T23	CI 60% + BF	10	22	0	0	-4	-6	0	5	-5	1	11	-2	-7	11	4	40
T24	CI 40% + BT	9	14	0	0	-2	-4	0	3	-5	-1	11	-2	-7	11	3	30
T25	CI 60% + BT	9	18	0	0	-3	-6	0	5	-5	0	11	-2	-7	11	4	35
T26	OI 40% + BF	9	6	-2	0	1	-5	5	-3	0	4	11	-1	-6	11	2	32
T27	OI 60% + BF	14	9	0	0	1	-6	9	-3	1	4	11	-1	-6	11	3	47
T28	OI 40% + BT	9	7	0	0	2	-5	4	-3	0	3	11	-1	-6	11	3	35
T29	OI 60% + BT	12	7	0	0	2	-5	6	-4	-1	3	11	0	-4	11	3	41
T30	AR 40% + BF	14	4	0	0	7	4	13	14	7	16	11	4	0	11	-3	102
T31	AR 60% + BF	14	6	0	0	7	5	16	18	7	22	11	5	1	11	-3	120
T32	AR 40% + BT	14	4	0	0	5	5	10	12	6	14	11	4	0	11	-3	93
T33	AR 60% + BT	14	4	0	0	8	7	20	18	8	24	11	5	2	11	-3	129

Regarding the availability of Al and aluminum saturation (m), no significant quantitative differences were observed between the different treatments under study, regardless of the inoculation of microorganisms BF or BT.

## Discussion

The comparison of the results obtained from the analysis of the by-products as potential soil remineralizers with the rules on the definition, classification, specifications, guarantees, and tolerances provided in Normative Instruction, showed that all meet the minimum and maximum requirements provided in the legislation, among which: (i) the physical nature specifications of the remineralizers (Table 1); (ii) the chemical nature specifications of the remineralizers (Table 2); (iii) the free silica contents (Table 3); and (iv) the presence of potentially toxic elements (Table 4).

None of the analyzed residues meet the provisions of the NI concerning the minimum contents of P and micronutrients, which does not prevent their use but restricts the declaration of these as guarantees of the marketed product, through labeling.

In the analysis of the agronomic protocol, in which the objective is to understand the potential of the different by-products in the release of macro and micronutrients, as well as in the improvement of several soil variables, the methodology proposed in this work meets the provisions of the current Brazilian legislation, proposed by EMBRAPA (SILVEIRA et al., 2019a,b).

The sum of the scores assigned after 150 DAA points out the AR by-product as the best material, among those analyzed, to be used agronomically (661 points), followed by the residues CB (495 points), OI (286 points), CI (188 points) and MG (157 points).

The naturally finer particle size of the AR waste may have contributed to its better score for agronomic use, as also observed by Basak et al. (2018) in studies with alkaline volcanic rock waste at different particle sizes and by Dalmora et al. (2020) in nutrient release studies from Andesito and Dacito rocks. According to Priyono and Gilkes (2008), in studies on the dissolution kinetics of multinutrients from silicate rocks, the dissolution rate of cations, for example, increases the smaller the particle size. This fact is important, because the changes resulting from the comminution process in the surface area of the particles assist in the release of elements that were previously fixed in the crystalline structure of minerals (DUARTE et al., 2021).

All by-products analyzed have agronomic potential for soil pH correction, known as 'liming effect', being the by-product AR the most promising also in this case. The pH

elevation with the use of the residue was also reported by Aquino et al. (2020) in studies with alkaline rocks from the volcanic province of Fortaleza (Brazil). The AR by-products also stood out for the variables 'total soil acidity' and 'base saturation index'.

The availability of phosphorus (P) was observed in all analyzed by-products, with CI showing the highest release. It is worth noting the importance of P for plant growth as it participates in essential processes, such as respiration, photosynthesis, root growth, energy storage and transfer, cell division, and fruit and seed formation, as well as precocity of production (TELES et al., 2017).

All analyzed by-products, except for AR, obtained positive results for the Prem variable. Remaining Phosphorus (Prem) is related to the soil's retention capacity of P, and the higher the retention capacity, the lower the value of Prem, which is related to the soil's clay content.

Since Brazil is the world's largest importer of potassium (K) (COMEXSTAT, 2022), a nutrient highly demanded by plants for maintaining and increasing crop productivity (CARVALHO et al., 2018), it becomes crucial to obtain new materials that can be widely used in the agricultural production system, aiming to reduce external dependence and production costs. Among the by-products analyzed in this study, only CB was found to have the ability to make K available to the soil-plant system, as observed for the 'cation exchange capacity at pH 7.0'.

The presence of sodium (Na) in the soil nutrient solution can benefit plant growth when present in small concentrations but can lead to soil salinization and sodification when in higher concentrations (AQUINO et al., 2020). Among the by-products analyzed in this study, only the CB by-product can make Na available to the soil-plant system. Similar results were obtained by Duarte et al. (2021) in studies on the release of nutrients from by-products of ornamental stone processing, including one quartzite, two quartz-diorite, and one syenogranite. The recommended concentration of this by-product will depend, for example, on the management of tillage, especially with regard to the nutrient content of the soil and the presence or absence of irrigated crops.

Regarding the availability of Ca in the soil, an element responsible for regulating the transport of nutrients and present in various enzymatic functions (GILLIHAM et al., 2011), the by-products OI and AR obtained satisfactory results, with emphasis on the AR by-product. The same occurs for the availability of Fe and Mn.

Copper (Cu) is a cofactor in numerous biochemical and physiological reactions of plants. However, excess Cu can reduce root vigor and growth, decrease P uptake,

and cause Fe deficiency. In this study, all by-products, except CB, showed release of this nutrient, with AR exhibiting the highest release.

The AR by-product was the only one to release Mg, an essential nutrient in the photosynthetic process (GUO et al., 2016). Positive results were also found for the availability of Zn and the 'sum of bases' variable, with emphasis on CB, OI, and AR by-products.

The use of BF or BT microorganisms did not significantly affect the solubilization of mineral nutrients present in the by-products under study. This may be attributed to the absence of plants in the incubation study system. All the aforementioned microorganisms are scientifically recognized as plant growth-promoting inoculants, and they depend on plants for proper colonization and development in the soil-plant system through complex inoculant-plant and inoculant-microbiota interactions.

The existing relationship between the soil microbiota and the solubilization of nutrients present in rocks was also reported by Basak et al. (2018) in studies on the significant positive correlation existing between K uptake by plants and K release by different chemical and biological processes, indicating that the rhizosphere of plants can accelerate the release of K from rocks for further uptake by plants, requiring an additional understanding of the biogeochemical processes existing in the rock-soil-plant system for the desired agronomic effectiveness to be achieved.

A relevant concern in the use of rock dust in agricultural soils, from the technique known as rock farming, is the potential availability of toxic elements naturally present in some rocks, which would represent environmental

risks (RAMOS et al., 2017; 2019). This was verified in the analysis of potentially toxic elements (Table 4) provided in Normative Instruction, showing that the contents of As, Cd, Hg and Pb in the samples, all below the detection limit, ensure that the use of the different types of by-products does not offer risks to human and animal health.

No traces of potentially toxic elements were identified in any of the by-products analyzed in this study, demonstrating compliance with Normative Instruction and highlighting the potential use of these by-products as soil remineralizers.

## Conclusions

All by-products analyzed in this research meet the minimum and maximum requirements outlined in Normative Instruction MAPA No. 05/2016 (BRASIL, 2016). The results obtained during the incubation stage, inherent to the Agronomic Protocol, show the potential use of the by-products MG, CB, CI, OI, and AR as soil remineralizers.

The CB by-product was the only one to make K available to the soil.

The sum of the scores assigned to the by-products after 150 DAA reveals, in general, AR as the most efficient by-product to be used agronomically, followed by CB, OI, CI, and MG.

The use of by-products incorporated into the soil has a high agricultural potential, which was not favored in the incubation stage with the inoculation of microorganisms 'BF' or 'BT'.

**Acknowledgments:** To the Center of Mineral Technology - CETEM, the Brazilian Association of the Ornamental Stones Industry - ABIROCHAS, the Espírito Santo Foundation for Research and Innovation - FAPES, the Brazilian National Council for Scientific and Technological Development (CNPq), the Federal University of Espírito Santo - UFES, and the companies Alto Liberdade Mármore e Granitos, Alto Vale Mineração, Gramarcá Granitos, Mármore e Calcários, Paraná Granitos Group, and MG2 Mármore e Granitos, there being no conflict of interest in the conduction and publication of this work.

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## PORPHYRY: A PRECIOUS STONE WITH EXCEPTIONAL EXPERTISE AT THE SERVICE OF URBAN DESIGN. NOW, A NEW TOOL IS AVAILABLE TO HELP DESIGN

G. Signori<sup>1\*</sup>, A. Angheben<sup>2</sup>

- (1) Ateneo di Scienze, Lettere ed Arti di Bergamo, \*[eurgeol.grazia.signori@gmail.com](mailto:eurgeol.grazia.signori@gmail.com)  
(2) Consorzio Italporphyry

**Summary:** *Even the most precious of resources, natural stone in particular, if used incorrectly, can be a source of failure and disappointment, with the negative effect of both distancing people from the product and wasting a non-renewable resource.*

*For this reason, the world of natural stone has always been looking for ways to communicate with the world of design.*

*In this context, it is essential to have technical documentation and updated manuals to support and guide the designer in defining the correct design solution.*

*Hence the project to create THE GUIDE TO PORPHYRY.*

*Following in the footsteps of some manuals from the 1990s, in a contemporary key and on a national and global scale, the Guide illustrates the product from a genetic, technical, regulatory and application point of view, showing the peculiarities of a sector that has recently rediscovered a new international destination, with exports to more than 50 countries.*

**Key words:** *porphyry, Porfido del Trentino, design, installation, guide, sustainability*

When we talk about "porphyry", we are talking about a product and a know-how that has withstood the test of time, chosen for its beauty, strength and durability. What's more, just like Carrara marble, it is one of the most popular Italian products abroad and one of the most widely used natural stones in architecture worldwide.

Rocks similar to porphyry are found all over the world, but only in the Trentino-Alto Adige region (Northern Italy) do they have the ideal characteristics to become a valuable product for paving city squares and streets, parks, villas and gardens, and are well established thanks to a century-old tradition of use.

### **A miracle of nature meets an industry's visionary clarity, a century ago**

From a petrographic point of view, Trentino porphyry is a rhyolite, i.e. the effusive equivalent of granite. It is composed of phenocrysts of quartz, feldspars and biotite embedded in a glassy groundmass.

Besides its composition, the peculiarity that makes Porfido del Trentino unique is its regular divisibility along three nearly orthogonal planes (Fig. 1).



*Fig. 1. The Porfido del Trentino typical and regular divisibility along three nearly orthogonal planes*

The extraordinary combination of composition and structure of Trentino porphyry is a true miracle of nature. It determines its durability and allows it to be split since time immemorial.

We owe this combination to the legacy left by a series of chance events that have occurred during the very long and peculiar history of this rock, from its origin as the last phase of the Piattaforma Porfirica Atesina volcanic system about 278 million years ago to the present, passing through the formation of the Alps.

The beginning of the quarrying of the Trentino porphyry is lost in the mists of time. Since the 20th century, however, an epochal change has taken place.

Since the '20s of the 20th century, in fact, the Trentino Porphyry sector has made an enormous and far-sighted effort to codify the phases of design and construction of stone paving, involving and instructing designers and public authorities, creating experimental fields and continuing to disseminate consolidated experience and successful know-how.

A century of experience and thousands of square metres of road paving in cubes, binders and porphyry slabs now provide a unique statistical basis for identifying and updating the approach to the design, construction and maintenance of stone roads to meet today's requirements.

For the paving world, this wealth of experience and knowledge is the unique asset that only Porfido Trentino can offer: a tested and analytical archive to meet today's challenges.

### **The aim of THE GUIDE TO PORPHYRY**

Porphyry from Trentino is an extraordinary resource, abundant but not infinite, a worldwide symbol of the value and beauty of Made in Italy.

For this reason, it is strategic to enhance it and guide it to its correct use through a specific and technical communication towards designers, users and Municipalities telling them clearly why Trentino Porphyry is so special, how the paving shall be designed, how it shall be laid, how the production cycles, the circular economy and product sustainability are pursued and implemented from the cradle to the gate and from the cradle to the grave.

The paving of open spaces is a key element for the cities of today and tomorrow, and must continuously innovate to face the challenge of usage requirements, the needs of smart cities, the management of global warming, demonstrating with numbers their contribution in terms of sustainability.

Hence the project to create THE GUIDE TO PORPHYRY, a valuable publication in terms of content and approach, properly designed to be the professional knowledge tool for the sector.

### **The contents of THE GUIDE TO PORPHYRY**

The contents of the guide deal with the various themes related to Porfido del Trentino.

Starting from the history of porphyry as a natural stone for building and paving, the guide explains its geological nature, its occurrence and the production processes.

All the different stone products produced, such as slabs, tiles, blocks, binders and smoller, are described in detail and discussed according to the technical standards for testing methods and stone laying.

Then it's the turn of the design, installation and maintenance of stone paving, according to paving units and paving load classes/final destination.

Finally, the durability of porphyry is discussed as an expression of sustainability.

### **The targeted audience of THE GUIDE TO PORPHYRY**

The addressees of the guide are architects, engineers, designers, specifiers, users, public authorities and installers.

Since Trentino Porphyry is specified and installed all over the world, the guide is in Italian and English.

### **The authors of THE GUIDE TO PORPHYRY**

The authors of the guide are Andrea Angheben and Grazia Signori.

Andrea Angheben has been working for the porphyry industry since early '90s, and is the main expert of porphyry worldwide, either for product specification, design, installation and maintenance.

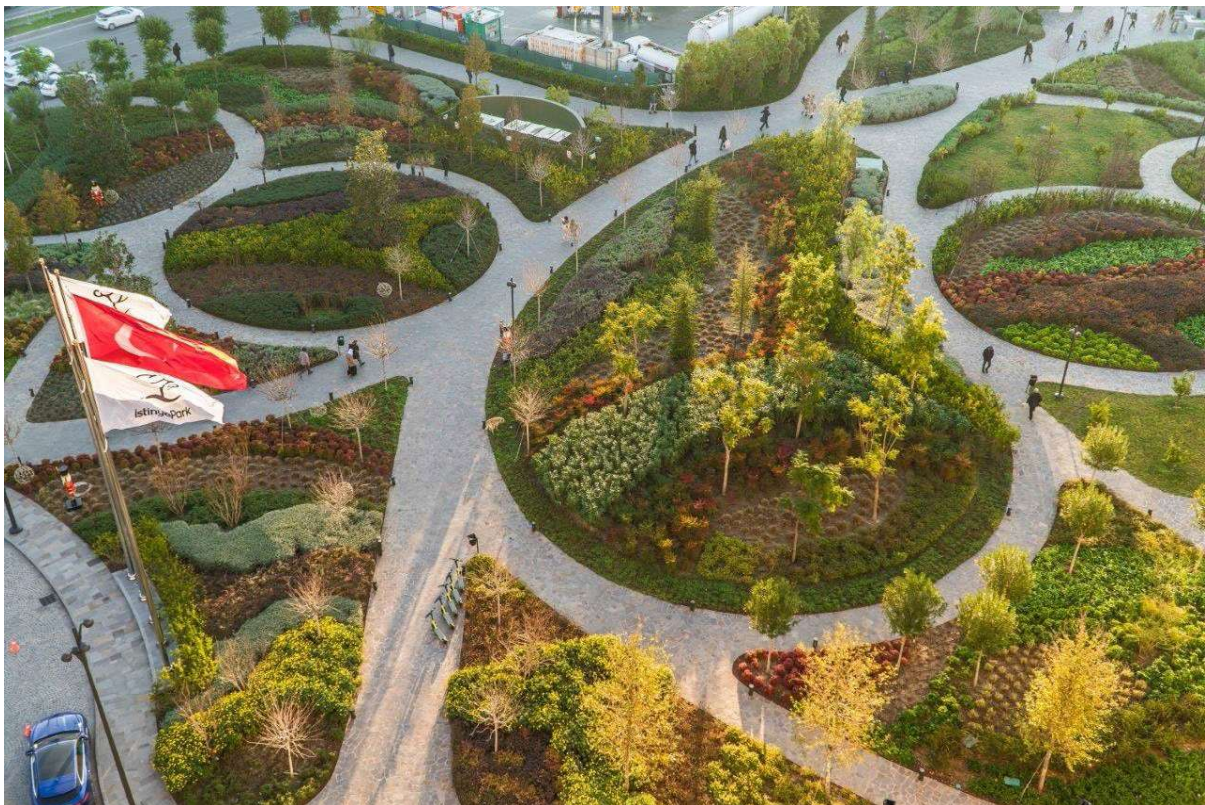
Grazia Signori has been working for the stone industry since 2001 and her expertise spans from geological topics to stone characterization, technical standardization and stone paving.

### **Conclusions**

THE GUIDE TO PORPHYRY will be officially presented to the stone sector next September 2023 in Verona, during Marmomac International Stone Fair.



*Fig. 2. Paris: Les Halles*



*Fig. 3. Izmir: Istinye Park Izmir*



*Fig. 4. La Valletta: Government Palace*

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# ADDITIVE MANUFACTURING OF NATURAL STONE BASED MATERIALS: ASSESSMENT OF BINDER-JETTING CAPABILITIES

Hélio Jorge

CTCV – Centro Tecnológico da Cerâmica e do Vidro, Parque Tecnológico de Coimbra, Lote 6 e 7, 3040-540 Antanhol-Coimbra, Portugal, [hrjorge@ctcv.pt](mailto:hrjorge@ctcv.pt)

**Summary:** Additive manufacturing technologies (AM) are well known since they make possible the production of objects directly from 3D computer files and, comparing to traditional processes, they present remarkable advantages in the replication of complex part geometries with reduced manufacturing time, no need for moulds, the use of much less raw material and less material waste.

One of the technologies under greater development is the binder-jetting (BJ), which uses powdered raw material. It is seen as very productive manufacturing technology and it has the ability to produce objects with complex geometry without construction support structures, which would be removed very cautiously. So, it is quite competitive in terms of production cost due to the low equipment use time and the reduced part finishing time.

The development of AM technologies of parts for stone projects is a hot topic, whether they process stone-mimetic materials, such as polymers, whether they use real stone materials. This work presents the first results of the assessment of binder-jetting capabilities for the production of stone-based products. 3D printing tests were carried out in a commercial printer using a specific stone powder and a binder formulation, composed with waste powders from the stone industry. Therefore, it will be possible to create sustainable, water resistant, fully inorganic materials, without plastics, glue or resins. Printing process procedure will be described and material characterisation results will be shown and discussed. The aim is to develop and to demonstrate the potential of that technology in order to shown to stone companies and their costumers the potential for innovative applications, such as in construction and architecture, product design, restoration or art.

**Key words:** Additive manufacturing, binder jetting, stone powder

## Introduction

Additive manufacturing makes possible the production of three-dimensional physical models from a virtual model. Based on computer-aided design (CAD), all additive processes currently available on the market are based on the deposition of material, layer upon layer, until a three-dimensional product is obtained [1].

Since the early 1980s, additive manufacturing technologies, initially called rapid prototyping, have been tools to support the product development process [2]. There is a set of automated manufacturing methods that play an important role in helping the development of models with complex and irregular geometries. Binder jetting (BJ), plastic extrusion deposition (FDM), stereo lithography (SL) and selective laser sintering (SLS) are four of the most commonly used commercial additive manufacturing techniques for producing artefacts from a virtual computer model. These

manufacturing techniques are capable of processing different types of materials, from ceramics, polymers or metals, in the form of three-dimensional artefacts without the use of a mould or machining and at a reasonable speed. Due to the impact that these technologies have had on industry, technological development and society, refined additive manufacturing techniques have emerged, more suited to certain types of raw materials. This is the case of rapid manufacturing systems that uses powders and is commonly referred to as Binder Jetting [3].

The use of these techniques makes possible to manufacture artefacts with complex geometries that are not possible to obtain through conventional production techniques. The reproducibility and computerized control of the design characteristics of these products make additive manufacturing techniques present several advantages for the industry and add a potential in the manufacture of applications that

respond to the needs of very specific market segments [4].

Binder-jetting is an additive manufacturing technique based on the use of powders, which consists of depositing a liquid binder material, from a print head, on pre-prepared layers of powder. Several materials can be processed by this technique, such as ceramics, polymers, metals and composites. The resolution of the printed models depends not only on the binder and the powder particle size used, but also on the accuracy of the binder droplet deposition and the interaction between the binder and the powder particles. This process has some points for improvement, namely the rough surface finishing, the porous material obtained, the brittleness of the green pieces and the part de-powdering, which could be very hard and time consuming when intricate shapes are present [5].

This paper presents preliminary results of an ongoing study about material characterization, both raw materials and printed materials, and the relationship with the printing parameters. The aim is to assess to the technology capabilities and seek for potential market applications.

### Experimental description

A Concr3de Armadillo White binder jetting printer was used to manufacture several parts (Fig. 1). The printer has a print volume dimensions of 370 x 290 x 250 mm, a print head resolution of 400 dpi and the layer height can be selected within the range of 40 to 500  $\mu\text{m}$ . The printing process can be described generically by Fig. 2. The printer has two powder beds. In each layering step, a powder transfer is carried from the feeding bed to the printing bed to form a thin layer. Then, a piezoelectric actuator holed printing head jets binder droplets to glue selectively the powder of the layer just deposited, so

that a 2D solid slice is formed. That layered process is repeated until a glued layer group forms the final 3D part.

The powder, supplied by the printer manufacturer, is composed with waste powder from the stone industry. It is a dried powder (humidity of 0.19% wet based) with an apparent and tap density of 1.26 and 1.78  $\text{g}/\text{cm}^3$ , respectively.



Fig. 1. Binder jetting printer -Armadillo White.

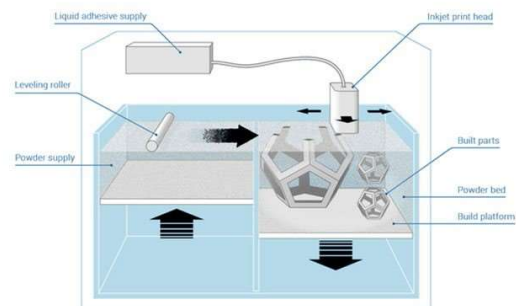


Fig. 2. Simplified process of powder bed binder jetting. Two powder beds (powder feeding bed and printing bed) and a 2-axis printing head are the main sections [6].

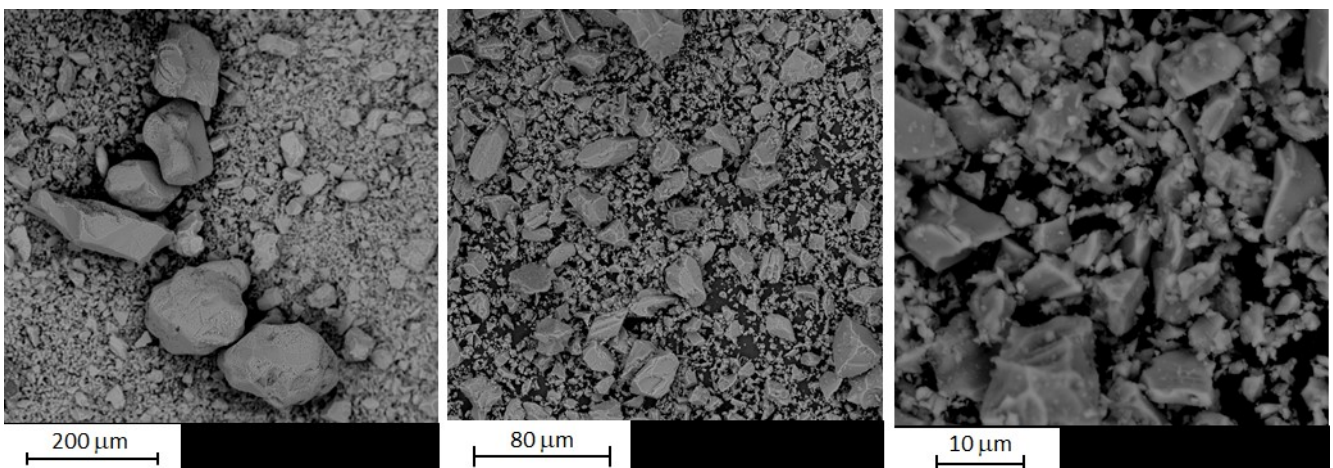


Fig. 3. SEM micrographs of the powder. A wide particle size was found from ca. 1 to 180  $\mu\text{m}$ .

By morphological observation carried with a Phenom Pro desktop SEM, it was possible to estimate a particle size range of the powder of 1 to 180  $\mu\text{m}$  (Fig. 3). Powders with such a wide change of particle size are often selected for binder jetting, because it offers a good balance between flow and reactive behaviour. Coarse particles provide better flow to ensure a good spread ability necessary for the powder layer forming in the printer, and fines are better to enhance wetting by the jetted binder and improve green strength.

An aqueous solution from the same supplier was used as the adhesive binder, which is jetted on the powder from the printer head. The viscosity, measured with a Netzsch

Kinexus lab+ rheometer, using a cone and plate geometry with 40 mm diameter, was constant in the range of 4 to 250  $\text{s}^{-1}$  shear rate with a value of 10 mPa.s (Fig. 4). The viscosity is in the range required for the printer head used (8-20 mPa.s)

Part demonstrators were printed to assess the geometry reproduction capability of the binder jetting technology and show some examples of viable applications. As recommend by the supplier, parts can be produced with that powder and binder with a layer height from 100 to 300  $\mu\text{m}$ . For this work parts were produced with 230  $\mu\text{m}$  layer height. Three application areas were chosen based on bibliography search and market scouting: a) sculpture, b) aquarium ornaments and c) sculptural building parts. Bar (150 x 30 x 15 mm) and cubic (30 x 30 x 30 mm) specimens were also printed for flexure and compression mechanical testing.

After printing, the so-called green parts are obtained. They have low mechanical strength because of the weak adhesion forces of the aggregation of powder particle by the liquid binder. Therefore post-printing steps are usually carried to improve the parts strength. The powder and binder used are designed to obtain hardened parts by dipping in water. Water dipping is claimed by the supplier to promote a hydration reaction of the powder, and the particle links become stronger [7]. Parts were dipped for 3 hours. Then drying was carried at 60  $^{\circ}\text{C}$  in an oven.

## Results and discussion

Building parts can be produced for new buildings as well for restoration works. Old damaged parts can be 3D scanned, from which the missing part can be designed on using a CAD software. Then the part can be 3D printed to produce a prosthesis to be assembled on the building damaged part. To implement this idea, assembling method, colour matching and material properties and durability should be investigated.

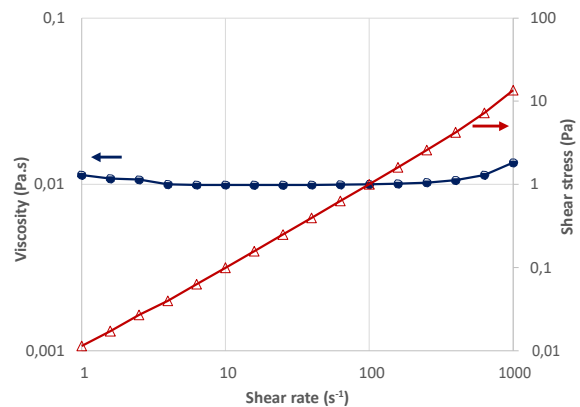


Fig. 4. Flow curve of the binder – viscosity and shear stress vs. shear rate – presenting a similar behaviour as Newtonian fluids.

Sculpture parts were successfully printed despite of some thin edges, more likely to brake (Fig. 5). Printing resolution, also considered as the minimum edge radius or minimum feature size, can be relatively accepted. They showed very well shape reproduction, but higher resolution could be desired. Therefore, future efforts will be carried to increase part print resolution, for example by printing with lower layer thickness and using a lower particle size powder.

The ornamental parts obtained show perfect aesthetics for aquariums, since they have old and worn stones appearance or similarities with corals (Fig. 6).

The building sculptural parts design complexity and thinness of the geometries have the appearance as it was been hand craving was achieved (Fig. 7). Printing resolution is not a handicap, as often the part is far enough out of sight to be to be noticed the layer effect.

Fig. 8 show the bar and cubic specimens. After hydration, low density and high water absorption was obtained comparing to a cement or a stone (Table I). Low values of compressive strength is an expected consequent. Therefore second post processing operations in order to make the material harder is a possible way to be tested in the on-going work plan.





*Fig. 5 Sculpture parts.*



*Fig. 6 Aquarium ornaments.*



*Fig. 7 Sculptural building parts.*

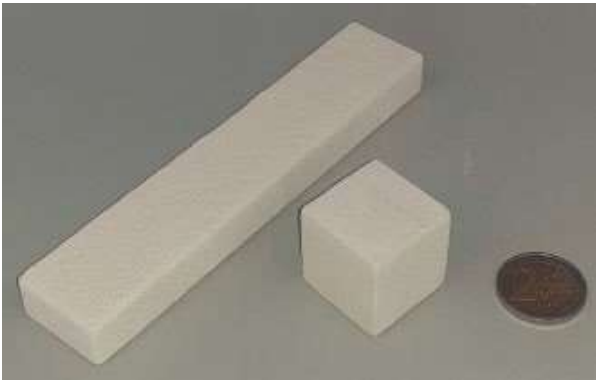


Fig. 4. Sculpture parts.

Table I. Material properties after the first step of post-processing

Property	Value	Specimen geometry
Density	$1,40 \pm 0,03 \text{ g/cm}^3$	Cube
Water absorption	$21,8 \pm 0,2 \%$	Cube
	$24,2 \pm 0,5 \%$	Bar
Compression strength	$8,0 \pm 1,3 \text{ MPa}$	Cube z-axis
	$9,1 \pm 1,1 \text{ MPa}$	Cube x/y-axis

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## Conclusion and future work

First results of a preliminary phase of a study about binder jetting technology applied to cementitious and stone powder based materials. Using a combination of powder and binder from the market it was possible to print some parts that demonstrates with success some examples of product application and opens up new perspectives on the innovation potential of the technology.

Due to the high porosity of printed materials, post processing steps should be tested in order to increase density and mechanical cohesion of the material. The first results of post processing testing of printed green parts were presented. Hardening step testing and a more extensive material characterisation not only of the final material but also of the raw materials are undergoing.

# Developing a Conceptual and Innovative Framework for Circular Economy in the Ornamental Stone industry

*Isabel Duarte de Almeida (isabel.cristina.almeida@iscte-iul.pt)*  
*Instituto Universitário de Lisboa (ISCTE-IUL), IBS, Lisboa, Portugal*

*Agostinho da Silva (a.silva@zipor.com)*  
*CIIC, Politécnico de Leiria, Leiria, Portugal*  
*ISG- Instituto Superior de Gestão, Lisboa, Portugal*

**Keywords:** Circular Economy; Ornamental Stone sector; Sustainability

Resource scarcity and increasing consumption are forcing companies and governments to rethink today's prevailing linear framework, in which products are discarded after their useful life. As a result, a different business model emerges - the circular economy (CE) - which allows economic growth to be decoupled from waste generation, environmental protection, pollution prevention and sustainable development. In CE, every part of a given value chain must realise that once an indispensable resource is gone, the chain loses. Like each component, the supply chain (SC) must operate within economic, environmental, and social thresholds. SC managers need to develop metrics that consider the sustainability context and build relationships with SC actors. Furthermore, the concept only works if SC and sector's partners, such as that of Ornamental Stones (OS), build mutual trust in each partner's sustainability information. Finally, CE requires transparency about the materials, financial flows and components associated with the product during their life cycle. Given the cyclical nature of this new economic trend, CE optimises the flow of goods and materials throughout the production process. In the OS industrial sector, companies can benefit from circular business models by modernising them and bringing them to a higher technological level.

From the above, the following research questions arise:

1. How can the environmental costs of the OS industry be reduced, from extraction to final product and at the level of waste from the manufacturing process itself?
3. What are the drivers and barriers for key stakeholders to engage in a stronger CE in the OS industry? What measures might be needed to improve this CE?

This article aims to search, analyse, and assess the circular models' efficiency by organising data on experiences of rational resource use in the OS industry and converting basic circular models into ones adapted to suit the environment in which Portuguese OS companies operate. This study will review management practices, operational processes, and production environments within the OS industry, that facilitate and promote the transition to a circular business model. This study contributes to developing a new conceptual framework in organisational capabilities to support the sustainable transition in the OS sector. It uses Innovation Theory to support this effort. The proposed framework for circular business models considers linkages between management practices and operational processes. It addresses the nature of OS production chains as a strategy for improving sustainable development indicators, reducing production costs, extending product ranges, and increasing the value of OS companies and OS sector robustness. Additionally, the framework contemplates the specificities of stone companies and the principle that a resource is valuable throughout its life cycle.

Portuguese stone companies can use the proposed conceptual framework for circular business models to design business strategies and long-term development plans.

# The Eurolithos project and its main outputs

*Jorge Carvalho, LNEG – National Laboratory for Energy and Geology, jorge.carvalho@lneg.pt*  
*Tom Heldal, NGU - Geological Survey of Norway, tom.heldal@ngu.no*  
*Konstantinos Laskaridis, HSGME - Hellenic Survey of Geology & Mineral Exploration, laskaridis@igme.gr*

Keywords: Eurolithos Project, Results, Atlas, Identity Card, Ornamental Stones

Ornamental stone has contributed significantly in shaping our rural and urban landscapes, through its use in our built heritage. Today, ornamental stone is a raw material produced with great skills all over Europe, exploiting the vast diversity of European natural stone resources. Yet, the actual use of local and regional stone resources in Europe is decreasing, and so is the knowledge of the resources, traditions and skills. Under the auspices of the European GeoEra Programme (<https://geoera.eu/>), Eurolithos (<https://www.eurolithos.org/>) was a project dedicated to European ornamental stones. It ran between July 2018 and October 2021, with 15 partners from 14 countries.

EuroLithos was built on the idea that increasing knowledge of the geology, quality and history of ornamental stone use in Europe will both stimulate more sustainable use of stone resources in Europe for the benefit of SMEs and our cultural heritage and will contribute to a sound land use management for the safeguarding of ornamental stone deposits. Establishing a framework for the development of a first edition of an Atlas of European Ornamental Stones was one of the core objectives of the project. The Atlas intended to be a science-based information system to identify, collect and harmonize existing data on the provenance of European Ornamental Stones. Another core objective of Eurolithos was the development of a Directory of Ornamental Stone Properties by establishing the framework for an “identity card” of each European ornamental stone providing harmonized data regarding their composition, physical properties and “performance in use” criteria.

Delays related to Covid-19 pandemics and difficulties to overcome INSPIRE compliance issues meant that the Atlas did not integrate all the required fields of data. What is currently available to the public through the EuroGeosurveys’ EGD platform (<https://www.europe-geology.eu/scientific-themes/mineral-resources/eurolithos/>) are only the data relating to:

- Name of Stone according to EN 12440;
- Alternative Name 1 and 2;
- Commodity Code Value from the new Eurolithos code list: Basalt, Granite, Limestone, Marble, Miscellaneous ornamental stones, Sandstone, Slate;
- Colour value from the new Eurolithos colour code list: Beige, Black, Black-green, Blue, Blue-grey, Brown, Dark grey, Green, Grey, Grey-brown, Grey-green, Light-grey, Light-pink, Multicoloured, Pink, Red, Redish brown, White, White-black, Yellow;
- Name of place of origin (and respective WGS84 geographic coordinates)
- Municipality
- Country

For the stones for which the Identity Card was uploaded to the EGD platform during the time-life of the project, a link to it is also provided (e.g. <https://repository.europe-geology.eu/egdidocs/eurolithos/lioz.pdf>). Currently, the project partners are still able to upload new datasheets.

A printable version of six country atlases (Cyprus, Emilia-Romagna (Italy), Greece, Italy, Portugal, and Slovenia) including the uploaded identity cards, was also prepared and can be downloaded at: [https://www.eurolithos.org/files/ugd/2b8de6\\_1cb3afa8fde14250994511d8e400fc4f.pdf](https://www.eurolithos.org/files/ugd/2b8de6_1cb3afa8fde14250994511d8e400fc4f.pdf).

## Sustainability in Dimension Stone Industry

J. Góis<sup>1\*</sup>, L. Lopes<sup>2,3</sup>, R. Martins<sup>2</sup>

<sup>1</sup> Ordem dos Engenheiros (Portuguese Association of Engineers), Faculty of Engineering – University of Porto, \*jgois@fe.up.pt

<sup>2</sup> University of Évora - School of Science and Technology. Institute of Earth Sciences

<sup>3</sup> Portuguese Association of Geologists

The dimension stone industry represents a unique sector and an important economic and social asset for many countries in the world. The global dimension stone mining market is expected to grow from \$8.51 billion in 2022 to \$9.12 billion in 2023 at a compound annual growth rate (CAGR) of 7.2%. The dimension stone mining market is expected to grow to \$11.89 billion in 2027 at a CAGR of 6.9%.

In 2020, 318 million tons were extracted from quarries, but only 155 million tons went into further processing figure 1. Later, when the blocks are cut, a lot of stone powder is lost, so in the end, only 92 million tons are utilized. These ratios have not changed in the last two decades and around 51% of the extracted material is dumped in the quarry, while 20% is lost during processing in the factories. Is it possible to promote sustainability in the dimension stone industry?

# Structural, physical and compositional proprieties analysis of marbles in a quarry face: a study case application on Estremoz Anticline

*Madrinha, João (1); Moreira, Noel\* (2,3); Lopes, Luís (3), Sitzia, Fabio (4), Mirão, José (5), Dionísio, Maria Amélia (6), Neves, Samuel (7)*

- (1) MSc student in Engenharia Geológica e de Minas - Instituto Superior Técnico, Lisboa; Research fellow in I4STONE project – Universidade de Évora.
- (2) Instituto de Investigação e Formação Avançada – Universidade de Évora. \*[nafm@uevora.pt](mailto:nafm@uevora.pt)
- (3) Universidade de Évora, Instituto de Ciências da Terra (ICT) - Polo de Évora, Departamento de Geociências - Universidade de Évora, Colégio Luís António Verney, Rua Romão Ramalho, 59, 7000-671 Évora, Portugal.
- (4) HERCULES Laboratory; Instituto de Investigação e Formação Avançada - Universidade de Évora, Palácio do Vimioso, Largo Marquês de Marialva, 8, 7000-809 Évora, Portugal
- (5) Departamento de Geociências - Universidade de Évora; HERCULES Laboratory - Universidade de Évora, Palácio do Vimioso, Largo Marquês de Marialva, 8, 7000-809 Évora, Portugal
- (6) Departamento de Engenharia de Recursos Minerais e Energéticos, Instituto Superior Técnico; CERENA- Centro de Recursos Naturais e Ambiente, Lisboa, Portugal
- (7) ARROW4D - Consultores de Engenharia e Geofísica, LDA, Évora, Portugal

## Abstract

The application of scientific and technological research activities to innovation and to social and economic development is one of the great challenges of contemporary times. Such aspects are even more significant in the industrial sector, because the R&D activities can have a remarkable impact on the sustainable production of raw materials and on the economic viability of companies. Indeed, the development of innovative and expeditious scientific and technological methods, which can reply to the usual and specific challenges of the exploitation, manufacture and transformation of dimension stones, are extremely important in the business dynamics.

The Estremoz Anticline is one of the most significant active poles of the extractive industry in Portugal, being one of the main marble-producing poles at the European scale. The extractive activity takes place in the three municipalities of the so-called Marble Triangle (Borba, Estremoz, Vila Viçosa), exploiting calcite marbles from the Estremoz Volcano-Sedimentary Carbonated Complex, whose geological age has not been fully clarified (Cambrian to Devonian ages were attributed to this unit). The marble exploitation, manufacture and transformation is one of the main drivers of development in the Marble Triangle, presenting great local and regional socio-economic significance. Beyond this, it is also central to increase the rock coefficient of utilization (20-25% in Estremoz marble quarries) in order to decrease the volume of wastes and increase the marble productivity in this region and therefore marble's production sustainability.

Indeed, in order to support the raw material exploitation process, the management of mineral masses and the potential rock coefficient of utilization and blockometrics, detailed geological mapping of quarry faces were carried out. These detailed geological mapping include: (1) Lithological and structural characterization of quarry faces, including detailed characterization of the existing lithotypes, their mineralogical composition and lateral variations of marble typologies, as well as the geometric and kinematic detailed characterization of the main structures, namely foliation, shear zones and fractures, including the density characterization of fracturing; and (2) In situ evaluation of aesthetic, compositional and physical-mechanical features of marbles, using multiple portable equipment's (e.g. X-ray fluorescence, colorimeter, Schmidt sclerometer). The application of this innovative and expeditious methodology, using a fine sampling matrix of the vertical and horizontal quarry faces, allowed the construction of detailed two- and three-dimensional maps, which could allow better management of the extractive activity.

## Acknowledgements

This paper is a contribution to the project I4STONE (REF: ALT 20-03-0247-FEDER-072231), financed by the European Regional Development Fund (ERDF) and Regional Operational Programme of Alentejo (ALENTEJO 2020). Noel Moreira thanks the financial support provided by the ICT, under contract with FCT (Fundação para a Ciência e a Tecnologia) (REF: UIDB/04683/2020 e UIDP/04683/2020).

# THE USE OF VOLCANIC STONE IN THE ARCHITECTURAL HERITAGE OF THE ISLAND OF TENERIFE (CANARY ISLANDS)

J.A. Álvarez<sup>1\*</sup>, A.J. Sánchez<sup>1</sup>, J.A. Valido<sup>2</sup>

(1) Dpt. of Fine Arts, University of La Laguna, La Laguna (Spain) \*[jalvarer@ull.edu.es](mailto:jalvarer@ull.edu.es)

(2) Dpt. of Industrial Engineering, School of Engineering and Technology, University of La Laguna (Spain)

**Summary:** *The volcanic origin of the Canary Islands has given the islands a great lithological diversity. The historical use that has been given to this type of material has been determined by its specific characteristics: hardness, cohesion, compactness, softness, textural heterogeneity, colour, etc. These volcanic materials, thanks to their intrinsic characteristics, give the architectural heritage of the islands a unique and differentiated character compared to the rest of Spain. Some of the most unique historical works on the island of Tenerife are highlighted (most of them have been declared of Cultural Interest) according to the lithology of the stone material used and the quarries from which these materials were retrieved. Nowadays, these quarries have been either abandoned or their extraction is restricted or prohibited. Knowing the origin of the stone used and its pathologies is of vital importance for the correct conservation and restoration of the heritage, as it allows, among other things, the replacement of a material that is compatible in behaviour with the deteriorated elements.*

**Key words:** *Tenerife, volcanic stone, heritage, architecture*

## 1. Introduction

There are no bibliographies of the quarry's locations in the history of Tenerife in general, although there are documents of great interest that deal with partial aspects of the subject. Firstly, we should mention a text by Larraz (1999), in which he analysed quarrying at the beginning of the Conquest, pointing out that "the determined policy of the council which favoured the creation of an open system of quarrying and commercialization of the material" and he also noted that the three types of stone used at this early stage, were the "Piedra Colorada", mainly taken from the area of "Tegueste" ("Pico Bermejo" or "El Peñón"), the "Piedra Blanca" from "Portechuelo" and the slope of "San Lázaro" and the "Piedra Jabaluna" in the "Camecerías" ravine. Isolated references that we have found in multiple documents (articles, papers, books of works and minor contracts), which allowed the typology and extraction sites to be extended considerably. In summary, these are as follows:

### *i. Tuffs*

- "Piedra Colorada", also known as "Bermeja" or red, of which the following quarries were mentioned in the area around "San Cristóbal de La Laguna", in the north-east of the island, the quarries of: "Pico Bermejo", "El Peñol", "Nombre de Dios", "Obispo", "Taganana", "Valle de Salazar" and in the north-west, the ones near "Masca", in "Teno" massif.

- "Piedra Blanca" of which the following quarries were cited: "Roque Maldonado", "Roque del Peñón", and on the slopes of "San Lázaro". There are also references to a quarry located in the "Portezuelo", probably on the mountain of "El Pulpito".

- "Piedra Tosca", abundant mainly in the southwest. Currently, it is exploited and marketed by "Cantos Blancos del Sur" (Arico).

### *ii. Ignimbrites*

- "Piedra de San Juan de la Rambla", matrix of golden gray tone with well adhered centimetric flames of darker tone, medium hardness.

- "Piedra Chasnera", matrix with different colours, well adhered centimetric flames, compact and resistant. "Chasneras" are currently exploited and marketed by "Guamarico S.L.", distinguishing four varieties: "Brown", "Violet", "Grey" and "Green". The physical properties of these stones have been studied by Valido et al. (2023). Historically this stone has been widely used as paving, transported by boat from "Tajao" to the main historical centres, so references to it can be found as "Losa chasnera".

### *iii. Basalts and trachybasalts*

- "Piedra Molinera", so called because the aborigines used them to build grain mills. In the ornamental heritage, we have found basically fine-pored vacuolar varieties. Found in the northern part in the "Mocán"

quarry and in the south of the island in of "Granadilla" quarries. Currently, it is marketed by "Rocasa S.A."

- "Piedra Jabaluna", a term used by the first stonemasons who arrived on the island to refer to a stone that must have been similar in appearance and properties to the dark-coloured slate quarried in Tarifa (Cadiz) known as "Piedra de Tarifa or Piedra Jabaluna". There is little documentation on the quarries where this type of stone was extracted. Some references mention the quarry "La Machada", possibly located in the ravine of "La Carnecería".

#### iv. *Trachytes and phonolites*

- "Piedra gris - dorada" (its light gray tone turns golden due to surface oxidation). The following quarries were mentioned: "El Viejo" (Tegueste), "Lomo Román" (Santa Úrsula) (Fig. 1), "Tigaiga" (Los Realejos) and "Buenavista del Norte".

- "Piedra Azul", of which the following quarries were mentioned in the surroundings of "La Laguna"; "Pedro Álvarez". In the northern area; "Acentejo", "El Esparragal", "La Solitaria de Taco", "Los Silos" and "Camino del Lugar" (Buenavista). In the southern area; mainly those of "Barranco de la Orchilla" (Abona) and those of "Sixto" (Granadilla), both in exploitation by "Talleres Sixto".



Fig. 1 Quarry "Lomo Román" (Santa Úrsula)

On the basis of these initial data, significant heritage examples have been selected for each type of stone. In presenting them, the possibilities for sculptural work will be discussed. Images and descriptions of quarries will also be presented.

## 2. Geological context

The origin of the Canary Islands has been the subject of study since the 1970s, and several hypotheses have been put forward since then. In the case of Tenerife, the most accepted theory is that the island was formed from a Central Shield, "Roque del Conde" (11.9 - 8.9 Ma) and two later constructions that grew on its flanks, "Teno" (6.2 - 5.6 Ma) and "Anaga" (4.9 - 3.9 Ma) (Guillou et al. 2004), this stage is called "Serie Basáltica Antigua". After a period of inactivity, a new eruptive stage began, mainly marked by the "Las Cañadas" volcano (3.5 Ma)

and in which three main cycles of activity can be distinguished, causing successive caldera collapses (Ancochea et al. 1990). This phase of formation is known as the "Edificio Cañadas". Subsequently, fissural volcanism initiated the construction of the "Edificio Dorsal" (1.0 Ma). After these formations, the first eruptions began, forming the "Complejo Teide-Pico" (0.2 Ma). The materials emitted during this period occupied a large part of the caldera and overlapped until they reached a height of 3718 metres, thus forming the Teide stratovolcano (third highest volcanic structure in the world).

#### i. *Tuffs*

The tuff quarries, from which the "Piedra Colorada" and "Piedra Blanca" were extracted, are mainly associated with the "Anaga" massif, although it is also possible to find some extraction areas in the "Teno" massif. This material occurs in levels interspersed between the basaltic lava flows. The lava flows being the most representative unit of this massif. Tuff is a pyroclastic rock formed by the lithification of lapilli and ash deposits (fall deposits) (de Vallejo et al. 2007), emitted in a volcanic eruption, usually of an explosive nature. This formation mechanism, typical of pyroclastic fall deposits, is equally valid for the "Piedra Tosca", as this is a type of tuff in which pumice fragments predominate (pumice tuff). This lithological unit, described on geological maps as "Undifferentiated felsic pyroclasts" outcrops mainly on the southern and south-western slopes, hence the extraction quarries are located in this area of the island.

#### ii. *Ignimbrites*

The ignimbrites of "San Juan de la Rambla" outcrop in the northern part of the island. With an estimated age of 1.24 Ma, they occur at depths of about 40 metres, forming poorly individualised beds with coarse columnar disjunction. The "Piedra Chasnera" belongs to the lithological unit "Arico ignimbrite's" (0.65 Ma), because it is on the slopes of this municipality in the south of the island where they outcrop. The term ignimbrite is sometimes used as a synonym for welded tuff, while other authors propose that the tuff is a non-welded ignimbrite (Hernández-Gutiérrez, 2017). The ignimbrites mentioned in this study correspond to pyroclastic flow deposits in which the lithic fragments are crushed and stretched (fiamme) following the flow direction, as described by de Vallejo et al. (2007).

It should be noted that the lithological units to which the "Piedra Tosca" and "Piedra Chasnera" belong have been extensively studied in the field of volcanology and geology, as they constitute, together with other



deposits, what is known as the "Bandas del Sur Group" (Dávila-Harris et al. 2013).

### iii. Basalts and trachybasalts

The historical quarries of "Piedra Molinera" have not been located exactly and although it is still marketed today, this stone material does not come from an active quarry but from occasional extractions taking advantage of the earthworks and clearings of large works. There is no doubt that the "Piedra Molinera" comes from basaltic lava flows (mainly of trachybasaltic composition), which are the most extensive lithological unit, so there can be outcrops of this stone in practically any area of the island.

Of the "Piedra Jabaluna", the only quarry of which we have reference is located on "aa" and "pahoehoe" type lava flows of porphyritic texture, fundamentally olivine-augitic or olivine.

### iv. Trachytes and phonolites

The "Piedra gris - dorada" extracted in the "Tigaiga" quarry belongs to trachyte - mafic phonolite flows of porphyritic facies. These basaltic flows are of low power with a surface area typical of "aa" basaltic flow. The stone extracted in the "Lomo Román" quarry corresponds to a phonolitic flow that appears sporadically interspersed in the basaltic flows and in the case of the "Buenavista" quarry, the stone is associated with the mafic phonolitic flows of the "Montaña de Taco" (0.7 Ma). They are generally quite massive rocks, although they are sometimes more porous, being similar in appearance to some trachybasalts.

The "Barranco de la Orchilla" and "Sixto" quarries, from which the "Piedra Azul" is extracted, are located on flows of trachyte-phonolitic composition. The lava flows that outcrop in the "Barranco de la Orchilla" correspond to a fairly extensive unit that occupies a large area of the slopes of Adeje and Vilaflor, in fact, this lithological unit is called "Vilaflor Phonolite's". In the "Sixto" quarry, the lava flows correspond to powerful phonolitic multi-lavic slabs that were emitted in the last phases of the "Edificio Cañada". The most notable difference between these two deposits arises at the mineralogical level. Most frequently, the stone that outcrops in "Barranco de la Orchilla" corresponds to a nepheline phonolite, while the outcrop where the "Sixto" quarry is located is composed of haüyne-phonolite.

## 3. Types of stone and their use in historical buildings

Depending on the characteristics of the volcanic stones in terms of hardness, density, colour, texture, etc., they were used to meet the aesthetic needs of their time. For example, if we look at the architectural elements that

present ornamentation in the historic centre of "La Orotava", we can see that a trachytic rock of a bluish grey colour, medium hardness and diffuse anisotropy predominates. It is a material that speaks to the cutting tool with good control over the material to be removed and responds to the different intensities of impact without conditioning. The shapes lend themselves to all types of finishes, both round and voluminous shapes and with wide or detailed enveloping shapes.

The anisotropy, being diffused and homogeneous, does not condition the way of directing. The cuts facilitate freedom of movement in the different tiling processes, although care must be taken with the finishing processes of the details.

The clearest example of the plastic possibilities of this type of stone can be seen in the ornamental repertoire, based on vegetal and animal motives, applied to the large corbel which gives the balcony a shape, located in the central part of the main doorway of the church of "Nuestra Señora de la Concepción" (Fig. 2), built in the 18th century by the master mason Patricio García (Tarquis, 1965).



Fig. 2 Ornamentation applied to the main doorway of the church of "Nuestra Señora de la Concepción" (La Orotava)

The quarries that supplied this type of stone material are located in the municipality of "Santa Úrsula", bordering the south-western slope of the "La Orotava" municipality. This type of quarry corresponds to very powerful flows, derived from the high pressure that unfolds and opens the surface of the dome, causing the material to slide slowly until it comes to a standstill. This stone material was used for the elaboration of the most outstanding architectural façades for the nobility of "La Orotava", being the means, to enhance their social power (Hernández, 2004).

Coarse-pored vacuolar basalt or "Piedra Molinera" was used for its characteristics of resistance to compression and abrasion for the manufacture of millstones and in construction, it was specifically used for buttresses, corners and foundations; although to a lesser extent we see it used to form prominent elements of a façade, such as the main doorway of a historical building. The main façade of the convent of "San Agustín" (La Orotava)

(Fig. 3) contains two doorways arranged at right angles, the entrance doorway to the convent made of millstone and the church doorway, the latter made of trachyte stone. Both façades were made in the 17th century by Juan González Agalet (Tarquis, 1965) and Lázaro de Miranda (Rodríguez, 2015), who were in charge of finishing the façade of the church of “San Agustín” as well as the doorway and bell tower of the convent. It is also known that part of the stonework was carried out by the official Diego Rodríguez (Hernández, 2004).

In this case, can the differences in colour, texture and even hardness between the two stones and their use be understood as an aesthetic resource. Since they were made at the same date and by the same craftsmen. Two architectural elements are shown, on the one hand the sober and rational image of the entrance doorway, marked by its compositional simplicity of broad, rounded forms, in contrast with the church doorway, which stands out for the beauty of its design and delicate ornamental decoration.



Fig. 3 Main façade of the convent of “San Agustín” (La Orotava)

The name millstone derives from the use that the aborigines gave to this lithic typology in the elaboration of mills and mortars to grind cereals.

Vacuolar basalt, compared to the tools of the time, lent itself, due to its hardness, to carving processes that resulted in broad and forceful shapes, in contrast to trachyte, which allowed all kinds of finishes.

In Tenerife this type of stone was quarried, according to the description of the stone used for the construction of the convent of “San Lorenzo” of “La Orotava”, Hernández (2004) commented: Another element used was the burnt stone of the “aa” lava of “Puerto de La Cruz”, with which they made the partitions of the dormitory and the new cells. We can deduce from this description that we are dealing with a vacuolar basalt. “Puerto de la Cruz” has been subjected to tourist construction overcrowding, which has limited the possibilities of exploiting this type of material.

Fig. 4 shows outcrops of vacuolar basalt which, when the time comes, could cover the need to replace losses in elements made of this type of stone.



Fig. 4 Vacuolar basalt outcrop (Buenavista del Norte)

Two types of stone are used in the Historic Centre of La Laguna. Most of the 16th century buildings were built with basaltic conglomerate extracted from the quarry on the “Nombre de Dios” (Fig. 5), in the village of “Tegueste”; it is a polychromatic stone reddened by the oxidation phenomena, its texture is irregular and the lithic fragments are welded together and have a good level of structural cohesion. It is a suitable material for carving, although in general it lends itself to large forms and a certain level of detail.



Fig. 5 Quarry on the “Nombre de Dios” (Tegueste)

Within the formal repertoire made in this type of stone, two façades stand out: the façade of the house of “Corregidor” (Fig. 6, left) from the mid-16th century and the façade of the “Alvarado Bracamonte’s” house (Fig. 6, right) from the 17th century. Due to its chromatic and textural characteristics, this type of stone has historically been given different names: “Encarnada”, “Roja” and “Bermeja” stonework. Martín (1978) commented on the façade of “Corregidor’s” house in his book “Arquitectura Doméstica de Canarias” as follows: The only thing that remains of the original building is its excellent façade of coloured stonework - the oldest example of Plateresque

architecture on the islands - and the best, together with the “Cabildo de Santa Cruz de La Palma”.

Almost all of the “Lagunera” doorways of the 17th and 18th centuries were made of trachybasalt from the “Pedro Álvarez” quarry, also in “Tegueste”. The material is bluish in colour, has uniform grain, is dense and compact, although relatively soft against the tool, lending itself to detailed forms.



Fig. 6 “Corregidor’s” house (left) and “Alvarado Bracamonte’s” house (right) (San Cristóbal de La Laguna)

There are several outstanding examples of façades made with this type of stone in “La Laguna”, such as the main façade of the “Salazar Palace” (Fig. 7), made entirely of stonework. It is known that its construction began around 1629, with the last building work in the middle of the 17th



Fig. 7 Façade of the “Salazar Palace” (San Cristóbal de La Laguna)

Another example that maintains formal and ornamental parallels with “Palacio Salazar” is undoubtedly the “Palacio Nava” (Fig. 8), whose main façade is also entirely covered in stonework. Its construction underwent various modifications between the end of the 16th century and the middle of the 17th century.



Fig. 8 Main façade of the “Palacio de Nava” (San Cristóbal de La Laguna)

We can see that the stone material used in the elaboration of both portals lends itself to detailed finishes as well as to blunt and rationed forms.

In the municipalities of “Buenavista del Norte” and “Garachico” there are two doorways made of dark grey trachyte stone: the entrance doorway to the Convent of “San Francisco” (Buenavista del Norte) (Fig. 9, left), founded in the first half of the 17th century, and the doorway of the main façade of the palace of the Marquises of “Adeje” and Counts of “La Gomera”, built in the second half of the 17th century and known as the “Casa de Piedra” (Fig. 9, right).



Fig. 9 Main façade of the “Casa de Piedra” (left), Main façade of the convent of “San Francisco”

The trachyte used in these two doorways is an excellent stone for carving, it lends itself to all kinds of finishes, its low hardness facilitates a smooth and soft cut that allows modeling without difficulty. In “Buenavista”, the best stone was found between the “Cejas” ravine and that of “Don Bartola” (Ruíz-Martín 2009) (Fig. 10)



Fig. 10 Quarry in “Buenavista del Norte”

The main doorway of the church of “San Antonio de Padua” (Fig. 11) in the municipality of “Granadilla de Abona”, dating from the early 17th century, is made entirely of phonolite stone, extracted from the “Sixto” quarry located in the same municipality (Fig. 12). This stone is of medium-hardness, which requires a slow and careful process to withstand the impacts of the tool, resulting in less control when it comes to fitting the shapes, but in terms of modeling it offers good qualities.



Fig. 11 Façade of the church of “San Antonio de Padua”



Fig. 12 Sixto Quarry (Granadilla de Abona)

#### 4. Deterioration

The aspects that influence the repertoire of lesions affecting the stone range from its own composition, the quarry, the construction systems, its environmental context and the evolution of the cultural property to which it belongs. In other words, intrinsic and extrinsic factors. Other elements that must be taken into account in the conservation of cultural property built in stone are the mechanisms of alteration, which can be chemical, physical or biological in nature (Gómez de Terreros and Alcalde, 2000; Alonso et al., 2006; Prado, 2019). The set of deterioration agents can trigger lesions of different nature. In this way, the main groups of damage are described from the point of view of the practice of conservation-restoration of cultural property (CNR-ICR, 1990; Garcia de Miguel, 2011; Laborde, 2013):

##### i. Lesions due to loss of matter

Erosion involves a reduction of the primitive surface, with a smoothing of forms due to chemical, physical and/or biological processes. Similarly, dissolution is mainly due to chemical processes that separate certain components without generally causing the surface to decohere. If the condition persists over time, selective dissolution phenomena may occur. In disintegration, there is an obvious detachment of particles from the substrate. Detachments involve a separation of material with various morphologies (blistering, exfoliation, etc.) and causes (soluble salts, defects in the stone, static problems, rusted iron elements, etc.). Finally, missing material is a localized and appreciable loss.

##### ii. Material-induced lesions

This group of lesions includes surface deposits (accumulations of foreign matter) and alteration of products (as a result of the interaction between extrinsic agents and the constituent material). In this sense, they cause both morphological changes and surface chromatic alteration.

##### iii. Superficial lesions of colour or gloss

In these pathologies, in which chromatic changes occur, no losses are perceptible and the possible contribution of matter is hardly noticeable. There is a change in the tone or intensity of the colour. On the other hand, discolouration is a loss of colour vibrancy and implies an increase in clarity. Further, stains are a circumstantial and localized variation of colour on the surface. They may be due to humidity, contact with metals, other organic substances, etc. Likewise, graffiti (with or without historical relevance) are chromatic alterations of anthropic origin.

iv. *Damage due to deformation and breakage of the material*

Stone materials can fracture without externalizing deformation, although, depending on their nature or geometry, they can develop a low degree of deformation. Thus, these deformations are associated with flat or linear constitutions and can be distinguished by buckling, warping and twisting. On the other hand, increasing stresses, whether natural or due to structural loads, can lead to cracking. The rupture is generated in the direction(s) of weakness of the material, and can be seen from discontinuities. Similarly, their orientation will depend on their textural components and/or structural stresses. Their size defines the terms fissure, fracture and crack.

## 5. Conclusion

The different types of stone used in the Artistic Heritage of the island of Tenerife are the result of responses in terms of their performance or complexities that may have been experienced in the working processes, a conditioning factor that has defined the choice of each type of stone for the execution of the carved elements.

The specific objective is the identification of the materials used in each heritage element, as well as the location of historical quarries or geological outcrops that show the viability of obtaining materials compatible with those used in the heritage assets in future restorations.

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**Acknowledgments:** The author J.A. Valido has carried out this study with the support of the Spanish Ministry of Universities, through a pre-doctoral contract (FPU16/05739).

## INNOVATIVE SOLUTIONS TO IMPROVE BUILDING STONE DURABILITY

L. Dias<sup>1,2\*</sup>, S. Martins<sup>1,3</sup>, H. Hashim<sup>1</sup>, A. Carrapiço<sup>1</sup>, F. Sitzia<sup>1,2</sup>, V. Pires<sup>1,2</sup>, M.R. Martins<sup>1,4</sup>, J. Mirão<sup>1,2</sup> and P. Barrulas<sup>1,3\*</sup>

<sup>1</sup>HERCULES Laboratory and IN2PAST, Institute for Research and Advanced Training (IIFA), University of Évora, Portugal

<sup>2</sup>Department of Geosciences, School of Science and Technology, University of Évora, Portugal

<sup>3</sup>Department of Chemistry and Biochemistry, School of Science and Technology, University of Évora, Portugal

<sup>4</sup>Health and Human Development School, University of Évora, Portugal

\*luisdias@uevora.pt, pbarrulas@uevora.pt

Portugal is rich in high-quality Natural Stone, whose reputation has placed it as a traditional producer and exporter of this material. Inherent to natural materials, stone is vulnerable to deterioration phenomena right after its extraction from the quarry, either through physical, chemical or biological agents. Additionally, during the last few years, the demand for synthetic construction materials has increased for their application in contemporaneous projects. Therefore, and to circumvent this trend, is of utmost importance to develop novel solutions to mitigate these deleterious effects on Building Stone and increase the lifetime of this construction material par excellence. Combining innovation with creativity, the HERCULES Laboratory and LITHOS (University of Évora) have sought to tackle actual concerns identified by the industry of the Natural Stone sector, whose procure to increase the certification of the materials they commercialize. The current solutions under development will mitigate the deteriorative effects caused by two key agents: water and biological colonization.

The preliminary results will be presented on:

- a) The design and synthesis of novel and greener compatible solutions with high hydrophobic properties;
- b) Biosynthesis of compounds with high antimicrobial potential.

The technology employed is based on the use of environmentally sustainable and cost-effective approaches, enabling the products' competitiveness regarding solutions currently existing on the market. The newly developed solutions will therefore contribute to the improvement of services provided by the industry of the Portuguese Natural Stone sector, outlining them from other players.

Keywords: Natural Stone; sustainability; hydrophobic properties; antimicrobial potential; "green" approaches.

Acknowledgements: This work has been financially supported by the Eco-STONEPROTEC – Eco-friendly superhydrophobic hybrid coatings for STONE PROTEction project – (EXPL/CTA-GEO/0609/2021), PhD research grant UI/BD/153583/2022, and by the UIDB/04449/2020 and UIDP/04449/2020 projects, which were funded by Fundação para a Ciência e Tecnologia (FCT) and by the European Regional Development Fund. The work is also supported by the project Sustainable Stone by Portugal - Valorization of Natural Stone for a digital, sustainable and qualified future, nº 40, proposal nº C644943391-00000051, co-financed by PRR - Recovery and Resilience Plan of the European Union (Next Generation EU).

# DEVELOPMENT OF A METHODOLOGY BASED ON NON-DESTRUCTIVE METHODS APPLIED TO THE CHARACTERIZATION AND RESEARCH OF HISTORICAL HERITAGE

M. Reyes<sup>1\*</sup>, A. Espín<sup>1</sup>, A. Gil<sup>1</sup>

(1) Technological Center for Marble, Stone and Materials, Ctra. of Murcia. Cehegín (Murcia) Spain. \*[monica.reyes@ctmarmol.es](mailto:monica.reyes@ctmarmol.es)

**Summary:** The PATRIGEO project tries to demonstrate that the combination of geophysical techniques can help to know in a very important way the archaeological sites and certain historical monuments, which need to be monitored to know their real state, on the surface and in depth. To test the degree of effectiveness of the proposed methodology in enhancing the knowledge provided versus the cost in resources (economic and material), it has been applied to different sites in the Region of Murcia. These sites are different from each other, which provides a great variability of characteristics to be studied with these techniques. The use of geophysical techniques such as ground-penetration radar 3D has made it possible to obtain geometries of buried structures in diverse archaeological sites such as the Roman villa of Los Cantos and Los Villaricos.

**Key words:** geophysical, archaeological sites, ground-penetration radar 3D, magnetometry, electrical tomography.

## Introduction

Lack of knowledge of non-destructive techniques and what they can contribute implies the use of techniques that can potentially cause damage to archaeological remains. The application of non-destructive evaluation techniques allows analysing the whole results (Boucher, 1996).

The techniques non-destructive have been applied to two archaeological sites in the Region of Murcia (Spain). In this research it's going to expose the most enlightening results.

The two locations were "Villa Romana de Los Villaricos" (Mula) and "Villa Romana de Los Cantos" (Bullas). For each case, different geophysical and topographic techniques have been used to analyze the archaeological remains.

## Objectives

The main objective of the PATRIGEO project is to serve as a guide of non-destructive evaluation techniques. This guide can be applicable in different situations and for all types of archaeological research.

The aim is to obtain a standardised tool that can be used for all types of archaeological sites using non-destructive methods.

In the project it's going to classify the most common materials (marble limestone, dolomite, travertine, sandstone and calcarenite), the different structures of the studied materials (anisotropy planes, stratification,

lamination, etc.), current status (buried, weathered, etc), it's different applications (chairs, base, shaft/pillar, cornice), sizes and geometries. And how these characteristics affect the results obtained after the application of the previously mentioned techniques will be evaluated.

## Study areas

The studied areas are in the Region of Murcia on the southeast of Spain (Fig.1). The first of this area is "Villa Romana de Los Villaricos", and the other area is "Villa Romana de Los Cantos".

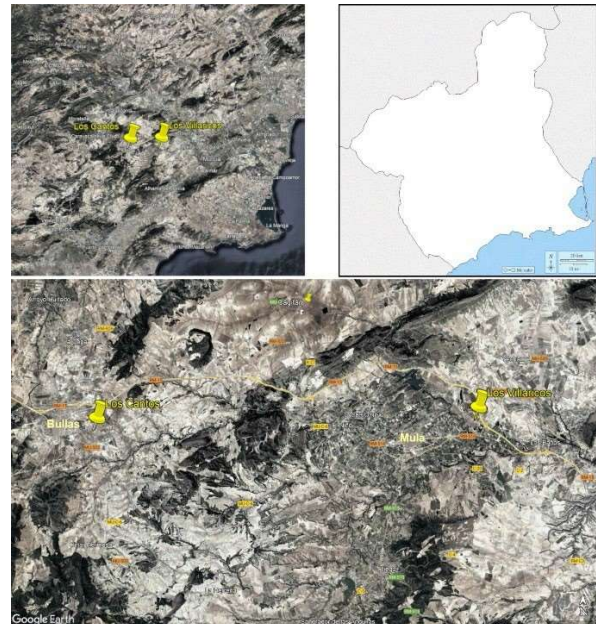


Fig. 1. Location map of the Roman villas.

- “Villa Romana de Los Villaricos” (Fig.2) is in the east of Mula. The excavation site has around 4,000 m<sup>2</sup> and sited on the upper part of the mountainside near to the Mula river. This is one of the most important villas in the Region of Murcia, which has been studied by many archaeological interventions by the University of Murcia since the 1980s. Is a substantial structure with evidence of several hundred years of occupation.

The villa is a single-family unit for agricultural exploitation. Is divided into the place of residence with a thermal area and the working area with a wine press and an olive mill. Once the villa was abandoned, it was used for religious purposes with a Basilica and a graveyard built by the first Christians (Galindo *et al*, 1991).

It is an extensive site, there are still large areas of the villa remaining unexcavated to date.

There are the remains of many Roman villas across the region of Murcia, the term “villa” being generally understood as a single-family unit for agricultural exploitation on a plot of land of varying size (Puche, 2017).



Fig. 2. General view of “Villa Romana de Los Villaricos”.

- “Villa Romana de Los Cantos” (Fig.3), is located in Bullas (Murcia). The archaeological area is on the top of a small hill occupying an area of approximately 10,000 m<sup>2</sup>, on the upper catchment area of Mula River. This Roman villa was discovered in 1867 as result of excavations carried out by Bernardino García, on land owned by the Marquise de las Almenas, wife of the Marquis of Corvera. (López, 1999) y (Ramallo, 2002).

That the Romans found agriculture a productive activity in the countryside around Bullas is evident from the fact that artifacts have been found here dating from as early as the second century BC and as late as the 5 th century AD. It can thus deduce that they stayed for around 700 years, and the complex includes not only a residential area, also a thermal bath area and the actual farm

installations where production was carried out (Porrúa, 2011). It can also be assumed that grape-growing and wine production were already an important part of the economic activities in Bullas during the period of Roman occupation.



Fig. 3. General view of “Villa Romana de Los Cantos”.

## Methodology

Specifically, for each archaeological location, it’s necessary provide information obtained by geophysical techniques for monitoring the subsoil for determining the existence of different constructive elements and buildings. The following techniques that are going to use are magnetometry, ground-penetration radar 3D (GPR3D), electric tomography and spectroradiometer, also is obtained an accurate topographic model and orthophotomaps (Fig. 4 y 5) of the surface of each area using drone model Trimble UX5 HP drone. The model of orthophotomaps is a tool for planning geophysical techniques.

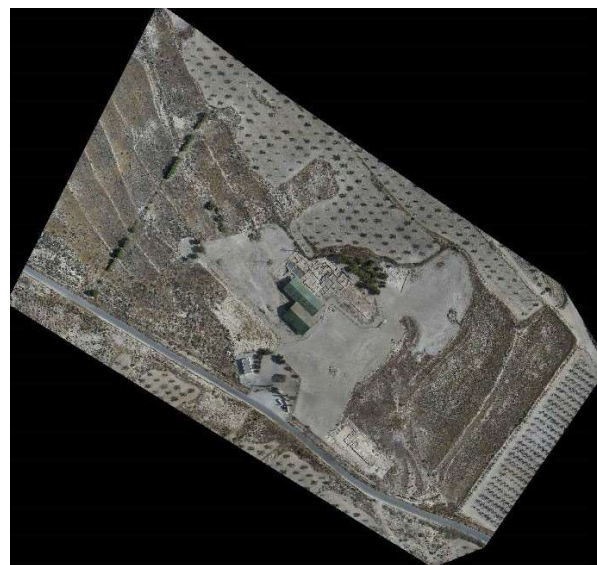


Fig. 4..Orthophotomaps of “Villa Romana de Los Villaricos”.





Fig. 5. Orthophotomaps of "Villa Romana de Los Cantos".

### - Magnetometry

A magnetometer measures magnetic anomalies on the earth's surface, which could be the product of a reservoir.

In this project has been used the portable proton magnetometer: Geometrics, model G-857 with gradiometer model G-857AX (Fig.6). The data was processed with the MAGMap 2000 software.

Different tracks have been made, where at each point (station) a value is measured with the upper detector and another measurement with the gradiometer that serves to correct the drift of the magnetic field during the measuring.

Each of these measurements has been georeferenced using the Trimble GPS centimetric equipment to the UTM ETRS89 system.



Fig. 6. Data collection with the magnetometer in Los Cantos.

### - Ground-penetration radar 3D

Ground-penetration radar (GPR3D) is a non-invasive electromagnetic method, uses radar pulses to image the

subsurface. It's fully used by his speed in data collection and the degree of detail, obtained from the terrain analysis. This non-destructive method uses electromagnetic radiation in the microwave band of the radio spectrum and detect the reflected signals from subsurface structures (Economou *et al.* 2015).

3D equipment uses multiple antennas in the same device, allowing to cover larger areas in less time and facilitating the interpretation of recorded anomalies as it logs in all directions.

For the acquisition of GPR profiles, a model DX1820 antenna from the Norwegian manufacturer 3D-Radar has been used mounted on a vehicle (Fig.7). The antenna has 20 sensors spaced every 7.5 cm, so that in each pass, it does a sweep that covers a width of 142.5 cm.

The 3D-GPR system emits a step-frequency continuous-wave (SFCW) waveform, which is a sinewave with constant amplitude and stepwise frequency variation. The waveform is specified by determining a start frequency, a stop frequency, a frequency step ( $\Delta f$ ), and a dwell time. The time-domain signal is then calculated from frequency information using the inverse Fourier-transformation. The wider bandwidth of this system ensures a better resolution at most depth levels.



Fig. 7. Data collection with multi-frequency 3D GPR.

For the GPR sweeps, the antennas are carried by a vehicle to make easier the data collection, and for non-accessible areas the antennas are being moved manually (Fig.8).



Fig. 8. Working with the three-dimensional GPR antenna moved manually.

In the studied areas, this technique is used for detecting walls and other architectural elements in depth, to guide the following archaeological excavations. Figures 9 and 10 shows the cover area made with the GPR in Los Villaricos and Los Cantos.



Fig. 9. Profile coverage in Los Villaricos by means of the multi-frequency 3D antenna (green lines).



Fig. 10. Profile coverage in Los Cantos by means of the multi-frequency 3D antenna (green lines) and location of the electric tomography meshes (C1 to C5 sites).

Villa Romana de Los Cantos, as in Los Villaricos, the maximum possible coverage is achieved with the multi-frequency 3D antenna.

It is important to point that the GPR results are worthless if a bad positioning system is used. Our system uses a triple positioning composed by an odometer, three GPS receivers (one internal inside the GPR antenna for accurate time synchronization plus two external ones, -SBAS aided-) and an IMU to provide coverage in case of GPS signal loss. The combination of all three systems yields excellent positioning accuracy to our GPR images.

#### - Spectroradiometer

The spectroradiometer is used for the measurement of the spectral radiance of the covers, which transform into

#### - High-definition electrical tomography 2D and 3D

It is a geophysical technique whose specific objective is to determine the real distribution of the electrical resistivity of the subsoil. This is measured from a certain range of depth along a profile (Fig. 11), by means of measurements carried out by conventional direct current methods apparent values of resistivity are obtained. The technique provides the resistivity changes present in the ground between the archaeological materials and the rocks present in the substrate.



Fig. 5. Data collection with 3D electrical tomography.

In "Villa Romana de Los Villaricos", eight 3D tomography are performed (Fig. 12), in places where other techniques previously indicate anomalies. A total of 144 electrodes are arranged, it reach depths of 3.4 m and the total investigated area are 120 m<sup>2</sup>.



Fig. 6. Location of electrical tomography meshes in Los Villaricos.

Considering the anomalies found by other techniques, in "Villa Romana de Los Cantos", five locations (Fig. 10) throughout the site have been selected for 3D tomography meshes (C1-C5). The dimensions of the 3D mesh have been for 144 electrodes of 15x16 m with a total investigated area of 240 m<sup>2</sup> and a maximum reach depth of 3 meters. (Brito-Schimmel, 2005).

reflectance measures to characterize the type of material.

Various measures have been carried out in the areas of the archaeological sites of Los Cantos and Villaricos and have been selected field samples with the aim of compare the

obtained spectral measures with that samples. This samples will be analysed with the Spectral Electronic Microscope (SEM).

It has been carried out with the FieldSpec4 spectroradiometer, manufactured by Analytical Spectral Devices, Inc. (Boulder, CO, USA), owned by the University of León.

The spectral measurements are sampled through a contact probe with its own light source. The reflectance is calibrated using a white panel (spectral on diffuse reflectance panel), both in the field and in the laboratory.

In the “Villa Romana de Los Villaricos”, have been made a total of 66 measurements in the different materials for their characterization. And in “Villa Romana Los Cantos”, were made a total of 31 measurements in the different materials also for their characterization.

### Results and discussion

The main results obtained during the research for each technique are described below:

#### - Magnetometry

Magnetometry has been used in the “Villa Romana de Los Cantos”, covering almost the entire site. The obtained data from this place is currently being analysed.

The magnetometry technique has limitations, especially when there are coverages with metallic content, it may cause interferences and provide erroneous data. Therefore, has not been possible to perform it in the “Villa Romana de Los Villaricos”.

#### - Ground-penetration radar 3D

Important anomalies can be detected in the obtained and interpreted radargrams, these anomalies define the presence of buildings, walls, and isolated remains.

The radargram of figure 13, shows different anomalies at the depth of 30 cm (red marked area), that could be

related to the presence of constructions, belonging to building walls.

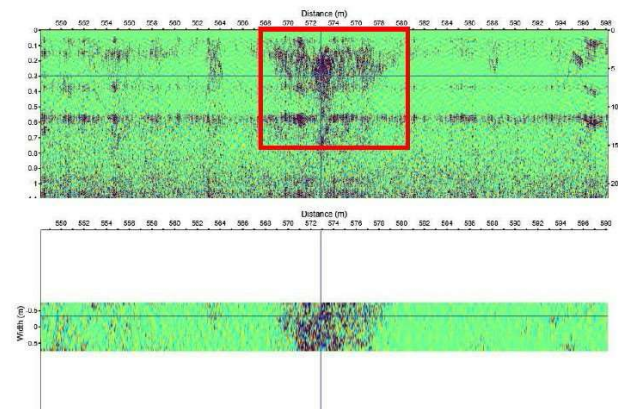


Fig. 7. Radargram in Los Villaricos (vertical and horizontal section) with the presence of reflections.

In the figure 14 of Los Cantos archaeological site, also show anomalies corresponding to possible structures as could be walls of the buildings (Scollar, 1962). The red marked area, at 30 cm of depth also indicate the presence of important structures.

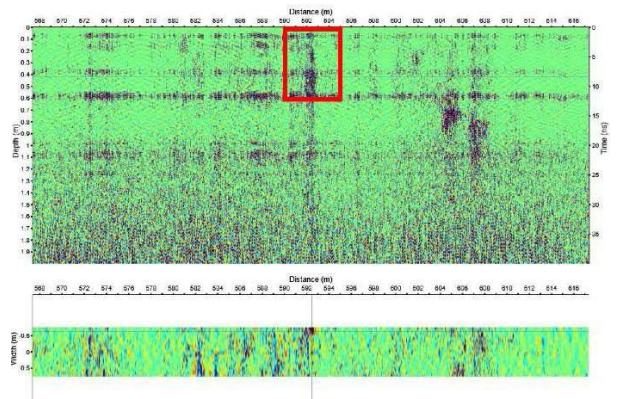


Fig. 8. Radargram in Los Cantos (vertical and horizontal section) with the presence of reflections.

With the results that will be obtained, could serves to design future accurate campaigns of techniques that are more expensive and slower to carry out.

#### - High-definition electrical tomography 2D and 3D

“Villa Romana de Los Villaricos” (profile V3):

In the profiles once processed, the existence of an area with abundant anomalies of high resistivity up to 1.5 m deep is observed, but it is from the surface to 1 m where can be shown geometries corresponding to building walls.

In the figure 15, horizontal cuts made to the 3D model can be observed and in this model at 0.45 m resistive anomalies are developed around electrode 1 (left-down corner), possibly being the beginning of the walls of the building.

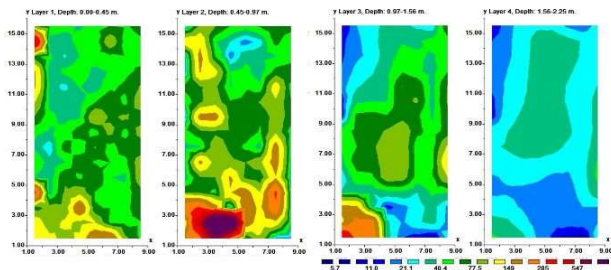


Fig. 9. Electrical tomography (V3) with horizontal sections X-Y in Los Villaricos at different deeps.

In the transversal and longitudinal sections (Fig. 16), it can be seen how the depth where the archaeological remains are located barely exceeds one meter deep. (Watters, 2004).

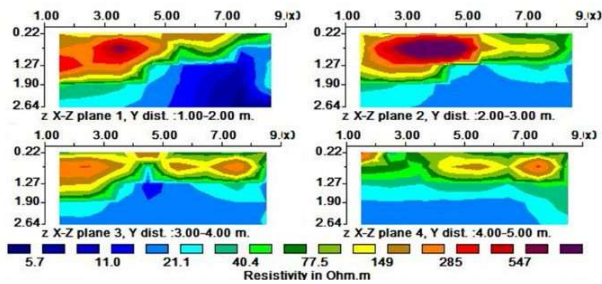


Fig. 10. Electrical tomography (V3) with transversal sections X-Z in Los Villaricos at different deeps.

“Villa Romana de Los Cantos” (profile C2):

The C2 profile (Fig. 17) shows the existence of an area with abundant high resistivity anomalies up to 2 m deep, but it's from the surface to 1.5 m where the most interesting geometries corresponding to building walls can be seen.

In the image obtained at 0.5 m, a resistive abnormality develops around electrode 1 (left-down corner) that may correspond to the presence of a new basin like the one found to the NW. This anomaly is very striking due to its very marked rectangular shape.

In addition, there are more anomalies to the S that may correspond to walls and buildings. Below 2.5 m, the existence of structures is not appreciated, even detecting the rocky substrate.

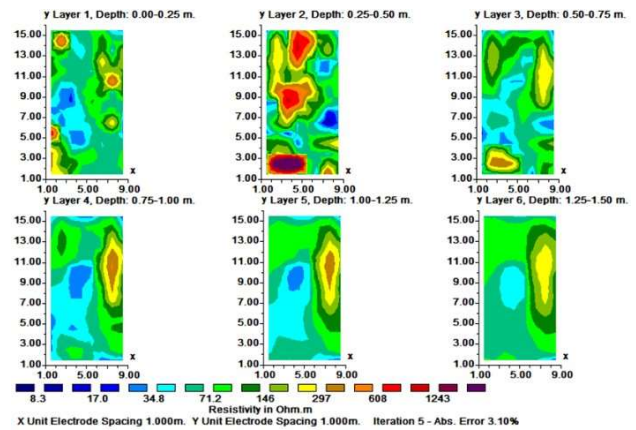


Fig. 11. Electrical tomography (C2) with horizontal sections X-Y in Los Cantos at different deeps.

In all the transversal profiles studied (Fig. 18), different high resistivity anomalies are located, most of them correspond to the stone walls and ashlar.

Also, low resistivity anomalies have been detected, that will be necessary to investigate to see its archaeological content, some of that low resistivity anomalies may correspond to graves. (Noel, et al, 1991).

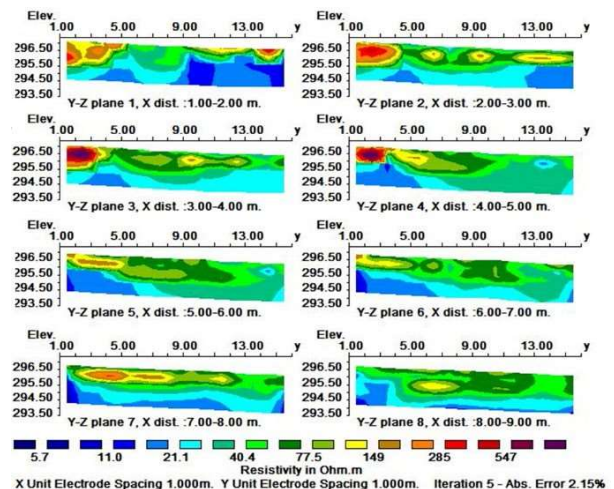


Fig. 12. Electrical tomography (C2) with transversal sections X-Z in Los Cantos at different deeps.

### - Spectroradiometer

The figure 19 and 20 shows the graphs with the representative spectral curves, taken from several samples of the field campaign carried out.

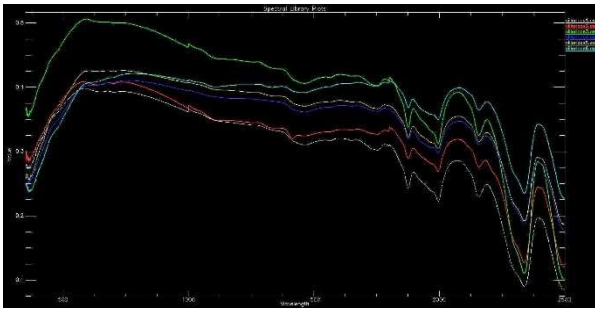


Fig. 19. Representative spectral curves corresponding to the marble source of the Los Villaricos samples.

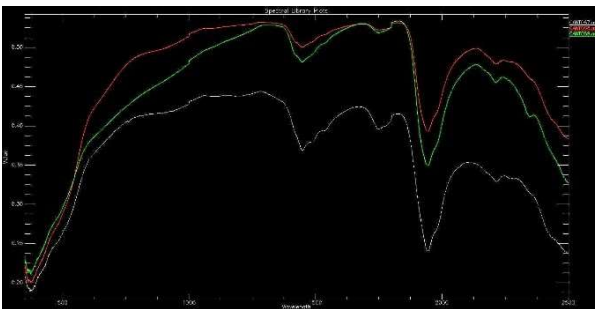


Fig. 20. Representative spectral curves corresponding to the Los Cantos samples.

In the absence of results about the mineralogical composition made by X-ray diffraction and SEM, only is shown the spectral curves taken in different places of each archaeological sites.

## Conclusions

It is revealed that the combination of drones, 3D scanners and geophysical techniques is an exceptional methodology for the study of archaeological sites, mainly where it is necessary to obtain information about their state quickly and without affecting the monument.

Through the combined use of drones and 3D scanners, it's possible generate a three-dimensional model of the surface of the studied areas that improves the knowledge of the archaeological deposits, as it serves as a basis of the planning of the subsequence's methodologies.

GPR3D has proven to be a fundamental tool in the knowledge of the subsoil at Los Cantos and Villaricos archaeological sites. The use of multifrequency antennas on GPR, delimit the detected structures in three dimensions, which was useful to identify the type of structure.

**Acknowledgments:** This project has been possible thanks to the collaboration and equipment used by the company Everest Geophysics S.L., External Geodynamics Area of the León University and INFO PATRIGEO project 2019.08. CT01.0031.

The GPR3D has also helps to design more precisely the location of the different electrical tomography profiles, so it has been possible to adequately optimize the resources.

The electrical tomography applied has allowed to identify the different geometries of the buildings and present elements. It has also been possible to verify its versatility when it comes to reaching different research surfaces and depths adapting to the studied area. Due to the presence of more potent fillers, as has happened in Los Cantos, surged the need to deep and increase the research surface.

The use of the 3D tomography mesh allows the identification, sizing and observe the geometry of the structures, that will guide future archaeologists research campaigns.

With the spectroradiometer, it's possible to obtain a mineralogical composition of the monitored materials without affecting the monument.

In conclusion, the combination of non-destructive techniques in archaeological sites is essential for improve it knowledge. The proposed methodology helps to improve both the historical-archaeological knowledge and the restoration-rehabilitation project.

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# INVESTIGATION OF CONSOLIDATION OF LIMESTONES USED IN HISTORICAL BUILDINGS IN İSTANBUL BY *MYXOCOCCUS XANTHUS* BACTERIAL STRAIN

Ö. Boso Hanyali<sup>1\*</sup>, A. Tuğrul<sup>2</sup>, g. Altuğ<sup>3</sup>, s. P. Çiftçi Türetken<sup>3</sup>, S. Yılmaz Şahin<sup>2</sup>, M. Yılmaz<sup>2</sup>

- (1) Mimar Sinan Fine Arts University, School of Conservation and Restoration Cultural Heritage\* [ozge.boso@msgsu.edu.tr](mailto:ozge.boso@msgsu.edu.tr)  
(2) Istanbul University-Cerrahpasa, Department of Geological Engineering, [tugrul@iuc.edu.tr](mailto:tugrul@iuc.edu.tr) , [sabahys@istanbul.edu.tr](mailto:sabahys@istanbul.edu.tr)  
[yilmazm@iuc.edu.tr](mailto:yilmazm@iuc.edu.tr)  
(3) Istanbul University, Department of Marine Biology, [galtug@istanbul.edu.tr](mailto:galtug@istanbul.edu.tr) , [pciftci@istanbul.edu.tr](mailto:pciftci@istanbul.edu.tr)

## Abstract

Many historical structures and items in cultural heritage are built of natural stone, and as time passes, they are exposed to the dangers of weathering owing to the growth of micro cracks in the stones. Carbonated materials, such as limestone, are more easily influenced than other natural stones due to their porous nature. This study aims to consolidate the limestone samples, which have different properties, by using the carbonate precipitation feature naturally performed by microorganisms. As a result of Scanning Electron Microscopy (SEM) analysis, it was determined that calcium carbonate precipitation occurred in all stones. Bacterial cells completely covered the pore walls of the stones at the end of the 27th day. Bacterial cells are attached to the calcite grains without blocking the pores. The results showed that the *M. xanthus* bacterial strain can potentially to consolidate on selected limestones.

**Key Words:** *Bacterial biomineralization, Stone conservation, Myxococcus xanthus, Limestone*

## 1. Introduction

Limestones have been used extensively in Istanbul's historic structures and artifacts throughout time. Air pollution, temperature fluctuations, freezing-thawing, and crystallization-dissolution of water-soluble salts (sulfate, nitrate, etc.) cause the limestones to deteriorate with time, destroying buildings and artifacts. If no precautions are taken, the historical buildings or artifacts will be at risk of deterioration.

The extensive use of limestone in our country's ancient structures and buildings, notably in Istanbul, consolidating the stones is crucial. For this reason, it is vital to improve the weather resistance and durability of these stones. Using the carbonate precipitation characteristic that it naturally possesses, bio-consolidation is accomplished using microbial consolidation. As a result of the interaction between the bacteria and the stone, it is anticipated that the new cement product, which is formed by the natural calcium carbonate precipitation induced by bacteria, will settle in the stone's microcracks and strengthen the stone's internal structure.

Microbial calcium carbonate (CaCO<sub>3</sub>) formation is common in soil, water, and marine sediments. One of the microbial precipitation formation mechanisms is ammonification. Ammonification is the phenomenon of the emergence of NH<sub>3</sub> from the breakdown of proteins in various organic wastes, dead plants, and animal remains. In this, NH<sub>3</sub> is

made, raising the environment's pH. At the same time, CO<sub>3</sub> (carbonate) ions and Ca<sup>2+</sup> ions join together and form crystals. *M. xanthus*, which provides crystal formation with this mechanism, breaks down amino acids in the environment and constitutes NH<sub>3</sub> and CO<sub>3</sub> ions. *M. xanthus* bacteria penetrated deeper and carried the newly produced calcium carbonate to the pore system, healing the stones and strengthening them for a long time. If many bacteria are in an environment with calcium and urea, they carry out significant calcium carbonate (CaCO<sub>3</sub>: calcite, aragonite, vaterite) mineralization (Spanos ve Koutsoukos. 1998; Al-Thawadi, 2001; Pinar, 2010; Yildirim et al. 2016). The metabolic process of the *M. xanthus* bacterial strain involves the oxidative deamination of amino acids, which is the process of taking amino acids out of a molecule. This process makes NH<sub>3</sub>. NH<sub>3</sub> creates an alkaline environment according to the following reaction.



With this reaction, while the pH is 8 at the beginning, it can go up to 8.5 and 8.7. (Rodriguez-Navarro ve et al. 2003).

Bacteria are the sources of CO<sub>2</sub>. CO<sub>2</sub> dissolves, and both HCO<sub>3</sub><sup>-</sup> and CO<sub>3</sub><sup>2-</sup> transform at high pH. Carbonate ions are provided by K<sub>2</sub>CO<sub>3</sub> in the culture medium. After sufficient saturation heterogeneous nucleation occurs in bacterial cell walls, and calcium carbonate formation occurs in calcite crystals. Calcium carbonate precipitation by bacteria is

known as “induced” because the produced calcite is highly dependent on environmental conditions.

The study aims to investigate how different types of limestone, which are often used in old buildings and artifacts in Istanbul, become solid. This will be done by looking at how microorganisms naturally precipitate carbonate.

## 2. Materials and Methods

The bacterial strain used in the study was determined to be *M. xanthus*, which has the highest calcium carbonate production ability and has been found to give the most effective results in the consolidation of limestones. *M. xanthus* is a Gram-negative, non-pathogenic, common soil bacterium belonging to the decomposition of Proteobacteria. (Jimenez Lopez et al. 2008). The *M. xanthus* bacterial strain ATCC 25232 (American Type Culture Collection) used in the study was commercially available.

The samples used in the study were obtained from limestones with different characteristics in dimension stone quarries located in Süloğlu (Edirne) and Sazlıbosna (Hadımköy). The other group of limestone samples used in Karadeniz Madrasa was obtained from the stones released during the restoration process of the building. samples taken from Karadeniz Madrasa were used as KM-coded Küfeki limestone samples; from Hadımköy were used as H-coded Sazlıbosna limestone samples; and samples from Edirne Süloğlu were used as E-coded Süloğlu limestone samples.

With the study, the characteristic features of three different limestone samples were determined first. In this context, mineralogical, petrographic analyses were carried out on the samples. The mineralogical composition and textural properties of the stones were determined. A mercury porosimetry test was carried out within the scope of physical analysis on samples. Within the scope of further analysis, the morphological features of the stones were determined by performing SEM analysis on the stones. The limestone samples were incubated for 27 days in a medium containing the *M. xanthus* bacterial strain at a density of 0.5 MacFarland ( $1 \times 10^8$  cfu/ml). The stones were immersed in the culture medium prepared with the *M. xanthus* bacteria strain without any mechanical mixing, to be incubated for 27 days. The incubation period of 27 days selected in bacteriological analyses was determined according to literature research. At this stage 18 limestone samples were prepared for each of the three stone groups, with dimensions of 2\*1\*1 cm. Half of these samples (9 pieces) were sterilized,

and the other half were used in experimental studies without sterilization. A sterilization process was carried out to remove fungi and microorganisms in the stones. Sterilization was carried out at 1 atm pressure and 121°C for 15 minutes. One sample from each group of stones was separated as a control. Limestone samples were removed from sterile flasks that had been dipped on the 27th day. A mercury porosimetry test and SEM analyses were performed on stones incubated for 27 days in a culture medium containing a bacterial strain. The resulting values were compared to the control group's, and any alterations in the stones were determined.

## 3. Analysis

### 3.1. Bacteriological Analyzes

#### Preparation of Media

Solid (nutrient agar) and liquid (nutrient broth) media that fed the bacterial strains were prepared. For the (nutrient broth) medium, 1.6 g of nutrient broth was added to 200 ml of distilled water, and then the medium was homogenized by mixing with a magnetic stirrer. After preparation, it was distributed into glass closed tubes with a volume of 9 ml and sterilized in an autoclave.

For solid media, 5 g of nutrient agar was added to 250 ml of distilled water and mixed with a magnetic stirrer. After being sterilized in an autoclave, nutrient agar was poured into petri dishes under aseptic conditions and left at room temperature until became solidified. The solidified media were stored at +4 °C for later use.

#### Preparation of Bacterial Culture

Nutrient agar, which was kept at +4 °C until the *M. xanthus* bacterial strain was prepared, was used after keeping it at room temperature. Since this commercially purchased bacterial strain is in lyophilized form, it was first enriched by inoculation in a liquid medium and then inoculated into a solid medium by the spread plate method under aseptic conditions. It was then incubated at 37 °C for 24 hours.

Bacterial suspensions of 0.5 McFarland (1 liter,  $1 \times 10^8$  cfu/ml) density were obtained from 24-hour *M. xanthus* bacterial strain cultures. Three groups of limestone samples (coded as KM, E, and H) in bacterial solutions were placed in 250 ml sterile flasks. The prepared flasks were placed in an oven with room agitation at 100 rpm (rotation speed per minute) at 25 °C.



### 3.2. Experiments and Analyzes on Limestone Samples

The experiments and analyses on the limestone samples used in the study (Table I) and their results are explained in detail. In order to make sure the experimental study is clear, the samples used were tagged according to the regions from where they were collected.

Sample Code	Explanation
KM-St	Sterile Küfeki Limestone Sample
H-St	Sterile Sazlıbosna Limestone Sample
E-St	Sterile Süloğlu Limestone Sample
KM-NSt	Non-sterile Küfeki Limestone Sample
H-NSt	Non-sterile Sazlıbosna Limestone Sample
E-NSt	Non-sterile Süloğlu Limestone Sample

#### Mineralogical and Petrographic Analyses

The mineralogical composition, microcracks, and textural properties of the limestone samples used in the study were determined by using a polarizing microscope. Petrographic analyses are based on the TS EN 12407/2013 standard. The polarizing microscope used in the studies is a Nikon brand 50iPol model.

According to the petrographic analysis, as a mineralogical composition, The Küfeki sample consists of mostly carbonate minerals (calcite and dolomite) and a small amount of quartz and clayey levels. It is a limestone, generally in sparitic calcite composition, in crystalline structure, with occasional cavities and named as "Biosparite" according to Folk (1964). Parts containing micritic texture in places consist of clay-sized calcite mud. Sazlıbosna sample is a limestone that contains some quartz grains and defined as "Intrabiosparite" in the crystalline structure. There is very little clay in the micritic part. It contains coral and nummulite fossils. Süloğlu sample is a limestone defined as "lithosparite" with a crystalline structure containing traces of quartz and micritic shells at a very a meager rate and containing quartz in places and minimal detrital material. Sterile limestone samples are more porous than non-sterilized stones, presenting a more porous structure due to partial melting-dissolution of limestone (Figure 1).

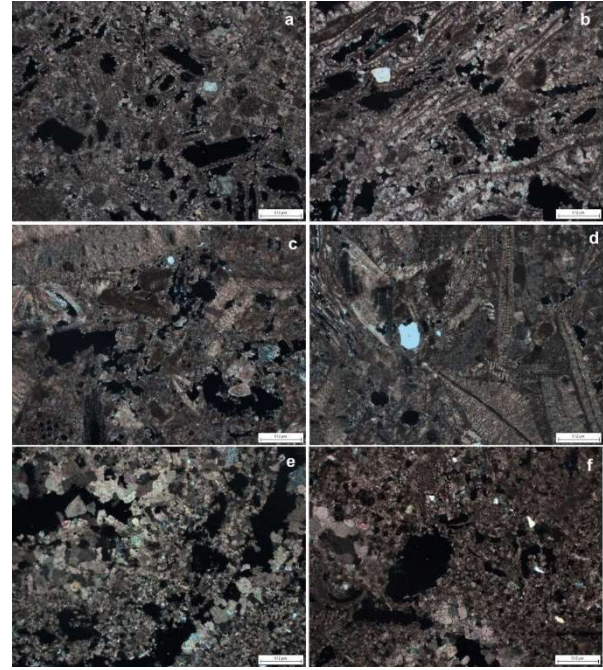
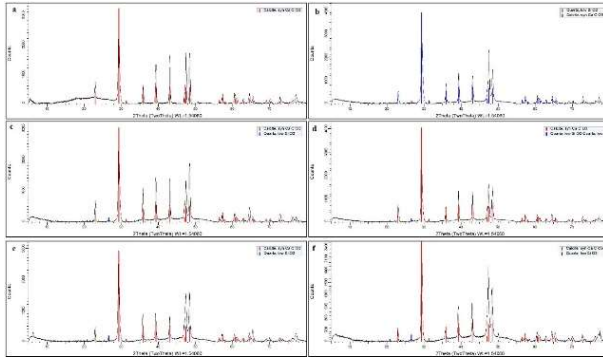


Figure 1. All of the limestone samples present more or less porous structure under the microscope (cross polar, scale, 4x10). a: Sterile Küfeki b: Non-sterile Küfeki, c: Sterile Sazlıbosna d: Non-sterile Sazlıbosna e: Sterile Süloğlu f: Non-sterile Süloğlu.

#### X-Ray Diffractometry Analysis

The mineralogical properties of the limestone samples were determined by the X-ray diffractometry (XRD). X-ray diffraction analyses of the samples were performed by Bruker D8 Discover XRD, using Ni-filtered CuK $\alpha$  radiation (40 kV, 30 mA) with a 2-dimensional Vantec 500 detector. All the measurements were carried out in the range 3° b 2 $\theta$  b 80°, with 10 increments and a step size of 0.02° in the step scan mode. X-ray diffraction Diffractometry (XRD) analysis was conducted at Mimar Sinan Fine Arts University, Cultural Heritage and Art Works Material Application and Research Center. As a result of XRD analysis on sterilized and unsterilized limestone samples, although no difference was observed in the mineralogical composition, the dominant mineral phase was determined as "Calcite". Apart from the sterilized limestone, a small amount of quartz mineral was also detected in other limestone samples (Figure 2).



**Figure 2.** Mineralogical compositions of the stones used in the study in X-ray Diffractometers;

a: Sterile Kufeki b: Non-sterile Kufeki , c: Sterile Sazlıbosna d: Non-sterile Sazlıbosna e: Sterile Süloğlu f: Non-sterile Süloğlu.

### Mercury Porosimetry Analysis

The effective porosity values of the limestone samples were determined with a mercury porosimetry device. The mercury porosimetry analysis was carried out based on the TS 699/2009 standard. Measurements were taken with a Quantachrome brand, Poremaster 60 model device at Yıldız Technical University, Central Research Laboratory. The mercury porosimetry results are given in Table II.

**Table II.** Effective porosity values obtained before and after the application of the microbial method.

Sample Code	Effective Porosity Value %		
	Before microbial method	After microbial method	Percentage Change %
KM-St	3,34	1,81	-45,81
H-St	5	2,06	-58,80
E-St	3,64	0,63	-82,69
KM-NSt	2,37	2,45	3,38
H-NSt	6,13	6,25	1,96
E-NSt	3,28	1,82	-44,51

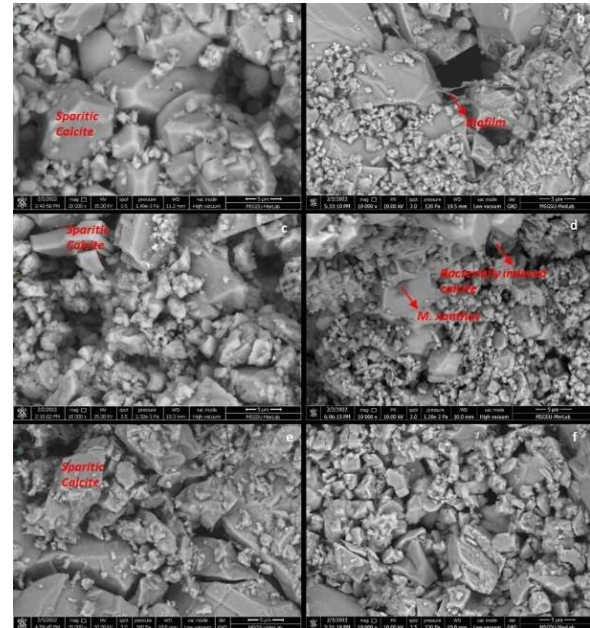
According to the mercury porosimetry experiments results, there was a percent decrease of 45-82% in the effective porosity values of the sterile limestone samples. In non-sterile stones, a percentage reduction of 44.51% was achieved only for the Süloğlu limestone (Table II).

### Scanning Electron Microscopy (SEM) Analysis

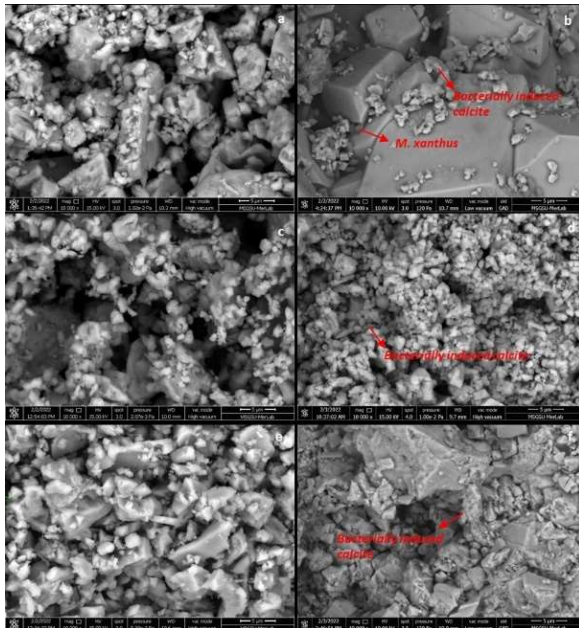
It is aimed to reveal all the changes that have occurred on the limestone samples by determining the pore structure, surface morphological properties and crystal structure of the limestone samples before and after applying the microbial consolidation method with SEM analysis. Especially

after the microbial method was used, it was possible to figure out how the stones' surfaces and void structures might have changed. Analyses were performed with the FEI Quanta 650 FEG SEM microscope. SEM analyzes were carried out at Mimar Sinan Fine Arts University, Cultural Heritage and Art Works Material Application and Research Center.

Calcite crystals of sterilized and unsterilized limestone samples were clearly observed. Sparitic and partly micrite parts were observed in the Kufeki and Sazlıbosna limestone samples. (Figure 3 and 4a,c,e). Calcite forms developed by bacterial strains are evident in stones inoculated with the *M. xanthus* bacterial strain (Figure 3 and 4d). *M. xanthus* bacterial strain settled in the stone's pore and deposited calcium carbonate in the cavities. After the bacteria application, some formations that indicate the onset of biofilm formations are observed in SEM images of sterilized limestone sample. (Figure 3b). Particularly within the scope of the formation mechanisms of biofilm forms, filaments that allow bacterial strains to adhere are recorded. Exopolysaccharide (EPS) formation was observed in unsterilized stones.



**Figure 3.** Scanning electron microscope (SEM) images of sterilized limestone samples (10.000 x magnification) a: Calcite crystals in Kufeki sample before microbial method b: The formation of biofilms on the Kufeki sample after the microbial method, c: Calcite crystals in Sazlıbosna sample before microbial method d: The appearance of *M. xanthus* and bacterial calcite crystals in Sazlıbosna sample after microbial method e: Calcite crystals in Süloğlu sample before microbial method f: The appearance of new calcite formations in Süloğlu I sample after microbial method.



**Figure 4:** Scanning electron microscope (SEM) images of non-sterilized limestone samples (*10.000 x magnification*)*a:* Calcite crystals in Küfeki sample before microbial method *b:* Bacterial calcite crystals in Küfeki sample after the microbial method, *c:* Calcite crystals in Sazlıbosna sample before microbial method *d:* Bacterial calcite crystals in Sazlıbosna sample after microbial method *e:* Calcite crystals in Süloğlu sample before microbial method *f:* Bacterial calcite crystals in Süloğlu sample after microbial method .

#### 4. Discussion and Conclusions

On the limestone samples where the microbial method is applied, a pore reduction of up to 80% has occurred, especially on the sterilized samples. This means that the pores of the sample are closed. Although the best results are obtained for sterile stones, it can be said that the *M. xanthus* bacterial strain alone is more successful in providing calcium carbonate precipitation.

As a result of SEM analysis, bacterial cells covered the pore walls of the limestone samples at the end of the 27th day. In stone samples immersed in the culture medium for 27 days, abundant calcined bacterial cells developed with the newly formed carbonate aggregates. Bacterial cells are attached to the calcite grains without blocking the pores. SEM analyses showed that the content of newly formed crystals is mainly  $\text{CaCO}_3$ . Rodriguez-Navarro et al. (2003) stated in their study that as the bacteria bind to the carbonate grains of the stone, the newly formed carbonate forms cement that bonds firmly with the substrate. According to Mc Bridge et al. (1993), the *M. xanthus* bacterial strain showed a very high biomineralization ability and generated a novel cement suitable with the substrate. Rodriguez-Navarro et al. (2003) stated that the *M. xanthus* bacterial strain can colonize not only on the stone surfaces but also more profound into the

pores of the stone, allowing the newly formed cement to root into the pores of the stone and increase the consolidation effect of the stone. Bacterial transfer occurs in the pores. Pores are about twice the size of bacteria. Bacterial transport in the pore of the stone depends on the pore structure of the stone, mineral composition, and bacterial adsorption (De Muynck et al. 2011). In the study, the ability of the *M. xanthus* bacterial strain to precipitate calcium carbonate on sterilized and unsterilized limestone samples was determined by SEM analysis.

Bacterial biofilms are formed when bacteria are irreversibly attached to a surface by producing extracellular polymeric substances (EPS). The short and flat fimbriae allow bacteria to attach to various surfaces and twitch. Biofilms bind ions in solution and act as heterogeneous nucleation surfaces for mineral deposition. It has been suggested that since biofilms retain moisture and provide nutrients, they are the most suitable medium for further bacterial colonization (Rodriguez-Navarro et al. 2003). The study observed extracellular polymeric substances (EPS) formation and fimbriae for bacterial biofilm formation in non-sterilized limestone samples.

This study provided data regarding the potential of the *M. xanthus* bacteria strain for stone consolidation. In addition, it was determined that the microbial method applied in the trials in which the microbial communities in the stone were not eliminated, increased the durability of the stones, indicating that the *M. xanthus* bacterial strain did not form an antagonistic relationship with other microorganisms in the stone that would negatively affect the increase in durability. However, further studies are needed to investigate the antagonistic and synergistic interactions between the bacterial strain used in trials for durability-enhancing microbial methods and other microorganisms naturally found in stones. Therefore, future studies should be conducted using components or variables of the *M. xanthus* bacterial strain to increase the durability of stones.

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# APPLICATION OF RESINOUS BINDERS WITH INCORPORATION OF CARBONATED SLUDGES from the dimension stone industry in the production of stone composites

P.A. Afonso<sup>1\*</sup>, A.S. Azzalini<sup>1</sup>, L. Lopes<sup>1,2</sup>, P.A. Faria<sup>1,3</sup>, P.A. Mourão<sup>1,4</sup>, R.V. Martins<sup>1</sup>, V.C. Pires<sup>5</sup>

- (1) Departamento de Geociências, Universidade de Évora, Évora, Portugal, \*pafonso@uevora.pt
- (2) Instituto Ciências da Terra, Polo de Évora
- (3) GeoBioTec, Departamento de Geociências, Universidade de Aveiro
- (4) Departamento de Química e Bioquímica, Universidade de Évora, Évora, Portugal and CHANGE & MED
- (5) Laboratório HERCULES — Herança Cultural, Estudos e Salvaguarda e IN2PAST — Laboratório Associado para a Investigação e Inovação em Património, Artes, Sustentabilidade e Território, Universidade de Évora

**Summary:** *The main objective of the Calcinata Project - Production of lime-based mortar from the calcination of carbonated sludge from the ornamental stone industry (marbles and limestone), was to present an alternative use for waste (slurries) from cutting and polishing carbonated ornamental rocks. The industrial application of this waste makes it possible to add value, transforming it into a by-product, thus contributing to the framing of the limestone and marble extractive and processing sub-sectors, in the “Action Plan for the Circular Economy”, promoting the sustainable growth. This research showed that recycling slurries has relevant potential since they can be used as a raw material integrated in binders production used in the manufacture of ornamental stone composites materials partial or totally replacing the epoxy resins traditionally used in this type of products.*

**Key words:** *sludge, marble, limestone, binder, composites*

## 1. Introduction

The carbonated dimension stone extractive and processing industry produce high amounts of wastes that are deposited in the open in heaps and deposits of carbonated sludge (Fig. 1).

Environmental impacts are unavoidable, such as: i) reduction of vegetation cover, ii) decrease in agricultural activity, iii) soil sealing, iv) alteration of water lines with a significant reduction in its quality, v) alteration of ecosystems, vi) decrease in air quality, vii) reduction in the photosynthetic process of plants and visual impact, the latter being quite striking, as the white color of the deposits is very contrasting with the mainly rural surrounding environment (Afonso et al, 2023).

The industrial application of this waste makes it possible to add value, transforming it into a by-product, thus contributing to the framing of the limestone and marble extractive and processing sub-sectors, in the “Action Plan for the Circular Economy”, promoting the sustainable growth. By this means, this research has shown that the recycling slurries as a raw material are possible to integrate binders production through its incorporation on the manufacture of ornamental stone composite materials, partial or totally replacing the epoxy resins traditionally used in this type of products.



Fig. 1 Deposit of carbonated sludge close to Vila Viçosa village.

## 2. Materials and Methods

The slurries were sampled in the filter presses plants at the companies: *António Galego & Filhos – Mármore SA*, referred to as M(AGF) and *A.L.A. de Almeida SA*, referred to as M(A). The limestone carbonated sludge was collected at *Solancis - Sociedade Exploradora de Pedreiras SA*, referenced as C(S) (Fig. 2) and *MVC - Mármore de Alcobaça Lda.*, referenced as C(MVC).

The laboratory work on the physical, chemical and mineralogical characterization of the slurries was carried out at the laboratories of the University of Évora, namely Geosciences Department, Mechanical Testing, Hercules and Ambiterro laboratories.



Fig. 2 Sampling of limestone carbonated sludge in Solancis factory.

After the slurry's characterization phase, a new stage began with binder formulations composed with different percentages of sludge and polyester resin, Recapoli 2196 by the Castro Composites Company, and consequent physical-mechanical evaluation at three curing times: 7, 14 and 28 days.

Once the best performing binder formulation has been determined, composites were prepared using marble aggregates supplied by *Marvisa, Mármores Alentejanos Lda.* Company. The mixture of the binders and aggregates were mixed and poured into 55 cm x 15 cm x 15 cm moulds to produce specimens' for subsequent stone composite physical-mechanical characterization.

### 3. Sludges - Physical and Chemical Characterization

Among the physical and chemical characterization, granulometric analysis (through particle sieving and sedigraph methods) and chemical and mineralogical composition of the samples stand out. From the point of view of granulometric distribution, the samples of limestone and marbled sludge showed similar distributions with the particularity of the limestone sludge having a finer granulometry than the marbled sludge, as shown in Figure 3.

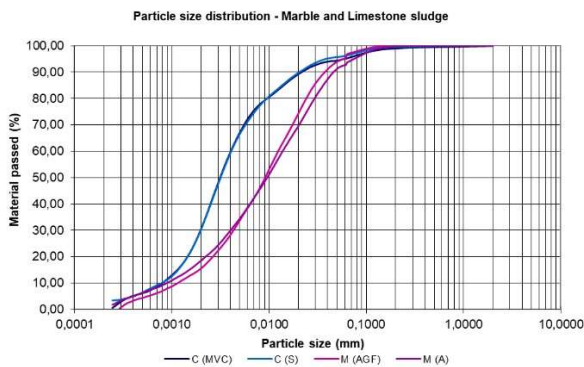


Fig. 3 Particle size distribution on the selected marble and limestone sludge samples.

The chemical analyses carried out using atomic spectrometry revealed a fundamentally carbonated material. All carbonated sludges have significant

percentages of CaO and show relevant percentage of loss of ignition. M(AGF) and M(A) depicted small percentage of SiO<sub>2</sub> (2.5 – 3.5%) and M(AGF) show the presence of magnesium (MgO) (Table 1).

Table 1 – Chemical analysis.

Samples	CaO (%)	SiO <sub>2</sub> (%)	MgO (%)	Loss on Ignition (%)
M(AGF)	45,504	2,549	3,156	42,97
M(A)	51,555	3,537	0,829	42,15
C(MVC)	52,580	0,357	0,337	43,50
C(S)	54,189	0,297	0,301	43,30

Mineralogical analysis corroborated the carbonate character of the sludges, with a well-defined peak in calcite on x-ray diffraction.

### 4. Formulation of Binders Consisting of Carbonated Sludge and Resin

Formulations were made with different percentage contributions of marbled sludge and polyester resin, the same being done with limestone sludge. The characterization was carried out at different times:

- i. Preliminary tests with manual mixtures in order to visually observe the final products after drying;
- ii. Physical characterization based on the viscosities studied from the formulations made only with manual mixtures;
- iii. Mechanical characterization based on tests of mechanical resistance to compression of cubic specimens, obtained from formulations prepared into molds measuring 15 cm x 15 cm x 15 cm.

For the evaluation of viscosity, an innovative experimental methodology was developed that allowed to perform a comparative evaluation on the viscous behavior of different binders. This methodology is based on an experimental device (Fig. 4) consisting of a glass plate that is positioned at a 45° inclination, to which a metal ruler is associated that allows measuring the displacement of the binder through time. In this way, it is possible to assess the fluidity of the binder when it is placed on the glass.



Fig. 4 Experimental set-up for viscosity and rheological measurements

To carry out this test, times were pre-established for: i) mixing the resin with the conditioner; ii) filling an adapted syringe and iii) emptying it at the top of the set-up ramp. After this, sliding times were measured.

Figure 5 shows the viscosity graphic representation of the four formulations with limestone sludge and another four with marble sludge. The fractional number represents the percentage of sludge in the numerator and the percentage of polyester resin in the denominator.

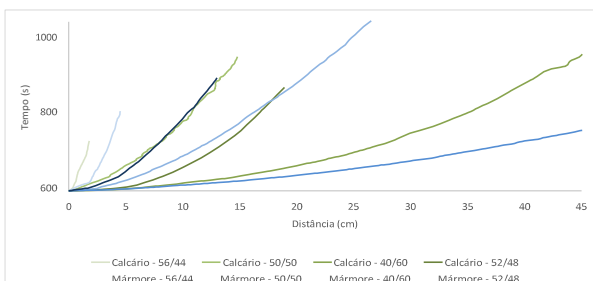


Fig. 5 Viscosity graphic representation of binder formulations.

Analyzing the graph from Fig.5, identical behavior can be observed for the formulations with the same percentages of sludge and resin. The higher the

percentage of resin, the more fluid the binder becomes. The aim of this research being to reduce resin and increase the contribution of sludge.

The mechanical characterization of the binder was carried out by evaluating the resistance to uniaxial compression of the different formulations with different percentage contributions of limestone and marble sludge and resin. The tests were carried out on cubic specimens with a 5 cm side and using a Pegasil compression press. After demolding, the specimens kept for curing in air and were tested at 7, 14 and 28 days, with increasing uniaxial compressive strength over this period, reaching higher uniaxial compression values at 28 days. Test were made according to the NP EN 1926:2008 standard. These results are shown in Table 2 (Afonso, P. et al, 2023).

Table 2: Results of uniaxial compressive strength tests after 28 days of curing.

Formulations %	R (MPa)
ANM3 – 54,43%NM / 45,57%Res.	102.73
ANM4 – 50%NM / 50%Res.	98.35
ANM5 – 47%NM / 53%Res.	96.23
ANM6 – 52%NM / 48%Res.	106.37
ANC3 – 52,31%NC / 47,69%Res.	103.20
ANC4 – 50%NC / 50%Res.	102.12
ANC5 – 47%NC / 53%Res.	96.04

NM – Marble sludge; NC – Limestone sludge; Res – Resine.

The proportion of 52% carbonated sludge and 48% resin, the formulation with marble carbonated sludge showed an increase of 3MPa in compression strength when compared to limestone, these two binders showed the best performances in terms of mechanical compression strength. The results obtained in the tests carried out allowed us to establish the ideal binder, with a proportion of 52% sludge (limestone / marble) and 48% polyester resin.

## 5. Composite Formulations with Binder 52/48 and Marble Aggregates

With the formulation of stone composites, it was intended to obtain prototypes with different compositions of aggregate and binder 52/48 (Table 3). The marble aggregate, supplied by the company *Marvisa, Mármore Alentejanos Lda.*, consisted of three types, referred to as BA, B1 and B2, with the following

granulometric intervals: BA (4 mm / 6.3 mm), B1 (8 mm / 14 mm) and B2 (14 mm / 25 mm).

Table 3: Stone composite formulations.

Formul.	Aggregates			Binders		Res
	BA	B1	B2	NC	NM	
F1	30%	30%	40%	52%		48%
F2	20%	20%	60%	52%		48%
F3	35%	15%	50%	52%		48%
F4	40%	40%	20%	52%		48%
F5	30%	30%	40%			52
F6	20%	20%	60%			52
F7	35%	15%	50%			52
F8	40%	40%	20%			52

Using a light mechanical mixer, the binder was mixed (sludge / resin + catalyst), subsequently introducing the aggregates (Fig. 6). After the period necessary to ensure complete homogenization of the entire mixture, it was poured into metal molds measuring 15 cm x 15 cm x 15 cm and 55 cm x 15 cm x 15 cm, with a view to obtaining specimens for different characterization tests. Adopting the same criteria used in the study of the binders, the objective was to study the specimens with three curing times: 7, 14 and 28 days.

After the uniaxial compressive strength tests (NP EN 1926-2008) in specimens subject to the three curing times, strength results were obtained in the specimens tested at 28 days (Table 4).

By analyzing the data, it can be seen that the formulation made up of F(4) limestone sludge and 52% Aggregates (40%BA/40%B1/20%B2) + 48% Binder (52%limestone sludge+48% Resin) was the one that presented the best results, reaching 91.96 MPa. The best formulation with marble sludge was F8 with 88.19 MPa.

Having defined the formulations with the best performance, in terms of compression strength, the remaining characterization tests were made, namely: i) determination of flexural strength under concentrated load (Table 5); ii) apparent density and porosity (NP EN 1936-2008) (Table 6); iii) absorption of water at atmospheric pressure (NP EN 13755) (Table 7) and iv) absorption of water by capillarity (NP En 1925-2000) (Table 8).



Fig. 6 Stone composites formulation mixing and vibration in the mold.

Table 4: Uniaxial compressive strength test results for the several formulations.

Formulations	Average uniaxial Compression Strength (MPa)
F1	73.30
F2	69.06
F3	61.11
<b>F4</b>	<b>91.96</b>
F5	52.26
F6	76.33
F7	81.20
<b>F8</b>	<b>88.19</b>

The specimens for the flexural strength test were obtained from the block produced in the 55 cm long molds, thus allowing them to have the dimensions of 30 cm x 5 cm x 5 cm (Fig. 7), required by the NP EN standard 12372.





Fig. 7 Specimens obtained from cutting the block with a marble sludge binder.

Table 5: Flexural strength.

Curing (Days)	Binder	Flexural strength (MPa)
28	Marble	1549 ± 1.38
	Limestone	13,49 ± 0.97

Table 6: Apparent density and open porosity.

Binder	Volume of open pores (ml)	Apparent volume (ml)
Marble	0.22 ± 0.03	117.0 ± 7.0
Limestone	0.21 ± 0.04	104.6 ± 7.4
Binder	Apparent density (g/m <sup>3</sup> )	Open porosity (%)
Marble	2.279 ± 0.014	0.19 ± 0.02
Limestone	2.263 ± 0.017	0.20 ± 0.03

Table 7: Water absorption at atmospheric pressure.

Binder	Water absorption at atmospheric pressure (%)
Marble	0.2 ± 0.03
Limestone	0.1 ± 0.03

Table 8: Water absorption through capilarity.

Binder	Average (g/m <sup>2</sup> .s)
Marble	0.088 ± 0.010
Limestone	0.062 ± 0.019

## 6. Future Works

To better characterize the formulations, freeze-thaw tests will be carried out at the Mechanic Testing Laboratory (LEM), according to Standard NP EN 012371\_2015 and using a FitoTerm-700GD25 climatic chamber, repeating the tests. The following tests on polished specimens will also be considered: thermal shock, brightness, roughness and color.

## 7. Conclusions

Atendendo aos resultados obtidos conclui-se o seguinte:

In view of the results, the following is concluded:

1 – The use of polyester resin in replacement of epoxy resin seems feasible. The price of epoxy resin is on average four times higher than polyester resin, which makes stone composite products substantially more expensive.

2 – As one goal of the investigation was the reduction of resin and its partial replacement by carbonated sludge, the investigation gradually incorporated successively higher percentages of solid load, without compromising the strength of the specimens. Thus, the ideal binder percentage was: 52% carbonated sludge and 48% polyester resin.

In the formulations with marble aggregate, the ideal percentage was achieved with 52% of aggregate and 48% of binder, which leads to a substantial reduction of resin in the formulation of a stone composite.

3 – The results obtained in the compressive and flexural strength tests are promising, given the fact that the formulations were carried out manually using a manual mixer. Execution in an industrial context using a suitable mixer with a vacuum chamber will allow a more homogeneous product, with expected higher density and without structural defects, allowing improved values of compression and flexural strength.

3.1 - A comparison with values of mechanical compression strength, traditionally found in different lithotypes sold as ornamental rock, considering the lowest and highest values of each stone type (Catalogue of Portuguese Ornamental Stones) show that the developed stone composites are within an equivalent range of performance:

Limestones: Pedra de Ançã (23 MPa) – Banco de Baixo (167 MPa).

Marbles: Rosa Venado (70 MPa) – Branco Venado (102 MPa).

Granites: Amarelo Vila Real (70 MPa) – Cinzento Alpalhão (253 MPa).

Recalling the highest values obtained in the compressive strength tests on formulations (F4 – 91.96 MPa; F8 – 88.19 MPa), it can be concluded that they are within the range of values for natural stone.

3.2 - Regarding flexural strength, to establish a comparison with different lithotypes marketed as ornamental rock, the example of some reference values is given (Catalog of Portuguese Ornamental Rocks):

Limestones: Olho de Sapo (Arrimal) (9 MPa) – Branco do Mar (31 MPa).

Marbles: Rosa (15 MPa) – Creme Venado (29 MPa).

Granites: Amarelo Figueira (6 MPa) – Azulália (35 MPa).

Similarly, to compression strength, flexural strength are also within the range of a large part of natural dimension stones.

4 – With regard to water absorption at atmospheric pressure, natural stone vary between: i) 0.2% and 9.6% on limestones; ii) 0.2% and 1.05% on granites and around 0.1% on marbles. The stone composites are also within this range of absorption values.

5 – Regarding open porosity, the two stone composites present values of approximately 0.2%, equivalent to the open porosities detected in marbles. Composites open porosity is lower than the one usually seen in granites (0.4%, 0.7%) and limestones (0.4%, > 1%) according to the Portuguese Ornamental Stones Portal, LNEG.

6 – Despite the studies not yet being completed, the possibility of using polyester resin in stone composites is very promising, as well as the incorporation of carbonated sludge from the processing of limestone and marble, thus contributing to the reduction of environmental impacts caused by the accumulation of these materials in the open.

#### **Acknowledgments:**

- Research developed within the scope of the project “CALCINATA – Production of lime-based mortar from the calcination of carbonate sludge from the ornamental stone industry (marble and limestone)” with the reference ALT20-03-0247-FEDER-072239. Project co-financed by the European Regional Development Fund (ERDF) within the framework of ALENTEJO 2020 (Programa Operacional Regional do Alentejo).
- Special thanks to Associação Cluster Portugal Mineral Resources, co-manager of the project and to the Project Support Office of the University of Évora.
- Special thanks to the Science and Cooperation Services and Administrative Services of the University of Évora.
- Special thanks to the Technical Services of the University of Évora and in particular to Mr. Artur Calhau for providing the mixers, without which it would not have been possible to carry out the formulations.

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**Topic:** Climate change impact on geomaterials

**Title:** High temperature impact on several Portuguese limestones

**Authors:** Lobarinhas R., Paneiro G., Dionísio A.

**Filiation:** CERENA, DER, Instituto Superior Técnico, Lisbon University, Av. Rovisco Pais, 1049-001, Lisbon, Portugal

Future projections in temperature extremes as consequence of climate change will lead to an increasing number, duration, and intensity of heat waves. This phenomenon is associated with droughts and devastating wildfires. In the Mediterranean this alarming trend is also confirmed, being projected a future increase in fire danger and fire season length in southern Europe, particularly in the countries of the northern arch of the Mediterranean Sea.

In Portugal, wildfires have been increasing in the last decades. According to the EFIS - European Forest Fire Information System, Portugal is the second European country with the highest annual total number of fires between 2006-2021 and the first with the highest annual total of burned area during the same period. This context becomes an important threat to, among others, Built Heritage. In this national context is critical to direct attention to the problem of rock vulnerability to high temperatures, which is a consequence of fire events.

In such an alarming scenario and being a country with a very rich stone cultural heritage, it's crucial to study the impact of fire on national lithotypes. Notorious examples of such damage, like the April 2019 fire at Notre Dame Cathedral in Paris, have brought attention to this problem. For that, is important to identify and geologically characterize such heritage stones, continuing the process of valorization of these resources. Besides the protection of the Built Heritage, the study of rock vulnerability to high temperatures is one of the main issues faced in mining and construction contexts where the stone is the main material, and this comprehension would be very useful.

This paper presents some studies conducted to assess the impact of high temperatures on different Portuguese limestones. The lithotypes were selected considering the variability of petrological properties such as grain size, mineralogical content, and the presence of fossils.

Samples were heated in a furnace at 300°C and 600°C for 3 hours. The samples were immediately cooled in water to also mimic the fire extinguishing scenario. Afterward, some physical tests were performed, and the results are compared to reference samples (non-heated). Properties such as open porosity and capillarity suction were evaluated. This range of temperature was chosen to mimic the major temperature range in real-case fires in larger and smaller degrees.

With these studies, it is possible to conclude how different petrographic aspects can influence limestone behavior at high temperatures.

# MINERAL ALTERATIONS CAUSED BY LIGHTNING IN THE CHRIST OF THE REDEEMER SURFACE, RIO DE JANEIRO, BRAZIL

Roberto Carlos Ribeiro\*, Nuria Fernández Castro and Rosana Elisa Coppedê Silva

Researchers from Centre for Mineral Technology – CETEM, Av. Pedro Calmon, 900, Ilha da Cidade Universitária, Rio de Janeiro, RJ, Brazil (55) (21) 38657264, [rcarlos@cetem.gov.br](mailto:rcarlos@cetem.gov.br); [ncastro@cetem.gov.br](mailto:ncastro@cetem.gov.br) and [coppede@cetem.gov.br](mailto:coppede@cetem.gov.br)

## Summary:

Christ the Redeemer is an Art Deco statue of Jesus Christ with open arms in Rio de Janeiro, Brazil, built between 1922 and 1931. The statue is located at the peak of the 700 m height Corcovado's Mountain in the Tijuca Forest National Park. It is a hollow structure of reinforced concrete, covered with small pieces (tesserae) of steatite (soapstone), which protect the monument structure against water infiltration. This stone was chosen due to its already-known characteristics of hydrophobicity and high resistance to extreme temperatures. The statue, by its location, at one peak of a coastal and urban city, is exposed to several natural agents like solid winds, solar radiation, rain (acid due to the sea-salt aerosols and pollution), and especially to lightning.

The monument has a lightning arrester system in the form of a crown over the statue's head, but it is hit by lightning several times yearly. In a technological research carried out in 2010 to analyse a proposed water-repellent's effect on the monument covering, samples of the monument's original tesserae (1931), substitutes placed in 2000, and fresh ones extracted from the quarry in 2010 were characterised. It was found that the stone's porosity increased from 0.41% (fresh samples) to 3% (original samples). The samples were extracted from the monument's body, below the arms. It was concluded that the microbiological action was the primary decay agent on the soapstone, and the proposed water-repellent was tested on all the samples with satisfactory results.

During that research, it was observed that the arms and head of the statue were the most deteriorated areas, with many missing parts and tesserae showing more mass losses and water absorption of around 20%, strikingly higher than in the rest of the monument. As those areas were not prone to microbiological action because of the washing effect of rain and solar exposure, it was thought that a possible cause for the deterioration could be the lightning. XRF, XRD, SEM-EDS, FTIR, and TGA analyses showed new calcium and magnesium carbonate minerals formed on the samples and a porosity of 8%. These results corroborate that lightning changed the mineral composition and consequent hydric properties of the steatite, so conservation treatments and products tested on or specific for soapstone may not be effective on the head and arms tesserae where the stone composition and properties differ from those in other parts of the monument. Technological support to properly characterise the tesserae cover of the statue's upper part and treatment products' compatibility will be necessary to prevent further damage to the monument.

**Key words:** *Christ The Redeemer, soapstone, mineral alteration.*

## 1. Introduction

### 1.1 Soapstone

Soapstone is a metamorphic rock, rich in magnesium, with essential mineralogy of talc and chlorite. This rock has a high density and shades of colour, varying from green to grey. Besides talc and chlorite, usually, the soapstone contains amphiboles, micas and some carbonate. This rock can be originated from ultramafic rocks, magmatic rocks, volcanic rocks and dolomite. The steatite's properties vary depending on the protolith and geological environment. For example, soapstones derived from ultramafic rocks have homogeneous structures and serpentine in their mineralogical composition, which does not happen when soapstone originates from some intermediate rocks. Steatite has some properties that make this stone suitable for monuments. For example, the large amount of talc and softness are characteristics that make the stone very easy to carve [1]

### 1.2 Christ the Redeemer

Christ the Redeemer is an Art Deco statue of Jesus Christ in Rio de Janeiro, Brazil, created by French sculptor Paul Landowski and built by the Brazilian engineer Heitor da Silva Costa in collaboration with the French engineer Albert Caquot. It is 30 m tall, not including its 8-meter pedestal, and its arms stretch 28 meters wide.

The statue, weighing 635 metric tons, stands at the peak of the 700 m Corcovado Mountain in the Tijuca Forest National Park overlooking the city of Rio de Janeiro. As a symbol of Brazilian Christianity, it has become an icon for Rio de Janeiro and Brazil. The statue was constructed in reinforced concrete and dressed with a soapstone mosaic between 1922 and 1931.

Landowski produced a miniature of the statue and the actual head and hands and shipped them out to Rio de Janeiro. Considering that the real-life statue is 38 meters

high, it is remarkable that Landowski could produce even these few sections at his Paris studio.

One hand alone weighed eight tons. Interestingly, the sculptor modelled Christ's hands after a woman's – those of his assistant.

Since there was already a rack railway leading up the steep, hunchback-shaped Corcovado, from which it derives its name, the building materials could easily be brought all the way up to the summit so that the main body of the statue could be constructed in situ. The monument was made essentially of concrete, its surface studded with thousands of small pieces of shimmering green or grey soapstone [2].

Small pieces of soapstone (Figure 1) (tesserae) cover the monument functioning as protection for the structure because that rock is hydrophobic, preventing water entrance.

### 1.3 Alterations in the monument

With time taking its toll, the mosaics gradually worked loose and whole sections of the concrete detached. Corrosion of the inner metal structure was to blame.

However, before the 2010 restoration intervention, it was perceived that both the steatite tesserae and the mortar water absorptions had substantially increased, allowing the water percolation, which damaged the monument's concrete structure [3]. Soapstone weathering and decay patterns in monuments of the State of Minas Gerais and studies aiming at that stone conservation had already been developed at that time [4, 5], as it is the most representative heritage stone of that state [6]. Several other studies have been conducted since then [7, 8].

In the year 2010, during the last restoration, CETEM was responsible for the technological support and carried out a study that could detect that one of the causes of tesserae's degradation was the significant microbiological proliferation that generated acids in its metabolism, thus destroying the tesserae, which started to present water absorptions in the order of 6%, where it should be around 1% [9]. In addition, because of the monument's location, on one the highest points of Rio de Janeiro, at Corcovado Hill, the monument suffers constant attacks of electric discharges. They primarily affect the head, fingers and chin tesserae, possibly altering their structure or exposing composing minerals less resistant to weathering, thus creating susceptible points for degradation.

Specifically in those areas of the monument, the water absorption and porosity values, often reaching 20%, proved the high decay degree.

The Christ Redeemer is exposed to several phenomena, like heavy rain, sometimes acid, strong winds and salt spray, as Rio is a coastal city. Besides that, the monument's position at the top of Corcovado favours lightning strokes. The arms and the head of the statue are the most deteriorated. The monument's soapstone dressing, a hydrophobic rock, protected the structure over the years [10], but the high water absorption values found in the tesserae would undoubtedly lead to the reinforced concrete deterioration [11].



Figure 1: Christ of the Redeemer Surface. Figure 2: Several lightning in the monument (Source: Agência Brasil. [12]).

### 1.4 Importance of Technological Support in Restoration

Heritage stones or mortars' characterisation is vital to acquire knowledge about the material, determining the causes of changes, and proposing methods to avoid or delay the monument's degradation [13]. Several places in the world have already had their stones or mortars characterised, including those from the Byzantine period from the sixth to the tenth centuries [13]. Many techniques have been successfully applied in the characterisation, including X-ray diffraction [14–26], macroscopic observation [25], petrography [25, 26], physical analysis [25], infrared spectroscopy [13,17], chemical analysis [23-25], thermogravimetric analysis (TG-DTG) [13-25], mechanical tests [24], and granulometry [13,24,26].

## 2. Objective

This work aims to verify whether lightning in The Christ of the Redeemer causes mineral alterations in the monument's soapstone tesserae.

## 3. Methodology

For the research, 56 soapstone samples were used: 16 original tesserae from the 1930s, collected near the monument's feet, 16 tesserae used in the restoration of 2000, 16 tesserae used in the restoration of 2010 and 8 fragments of tessera present in the arms after a lightning strike. Tesserae from 2000 and 2010 were stored inside the monument.

In this paper, sample identifications are Tesserae 1930, Tesserae 2000, Tesserae 2010 and Lightning.



Figure 3: Tesserae's extraction.

### 3.2 Methods

The samples were first observed macroscopically for characteristics identifiable by the naked eye, such as colour, texture, shape and size.

The microscopic description used a Schneider magnifying glass with a Carl Zeiss lens, zoomed in from 6.5× to 60× and fluorescent light. Raman analyses were carried out using a BWTEK Raman device to support the previous observations. The equipment covers a spectral range in Raman displacement of 150-3000  $\text{cm}^{-1}$ , with a spectral resolution of 5  $\text{cm}^{-1}$ . Spectra were acquired using commercial software provided by B&W TEK.

The remainder of each sample was manually ground using an agate mortar and pestle, sieved up to 105 $\mu\text{m}$  and characterized using X-ray Powder Diffraction (XRPD), Fourier Transform Infrared Spectrometry (FTIR) and Wavelength-Dispersive X-ray Fluorescence Spectrometry Panalytical WDS-1 spectrometer.

X-ray diffraction spectra were obtained by a Bruker D4-Endeavor instrument (40 kV, 40 mA) with a  $\text{CoK}\alpha$  wavelength from 10 to 100° in  $2\theta$ , step size of 0.02° and 3.6 s/step scan.

For the application of the Fourier transform infrared spectrometry (FTIR) to the qualitative identification of compounds present in the samples, a Perkin Elmer Spectrum 400 was employed. The pellets, about 13 mm in diameter, contained approximately 2.0% of the sample; they were well mixed with 300 mg of potassium bromide.

The light staining area was also evaluated through SEM-EDX analyses. For this, a Hitachi scanning electron microscope was used. The instrument was equipped with a Bruker X-Flash energy dispersive X-ray spectrometer, with MIN SVE detector and scan generator connected.

The samples for WDXRF analysis were dried in an oven at 100°C; then, about 7 g of each sample were pressed into uniform pellets (20 mm in diameter) using a Vaneox automatic press machine under 20 tons of pressure with a standing time of 30 s, using boric acid as a base. All the

measurements were carried out on an AXIOS Panalytical WDS-1 spectrometer.

TGA was performed using NETZSCH thermogravimetric equipment. Approximately 12 mg samples were weighed in an aluminium crucible and heated between 40 and 560 °C (10 °C  $\text{min}^{-1}$  rate) under a nitrogen atmosphere, with a flow rate of 50  $\text{mL min}^{-1}$ .

### 4. Results and Discussion

The results of XRF (Table 1) indicate that the natural soapstone presents percentages of silica that reach ~60% and magnesium ~30%. The tesserae removed from the arms (lightning) present about 10% silica, the magnesium content is significantly higher (50%), possibly associated with the carbonate, since there is an increase in the PPC value to 26%.

Table 1: XRF results of the samples (%).

	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	PPC
1930	31.2	1.0	57.0	1.9	3.6	4.8
2000	33.2	0.6	58.9	0.0	2.5	4.6
2010	32.6	0.5	58.8	0.0	3.0	4.8
lightning	54.1	0.3	10.8	8.0	0.4	26.3

As illustrated in Figures 4-6, the X-ray diffraction analyses carried on the tesserae (1930, 2000 and 2010) present peaks of talc and tremolite. Figure 7 shows the X-ray diffraction of the arms (lightning) samples, where is possible to see peaks of calcite and dolomite. This is credited to the dissociation of magnesium or calcium silicate and transformation in magnesium or calcium carbonate, at the high temperature and pressure of storm. Samples from the regions of the monument that suffer from lightning rays (head and arms) present mineral alterations, with dolomite or calcite formation. It is possible that the high pressure and temperature of the rays, promote the chemical bonding of the magnesium present in the soapstone structure with the atmospheric  $\text{CO}_2$ , indicating the formation of calcium carbonate or magnesium.

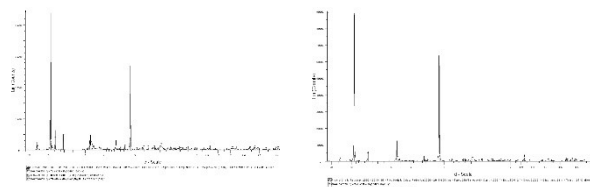


Figure 4: Tesserae 1930 XRD. Figure 5: Tesserae 2000 XRD.

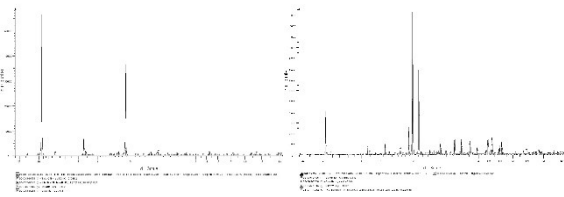


Figure 6: Tesseræ 2010 XRD. Figure 7: lightning XRD.

Figure 8 shows the structure of the natural soapstone, evidencing the presence of talc. In Figure 9, from the arms and head samples, the defragmentation of the talc sample and the visualization of new minerals such as dolomite and calcite are observed.

Studies in the area of High Voltage indicate that the action of the pulses caused by the rays are able to disorient and defragment several materials, allowing their better application in several industrial sectors, such as recycling and recovery of precious metals in electronic scrap and recovery of glass fibre sector. However, to date there are no papers in the literature on mineralogical changes caused by the action of lightning at high pressures and temperatures [10-13].

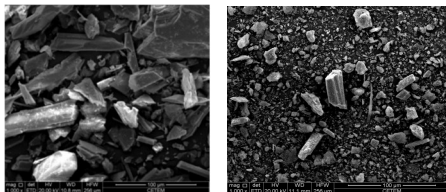


Figure 8: SEM image of tesseræ 2010 (1000x). Figure 9: SEM image before lightning (1000x).

Figures 10, 11 and 12 shows the SEM results for 1930, 2000 and 2010, respectively. They show the presence of Si, Al, Mg and Fe in the mapping and EDS corroborating the results of FRX and XRD.

Figure 13 shows the SEM/EDS result of the sample of lightning where calcium carbonate or magnesium formation can be inferred from the presence of carbon, magnesium, calcium and oxygen. This results agrees with the theory of those minerals formation in the monument.

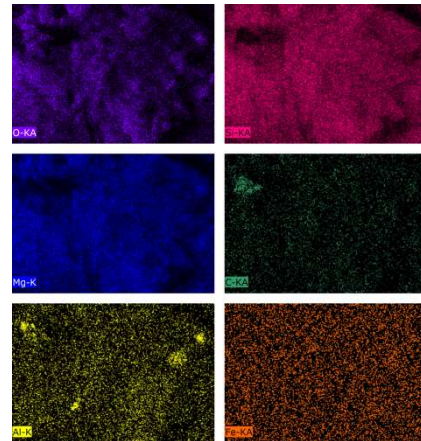
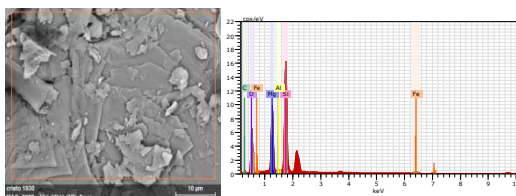


Figure 10: SEM/EDS mapping Tesseræ 1930.

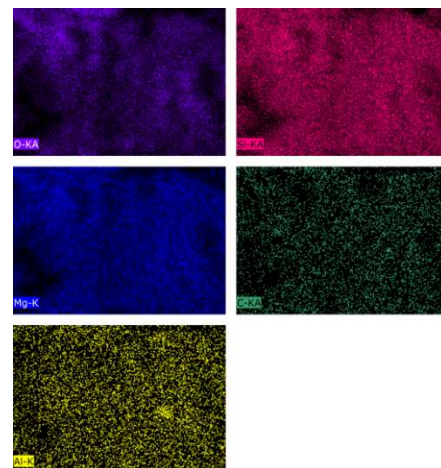
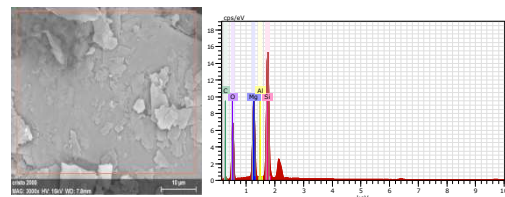


Figure 11: SEM/EDS mapping Tesseræ 2000.

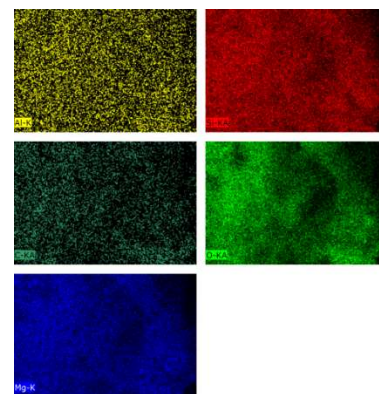
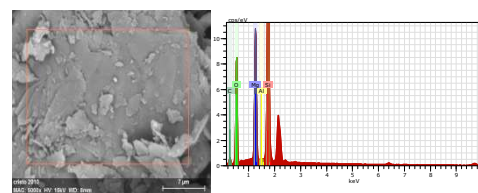


Figure 12: SEM/EDS mapping Tesseræ 2010.

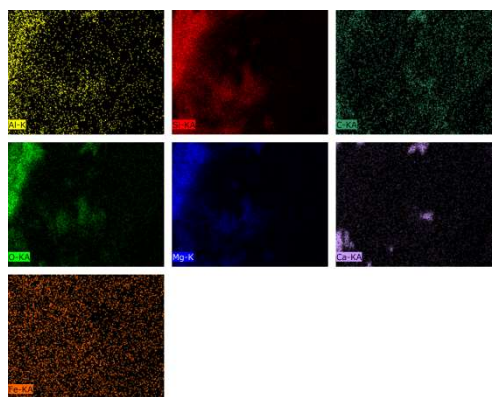
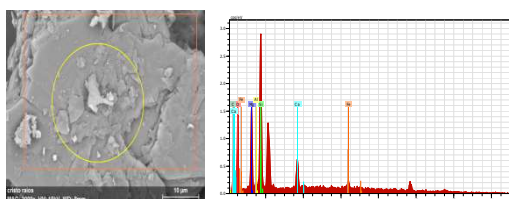


Figure 13: SEM /EDS mapping Tesseræ lightning.

Figure 14 shows the FTIR results where it is possible to verify, between 1,400 and 1,500, characteristic peaks of the formation of carbonates (Mg and Ca) in the sample that was attacked by the lightning-rod (green line), confirming the formation of these minerals due to the alteration of the soapstone at high pressures and temperatures.

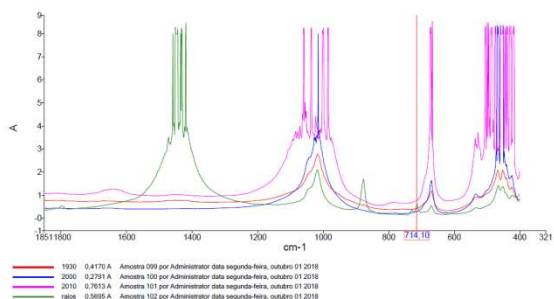


Figure 14: FTIR results of samples.

Figure 15 shows the results of the TGA evaluation of the samples, where it can be observed that the tesseræ of 1930, 2000 and 2010 are very stable, with a mass loss around 5% at 1,000 °C. On the other hand, the sample attacked by the lightning, presents a differentiated behavior, with mass loss of more than 60% at 900°C, supporting the previous results, once again, of the possible carbonates formation, and alteration of the soapstone.

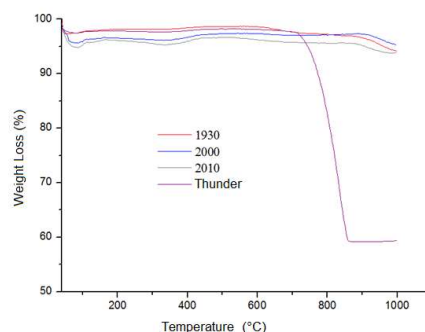


Figure 15: TGA results of samples.

Several authors have described mineral changes due to building stones weathering, especially regarding the roles of atmospheric sulfur and NaCl. Sulfur may form gypsum over the stones, where calcium from mortars or even other rocks is associated with that pollutant, generating the mineral and forming black crusts. In addition, NaCl's action on stones' deterioration is well known; this salt from the sea deposits on the rocks' surfaces and migrates to their interior through their pores, causing significant changes in the pore structure that can even break the stones [14-16].

The porosity and water absorption of tesseræ (1930, 2000 and 2010) are around 1% and 0.3%, respectively. The values of the tesseræ extracted from the arms and head of the monument (lightning) show values of 21 and 15%, respectively. As discussed in this paper, the formation of calcium and magnesium carbonates due to the electrical discharges of lightning on steatite could explain such water absorption values. The soapstone carbonates content increase in those parts of the monument by this alteration would make that stone much more absorbent than the original.

Some international studies on soapstone do not report these types of mineral transformation [17-18].

## 5. Conclusions

The main conclusion is that the rays reaching the soapstone may form or expose new minerals more susceptible to decay, facilitating the degradation of the monument's structure. In addition, the protective agents applied to the monument may not effectively prevent its deterioration. Those products' efficacy would have been explicitly tested for the original composition of soapstone, which could have changed significantly due to the formation of calcium and magnesium carbonates. A proper characterization of the steatite in the higher part of the monument shall be carried out before choosing the protective product.

All the results from this work point to the carbonates' neoformation by a lightning stroke on steatite. FRX, XRD, MEV, EDS, TGA and water absorption results of the



monument's head and arms tesserae, where the lightning strikes, support that theory.

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# Production Process Based on Additive Manufacturing Powder Bed Methods

Rafael Tavares<sup>1\*</sup>, Tomás Carvalho <sup>1</sup>, Florindo Gaspar<sup>1</sup>, José Fernandes<sup>3</sup>, Alexandre Vieira<sup>4</sup>, Jorge Caetano<sup>2</sup>, Artur Mateus<sup>1</sup>

- (1) Centre for Rapid and Sustainable Product Development – Polytechnic of Leiria, \*[rafael.m.tavares@ipleiria.pt](mailto:rafael.m.tavares@ipleiria.pt)
- (2) Sorgila
- (3) MVC – Portuguese Limestone
- (4) Mármores vigário

## Summary:

Powder Bed (PB) 3D printing is a type of additive manufacturing that involves building a part layer by layer using a powder material. In this method, a thin layer of powdered material (such as plastic, metal, ceramic, or stone powder, etc.) is spread evenly over a build platform. A laser, electron beam or binder aggregates is then used to selectively melt, fuse, or bind the powder particles in the desired areas to form a solid part, acting as a liquid or solid agent. The build platform is then lowered by a fraction of a millimeter, and the process is repeated with a new layer of powder on top of the previous layer until the part is complete. This article proposes an analysis of the application of unconventional materials such as geopolymers and cementitious in a variant of the PB process called Digital Selective Powder Deposition (DPSD) where selectively apply the materials, binder agents and aggregates to produce and solidify the layers of powder.

**Key words:** *additive manufacturing, 3D printing, Digital Selective Powder Deposition*

## Introduction

Additive manufacturing, also known as 3D printing, has revolutionized the way products are designed and manufactured. One of the most widely used 3D printing technologies is the PB methods, which involves selectively fusing or solidifying layers of powdered material to build up a 3D object. This method has gained popularity due to its ability to produce the geometries, use a wide range of materials, and achieve high precision and accuracy. This article explore PB methods used in 3D printing, including their principles, advantages, and limitations to contextualize the construction of parts made by new kinds of materials examining specific methods of powder deposition aimed at producing mineral-based products. The article mentions that experiments have been conducted using various materials such as ceramic, basalt, cement and geopolymers, but it's important to note that this particular research is still in its preliminary stage. It is also discussed the latest advancements in this technology to present their impact on the manufacturing industry, applying them on use of unconventional materials. Whether for design engineers, manufacturers, or simply interested people in the world of 3D printing, this article will provide a new comprehensive and approach of PB methods and their role in shaping the future of manufacturing.

### 1. Powder Bed Methods for 3D Printing

Powder Bed method is a type of 3D printing technique in which a layer of powdered material is spread onto a build platform, and a printer head selectively fuses or solidifies the powder in a specific pattern, layer by layer, until the final part is formed. There are several variations of PB 3D printing, including Selective Laser Sintering (SLS) [1], Digital Light Processing (DLP), and Electron Beam Melting (EBM).

In the SLS process, a laser is used to selectively melt or sinter the powdered material (usually a polymer or a metal), creating the desired part. The laser follows a path defined by the CAD file of the part and fuses the powder where it needs to be solidified. Once a layer is completed, the build platform is lowered and a new layer of powder is spread on top of the previous one, and the process is repeated, as all building processes by additive manufacturing, until the final part is formed.

In the DLP process, a digital light projector is used to selectively cure and solidify a layer of photopolymer resin. The projector projects a pattern of UV light onto the thin layer of resin, causing it to solidify in the horizontal slice of the shape part.

In EBM, an electron beam is used to selectively melt metal powder. In this case, the electron beam melts the powder in the target areas.

The principal advantages that can be highlighted in PB printing process over other methods, include the ability to create different shapes with high precision and accuracy, and to use a wide range of materials. However, they can be more expensive and slower than other techniques and may require additional post-processing steps to remove excess powder or to achieve the properly surface finish.

## 2. *Selective Powder Deposition and Digital Selective Powder Deposition*

Selective Powder Deposition (SPD) and Digital Selective Powder Deposition (DSPD) are both PB 3D printing techniques that use powder materials and a printer head to create the solid.

The SPD is a method of PB 3D printing that employs deposition of powder material and the support material with high precision to create an object using a digital model of the desired shape, created with CAD software. This model is then divided into thin cross-sectional layers, and powder material such as metal or plastic is selectively deposited on a substrate based on each layer's design. This is usually accomplished by a printhead or a similar device that regulates the flow of the powder material.

In addition, DSPD, uses a powder jet printer head to selectively deposit a layer of powder onto a build platform. Once the layer is deposited, a binder material is jetted onto the powder in a pattern that corresponds to the desired part geometry. The binder material selectively binds the powder particles together, creating the solid layer. The build platform is then lowered, and the action repeats until reach the final part.

Another difference between SPD and DSPD is the type of materials that can be used. SPD is typically used for printing with ceramics and metals, while DSPD can be used for printing with composites polymers, ceramics, cement, geopolymers, among others.

In terms of resolution and accuracy, SPD offers higher resolution and accuracy compared to DSPD. Although SPD can create complex geometries and intricate

details with high precision, DSPD can be more appropriated for printing with certain types of materials, which may not be compatible with SPD.

Concluding, both SPD and DSPD are effective PB techniques that offer different advantages and may be more or less applicational suitable, depending on the specific material requirements and part characteristics.

## 3. *Application of DSPD methods, based on Iro3D machine*

Based on the production methods currently available on the market, additive manufacturing processes using PB methods demonstrate great differentials in the spheres of production of parts with complex shapes and enable a reduction in material waste [2].

In the current market, there are already equipment that work using this process. An example of this equipment, which through the combination of PB and material jetting (MJ) methods [1], the Iro3D machine shown in Figure 1 acts by depositing materials selectively [3], presenting the opportunity to work with various materials [4]. To complement the study, the concept of Multiple Materials Additive Manufacturing (MMAM) [5] and Dry Powder Printing (DPP), a technique for depositing fine powders, is added. In this method, the deposition dosage of the material occurs through ultrasonic vibration and a piezoelectric transducer, having significant advantages to deposit fine powders in a uniform and controllable way and possibility to deal with a wide variety of powder materials. Powder is dispensed by a vibration-assisted system using a glass nozzle as a funnel and a computer control system where drops of powder are discharged directly from the dispenser [6].



Fig. 1: Iro3D machine. Process of SPD through PB method.

In addition to being a PB-based process, Iro3D selectively deposits the powders in the areas delimited by the G-code, thus building the part, following the Drop-on-Demand method [6], represented in Figure 2.

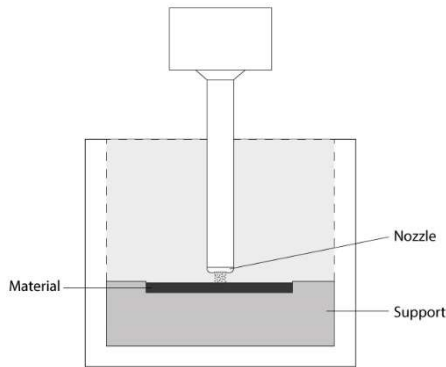


Fig. 2: Drop-on-Demand process, deposits the material precisely in the areas delimited for the construction of the support and part.

Developed by Sergey Singov, the Iro3D machine uses the concept of SPD in PB to print metal parts in crucibles and then take them to the oven to be baked, where a compounding process can also take place. Having four distinct compartments for the main and support materials, illustrated in Figure 3, these components are also connected to four nozzles, which deposit the materials according to the codes defined for the trajectory by the slicer software [7], presented in Figure 4. The machine demonstrated a promising potential in the sphere of development of parts purely or with metallic compounds.

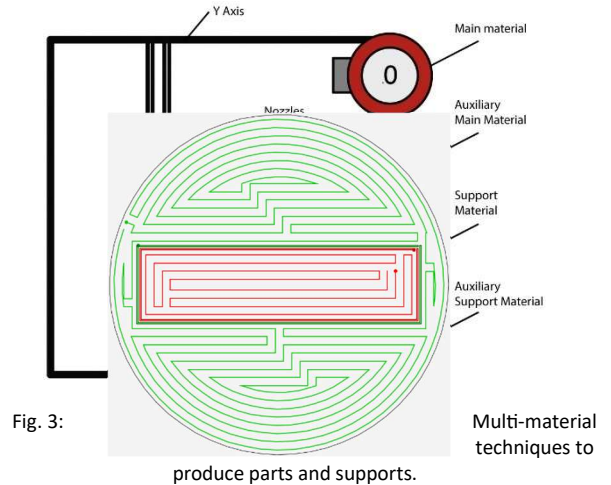


Fig. 3:

Fig. 4: In green: the trajectory calculated to deposit the support material. In red: the trajectory for depositing the final part material.

### 3.1. Unconventional materials: ceramics, glass and basalt

To expand the concepts and possibilities for the use of SPD, the use of non-conventional materials was experimented to produce shapes and samples in an exploratory and protocolized way using the sintering process. Using Iro3D and the PB process, ceramic materials such as stoneware, earthenware and porcelain were evaluated, as well as glass and basalt, exemplified in Figure 5.



Fig. 5: Production examples in porcelain, glass and basalt, in respective order from left to right.

The ceramics demonstrated relevant initial results. Figure 6 shows that after being baked in the kiln, the ceramic granules were able to maintain their grouping after removal from the mould, preserving their designed shape, with fragile yet precise resistance. Hand-milled, the ceramic powders showed a great ability to absorb moisture, tending to agglomerate into larger buds, sometimes causing clogging in the nozzle exit hole. To get around this problem, resting the powder in an oven and the use of industrial granules, improved the powder flow because they have more rounded granules that reduce agglomeration [7].

The application of vibration also enhanced the flow performance, being proved to be an effective reinforcement in the process.



Fig. 6: Experimental parts of ceramic materials, generated by SPD method in Iro3D.

In the case of basalt and glass, the granules flowed more efficiently, with the part preserving its shape considerably, after being baked in the oven. The behavior of basalt is similar to glass, considering its composition based on studies carried out by the American Space Agency NASA with regolith and volcanic glass [8]. The sintering process was effective, however, resulting in parts that were more resistant and viable than ceramics, with a slight change in shape, as shown in Figures 7 and 8.



Fig. 7: Experimental glass part, generated by SPD method in Iro3D.



Fig. 8: Experimental basalt part, also generated in Iro3D by SPD method.

### 3.2. Expanding beyond limits: cement and geopolymers

For the production of cement parts, a mixture of cement, sand and stone powder was used, with fine sand as support material. The proportion adopted was 20% of cement in the mixture.

In this case, aiming to explore the behavior of the materials and their viability for the process, the DSPD method was simulated manually, in order to avoid complications generated by humidity with the use of Iro3D. A rectangular acrylic container with holes was chosen for draining the water that will be applied later. For the deposition of materials, a first layer of sand was introduced as a support base for construction.

In the second layer, a mold is introduced, and the support sand is placed around it, to keep the mold fixed in position and finally, the filling with the cement is made.

Carefully, the mold is gently removed from the sand, always taking care so that the shape of the part does not distort during the action. After filling the mold and the support sand, the cover layer is included, being just sand, to involve the cement, finalizing the printing process.

It's time to introduce the water. The cement and sand tend little by little to be involved by the water and the reaction begins. It is at this moment that the vibrator is used. With the cement and sand covered in water, the vibration causes the particles to settle in a structurally organized way, in order to improve the connection between the particles, with the aim of creating a more solid and resistant part.

The acrylic crucible has holes in its base, to allow the water to drain later. While the construction process is underway, these holes are sealed with cling film or a rubber sheet to prevent materials from leaking. After the vibration process is finished, the holes are opened so that excess water can drain.

Finally, the container was left to rest for 4 days, until all the excess water had drained and the cement had solidified. After the established resting period, the sand is still wet, but most of the water has drained, the cement has hardened and can be removed from the container.



Fig. 9: Part of a specimen using cement simulating the DSPD process.

The formed part remains wet for two more days until it dries completely. As it is sand, the texture of the support material was reproduced on the surface of the part. In Figure 9 we can see the part in its final form, after removing the sand and drying.

In geopolymers, the same process was carried out in order to evaluate the usability of this material, using a solution of sodium silicate and sodium hydroxide as activating and solidifying reagent, the result is shown in Figure 10. Another objective of this study is to observe the behavior of the geopolymer, being a mixture of metakaolin and sand, also exposed to tests with the application of water to serve as an activator, seeking to evaluate its use as a substitute for the chemical solution.



Fig. 10: Part of a specimen using geopolymer with activator solution, simulating the DSPD process.

#### 4. Application of SPD and DSPD process through PB on a large scale

Based on existing concepts, a project was designed to improve this process, seeking to develop equipment that could, in addition to exploring new materials, build parts on a large scale. The scope of the project is to develop the equipment so that it can reproduce the DSPD process, selectively applying it in the delimited areas. Unlike the strategy used by Iro3D, the use of a descendant platform like those used in the SLS and EBM processes is proposed. For this, two hypotheses are suggested, being an endless screw or a scissor lift, as shown in Figure 11, which would be to enable the Z axis, going down to each layer, depending on its height, or delimited printing resolution. For the deposition of materials, two strategies for the mechanism would also be addressed, the first being that of a single nozzle for each material, similar to that seen in the Iro3D, which would act in the deposition of the powders alternately or jointly, covering the platform guided by the trajectories delimited by the slicing software.

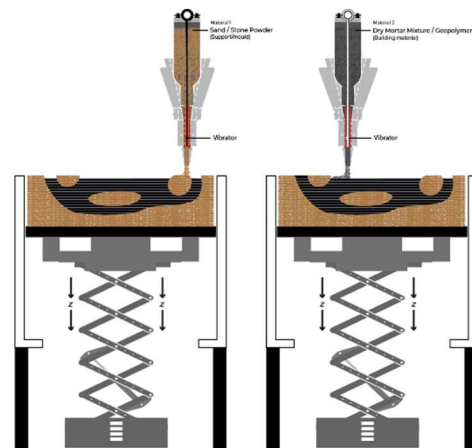


Fig. 11: Illustration of proposed large-scale DSPD process.

Another option is to use a comb-shaped mechanism, containing several nozzles that would work simultaneously, reducing the size of the mechanism, the space required for the deposition component and increasing the construction speed, compared to the first individual approach. In the second strategy, it would be possible to provide conditions to produce parts with even larger sizes. It is shown illustrations of these approaches in Figures 12-14.

To aid the initial solidification of each deposited material, the introduction of water or activator solution can be applied to each layer, increasing the

performance of the construction process. In addition, a compression mechanism can be deployed, acting at each layer or a certain number of layers, taking care not to move the position of the powder granules. The application of heat can also be tested, through a light power resistance, coupled to the deposition mechanism, acting as the layer construction takes place.

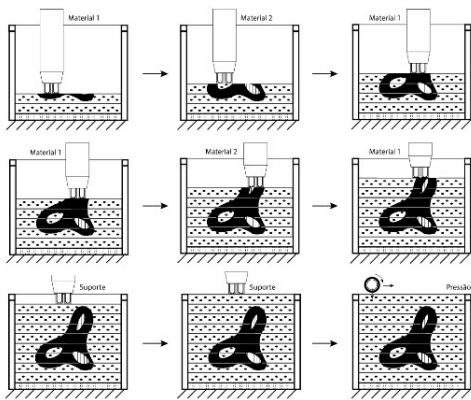


Fig. 12: Single depositor hypothesis.

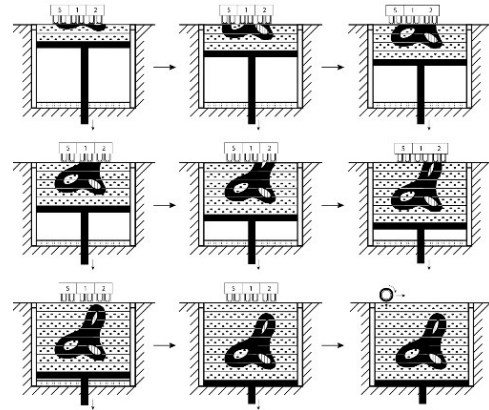


Fig. 13: Hypothesis of several comb-shaped deposits.

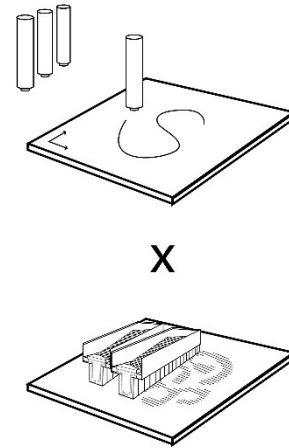


Fig. 14: Comparison of deposition strategies.

## Conclusion

In conclusion, both SPD and DSPD are advanced 3D printing techniques that use powder materials and printhead technology to create objects with high precision. These techniques offer advantages and disadvantages depending on the material and shape parameters. Additionally, SPD offers higher resolution and accuracy, while DSPD can accommodate new kinds of materials that may

expand the range of SPD performance. In the same way, DSPD can be adaptive, with several strategies, concerning the materials requirements such as heat or binder application, compression and vibration. However, the acquired results shown in this article, demonstrate that the two techniques presented good perspective in the area of industrial production of parts by additive manufacturing, open to be explored to better innovations in the future.

## Acknowledgements:

This work is supported by the FCT (Fundação para a Ciência e a Tecnologia) support of CDRSP-IPLeiria (Strategic Projects UIDB/04044/2020 and UIDP/04044/2020; and Associate Laboratory ARISE LA/P/0112/2020); and IAPMEI, through the Agenda Mobilizadora INOV.AM – Inovação em Fabricação Aditiva; and finally, IPLeiria (Institute Polytechnic of Leiria)



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# QUALITATIVE ANALYSIS OF THE LIMESTONE WASTE POTENTIAL FROM THE SERRA DE AIRE E CANDEEIROS QUARRIES

S. M. C. S. Monteiro<sup>1</sup>, M. Jorge<sup>2</sup>, N. S. Oliveira<sup>3\*</sup>, M. L. Alves<sup>4</sup>, A. Veiga<sup>5</sup>, A. Silva<sup>6</sup>

- (1) ESTG, Polytechnic Institute of Leiria, Leiria; LSRE-LCM - Laboratory of Separation and Reaction Engineering, Polytechnic Institute of Leiria; ALiCE - Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Portugal, [nelson.oliveira@ipleiria.pt](mailto:nelson.oliveira@ipleiria.pt)
- (2) School of Technology and Management, Polytechnic Institute of Leiria, Leiria, Portugal.
- (3) ESTG, Polytechnic Institute of Leiria, Leiria; LSRE-LCM - Laboratory of Separation and Reaction Engineering, Polytechnic Institute of Leiria; ALiCE - Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Portugal.
- (4) ESTG, Polytechnic Institute of Leiria, Leiria; INESC Coimbra - Institute for Systems Engineering and Computers at Coimbra, Polytechnic Institute of Leiria, Portugal
- (5) CGEO – Geosciences Center, University of Coimbra, Portugal
- (6) CEFAGE-UE, Center for Advanced Studies in Management and Economics, Évora; MARE, Marine and Environmental Sciences Centre, Polytechnic Institute of Leiria; CIIC, Computer Science and Communication Research Centre, Polytechnic Institute of Leiria, Portugal

**Summary:** *The mountains of Aires and Candeeiros are part of the Maciço Calcário Estremenho where the landscape is predominantly made up of limestone. The limestone explored in the region has been the driver of social and cultural development, contributing significantly to the local economy. This emerging concern has motivated public and private organisations to develop solutions to improve the sector's sustainability. The present work aims to evaluate the limestone waste potential to produce precipitated (PCC) and ground (GCC) calcium carbonate in the region by surveying the local extraction industries that fulfil the defined minimum requirements to produce high quality PCC and GCC. The results allowed to establish an estimate of the amount of waste resulting from the extraction process. The conversion of these wastes into PCC or GCC constitutes a possibility of recovery into by-products, allowing use in noble and profitable applications and contributing to the circular economy and the sustainability of the sector.*

**Key words:** *Limestone Waste, Precipitated Calcium Carbonate, Ground Calcium Carbonate*

## Introduction

The study area is located in the south-central region of Portugal (Santarem district), more specifically in the Serras d'Aire and Candeeiros Natural Park. From a geomorphological point of view, the area belongs to the Maciço Calcário Estremenho (MCE), an over-elevated geomorphological unit as a result of Cenozoic compressive tectonics, where geological materials of ages ranging from the lower Jurassic to the Cretaceous outcrop, predominantly carbonate formations of the middle and upper Jurassic (Carvalho *et al.*, 2012).

In the study area, limestone rocks of the middle Jurassic predominate, constituting layers of great thickness, revealing themselves of great interest for the exploitation of rock for ornamental purposes.

The extractive industry has been going on intensively since the beginning of the 80s of last century, comprising blocks of 3 to 5 cubic metres, which allow the

transformation into slabs of variable thickness, up to smaller fragments such as slabs and blocks for paving.

The limestone explored in the region has been the driver of social and cultural development, contributing significantly to the local economy. However, the environmental impacts of limestone extraction and transformation activities must be addressed.

Because of these assumptions, it is essential to reconcile the extractive industry with nature conservation, studying the management of waste generated and its economic value.

## Characterisation of the region under study

According to the information contained in the Geoportál Energy and Geology of the National Laboratory of Energy and Geology (LNEG), in the MCE, which includes the Aire

and Candeeiros Mountains, the region under study, there are several ornamental rock quarries, limestones, with a high percentage of calcium carbonate in their chemical composition, mostly calcite (LNEG, 2020-2023).

The region under study and the existing ornamental rock quarries in it, are represented in Figures 1 and 2.

### Methodology for the quarries selection

Figure 2 presents ten quarries in the region under study where the extraction of sedimentary rock is carried out and from which it is possible to extract rocks with a high percentage of calcium carbonate ( $\text{CaCO}_3$ ) in their composition. The percentage of  $\text{CaCO}_3$  in the extracted rock will be a condition for industrial application. The sedimentary rock of this region is rich in limestones and dolomites, which are among the rocks with the highest percentage of  $\text{CaCO}_3$ , so its commercialisation is relatively high.

The existing  $\text{CaCO}_3$  in the extracted limestone can be industrially applied through its crushing to finer granulometry, originating the so-called ground calcium carbonate (GCC) or through its precipitation, which allows obtaining fine or very fine calcium carbonate nanoparticles produced from high-purity carbonate rocks, originating the precipitated calcium carbonate (PCC), also known as purified or synthetic calcium carbonate.

The production sequence of PCC is based on three chemical processes: calcination of limestone, hydration of calcium oxide and precipitation of calcium carbonate.

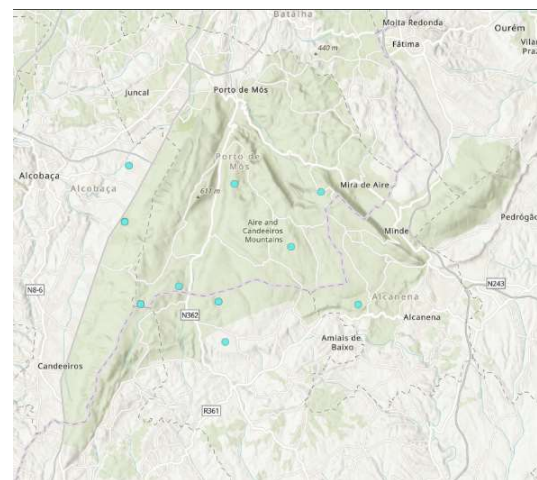
The PCC and GCC have the same chemical composition, even if PCC is purer than the GCC, usually with a lower percentage of silica, magnesium, and lead. Although GCC is widely used because it is produced more simply (without chemical processes), its market price per ton is much lower than that of PCC, whose industrial applications are more restricted, although nobler.

The difference in applications of GCC and PCC is fundamentally based on the difference in their properties, as illustrated in Table I.



Fig. 1. Location of the existing ornamental rock quarries in Portugal: ● metamorphic, ● sedimentary, and ● igneous rocks. (Data from LNEG, 2020-2023)

Fig. 2. Location of the existing ornamental rock quarries in the region



under study, the Aire and Candeeiros Mountains: ● sedimentary rocks. (Data from LNEG, 2020-2023).

Table I – GCC and PCC properties.

Property	GCC	PCC	References
Particles shape	Irregular shape of rhombohedral particles	Uniform shape of calcite polymorphs	Jimoh <i>et al.</i> , 2018; Mantilaka <i>et al.</i> , 2013; Chen & Nan, 2011
Particle size	Uniform	Non-uniform	El Sherbiny <i>et al.</i> , 2015
Smoothness	High	Low	D'Haese <i>et al.</i> , 2013
Abrasion	Low abrasion	High abrasion	
Porosity	Lower	Higher	
Specific contact area	Lower	Higher	
Chemical absorption	Lower	Higher	
Brightness	Lower	Higher	Hubbe and Gill, 2016
Opacity	Lower	Higher	
Purity	Lower	Higher	

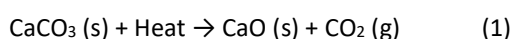
From an economic perspective, the minimum quantity of calcium carbonate in limestone to produce PCC can vary depending on several factors, such as the purity of the limestone, the cost of raw material, the cost of energy, the production efficiency, and the market demand for PCC.

Typically, limestone contains at least 90% calcium carbonate, but the percentage can vary from deposit to deposit. To produce PCC, the calcium carbonate in limestone is typically chemically processed through a precipitation reaction with other chemicals, such as calcium hydroxide and carbon dioxide.

Generally, the higher the percentage of calcium carbonate in the limestone, the more efficient the production process will be, leading to lower production costs and higher profitability (Minerals Technologies Inc., s.d.)

Most of the impurities present in GCC are silicates, resulting in a purity of around 98%, while PCC is almost free of impurities, thus reaching a purity of 99.99 % (Carreira, 2021) (Duarte, 2021).

In the calcination of limestone, calcium carbonate is decomposed into calcium oxide and carbon dioxide by heating the limestone. The calcium oxide is also known as quicklime, burned lime or unslaked lime, and the process is expressed as follows (Teir *et al.*, 2005):



During this process, the heating of the limestone releases carbon dioxide and produces calcium oxide, known as quicklime, which usually occurs at temperatures around 1000 °C, being an endothermic reaction.

To determine the quantity of calcium carbonate in limestone, it can be used several analytical methods, such as titration of the unreacted hydrochloric acid with sodium hydroxide after digestion of limestone with hydrochloric acid; Attenuated total reflectance Fourier transform infrared (ATR/FT-IR) spectrometry; X-ray Diffraction (XRD); analysis of the Lost on Ignition (LOI) by Thermogravimetric Analysis (TGA); chemical analysis of calcium oxide after ignition at 1000 °C. Based on the results of the LOI, calcium oxide and magnesium oxide content presented in the database of the geoportal of LNEG (LNEG, 2020-2023), the percentage of calcium carbonate in limestone was estimated. The determination of calcium carbonate by the LOI assumes that the limestone was previously dried, and the weight LOI is related to the loss of carbon dioxide from the calcination of calcium carbonate and magnesium carbonate present in the limestone. The percentage of calcium carbonate calculated based on the LOI is achieved according to equation (2):

$$\% \text{CaCO}_3 = \left( \frac{\text{LOI}}{M(\text{CO}_2)} - \frac{\% \text{MgO}}{M(\text{MgO})} \right) M(\text{CaCO}_3) \quad (2)$$

Where  $M(\text{CaCO}_3)$ ,  $M(\text{CO}_2)$  and  $M(\text{MgO})$  are the molecular weight of calcium carbonate, carbon dioxide and magnesium carbonate, respectively. The content of calcium oxide and magnesium oxide in the chemical analysis can also be used to estimate the calcium carbonate content, assuming that all calcium of the calcium oxide originated from the calcium carbonate of limestone. Equation (3) presents the determination of calcium carbonate based on the calcium oxide content in the chemical analysis of limestone.

$$\% \text{CaCO}_3 = \left( \frac{\% \text{CaO} \times}{M(\text{CaO})} - \frac{\% \text{MgO}}{M(\text{MgO})} \right) M(\text{CaCO}_3) \quad (3)$$

Where  $M(CaO)$  is the molecular weight of calcium oxide, based on the assumptions of equation (2) and equation (3), the real calcium carbonate content in limestone is between both results.

Regarding the purity levels of  $CaCO_3$  most suitable for the production of PCC, the values are above 98%, so the CaO values resulting from its precipitation reach levels above 55%.

Typically, the purity level of  $CaCO_3$  in commercial PCC is above 99 %. According to this purity and priority to the extraction of calcite, it was defined that the limestone should contain at least 98,2 % of  $CaCO_3$ , corresponding to at least 55 % of CaO in the chemical analysis of the limestone. For the highest percentage of  $CaCO_3$  (100 %), a maximum value of CaO is achieved according to the above equation, namely, 56%.

This minimum value was used as the basis for selecting the limestone commercial names according to the LNEG catalogue of Portuguese Ornamental Rocks selected within the scope of this study and which are considered as having the most significant potential for PCC production.

Therefore, from among the limestone commercial designations explored in the ten identified quarries belonging to the Serra de Aire e Candeeiros region, whose CaO percentage is higher than 55%, obtained through precipitation of  $CaCO_3$ , the following limestone commercial designations were identified in Table II.

In the study region, there is significant variability of limestones in terms of texture, colour and chemical composition leading to different commercial designations. Thus, there are quarries where ornamental limestones with different commercial names are exploited. Figure 3 identified, for each quarry, which of the explored rocks presents the most significant potential for PCC production.

Among the various companies exploiting the above-mentioned raw materials, some exploit only a few varieties, while other companies related to the quarries only focus on its transformation (Table III). These quarries are concentrated in five fundamental extraction poles.

Table II – Commercial designation of limestone in the region and the percentage of CaO and  $CaCO_3$ . Data from LNEG, 2020-2023

Commercial Designation	CaO (%)	CaCO <sub>3</sub> (%)
Vidraço Moleanos	55,69	99,4
Semi-rijo do Codaçal	55,37	98,8
Moca Creme	55,86	99,7
Moleanos Rijo	55,7	99,4
Semi-Rijo Cabeça de Veada	55,64	99,3
Semi-Rijo do Arrimal/Branco do Mar	55,91	99,8
Olho de Sapo	55,24	98,6
Moleanos Macio	55,76	99,5
Banco do Fundo	55,10	98,3
Moleanos Azul/Azul Moleanos	55,58	99,2

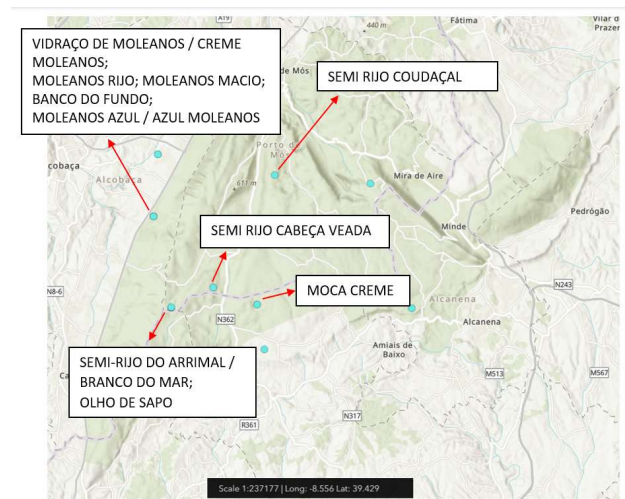


Fig. 3. Limestone quarries in the region by the commercial designation of limestone. (Data from LNEG, 2020-2023)

### Data collection proposal

The importance of data on quarries allows a characterisation of the exploitation of these resources at a national level. The data on quarries are compiled on the Direção-Geral de Energia e Geologia (DGEG) website from 1999 to 2021, where for the sub-sector corresponding to Ornamental Rocks - Marble and Limestone, exploitations of 1927812 tons have been identified, with the respective exploitation cost of 92.06 M€ for the year 2021 (DGEG, s.d.).

In 2020, in continental Portugal, 122 quarries in activity were identified (Number of Establishments with Statistical Bulletins) dedicated to the exploitation of ornamental rocks - marble and limestone.

Table III – Commercial designation of limestone by each extraction pole.

Extraction Poles	Commercial Designation	Exploration Companies	References
Cabeça Veada, Mendiga	Cabeça Veada/Semi-Rijo	6	Six companies confirmed at LNEG*/DGEG**
Moleanos, Prazeres de Aljubarrota	Vidraço Moleanos/Creme Moleanos; Moleanos Rijo; Moleanos Macio; Moleanos Azul; Banco do Fundo	11	11 companies confirmed at LNEG*/DGEG**
Codaçal, Cerro Ventoso	Semi-Rijo do Codaçal	12	11 companies confirmed at LNEG*/DGEG**; 1 company, according to its own information
Pé da Pedreira, Alcanede	Moca Creme	29	28 companies confirmed at LNEG*/DGEG**; 1 company, according to its own information
Salgueira, Arrimal	Olho de Sapo; Semi Rijo do Arrimal/Branco do Mar	10	Five companies confirmed at LNEG*/DGEG**; 5 companies, according to its own information

\* Data obtained in the Geoportal-Energia e Geologia of LNEG (<https://geoportal.lneg.pt/>)

\*\* Data obtained in the Direção Geral de Energia e Geologia (DGEG) (<https://www.dgeg.gov.pt/>)

Collecting information on quarries is essential for characterising the natural resources obtained from them, as well as their mapping in the various national regions. The quarry statistics refer to information on licensed mineral mass establishments (quarries) which, each year, report information under Article 51 of Decree-Law 270/2001 of October 6th, amended and republished by Decree-Law 340/2007 of October 12th (Decree-Law defining the regime for the exploitation of mineral masses - quarries).

To carry out this statistical operation, the quarry concessionaires send the DGEG the statistical data by the end of April each year through the annual completion of the "Single Quarry Survey", a procedure carried out electronically in the DGEG's Online Services.

The "Single Quarry Survey" aims to collect the following information about the Quarries:

1. Identification; Table nº 1 - Extracted Substances (at the exit of the dismantling front); Table nº 2 - Commercial Substances (tradable and/or used for own consumption); Table nº 3 - Consuming Industry; Table nº 4 - Employment; Table nº 5 - Investments and Operational Expenses; Table nº 6 - Energy Consumed; Table nº 7 - Materials Consumed and Water; Table nº 8 - Technical Information concerning Operation; Table nº 9 - Equipment; Table nº 10 - Environmental and Landscape Recovery Plan; Table nº 11 - Waste Incineration Plant; Table nº 12 - Backfilling; Annexes - Plants and topographic cuts in digital format; Concluding remarks.

Even so, this information must reach the detail required for a more in-depth study to characterise the region under study, the MCE. Issues related to the exploitation by the companies of the region corresponding to very different realities, both in terms of the extraction procedure, raw materials extracted, by-products produced by them inherent to the extraction process and the way these by-products are treated. Table IV shows some of these issues.

Table IV – Survey of the companies that explore quarries.

Q.1	How many quarries are operated by the companies?
Q.2	Which varieties are exploited by the companies?
Q.3	What is the approximate total mass of ornamental rocks (limestone) extracted (in tons) (in particular if they are first-choice blocks, second-choice blocks, or material sent to crushing or to other destinations)?
Q.4	In particular, in the first and second choice blocks, which are processed by the company itself, by other companies or are destined for export?
Q.5	If intended for export, identify the total mass exported.
Q.6	What by-products generated in the production process constitute chips from the cutting process, remains of blocks and non-viable blocks, carsified material or other types, and the corresponding total masses?
Q.7	Regarding these by-products, identification of which are sent for crushing by other companies or within the company itself, which are used as filler in the quarry, for rehabilitation of other quarries, or for other destinations.

As part of the present exploratory work, a survey of the various companies exploiting the quarries in the region under study is currently being developed/implemented, allowing us to fill some of these information gaps.

### Conclusions and final considerations

Among the ten limestone quarries in the Aires and Candeeiros mountains, 5 allow the extraction of different limestone commercial denominations, which present a high percentage of CaO and CaCO<sub>3</sub> in their chemical composition. It is estimated that these quarries generate a large amount of unused waste annually or are used for purposes of low economic value.

Although exact figures on the volume of stone waste generated are unknown, their high level of calcium carbonate purity allows us to conclude that they

present a potential for PCC and GCC production, contributing to the valorisation of these by-products.

Some governmental entities have already documented several data on the extractive process in Portuguese quarries, however, this information does not reach the detail required for a more in-depth study to characterise the region under study, in particular information that makes it possible to analyse the potential for recovery of the by-products produced in the quarries, both in qualitative and quantitative terms.

The by-products inherent to the productive process of extraction in the quarry can be subject to various destinations that allow them to add commercial value. This market-added value can be increased if its transformation/application is carried out within the quarry, which minimises logistics and transportation costs and environmental and social impacts, with the inherent promotion of environmental and energy sustainability.

The present research constitutes a decision support tool for companies that exploit raw materials in several quarries with high potential for PCC and GCC production and that intend to install new pilot plants to produce these materials in or near the quarries themselves.

**Acknowledgements:** The authors express appreciation to the StonebyPortugal- Plano de Recuperação e Resiliência «Agendas para A Inovação Empresarial», Portaria n.º 43-A/2022 January 19th, for the support on their work.

This work was financially supported by LA/P/0045/2020 (ALiCE), UIDB/50020/2020, and UIDP/50020/2020 (LSRE-LCM), funded by national funds through FCT/MCTES (PIDDAC), UIDB/00308/2020 (INESCC) and UIDB/00073/2020.

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# SUSTAINABILITY STUDY OF LIMESTONE QUARRY WASTE INTO VALUE-ADDED PRODUCTS: PCC AND GCC

S. M. C. S. Monteiro<sup>1</sup>, M. Jorge<sup>2</sup>, M. L. Heleno<sup>2</sup>, N. S. Oliveira<sup>3\*</sup>, M. L. Alves<sup>4</sup>, A. Veiga<sup>5</sup>, A. Silva<sup>6</sup>

(7) ESTG, Polytechnic Institute of Leiria, Leiria; LSRE-LCM - Laboratory of Separation and Reaction Engineering, Polytechnic Institute of Leiria; ALiCE - Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Portugal, [nelson.oliveira@ipleiria.pt](mailto:nelson.oliveira@ipleiria.pt)

(8) School of Technology and Management, Polytechnic Institute of Leiria, Leiria, Portugal.

(9) ESTG, Polytechnic Institute of Leiria, Leiria; LSRE-LCM - Laboratory of Separation and Reaction Engineering, Polytechnic Institute of Leiria; ALiCE - Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Portugal.

(10) ESTG, Polytechnic Institute of Leiria, Leiria; INESC Coimbra - Institute for Systems Engineering and Computers at Coimbra, Polytechnic Institute of Leiria, Portugal

(11) CGEO – Geosciences Center, University of Coimbra, Portugal

(12) CEFAGE-UE, Center for Advanced Studies in Management and Economics, Évora; MARE, Marine and Environmental Sciences Centre, Polytechnic Institute of Leiria; CIIC, Computer Science and Communication Research Centre, Polytechnic Institute of Leiria, Portugal

**Summary:** *Calcium carbonate can be obtained naturally from limestone, chalk, marble, and other sedimentary rock forms. Ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC) are two materials that can be produced from natural calcium carbonate. The future of GCC and PCC is promising since they are linked to industries with high demand, such as packaging, building & construction, transportation, and industrial applications, with an expected compound annual growth rate higher than 4% until 2027. This research focuses on the production of GCC and PCC through a comparative analysis that identifies the macro conditions that become advantageous to produce and commercialising PCC in a quarry context. This allows a valorisation of the limestone waste resulting from the extraction operations, converting it into by-products of the process. Not all existing limestone quarries in the Serra de Aire e Candeeiros region are suitable sources of raw material to produce PCC. The five extraction poles were identified with the potential for extracting suitable raw materials, associated with several companies dedicated to the extraction operation in these quarries.*

**Keywords:** *Precipitated calcium carbonate, Ground calcium carbonate, Limestone waste*

## Introduction

References to calcium carbonate are extensive and diverse, with a long commercial history. Among the earliest records associated with human activity is using calcium carbonate in prehistoric cave paintings between 40,000 and 10,000 BC. In the Middle Ages, chalk was applied to the land as a fertiliser - a practice still routinely carried out by farmers today. Regular calcium carbonate applications help reduce soil acidity and help optimise conditions for crop growth. With the industrial revolution, the commercial applications of calcium carbonate grew in quantity and diversity, prompting the industrial production of precipitated calcium carbonates (PCC) to complement natural calcium carbonate (GCC). PCC was industrially produced and commercialised for the first time in 1841 by the English company John E. Sturge Ltd, called precipitated chalk. This new product, which has a high purity and uniform grain size, was used

in the 19th century as an additive in paints, building materials, cleaning agents and the glass industry. In the 20th century, parallel to the invention of modern toothpaste, PCC was fundamental in the development of several industries, such as the paper industry, adhesives and sealants industry, plastics and rubber industry, paints industry, food and pharmaceutical industry (Erdogan & Eken, 2017) (BICCF, 2023) (STT, 2023).

## Extractive industry waste and by-products

Waste produced by the extractive industry can be divided into two main types: mining waste and non-mining waste resulting from ancillary operations to the extractive activity. Mining waste results directly from quarrying activities, such as extraction, treatment or transformation. Mining waste is produced in large quantities in the case of ornamental rock, up to 3 m<sup>3</sup> of waste is produced for each m<sup>3</sup> of marketable

ornamental rock (Assimagra, 2015). Other authors refer that in the marble industry, waste can represent 80-90% of the total soil and stone extracted (ANIET, 2018).

This waste is mainly produced at the blasting front, namely as a consequence of block chips and non-viable material from the fall of the carving. Currently, the different extractive industries have "exclusive" strategies that aim to reduce the amount of this waste, betting on procedures associated with the fall of the carving, allowing to achieve profitability of almost 50%.

In the scope of the environmental impact statement concerning the integrated project of the Pé de Pedreira Quarrying Nucleus, corresponding to an area of 391 hectares, 32 ornamental limestone exploration companies were considered. The ornamental limestone reserves in this cluster are estimated at 59 533 400 m<sup>3</sup>, the usable reserves about 50%, or 29 766 700 m<sup>3</sup> (APA, 2018).

Considering the physical characteristics of these residues, they can be divided into (1) soils and rocks (vegetable land and materials resulting from karstification), (2) unfeasible stone blocks (stones of variable dimensions without ornamental aptitude, produced in large quantities on the dismantling fronts), and (3) sludge (Assimagra, 2015).

Improving performance in the management of extraction residues must involve the prevention, reduction and recovery of these residues. Considering its definitive deposition in heaps, the disposal should be the last option, justified only when its recovery is technically and financially unviable. In Table I are the areas of specific intervention (AI), the area intervened by quarrying (AQ), the volume of existing extraction waste in heaps (VW) and the relationship between the area of heaps and the area intervened by quarries (R) of 475 heaps in the five areas of specific intervention (Assimagra, 2015).

The R value for the Moleanos-specific intervention areas is much lower due to the integrated waste management solution near these specific intervention areas, like the *Vale Grande* landfill. The locations of the heaps, such as the accesses for transporting the waste, should have considered factors such as the natural heritage, landscape impacts and good management of the mineral resource. Allied with the fact that there are high volumes of waste from extraction in the heaps, there is a significant proliferation of heaps, some of the oversized dimensions, and this deposition deficiency occurs without any separation by typology (land or blocks) and any concern in terms of security or land use (Assimagra, 2015).

Table I. Characterization of the heaps existing in the areas of specific intervention (Assimagra, 2015).

Extraction Poles	AI (ha)	AQ (ha)	VW (m <sup>3</sup> )	R (%)
Moleanos	147	17,0	95 700	11
Protela das Salgueiras	63	45,5	138 700	16
Pé da Pedreira	1374	510,4	3 402 000	30
Cabeça Veada	29	20,3	252 700	31
Codaçal	98	71,8	898 940	33

Reducing or preventing waste production is part of the circular economy strategy, which should increasingly be a priority for this sector. The promotion and implementation of new technologies associated with the procedures at the blasting front in order to obtain the most significant possible number of viable blocks, such as using unfeasible materials to cushion the fall of the cut, and replacing explosives, are examples of good practices in this scope of waste management.

Valorisation is already a current practice, namely with the forwarding of the raw material to other industries, such as the production of lime and aggregates for supplying the construction industry and public works for the cement industry. For each area of a specific intervention, there are currently several mobiles and fixed crushing facilities, such as crushing and screening for the production of gabion, riprap, *tout-venant*, stone powder, rice grain and gravel. In 2015, the installed capacity for aggregate limestone production was 603 000 t/year. However, valorisation still has great potential (Assimagra, 2015).

The forwarding of extraction waste for the production of GCC and PCC is also considered a valorisation alternative, which presents a higher added value. Furthermore, the deposition of the finer extraction material to fill the excavation voids, within the scope of the landscape recovery of the quarry, is understood in the Extraction Waste Management Plan prepared by the Portuguese Association of the Mineral Resources Industry as a waste recovery operation. This raw material is considered a by-product in these cases (Assimagra, 2015).

The law on the general regime for waste management, Decree-Law 102-D/2020 of 10 December, establishes the requirements for substances or objects resulting from a production process to be considered as by-products and not waste, any substances or objects resulting from a production process whose main

objective is not its production, and an application must be submitted to the National Waste Authority, individually or through the respective sector association.

In parallel, the incorporation of ornamental stone waste in the sector itself or other sectors is an environmentally correct way of managing that waste, promoting circular economy strategies and industrial symbiosis, where a "waste" of an industry "transforms" into "secondary raw material" of another industry, contributing to sustainability and environmental preservation.

### Precipitated calcium carbonates (PCC) and ground calcium carbonate (GCC)

There are two sources of calcium carbonate used in different industrial applications, namely ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC). Both GCC and PCC can be obtained from limestone, a common sedimentary rock composed mainly of the mineral calcium carbonate, calcite ( $\text{CaCO}_3$ ) and rarely contains more than 95%  $\text{CaCO}_3$ . Limestone constitutes approximately 10% of all rocks on Earth's surface (Parker, 1967).

Limestone is extracted from the Earth in varying quantities in calcite, aragonite, vaterite, limestone, chalk, marble or travertine. GCC is also known as natural calcium carbonate (NCC) as it is taken from nature and ground according to the desired granulometry, not involving any chemical transformation. GCC is also referred to as fine-ground calcium carbonate (FGCC) since current markets consume predominantly fine grains of calcium carbonate with sizes below 45  $\mu\text{m}$ .

The PCC can be defined as a dry, white, light, porous, odourless, tasteless powder with low oil absorption obtained by suspension or solution of hydrated lime or calcium salt by carbon dioxide or carbonate. It consists of chemically pure, micro-crystallised calcium carbonate in calcitic or aragonitic form, with a minimum of 90%  $\text{CaCO}_3$ . Generally, the purity of the PCC produced is greater than 99% and has a density of approximately 2.7  $\text{g/cm}^3$ .

The anhydrous  $\text{CaCO}_3$  can be classified as rhombic calcite, needle-like aragonite or spherical vaterite. The formation of any of these three polymorphs strictly depends on parameters such as the temperature, supersaturation and pH of the reaction solution (Erdogan & Eken, 2017) (Jimoh, Ariffin, Temitope, & Hussin, 2018). Calcite is considered the most stable phase under ambient atmospheric conditions.

There are different types of PCC morphology and particle sizes, each with different properties. PCC can be marketed in more than one grade by varying the particle size, particle size distribution, surface area, and particle morphology, among other characteristics, which may condition its use in different industrial applications (Jimoh, Ariffin, Temitope, & Hussin, 2018). Table II lists some properties of calcium carbonate relevant to its use in different industrial applications, whether GCC or PCC.

Table II. Typical properties of calcium carbonate (PCC and, or GCC) in different industrial applications

Industrial Applications	Purity ( $\text{CaCO}_3$ %)	Granulometry ( $\mu\text{m}$ )	Whiteness (%)	References
Glass	97.8	100 to 500		(Assimagra, 2015)
Paper	> 98	3 to 10 / 0.7 to 2	> 90	(Assimagra, 2015)
Ink	>97 to 98	3 to 10 / 0.7 to 2	<94	(Assimagra, 2015)
Paint		5-14 / 5-1		(Jimoh <i>et al.</i> , 2018)
Pharmaceutical	98.5 to 100	1 / 3	93	(Donnadio <i>et al.</i> , 2020)
Plastics	> 96	< 1.5	> 90	(Assimagra, 2015)
Adhesives and sealants		1-15/30-45		(Jimoh <i>et al.</i> , 2018)
Agri-food and food	> 99	5 / 100 to 500	> 95	(Assimagra, 2015)

Compared to GCC, PCC has better physical properties, including high brightness, opacity and purity. PCC has internal porosity, a higher specific area, and excellent chemical absorption and binding performance.

However, PCC has a high degree of aggregation, with several crystals growing together, forming a single particle. Particle size distribution is also more uniform than GCC, providing smoothness and low abrasion (Jimoh, Ariffin, Temitope, & Hussin, 2018).

## Marketing of GCC and PCC

The markets that consume fine grades of calcium carbonate, less than 45  $\mu\text{m}$  in particle size, are presented in Figure 1. These results relate to the world consumption of GCC and PCC in 2022. Asia is the highest regional world consumer of GCC and PCC, and China tops the world in PCC usage.

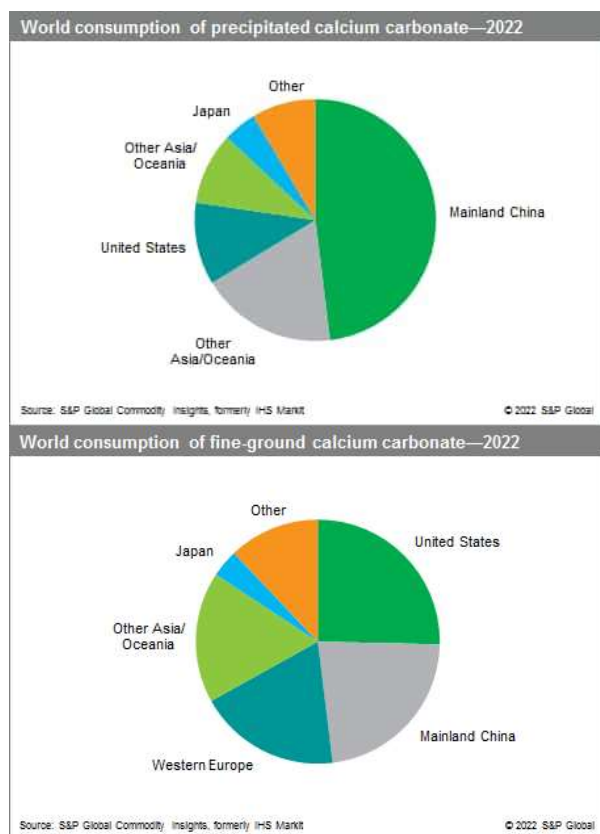


Fig. 1. World consumption of GCC and PCC in 2022 according to S&P Global Commodity Insights (Global, 2022).

It is possible to find data about the consumption of GCC and PCC in Europe in 2013 (Figure 2), verifying that the paper industry is the one that has the most impact on this market. This study also states that the growth of PCC consumption in Europe will depend on implementing new local PCC production units that are competitive in terms of costs with GCC producers (Jimoh, Ariffin, Temitope, & Hussin, 2018).

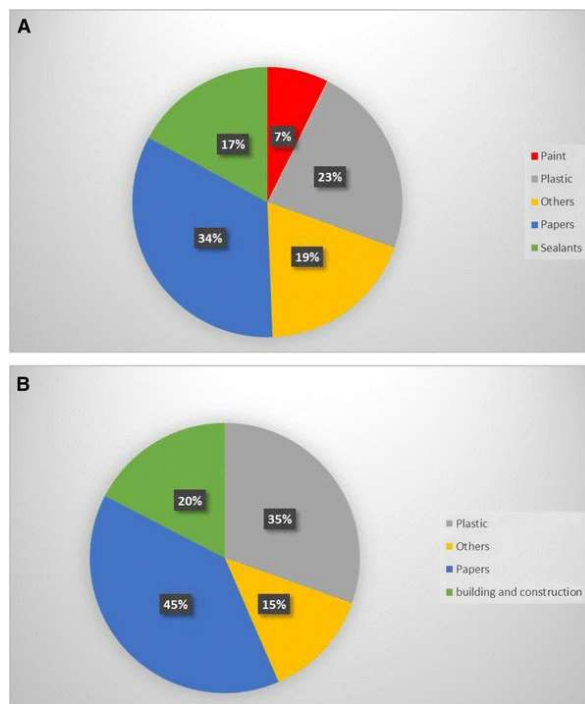


Fig. 2.: (A) Estimated global consumption of PCC by 2013 and (B) Estimated global consumption of GCC by 2013 (Jimoh, Ariffin, Temitope, & Hussin, 2018).

In the market for calcium carbonate in Western Europe, the GCC, which is produced locally, is of very high purity, and the competition between GCC and PCC is quite intense. There needs to be more extra-regional trade in GCC and PCC, as most material is sourced locally to avoid high transportation costs, especially for paper applications. The choice of GCC versus PCC in the paper industry depends on local market conditions and the cost of raw materials. In most regions, limestone, the mineral required for GCC and PCC production, is readily accessible. The quality of the limestone plays a significant role in the end quality of the GCC (Aresta & Eldik, 2013) (Global, 2022).

The 2021 annual report of “Mineral Technologies Inc.,” the world's largest PCC producer, reported PCC sales of USD 427M, a significant increase of about 13% on sales of USD 377.7M in 2020 (MTI, Mineral Technologies Inc, 2021). The paper industry is the primary recipient of this company's PCC products. However, the company also produce PCC products for sale to the polymer industry for use in automotive and constructions applications and to the adhesive and printing inks industries, to use by the food and pharmaceutical industries as a source of calcium in tablets and food applications, as a buffering agent in tablets and as a mild abrasive in toothpaste. Although this company also markets high-quality GCC, the turnover of this product is much lower (Figure 3).

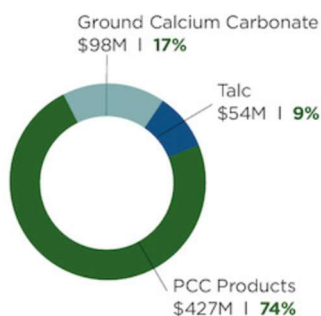


Fig. 3. 2022 PCC and GCC sales volume from one of the world's largest producers (MTI, Mineral Technologies Inc, 2021).

### Some applications of PCC and GCC

In food and foodstuffs industries, calcium carbonate is utilised not only because it provides the body system with an important nutrient (calcium), being used both in the human and animal food industry, but also useful as a conditioner in the prevention of caking in food powders. It also is found on many grocery store shelves in products such as baking powder, dry-mix dessert mixes, dough, and wine.

Apart from food products, precipitated calcium carbonates are also used to a great degree in dentifrices, particularly toothpaste, where they serve as both abrasives and fillers. PCCs are much less expensive when compared to other dentifrice abrasives, such as silica and di-calcium phosphate (Jimoh, Ariffin, Temitope, & Hussin, 2018).

In pharmaceutical applications, calcium carbonate is used widely as an effective dietary calcium supplement, antacid, phosphate binder, or base material for medicinal tablets. In drug formulation,  $\text{CaCO}_3$  is a widely chosen expedient because it is a natural, non-toxic biomineral, and it is a safe, cheap and eco-friendly excipient able to dissolve in an acidic environment. So  $\text{CaCO}_3$  affect the drug release properties, and porous calcium carbonate has been proposed as an excipient to improve the dissolution of poorly soluble drugs (Donnadio, Corneli, Ricci, Bini, & Ambrogi, 2020).

PCC of nanometer-sized with rhombohedral morphology is highly effective for coating on paper production. In the paper industry, PCC is used as a filler in producing different coated and uncoated papers and as a coating pigment for papers and packaging. Adding PCC fillers to papers reduces cost and increases functional roles such as optical properties, smoothness, ink adsorption, durability and sheet formation. So, The PCCs provide brightness, bulk, porosity, and smoothness to papers, improve their runnability and printability, and lower their costs (Erdogan & Eken, 2017) (BICCF, 2023) (MTI, Minerals Technologies Inc, 2023) (MTI, Minerals

Technologies Inc, 2023) (Jimoh, Ariffin, Temitope, & Hussin, 2018).

In the Plastics industry, PCC has been used as filler with the primary purpose of reducing costs. The addition of PCC as fillers in plastic can also lead to greater impact resistance since it increases elastic modulus and thermal conductivity, shortening the production cycle (Jimoh, Ariffin, Temitope, & Hussin, 2018). It also increases the impact strength when used as filler in PVC formulation.

To reduce the consumption of the expensive  $\text{TiO}_2$ , which is responsible for the white colour, PCC has been used in various paint formulations. A typical formulation can include micronised talc or calcined clay. However, GCC is most used as filler in paint, and PCC is used when high whiteness and brightness are required. PCC also gives the paint good weather resistance, and it is ideal for low-solvent paint due to its good dispensability in water-based systems. It is well suited for interior decorative low-gloss paint. About 10-30 % by volume of carbonate filler is typically incorporated into a basic solvent or water-based emulsion paint, depending on the final application and the sheen required (Jimoh, Ariffin, Temitope, & Hussin, 2018).

The nano-PCC is used to control the flow properties, to provide a body, and maintain dispersion. Typically, 5–14  $\mu\text{m}$  PCC is used in flat and semi-gloss paints, while 5–1  $\mu\text{m}$  ultrafine sizes may be used in gloss finishes to help adjust consistency and minimise paint sag (Jimoh, Ariffin, Temitope, & Hussin, 2018).

PCC is used as filler in liquid systems such as sealants and adhesives to control the shrinkage, sag, and thixotropic properties. It is also used to reduce cost reduction, rheology modification and strengthening.

### PCC and GCC in the Serra de Aire e Candeeiros

Not all existing limestone quarries in the Serra de Aire e Candeeiros region are suitable sources of raw material to produce PCC. Generally, the higher the percentage of calcium carbonate in the limestone, the more efficient the production process will be, leading to lower production costs and higher profitability.

Therefore, five extraction poles were identified with the potential for extracting suitable raw materials. These are associated with several companies dedicated to extraction operation in these quarries, whose  $\text{CaCO}_3$  percentage is higher than 98,5%. These values were estimated from the information in the database of the geoportal of LNEG (LNEG, 2020-2023).

The five extraction poles identified are:

- Moleanos

- Cabeça Veada
- Codaçal
- Pé da Pedreira
- Salgueira

From one of those companies exploring some of the identified quarries, 48% of limestone extraction is waste. The waste between 200-400 mm is forwarded for the lime industry, and the thinner waste is forwarded for the recuperation of the quarries. By analysing the data of Table I and the results of surveys, the volume of heaps is still increasing, and the aggregate, GCC and cement industry cannot absorb this limestone waste. This work identified just two companies with the capacity to produce PCC, each with the capacity to produce 180 kton of PCC. Those companies are integrated into the main Portuguese paper industry.

### **Conclusions and final considerations**

Although the limestone extractive industries have new extraction methodologies and technologies, and there are current practices for waste valorisation, the quantity of waste not valued is still notorious, considering the volume of waste deposited in heaps. This situation is a consequence of its recovery is technically unviable, and of the inability of the lime and crushing industry to absorb all the waste, which are currently the main destinations for the valorisation of this waste. It should also be noted that these two recovery alternatives are sometimes financially unviable because their economic valuation could be higher, often not compensating for transport costs, for example.

In this sense, it is urgent to find economically attractive alternatives, promoting that these wastes are classified as by-products intended to obtain value-added products. In this context, the production of PCC arises, as a response to the increased sustainability of limestone quarry waste into value-added products, giving a positive solution to the three pillars of sustainability: environmental, social, and economical.

The recovery of the main waste from the stone sector can be carried out in this sector or other sectors of economic activity, improving the efficiency of the management of mineral resources and mitigating the waste generated in the extraction and processing of ornamental stone, thus leading to the reduction of environmental impacts generated and contributing to the circular economy in the sector.

**Acknowledgements:** The authors express appreciation to the StonebyPortugal- Plano de Recuperação e Resiliência «Agendas para A Inovação Empresarial», Portaria n.º 43-A/2022 January 19th, for the support on their work. This work was financially supported by LA/P/0045/2020 (ALiCE), UIDB/50020/2020, and UIDP/50020/2020 (LSRE-LCM), funded by national funds through FCT/MCTES (PIDDAC), UIDB/00308/2020 (INESCC) and UIDB/00073/2020.

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## THE ROLE OF THE IUGS HERITAGE STONE SUBCOMMISSION ON THE PRESERVATION OF GEOLOGICAL LEGACY

V. Cárdenes<sup>1\*</sup>, G. Kaur<sup>2</sup>, A. Ehling<sup>3</sup>

- (1) Geology Department, Oviedo University, Spain. [cardenesvictor@uniovi.es](mailto:cardenesvictor@uniovi.es)
- (2) Geology Department, Panjab University, India.
- (3) Federal Institute for Geosciences and Natural Resources, Germany.

**Summary:** The use of stone to build permanent settlements can be traced back to the Neolithic, around 10.000 years B.C., when agriculture and livestock spread among the different human societies. It was during this crucial point of the history when human communities developed the art and techniques of extracting and working stones, and erecting buildings with them. Since then, each society has left its own architectural heritage, which explains and preserves the social and historical context of the past cultures. This important legacy has been profusely studied by academia, but practically always from an architectural standpoint. The stones that were used to build this heritage have been frequently forgotten, despite they represent an essential part of history. The outcrops, methods, social structure, traditions, tools and communities that grew around the stone culture are archeological and cultural objects that should be preserved.

In response to the growing concern of preserving this vast tradition, the IUGS Heritage Stone Task Group was established in the 34<sup>th</sup> International Geological Congress, held in Australia 2012. The aim of this group is to put into value building stones as essential elements of the social and architectural heritage of mankind. Since then, up to 32 stones have been recognized as Heritage Stone by the IUGS. This work gives an overview of the aims and tasks of the Heritage Stone Subcommittee, reviewing three of the designated Heritage Stones.



# DIGITALIZATION IN THE MINERAL RESOURCES INDUSTRY: CHALLENGES AND NEW DEVELOPMENT STRATEGIES

V. Francisco<sup>1\*</sup>, A. Fabre<sup>2</sup>

- (1) Technological Centre for Ceramic and Glass (CTCV), Coimbra, Portugal, \*Victor Francisco, [victor.francisco@ctcv.pt](mailto:victor.francisco@ctcv.pt)
- (2) Technological Centre for Ceramic and Glass (CTCV), Coimbra, Portugal, Arabela Fabre, [arabela.fabre@ctcv.pt](mailto:arabela.fabre@ctcv.pt)

**Summary:** *The work presented aims to contribute to support the digital transformation of SMEs in the Mineral Resources Industry, by sharing solutions and best practices*

**Key words:** *digital transformation, digital tools, industry 4.0,*

**The Challenge** | “Industry plays a major role in European Union's economy, representing 15% of added-value in the economy (compared to 12% in the USA). Industrial activity remains a key lever for the development of research, innovation, productivity, skilled job creation and exports” (Roland Berger Consultants, in *Jornal de Negócios*).

Digitalization emerges with the perspective of promoting better business performance in productivity, management practices, innovation, growth, qualification of employees and better-paid jobs. Incorporating digital transformation processes is therefore essential for companies to remain competitive.

The Mineral Resources Industry, like any other industrial sector, currently faces several challenges to proceeding with the industry 4.0 (i4.0) concept implementation. So, the main question behind this work was: **how can we support companies to establish their path in i4.0?**

To answer this question we need to know, on the one hand, what companies know about i4.0 and, on the other hand, in what stage of maturity they are at. Only by knowing each one in more detail, it's possible to reflect on and propose a digital transformation strategy sustained by feasible and monitorable stages.

**Methodology** | Within the “Stone 4.0 Age” Project, promoted by the Portuguese Association ASSIMAGRA, with the support of COMPETE2020 - FEDER, and with the participation of CTCV - Technological Centre for Ceramic and Glass - a digital maturity diagnosis was carried out on Portuguese natural stone companies in order to perceive their current level of digitalization, identify obstacles to the implementation of the Industry 4.0 principles and establish proposals for actions so they can achieve a higher level of digital maturity.

The strategy for approaching this study consisted of three distinct steps:

- Conducting sectoral focus groups
- Meetings with Companies
- Bibliographic research and analysis

The focus groups were centred on the following topics: familiarisation with the i4.0 concept, strategy for digitalization practices; motivation to embrace the challenges of i4.0, skills needed, associated investments, operations and processes, follow-up and monitoring, constraints and expected impacts. These sessions allowed a first approach to i4.0 issues with SME from the mineral resources sector, which enabled to understand the level of knowledge of the participants on the concept and technologies of industry 4.0; promote the discussion on the state of the art of SME regarding the implementation of i4.0 principles; promote awareness on the constraints and difficulties felt by companies in the process of implementing i4.0 principles; explore the sector's potential for incorporating and increasing digitalization practices in its processes.

In order to increase and detail the knowledge on these topics, meetings were held with companies, which agreed to discuss individually a set of questions about their current reality. These meetings were conducted by a technical team and were based on the model developed by IW Consult of the Cologne Institute for Economic Research and FIR of the RWTH Aachen University (<https://www.industrie40-readiness.de/>) for IMPULS Foundation of the German Engineering Federation (VDMA). This model allows ranking companies in six levels of digital maturity [0 (outsider) to 5 (Leader)] after evaluating the six dimensions relevant to i4.0: Strategy and Organization, Smart Infrastructure, Smart Operations, Smart Products, Data-Based Services and Human Resources.

To complement the gathered information, bibliographical research and analysis on the topic were also carried out. Also, as an add-on to this study,

benchmarking with other sectors was also carried out, namely with the ceramics and glass sector.

**Main conclusions** | Based on the results collected from the companies which participated in this study, it emerges that all are **Newcomers** to the i4.0: 60% are beginners and 40% are outsiders. Therefore, there is a long way to go in terms of digital transformation in companies of the stone sector:

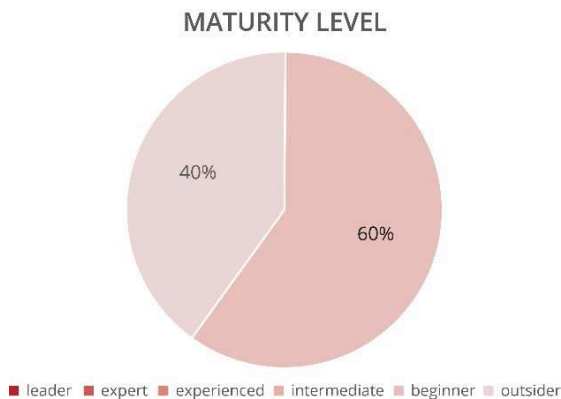


Fig. 1. Global Digital Maturity Level

The results per dimension evaluated:

- Strategy and Organization - about 70% of the companies are at level 1 (beginner), which already demonstrates some concern with this theme;
- Intelligent Infrastructure - 10% of companies are already at level 2 corresponding to an intermediate level of incorporation of the i4.0 principles. In this dimension, some companies have already done recent investments in technology – machinery and equipment – with integrated capacity for communication and transmission of information directly to central management systems;
- Intelligent Operations - 70% of participating companies are at maturity level 3 (Experienced). What most contributes to this assessment is the significant existence of management systems that make it possible to share information internally and externally with customers and suppliers, namely ERP, CRM, management of materials, contracts and projects, including management dashboards using Analytics;
- Intelligent Products - 70% of the companies participating in the project are at maturity level 1, which means that the company's products have at least IT functionality such as memory,

processing, integration, location, support service, monitoring or automatic identification. However, companies do not yet collect data in the product use phase.

- Services based on Data - 90% of the companies participating in this project are at maturity level 0, reflecting two levels of difficulty/constraints: On the one hand, the lack of creation of bigdata based on the collection of production process data; on the other hand (and above all) the fact that those data aren't used to adding value to organizations, they are only stored; this is one of the most important dimensions of digital transformation processes that in this case could contribute to improving companies' processes.
- Human Resources - 70% of participating companies are at maturity level 2, showing some proactivity and predisposition to innovation in this area, although there are difficulties noted on hiring qualified people and in the upskilling/reskilling processes.

**Benchmarking Intersectoral** | At the time of this study, there were several ongoing work, involving other organisations, which will allow us to characterize the "state of the art" in terms of digital transformation of each of the industrial sectors, letting to have a global vision of the national industry. However, it is already possible to identify some common issues across companies, which the most important ones are:

- Low level of digital culture;
- Absence of a strategy to implement i4.0 methodologies;
- Focus on technologies and associated investments to the detriment of other equally important areas.

The data available at the time of this study allowed us to compare the levels of digital maturity among companies from Stone sector, Ceramics sector and Glass sector.

The average companies of these three sectors present level 1 digital maturity – beginner level - however, depending on the evaluation dimension, we verified the existence of different levels of development and maturity (Fig.2).

The Intelligent Operations and Human Resources dimensions are the two stand-out dimensions, in which a greater level of implementation of i4.0 principles are observed.

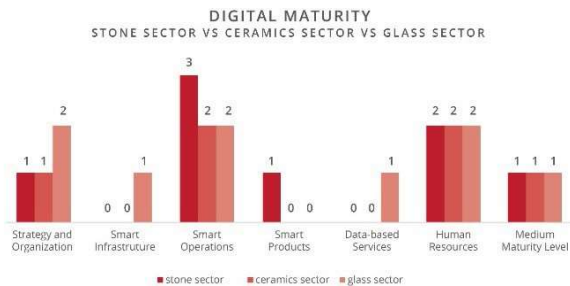


Fig. 2. Comparing the maturity levels in different sectors

**Development strategy proposal** | The results presented both in the study carried out by the Stone 4.0 Age project and in the benchmarking exercise carried out, show a reduced level of maturity presented by companies in the stone sector, so some initiatives are to be considered as a priority, namely:

- Raising awareness and empowering companies for the digital transition;
- Use of supporting tools and roadmaps;
- Cross-sectoral best practice benchmarking;
- Assessment of the potential for alignment with initiatives in support of digitalization in the start-up phase;
- To define and implement action plans for digital transformation in companies;
- Preparing employees and teams for a new paradigm of greater internal collaboration (between departments) and external (with the Academy and other stakeholders);
- Promoting the collaborative development of new internal ideas and market opportunities identified, for the development of technologies and business support mechanisms.

**Acknowledgments:** The authors acknowledge the support of the Project "Stone Age 4.0", promoted by ASSIMAGRA - Portuguese Association of Mineral Resources Industry, with the support of COMPETE2020 – FEDER, as well as all the companies that participated in the focus group activities.

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## Marble façades structural decay due to bowing – A comprehensive review

Pires, Vera<sup>1\*</sup>; Sitzia, Fabio<sup>2,3</sup>; Mirão, José<sup>2,3</sup>; Lopes, Luis<sup>3</sup>; Dias, Luis<sup>2,3</sup>; Neves, Samuel<sup>4</sup>

<sup>1</sup>HERCULES Laboratory, Institute for Advanced Studies and Research, LEM | Laboratório de Mecânicos da Universidade de Évora, University of Évora, Évora (Portugal)

e-mail: [vlcp@uevora.pt](mailto:vlcp@uevora.pt), \*Corresponding author

<sup>2</sup>HERCULES Laboratory, Institute for Advanced Studies and Research, Geosciences Department, School of Sciences and Technology, University of Évora, Évora (Portugal)

e-mail: [jmirao@uevora.pt](mailto:jmirao@uevora.pt), [fsitzia@uevora.pt](mailto:fsitzia@uevora.pt), [luisdias@uevora.pt](mailto:luisdias@uevora.pt)

<sup>3</sup>Geosciences Department, School of Sciences and Technology, University of Évora, Rua Romão Ramalho 59, 7000-671 Evora (Portugal)

e-mail: [lopes@uevora.pt](mailto:lopes@uevora.pt)

<sup>4</sup>ARROW4D | Engineering and Geophysics Consultants Évora (Portugal) e-mail:

[geral@arrow4d.pt](mailto:geral@arrow4d.pt)

### ABSTRACT

Natural stone has been used in façades for centuries. Initially, stone elements were rather thick when used as construction elements, and the durability was appropriate. Scientific research on the properties of marble began in the late 19th century. In the following years, the thickness of natural facade stones decreased from over 1000 mm (as in construction elements) to 20-50 mm (in cladding applications) because of new cutting technologies and equipment developed by the industry. Even though most marble claddings perform satisfactorily, durability problems have begun to appear at an increasing rate after some 50 years of using thin cladding. Well-known buildings such as the Amoco Building in Chicago, SCOR tower in Paris, and the Finlandia Hall in Helsinki have had their marble cladding replaced after less than 30 years at the cost of many millions of Euros. The deterioration gives a considerable change in the appearance of the panels. They bow, warp or break. Most cases of bowing involve Italian marble from the Carrara area, simply because it is the most widespread and used marble type. It is, however, vital to emphasize that most building facades with Carrara marble perform well, and marbles from other areas also exhibit durability problems. The bowing of marble is not only restricted to buildings and gravestones of marble are also known to bow. Nowadays, most of the well-known cases are from Europe or North America, most likely because of the much more widespread use of thin marble claddings in these regions. In Europe, there are building facades with bowed marble slabs in both the cooler climates of Finland and Denmark and the warmer climates of Portugal and Spain. Bowed slabs can also be found in Austria, Belgium, France, Germany, Greece, Italy, the Netherlands, Norway, Sweden, Switzerland, and the UK. Despite more than 100 years of research, the marble bowing problem has not been solved. Numerous works are entirely focused on the possible factors responsible for the deterioration of marble, and they clearly show that temperature variations and moisture are key factors in the degradation processes. The interaction of temperature and moisture must be a crucial external factor for the bowing and strength loss of certain marble types. Still, an updated and comprehensive review of marble façades structural decay due to bowing is not available. In this sense, there is a need to consolidate the understanding of marble bowing.

**KEYWORDS:** natural stone; Building Materials; Cladding; Marble; Bowing

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