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PERFORMANCE OF CARBONATED SLUDGE-BASED COMPOSITES (MARBLE AND LIMESTONE): UNIAXIAL COMPRESSIVE AND FLEXURAL STRENGTH UNDER CENTRAL LOADING AFTER FREEZE-THAW CYCLES AND THERMAL SHOCK

• **P. Afonso[1,2]*, L. Lopes[3,4], P. Faria[2,5] P. Mourão[6], R. Martins[3], V. Pires[2]**

(1) University of Évora, Institute for Advanced Studies and Research (IIFA), Department of Geosciences, School of Science and Technology, Évora, Portugal; *pafonso@uevora.pt

(2) University of Évora, HERCULES Laboratory and IN2PAST, Associate Laboratory for Research and Innovation in Heritage, Arts, Sustainability and Territory, Institute for Advanced Studies and Research, Portugal

(3) University of Évora, Department of Geosciences, School of Science and Technology, Évora, Portugal

(4) University of Évora, Institute of Earth Sciences, Évora Pole, Portugal

(5) University of Aveiro, GeoBioTec, Department of Geosciences, Aveiro, Portugal

(6) University of Évora, CHANGE & Mediterranean Institute for Agriculture, Environment and Development, Department of Chemistry and Biochemistry, Évora, Portugal

Calcinata Project

C@lcinata

UNIVERSIDADE DE ÉVORA
ESCOLA DE CIÊNCIAS E TECNOLOGIA
DEPARTAMENTO DE GEOCIÊNCIAS

ALENTEJO 2020 | PORTUGAL 2020 | UNIÃO EUROPEIA Fundo Europeu de Desenvolvimento Regional

The *Calcinata* project (reference no. 72239), co-financed by Alentejo 2020, Portugal 2020 and the European Union through the European Regional Development Fund (ERDF), was a co-promoted R&TD project that ended in June 2023.

Calcinata research project aimed to study the application of carbonate sludge from marble and limestone processing as a component of resin binders for incorporation into stone composites.

The Geosciences Department of the University of Évora was involved in the project, carrying out research into the potential for valorising this waste in different industrial applications.

Introduction

The carbonate dimension stone extractive and processing industry produces large amounts of wastes later deposited in heaps and deposits of carbonate sludge.

The waste and residues are divided into two types:

1. In quarries:

i) Rock fragments that contribute with **95%**
ii) Carbonated sludge with **5%** contribution

2. In processing units:

i) Rock fragments - **58%**
ii) Carbonated sludge - **42%**

Inevitable environmental impacts

- Reduction in vegetation cover;
- Soil sealing;
- Alteration of water lines with a significant reduction in their quality;
- Alteration of ecosystems;
- Reduction in air quality;
- Reduction in the photosynthetic process of plants;
- Visual impact.

Quarries and the circular economy

Creation of new products using waste from the extraction and processing of carbonate ornamental stone.

Calcinata Project

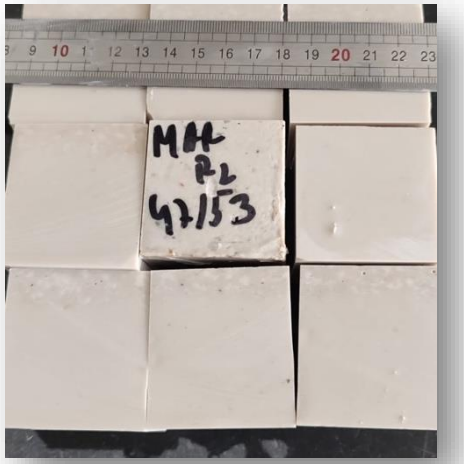
Ideal Formulation - Binder

The first goal was to define the ideal formulation for the composite, adjusting the proportions of carbonated sludge (marble and limestone sludge) with different resin percentages.

This work was carried out in the initial phase of the project, with results already published.

Afonso, P., Pires, V., Faria, P., Azzalini, A., Lopes, L., Mourão, P., & Martins, R. (2024). ***A novel approach for the reuse of waste from the extractive and processing industry of natural stone binders: development of stone composites.*** *Sustainability, 16(1), 64.*

Formulations %	R (MPa)
47%NC / 53%Res.	96.04
50%NC / 50%Res.	102.12
52,31%NC / 47,69%Res.	103.20
52%NM / 48%Res.	106.37
47%NM / 53%Res.	96.23
50%NM / 50%Res.	98.35
54,43%NM / 45,57%Res.	102.73

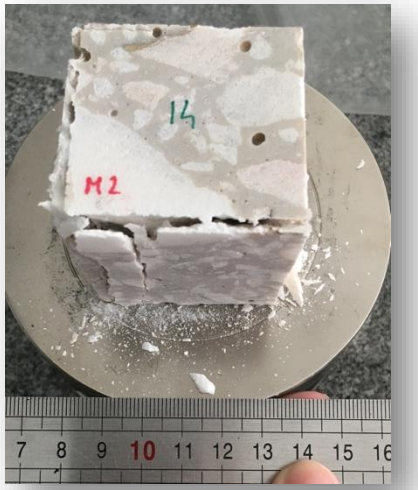


Composite Formulation - Binder & Marble Aggregates

Formul	Aggregates			Binders			Average uniaxial Compression Strength (MPa)
	BA	B1	B2	NC	NM	Res	
F1	30%	30%	40%	52%	-	48%	73.30
F2	20%	20%	60%	52%	-	48%	69.06
F3	35%	15%	50%	52%	-	48%	61.11
F4	40%	40%	20%	52%	-	48%	**91.96**
F5	30%	30%	40%	-	52%	48%	52.26
F6	20%	20%	60%	-	52%	48%	76.33
F7	35%	15%	50%	-	52%	48%	81.20
F8	40%	40%	20%	-	52%	48%	**88.19**

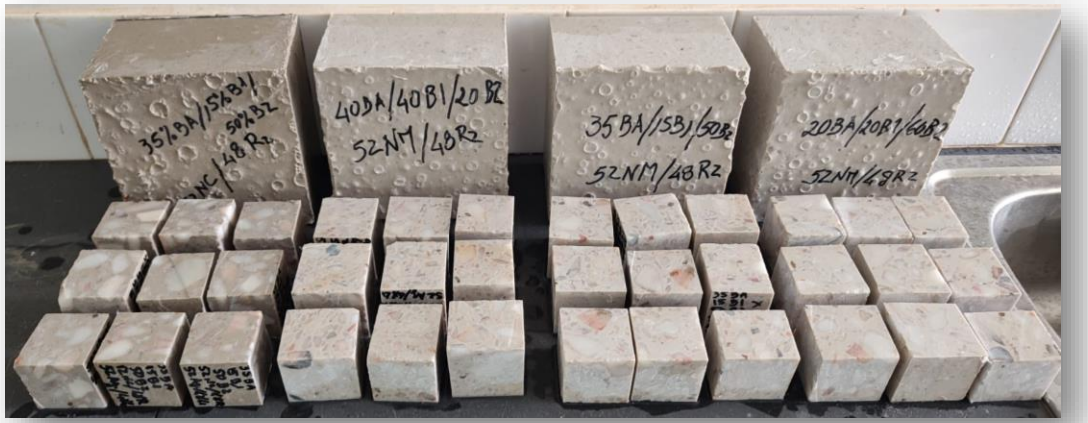
The marble aggregate, supplied by the company *Marvisa, Mármores Alentejanos Lda.*, consisted of three types, with the following granulometric intervals:

- BA (4 mm / 6.3 mm)
- B1 (8 mm / 14 mm)
- B2 (14 mm / 25 mm)



After analysing the best binder results, the next stage consisted of evaluating different percentages of aggregates (types BA, B1 and B2) in order to optimise the composite formulation.

Through uniaxial compression tests, the ideal percentage for each type of aggregate was determined, which was then adopted in all the tests that followed.



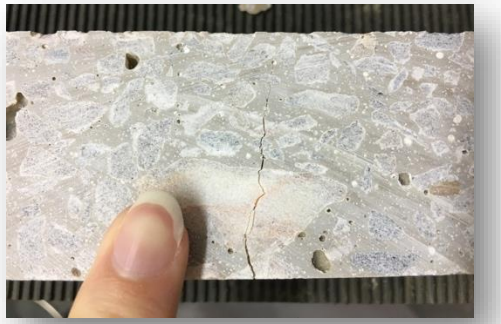
Results

Uniaxial compressive and flexural strength under central loading after freeze-thaw cycles and thermal shock

The mechanical properties of the marble and limestone sludge composites were evaluated before and afterwards:

56 freeze-thaw cycles (EN 12371)

Thermal shock ageing



Uniaxial compressive strength (EN 1926)

Freeze-thaw:

Reductions of **11%** (marble) and **26%** (limestone).

Thermal shock:

More significant decreases, **28%** (marble) and **36%** (limestone), possibly associated with thermal degradation of the resin.

Flexural strength under central loading (EN 12372)

Freeze-thaw:

Marble showed **no** significant changes and limestone showed a **24%** reduction.

Thermal shock:

Reductions of **41%** for marble and **43%** for limestone.

Tests	Composite	R (MPa)	After freeze-thaw	After thermal shock
Compression Strength	Marble	88,19	78,29	63,36
	Limestone	91,96	67,83	59,04
Flexural strength	Marble	15,49	15,70	9,09
	Limestone	13,49	10,25	7,69

Conclusions

- The composites with carbonated sludge maintained good mechanical performance, even after the accelerated ageing tests.
- Thermal shock was more aggressive than freeze-thaw, especially for flexural strength.
- The marble sludge composite showed better resistance to changes caused by thermal shock cycles, showing less degradation compared to the limestone sludge composite.
- The results confirm the technical potential of carbonated sludge as an alternative raw material.
- It is possible to use carbonated sludge in stone composites, providing economic value which allows it to be classified as a by-product instead of waste.
- The results confirm the potential of carbonate sludge as a raw material in the production of sustainable composites, in line with the “Circular Economy Action Plan” and promoting greater sustainability in the sector.

Acknowledgments

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Thank you all for your attention!