



Universidade de Évora - Instituto de Investigação e Formação Avançada

Programa de Doutoramento em História

Tese de Doutoramento

**The exploitation of plant resources in the human history: an
archaeobotanical insight**

Ginevra Coradeschi

Orientador(es) | Cristina Barrocas Dias
Fernando Branco Correia
Laura Sadori
Luís Gonçalves

Évora 2022



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A tese de doutoramento foi objeto de apreciação e discussão pública pelo seguinte júri nomeado pelo Diretor do Instituto de Investigação e Formação Avançada:

Presidente | António José Candeias (Universidade de Évora)

Vogais | Alan Crivellaro (University of Cambridge)
Alessia Masi (Università degli Studi di Roma "La Sapienza")
Ana Margarida Arruda (Universidade de Lisboa - Faculdade de Letras)
Cristina Barrocas Dias (Universidade de Évora) (Orientador)
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UNIVERSIDADE DE ÉVORA
INSTITUTO DE INVESTIGAÇÃO
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Scientific Outputs

Poster presentations:

- **Title:** “*Estudo arqueobotânico preliminar dos macrorestos vegetais do sítio de Paço dos Lobos da Gama (Évora)*”

Authors: G. Coradeschi, S. Mouzinho Valez, L. Dias, L. Sadori, C. Barrocas Dias

Name of the event: YABURA: uma cidade do Al-Andalus

Place and date: Convento dos Rimédios (organized by the Camara Municipal de Évora), Évora (Portugal), 15th April - 15th August 2015.

- **Title:** “*Investigação arqueobotânica do sítio arqueológico de Paço dos Lobos da Gama: um subúrbio Islâmico da cidade de Évora (século XI)*”

Authors: G. Coradeschi, A. F. Mourer, J. R. Santos, G. Lopes, C. Vignola, F. Branco, L. Sadori, C. Barrocas Dias

Name of the event: XI CONGRESO IBÉRICO DE ARQUEOMETRÍA

Place and date: Laboratório Hercules, University of Évora (Portugal), 14th - 16th October 2015.

- **Title:** “*Random gathering or intentional wood selection? Charcoal analysis of pit 16 deposit from Perdigões archaeological site*”

Authors: G. Coradeschi, C. Barrocas Dias, F. Branco, L. Sadori, A. Valera

Name of the event: International Meeting: Wood and Charcoal: Approaches from Archaeology, Archaeobotany, Ethnography and History

Place and date: University of Minho, Braga (Portugal), 15th-16th April 2016.

- **Title:** “*Análise carpológica dos sedimentos arqueológicos de Paço dos Lobos da Gama (Évora-séculos XI-XII)*”

Authors: G. Coradeschi, J. R. Santos, G. Lopes, L. Sadori, F. Branco, L. Gonçalves, C. Barrocas Dias

Name of the event: 1^a Mesa-redonda DOUBLE-U REPLAY (WR)

Place and date: Antigos Paços do Concelho, Torres Vedras (Portugal), 16th September 2017.

- **Title:** “*Estudo arqueométrico dos materiais de construção da Domus dei Dolia (Vetulonia, Toscana, Italia)*”

Authors: G. Coradeschi, S. Rafanelli, M. Beltrame, A. Candeias, P. Moita, J. Mirão, C. Barrocas Dias

Name of the Event: XI CONGRESO IBÉRICO DE ARQUEOMETRÍA

Place and date: CENIEH, Burgos (Spain), 25th - 27th October 2017.

- **Title:** *“The exploitation of plant resources in the human history: an archaeobotanical insight”*

Authors: G. Coradeschi

Name of the event: Congresso Histórias Da Arte em Viagem: Circulação de Ideias, Formas, Objectos. I Colóquio Luso-Brasileiro Arte Entremundos V

Place and date: CES (Colégio do Espírito Santo), University of Évora (Portugal), 04th - 06th June 2018.

- **Title:** *“Análisis antracológico y quimiométrico de carbones de la fosa 16 perteneciente al área arqueológica de Perdigões”*

Authors: G. Coradeschi, N. T. Jiménez-Morillo, R. M. Godinho, C. Barrocas Dias, A. Valera

Name of the event: XIII CONGRESSO IBÉRICO DE ARQUEOMETRIA

Place and date: Campus de Gambelas, University of Algarve, Faro (Portugal), 16th - 17th October 2019.

Oral presentations:

- **Title:** *“Presentation of the first results of the analysis of the building materials from the Domus dei Dolia “*

Authors: G. Coradeschi and M. Beltrame

Name of the event: Seminary

Place and date: Museo Isidoro Falchi, Vetulonia (Italy), 28th May 2016.

- **Title:** *“Preface: an archaeometric study from Domus dei Dolia and Vetulonia Etruscan Museum materials”/ “An archaeobotanical view into the wooden materials”*

Authors: G. Coradeschi and C. Barrocas Dias

Name of the event: *Domus dei Dolia: an archaeometric study of an Etruscan house – A patnership project between the Castiglione della Pescaia Municipality and the Hercules laboratory”*

Place and date: CES (Colégio do Espírito Santo) Room 131, University of Évora (Portugal), 14th November 2016.

- **Title:** *“Archaeometric study of waterlogged wood from the roman cryptoporticus of Lisbon”*

Authors: G. Coradeschi, A. Manhita, N. Mota, A. Caessa, C. Nozes, L. Sadori, F. Branco, L. Gonçalves, C. Barrocas Dias

Name of the event: Technoheritage 2017

Place and date: University of Cadiz (Spain), 21st - 24th May 2017.

- **Title:** *“A multidisciplinary study of waterlogged wood from the Roman cryptoporticus of Lisbon”*

Authors: G. Coradeschi

Name of the event: INTERNATIONAL SUMMER SCHOOL: Dendroecology, Quantitative wood anatomy and stable isotopes: from xylogenesis to tree rings

Place and date: University Federico II - Dept. Agriculture, Portici, Naples (Italy), 25th - 29th September 2017

- **Title:** “*The exploitation of plant resources in the human history: an archaeobotanical insight. Two new case studies*”

Authors: G. Coradeschi

Name of the event: Public presentation of phd in History, 2^o edition

Place and date: Webinar, 13th October 2017.

- **Title:** *The exploitation of plant resources in the human history: an archaeobotanical insight*

Authors: G. Coradeschi

Name of the event: 1^o Encontro Heritas Estudo do património - Investigações no âmbito do Programa de Doutoramento

Place and date: Faculdade de Belas-Artes, University of Lisbon (Portugal), 23rd March 2018.

- **Title:** *Presentations of the new results from the “Welcome Home Project” - Hercules laboratory*

Authors: G. Coradeschi, M. Beltrame, C. Barrocas Dias, J. Mirão, A. Candeias, P. Moita

Name of the event: Conclusion of the Archaeological campaign

Place and date: Museo Isidoro Falchi, Vetulonia (Italy), 28th September 2018.

- **Title:** “*An archaeobotanical view into the wood materials of Domus dei Dolia Etruscan House (3rd to 1st century BC)*”

Authors: G. Coradeschi

Name of the event: 18^o International Course on Wood Anatomy & Tree-Ring Ecology 2018, 25/11/2018-01/12/2018

Place and date: Kloster, (Swiss), 25^h November - 01st December 2018.

- **Title:** *Welcome Home – Risultati dello studio archeometrico dei materiali della Domus dei Dolia (Vetulonia)*

Authors: G. Coradeschi, C. Barrocas Dias, M. Beltrame

Name of the event: tourismA

Place and date: Palazzo dei congressi, Firenze (Italy), 22nd - 24th February 2019.

- **Title:** “*An archaeobotanical view into the wooden materials of Domus dei Dolia Etruscan House (3rd to 1st century BC)*”

Authors: G. Coradeschi

Name of the event: Summer School of University of Évora - HERITAS Meeting – Estudos do Património

Place and date: CES (Colégio do Espírito Santo) Room 102, University of Évora (Portugal), 21st June 2019.

- **Title:** “*Archaeological grape seeds from Torre dos Namorados: state of the art and methodology*”

Authors: G. Coradeschi

Name of the event: 2ª Reunião de Ampelografia Histórica

Place and date: Museu de Arqueologia de Fundão (Portugal), 16th - 17th October 2019.

- **Title:** “*Archaeological wood analysis: anthracological study of charred wood building elements and furnishings from Domus dei Dolia*”

Authors: G. Coradeschi

Name of the event: Ecological Plant Stem Anatomy and Wood Identification

Place and date: University of Évora (Portugal), Colégio Luís Verney - 3rd November 2020.

- **Title:** “*Gli elementi strutturali e gli elementi di mobilio e arredo in legno della Domus dei Dolia*”

Authors: G. Coradeschi

Name of the event: Terra, legno e materiali deperibili nell’architettura antica

Place and date: University of Padova (Dipartimento dei Beni Culturali), 3rd - 5th June 2021.

- **Title:** “*The exploitation of plant resources in the human history: an archaeobotanical insight*”

Authors: G. Coradeschi

Name of the event: Digital Techniques and Laboratory Practice in Archaeological Materials Science – Erasmus Mundus Joint Master in Archaeological Materials Science (ARCHMAT)

Place and date: University of Évora - Colégio Luís Verney (Portugal), 3rd November 2021.

Partecipation in the organization of scientific spreading events:

- **Name and type of the event:** Exhibition: YABURA: uma cidade do Al-Andalus

Place and date: Convento dos Rimédios (organized by the Camara Municipal de Évora), Évora (Portugal), 15th April - 15th August 2015

Organized by: Évora Municipality with the support of the followed institution: CIDEHEUS, Laboratório Hércules (Universidade de Évora), Governo de Portugal/Secretário de Estado da Cultura/Direção Regional da Cultura do Alentejo, Museu de Évora, Museu Arqueológico Provincial de Badajoz, Campo Arqueológico de Mértola, Câmara Municipal de Montemor-o-Novo and under the patronage of Turismo do Alentejo E.R.T.

- **Name and type of the event:** Workshop: Ecological Plant Stem Anatomy and Wood Identification

Place and date: University of Évora (Portugal) - Colégio Luís Verney , 3rd November 2021

Organized by: Hercules Laboratory in the framwork of Erasmus Mundus Joint Master in Archaeological Materials Science (ARCHMAT) with the experties of Dottor Alan Crivellaro (Research associate, University of Cambridge).

Articles:

Coradeschi, G., Beltrame, M. (2016). *Il progetto Welcome Home*. In Bentornati a casa La *Domus dei Dolia* di Vetulonia riapre le porte dopo duemila anni. Ara Edizioni, Siena, ISBN: 88-98816-33-2, p. 55.

Coradeschi, G., Mourer, A. F., Santos, J. R., Lopes G., Cristiano V., Sadori, L., Barrocas Dias, C. (2017). *Investigação Arqueobotânica dos sedimentos arqueológicos de Paço dos Lobos da Gama: um arrabalde islâmico da cidade de Évora (séculos XI-XII)*. Proceedings of the XI Congresso de Arqueometria Iberico (CIA XI 2015), digiTar Revista Digital de Arqueologia Arquitectura e Artes (on-line magazine) n4, University of Coimbra, https://doi.org/10.14195/2182-844X_4_4, pp. 33 - 40.

Coradeschi, G., Manhita, A., Mota, N., Caessa, A., Nozes, C., Sadori, L., Branco, F. L., Gonçalves, L., Barrocas Dias, C. (2018). *Archaeometric study of waterlogged wood from the roman cryptoporticus of Lisbon*. Proceedings of the 3rd International Congress on Science and Technology for the Conservation of Cultural Heritage (TechnoHeritage 2017), Balkema Book, CRC Press, London, pp. 323 - 325.

Coradeschi, G., Beltrame, M., Rafanelli, S., Quaratesi, C., Sadori, L., Barrocas Dias, C. (2021). *The wooden roof framing elements, furniture and furnishing of the Etruscan Domus of the Dolia of Vetulonia (Southern Tuscany, Italy)*. Heritage (4) – MDPI - Open access journal.

Coradeschi, G. *Gli elementi strutturali e gli elementi di mobilio e arredo della Domus dei Dolia*. In J. Bonetto, C. Previato (eds), *Terra, legno e materiali deperibili nell'architettura antica*, Atti del Convegno Internazionale di Studi (Padova, 3-5 giugno 2021), Roma.
Status: In press.

Coradeschi, G., Uccesu, M., Dias, E., Cunha, J., Baleiras Couto, M. M., Ângelo, M., Alegria Ribeiro, C., Barrocas Dias, C., Bacchetta, G. *A glimpse into the viniculture of the Roman Lusitania: morphometric analysis of charred grape seeds from Torre dos Namorados archaeological site in Portugal*. VHAA.
Status: Submitted.



Klosters (Swiss) - 30/11/2018

“The answer, my friends... is written in the rings”
(Fritz Hans Schweingruber 1936 – 2020)

“Doubt is not a pleasant condition, but certainty is an absurd one”
(Voltaire 1694 -1778)

*“Nothing in life is to be feared, it is only to be understood. Now is the
time to understand more, so that we may fear less”*
(Marie Curie 1867-1934)

To my family,

*To my parents Maddalena and Vezio and to my brother Lorenzo for
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Abstract

This thesis focuses on the interdisciplinary nature of Archaeobotany and as such, different types of interactions between past human communities and plant resources have been evaluated. Additionally, the analysis of different categories of macroremains, coming from different archaeological sites with different chronologies (Chalcolithic, Etruscan, Roman and Islamic) made it possible to collect various data sets that enable answering multiple questions within the different archaeological contexts.

Thus, the PhD thesis addresses several different issues, such as:

- the evaluation of human plant selection and use for ritual purposes;
- the analysis of charred wooden remains employed as building materials and furnishings of a residence;
- the identification of waterlogged wooden objects and the evaluation of their degree of conservation;
- the study of the viniculture process in the province of Roman Lusitania;
- the study of the plant-based diets of past human communities.

To address these different issues, different methodologies and analytical techniques were used. The traditional archaeobotanical methods, namely anthracological, xylological and carpological analyses were employed, together with analytical techniques, Pyrolysis-Gas Chromatography- Mass Spectrometry (Py-GC-MS) and FT-MIR Spectroscopy, image analyses, chemometric analyses and two different types of statistical analyses (PLS and LDA analyses). The thesis also presents a component of experimental archaeology, where contemporaneous samples of wood and grape seeds were carbonized to mimic the archaeological samples

Results obtained from this dissertation, regarding the relationships between humans and their use of surrounding environments, validate the importance of Archaeobotany as a discipline, professing its important role within the study of tangible and intangible Cultural Heritage. Finally, parts of the thesis results have been presented in scientific meetings and were published in conference proceedings and international peer reviewed journals. Moreover, some of the results obtained were also used in museum exhibitions and cultural events, bringing awareness to a wider audience of the importance of Archaeobotany.

Resumo

A presente tese centra-se no caráter interdisciplinar da Arqueobotânica. O principal objetivo do trabalho é a caracterização de vários tipos de relações entre as comunidades humanas do passado e os seus recursos vegetais. A análise de diferentes categorias de macro restos vegetais, provenientes de sítios arqueológicos diacrónicos (Calcolítico, Etrusco, Romano e Islâmico) permitiu a recolha de dados em contextos distintos.

Assim, a tese de doutoramento aborda várias questões, pretendendo, nomeadamente:

- avaliar a seleção humana de plantas e a sua utilização para fins rituais;
- analisar os restos carbonizados de madeira utilizados como materiais de construção e mobiliário de uma unidade habitacional;
- identificar objetos de madeira de zonas alagadas e avaliar o seu grau de conservação;
- recolher dados sobre o processo de vinicultura;
- reconstruir a dieta vegetal duma comunidade humana do passado.

Para o efeito, o trabalho adotou diferentes abordagens. Os métodos arqueobotânicos tradicionais, nomeadamente a análise antracológica, xilológica e carpológica conjugaram-se com técnicas analíticas (pirólise acoplada a cromatografia gasosa e espectrometria de massa e espectroscopia FT-MIR), técnicas de análise por imagem, análises quimiométricas e estatísticas (PLS e LDA). A tese apresenta também numa componente experimental.

Os resultados obtidos a partir deste trabalho sobre as relações entre os seres humanos e o ambiente envolvente, validam a importância da Arqueobotânica como disciplina, estabelecendo o seu papel central no estudo do Património Cultural tangível e intangível. Finalmente, parte dos resultados da tese foi apresentada em congressos científicos e publicada em atas de conferências e revistas internacionais. Além disso, uma parte dos dados foi ainda apresentada em eventos culturais abertos a um público mais vasto, em encontros em museus locais e internacionais.

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1st CHAPTER. Introductory section

The exploitation of plant resources in the human history: an archaeobotanical insight

1. 1. Introductory note

This PhD thesis intitled “*The exploitation of plant resources in the human history: an archaeobotanical insight*” has been developed in the framework of the HERITAS- Heritage Studies doctoral program. HERITAS is an innovative and multidisciplinary program, which aims to provide a common language of dialogue between History, History of Art, Architecture, Museology, Archaeology, Conservation and Restoration and Heritage Sciences. The program is jointly developed by the University of Évora (UE) and the University of Lisbon (UL) (Portugal) and involves different institutions (CHAIA, Centre for Art History and Artistic Research <http://www.chaia.uevora.pt/en/event/188/doctoral-programme-in-heritage-studies-jointlydeveloped-by.html>, consulted on 27/09/2021).

The PhD thesis was granted by the Foundation for Science and Technology (FTC - Portugal) (PD/BD/128278/2017) and the analyses were carried at the HERCULES laboratory - Cultural Heritage, Studies and Safeguarding (UE), under the joint supervision of the University of La Sapienza (Rome, Italy) (PhD course in Earth Sciences - Curriculum Applied Sciences for the Protection of the Environment and Cultural Heritage).

This PhD thesis was also part of a project resulted from an agreement between the Municipality of Castiglione della Pescaia (Tuscany, Italy), the Isidoro Falchi Archaeological Museum of Vetulonia (Tuscany, Italy) and the HERCULES Laboratory (Figure 1). The project titled "WELCOME HOME" included the analysis of organic and inorganic materials retrieved from the *Domus dei Dolia* (Vetulonia, Tuscany, Italy). The results of the project (some of which are part of this thesis) were presented at "tourisma" - International Fair of Archaeology (Congress Palace’s auditorium) the 24th of February 2019.



Figure 1: Signature of the agreement at the Municipality of Castiglione della Pescaia (GR, Italy).

Finally, this thesis also benefited from an agreement established between the Archaeobotany Laboratory of the Hortus Botanicus Karalitanus (University of Cagliari, Sardinia, Italy), the HERCULES Laboratory and the National Institute for Agricultural and Veterinary Research (INIAV)

(Dois Portos, Portugal), which made possible to carry out some interdisciplinary analyses on the archaeological grape seeds from Torre dos Namorados (Fundão, Portugal). The agreement also included a visiting program at the Archaeobotanical Laboratory of the Hortus Botanicus Karalitanus to develop part of the analyses.

The establishment of a joint doctoral supervision with University of la Sapienza of Rome and the collaboration with international research centres and public institutions highlights the interdisciplinary nature of this PhD thesis and how it represents an integrated strategy for enhancing and promoting the study of cultural heritage.

1. 2. Thesis structure

The first chapter of the thesis consists of a brief introductory note which frames the interdisciplinary nature of the thesis, followed by the state of the art of the discipline. The second chapter presents the research aims, divided into the general objectives of the thesis and the specific goals of each case study. The third chapter includes the materials and methods analysed for the thesis. The description of the materials is followed by a brief introduction to the methods, as some of these are common to several case studies, and a summary table of the methods employed is presented at the end of the chapter. The fourth chapter describes the five case studies analysed in the thesis. A brief preface introduces each chapter. Each individual case study is made up of an introduction, a state of the art, and a description of the archaeological site and/or the context. The description of the materials analysed and of the type of sampling and quantification method employed, which due to its specificity, had not been covered in the third chapter of the thesis, are followed by the discussion and the conclusions. The last chapter of the thesis, titled Synthesis, includes the final observations on each single case study and a more general conclusion of the thesis. At the end of the thesis a final note regarding the contribution of this PhD thesis to the HERITAS Doctoral Program is presented.

1. 3. Plant remains as tangible and intangible human evidence

The term ‘cultural heritage’ includes tangible and intangible human evidence and encompasses everything related to the past. Moreover, this term embodies various interpretations and multiple meanings, as it refers to everything related to the past, understood both in a material and immaterial sense, but also with the relationships and connections that belong to the present. In addition, material heritage, which in fact always contains references to the immateriality of man's actions in the past, can therefore be the subject of analysis but only because it serves as an effective vehicle to highlight and display the intangible aspects.

Among examples of tangible and intangible cultural heritage, archaeological plant remains represent a veritable archive of social, technological, and economic aspects of ancient human communities. This kind of evidence can be studied by Archaeobotany. UNESCO defines intangible cultural heritage as a “set of practices, representations, expressions, knowledge and expertise - as well as the instruments, objects, artefacts and cultural spaces associated therewith - that communities, groups and, in some cases, individuals recognize as part of their cultural heritage. This intangible cultural heritage, transmitted from generation to generation, is constantly recreated by communities and groups concerned in accordance with their environment, their interaction with nature and their history, and provides them with a sense of identity and continuity, thereby promoting the respect for cultural diversity and human creativity (UNESCO (ICH) GLOBAL, 2003). To create a research project focused on cultural heritage, the five areas identified by UNESCO should be considered. Among these areas, the immateriality of cultural evidence is manifest. Point number 5 refers to: “Knowledge and practices concerning nature and the universe” (UNESCO (ICH) GLOBAL, 2003). Namely, the study of knowledge, beliefs and experiences developed over the centuries by communities in their interrelationship with nature, which are often no longer practiced nowadays, but that can still be rediscovered and proposed again. Some research lines are the ancient knowledge of flora and fauna and their interaction with people, the traditional recipes linked to seasons and wild herbs, beliefs and rites. To develop this research project, which aims to contribute to the understanding of plant exploitation in human history, all these aspects have been considered by linking the past to the present, through Archaeobotany.

1. 4. The Archaeology of human – plant relations

Trees, flowers, seeds, and fruits have always played a primary role in human life. This simple truth is not so evident. In the 21st century world, plants seem somewhat forgotten in daily lives and out of fashion. Many people are unaware that they are dealing with plants and their derivatives when they are mixing flour and sugar in a fragrant sweet, or when they are drinking their favourite wine, and synthetic products have now replaced many of the oldest botanical products that were used in the past. We collect fruit from stalls without knowing if the produce was imported or not, and this seems to have no importance for us, modern hunter-gatherers who seek food in supermarkets as our ancestors did in the forests. Thus, common knowledge of the plant world is soon going to be lost, and with that we will lose the perception that caring for plants means taking care of our own lives. We must realize, however, that even today plants fulfil primary human needs such as food, medicinal remedies, clothing, manufacture of tools, furniture, and homes. They fulfil social needs as well, being

used in tattoos, makeup, and ornaments, and they are symbols in coats of arms and flags, and so, as in the past, plants retain their ‘metaphorical use’ in ceremonies and religious rituals.

Archaeobotany is a discipline which explores the history of the interaction with plants by humans, through the analysis of archaeobotanical remains, that have been preserved until the present day. Through the study of archaeobotanical material, it is possible to disclose how people used plants in the past e.g., for food, fuel, medicine, ritual purposes or for building. Their study could make a substantial contribution to the understanding of the evolution of man-environment relationships, which have always been an integral part of human culture. Moreover, through these studies the reconstruction of a past vegetation is achievable (Mercuri et al., 2009).

Archaeology is generally known as the discipline dealing with the discovery of temples, castles, tombs, and treasures but not plants. Although small and fragile, plant remains can be every bit as precious as, if not more so, than large permanent structures and precious artifacts, in supplying information concerning human life in the past.

The broadest, general definition of Archaeobotany was given by Greig (1989) (who is considered one of the fathers of the discipline) who defined Archaeobotany as “*the study of plant remains of any kind from archaeological sites, from Palaeolithic cave deposits to nineteenth century urban layers*”.

The term “archaeobotanical remains” encompass two main forms, namely macro-botanical and micro-botanical remains (Magid, 1989). The macro-botanical remains are those which can be seen by the naked eye or low power microscope (seeds, fruits, nuts, wood/charcoal, roots, tubers, leaves). The micro-botanical remains cannot be seen by the naked eye and require a high power microscope (pollen, phytoliths, spores, starch grains)(Vanhanen, 2010).

The importance of botanical remains originating from archaeological records has been acknowledged significantly later compared to the other types of archaeological materials. As a discipline, the history of Archaeobotany is linked with two traditions, one European, and one American. The American tradition concerns the use of the term “Paleoethnobotany”, instead of “Archaeobotany”. The older European tradition grew out of botany, while on the contrary, the American one grew out of Ethnobotany, the study of modern relationships between plants and people. This difference, both in terms of definitions and research content, has become increasingly blurred over time. Nowadays, most researchers use the two terms interchangeably. In this thesis the term Archaeobotany will be adopted, referring to both the recovery and the identification of plant remains, and their interpretation in term of past human-plant relationships.

The history of the European discipline can be traced back to the late 19th century, when several studies were made, most important being the finding of desiccated plant remains in ancient Egyptian

tombs and plant remains from Swiss Neolithic lake villages (Renfrew, 1973; Vanhanen, 2010). However, it was only during the 1970 's and 1980's that the introduction and development of archaeobotanical techniques and methodology were applied on a wider and more systematic scale in archaeological research. In turn, for the last three decades or so, such studies left the laboratory and became an integral part of many archaeological projects. Studies on archaeobotanical remains have undergone a dramatic increase from the late 90s, numerous advances have been made on several issues, among which: the identification of cultivated plants; the understanding of wood acquisition, use, and environmental impact; plants as indicators of ethnicity; analysis of agricultural practices; management and land use; the interpretation of crop origins and the evolution of agricultural systems. New themes were also approached, such as the use of wild foods by agriculturalists; the identification of ancient beverages and spices; the study of plants in ritual; the analysis of fibres and wooden artifacts; the study of plants in economic systems and the reconstruction of human past diet and subsistence (Cappers and Neef, 2012; Pearsall, 2015; Pearsall, 2019). It is evident that in the last 60 years archaeologists have realized the quality and the quantity of knowledge that can be gained from the collection and study of archaeobotanical remains, and have developed analytical techniques and research questions accordingly (Pearsall, 2015). However, the most innovative element was the changing of analytical approach from archaeobotanists, which includes their increasing presence in the field and a very close link with the archaeological process. It was crucial to promote a language that would allow researchers to plan and investigate those points at which plant science and archaeology overlap and to interpret the mutual significance of their interrelationship. More and more archaeologists and archaeobotanists have formed a language by which they can understand and communicate the questions to be asked, the plans to be made, the work to be carried out, analysis to be done and objectives to be achieved. In the archaeological field a (healthy) trend toward interdisciplinary research was developed in which biological science started to play an active role. Finally, archaeobotanical research focuses on a clear link between archaeology, taphonomy and biology much more than it did in the past (Cappers and Neef, 2012).

1. 4. 1. Xylology and Anthracology: the history and new methodologies applied

Xylology (from Greek *xilos*, wood) and Anthracology (from Greek *anthracos*, charcoal) are the two sub-disciplines that study assemblages of wood and charcoal macro-remains, in order to provide both paleoenvironmental and paleoethnobotanical information (Castelletti, 1990; Pearsall, 2015; Renfrew, 1973). These types of studies consider both the vegetational aspects, and more specifically, those related to human actions. Therefore, the technological and sociological aspects of human activities can be understood from the study of these remains.

These types of studies are commonly referred as “wood and charcoal analysis”. Some researchers have shown some dissension about the use of the term Anthracology with a general meaning of charcoal analysis (Thiébaud, 2008). In this thesis we assume that “Anthracology” and “Xylology” correspond to the study of charcoal and wood remains from archaeological contexts.

The first identifications of archaeological wood remains go back to the end of the 19th century (Heer and Passerini, 1865; Pigorini, 1865a; Pigorini, 1865b; Unger, 1849). From the first half of the 20th century, wood and charcoal identification methods were becoming more efficient. In particular, the use of reflected light microscopy gave rise to a true revolution, primarily in charcoal analysis. From that moment onward, charcoal could be identified simply by breaking its surfaces and observing them (Western, 1970). The use of this type of microscopy, avoided the use of tedious preparation which involved mounting charcoal samples on a slide. This allowed researchers to save a large amount of time and made it possible to analyse much larger numbers of fragments. Moreover, the cost of analyses was considerably reduced.

In the following decades, studies of woods and charcoal remains became more widespread. From the late 1960s, the adoption of reflected light microscopy become standard, enabling systematic analysis of large assemblages from a range of site and contexts (Couvert, 1968; Donoghue, 1979; Leney and Casteel, 1975; Vernet et al., 1979; Western, 1969). In addition, the increasing number of archaeobotanical reference collections (The Cecilia A. Western Wood Reference Collection Notebbok, created by Eleni Asoloti, <http://pcwww.liv.ac.uk/~easouti/Index.htm>, consulted on 28/09/2021), together with the development of the first publications of comprehensive wood anatomy texts and atlases (Fagerstedt et al., 1996; Greguss, 1955; Greguss, 1959; Grosser, 1977; Hoadley, 1990; Schweingruber, 1978; Schweingruber, 1990; Wagenführ, 1996; Wilson and White, 1986) facilitated and increased archaeological wood and charcoal studies significantly. With the beginning of the 21st century, these types of studies were accelerated by having more routine access to the most sophisticated and advanced microscope, the Scanning Electron Microscope (SEM). Nowadays, the study of archaeological woods and charcoals involve different types of analysis and methodological techniques. These include Fourier-transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy, applied in order to understand the chemical composition and alterations of ancient woods (Moosavinejad et al., 2019; Traoré et al., 2016), dendrochronological studies concerning the provenance and dating of archaeological woods and charcoals (Pichler et al., 2013; Rybníček et al., 2020), stable isotopes analyses of wood and charcoal for establishing wood provenance and climate changes (Hajj et al., 2017; Masi et al., 2013; Vignola et al., 2017), extraction of DNA from archaeological wood (Akhmetzyanov et al., 2020), X-Ray Fluorescence (XRF) analyses on marine archaeological wood (Fors, 2021), Pyrolysis Coupled with Mass Spectrometry (PY/GC/MS) analyses

for the study of degradation processes, and for the development of consolidation and conservation procedures for archaeological woods (Blanchette, 2000; Colombini et al., 2007; Colombini et al., 2009; Łucejko et al., 2012). This brief review aims to illustrate how a meaningful and versatile multidisciplinary approach, encompassing the natural sciences, humanities, and the arts is required to study of this type of remains (Soo Kim and Singh, 2015).

1. 4. 1. 1. Wood

The object of the xylo-anthracological analyses is wood or xylem, which in a strictly botanical sense is the anatomical region of the stem/branch of the secondary structure of plants. Secondary xylem is a vascular plant tissue with the function of transporting water and dissolved minerals within the body of the plant (Cluter et al., 2007; Dumbleby, 1978; Nardi Berti, 2006). It consists of a variety of specialized water-conducting cells called tracheary elements. This well-defined microscopic structure changes according to the plant from which it comes (Giordano, 1981). Wood is a naturally originating product, with extremely variable anatomical characteristics. The environmental conditions exert a strong influence on the growth of the plant from which the wood is obtained. The differences induced by these causes can be so marked as to exceed those found between different wood species (Berti et al., 2007).

There are two main types of wood, beginning with its biological origin. Softwood, resulting from gymnosperms (conifers), is predominantly constituted of axial tracheids. Hard- wood, derived from angiosperms (deciduous), is morphologically more complex, generally including up to five different axial elements counting parenchyma, fibres and, most notably, vessels (Cluter et al., 2007; Giordano 1981).

The anatomy, classification and nomenclature of the different wood species (not described in this thesis by the will of the author) are discussed by specialist literature necessary for the study of these remains (Abbate Edlmann et al., 1994; Crivellaro and Schweingruber 2013; Gale and Cluter 2000; Nardi Berti 2006; Schweingruber 1990).

1. 4. 1. 2. Archaeological wood: its use as a main raw material by past human generations, and its importance as cultural heritage

Wood is one of the most important raw materials used throughout human history. This has been proven by plant remains detected on stone tools dating from two million years ago (Dominguez et al., 2001) and from the few and rare artifacts made of wood dating back to prehistory (Aranguren et al., 2018; Rios-Garaizar et al., 2018). Wood has been continuously utilized by humans, having many uses. It was employed for construction and structural applications, as well as for decorative

purposes, the manufacturing of objects, tools and instruments, artworks, ritual, and religious purposes, and so on (Figure 2).

Wooden artifacts can be considered as important Cultural Heritage as they provide much information about human life and culture, and they are considered worthy of preservation for the future (Lo Monaco et al., 2018; Rowell and Barbour, 1990). Knowledge about human lives and values, as well as the skills and the ingenuity of past generations are embedded in wooden cultural heritage.

Despite its perishable and fragile nature, archaeological wood does not blow in the wind, does not stick to animal fur, and does not easily roll into sediments, in contrast to other types of plant remains such as pollen or seeds. This means that these type of remains are usually part of the archaeological record as a result of the intentionality of human activity in the past. Moreover, they can often be linked to a specific human purpose. A large amount of wooden remains has been retrieved from archaeological sites, although they are



Figure 2: Wooden statue of São Pedro from 18th century - Igreja de São Francisco (Évora). Image provided by Maria Festas.

regrettably few if compared to the widespread inorganic materials and considering the importance of wood. A greater number of wooden artifacts come from firewood as wood charcoal (Deforce and Haneca, 2011; Figueiral et al., 2017; Hazell et al., 2017; Marston, 2009; Moskal-del Hoyo, 2012; O'Donnell, 2016; Piqué and Anton Barcelo, 2002; Scheel-Ybert et al., 2014; Théry-Parisot, 2002). However, archaeological excavations bring to light wooden artifacts of various kinds. Tools, objects, furniture of any type (Aranguren et al., 2018; Costa Vaz et al., 2016; Moreno-Larrazabal et al., 2011; Rios-Garaizar et al., 2018), shipwreck frames (Abbate Edlmann et al., 1989; Allevato et al., 2010; Capretti et al., 2008; Giachi et al., 2003; Sadori et al., 2015), dwellings, settlement remains, and building elements (Albore Livadie and Cicirelli, 2012; Albore Livadie et al., 2012; Billamboz, 2014; Coradeschi, 2013; Giachi et al., 2010; Mavromati, 2020; Moser et al., 2013; Moser et al., 2018) have all been retrieved in the archaeological record of different periods. These copious remains represent

proof of the important role of wood throughout human history, in both its use as a main raw material and its significance as world cultural heritage, stressing the importance of their study.

1. 4. 1. 3. Chemical composition of wood

Wood material is exposed to the chemical recycling processes of nature (Thiébaud, 2008). The chemical and physical characteristics of archaeological wood are analysed mainly to understand the deterioration process of this material and/or to plan conservation treatments aimed to stabilize the wood. Therefore, the improvement of our knowledge concerning microorganisms and processes which affect archaeological woods, as well as concerning the structural and chemical changes that occur in their degradation is extremely important for an in-depth study of these remains.

The major chemical component of sound wood is water, but on a dry weight basis we can define wood as a three-dimensional biopolymer composite. Chemical constituent elements of wood are C (~ 48-51%), H (~ 6-7%), O (~ 43-46%) and N (\leq 1%), which mainly constitute cellulose, hemicellulose and lignin. These substances with a high molar weight (polymers) form the cell wall complex, constituting 95% percent of the dry weight of wood. Minor components of wood are extractives (a term for various organic compounds present in some timbers in relatively small amounts), inorganic compounds, pectin, starch, and proteins (Hedges, 1990; Ramage et al., 2017; Wiedenhoef, 2013). The proportions between cellulose, hemicellulose and lignin define the different types of wood, changing among conifers and angiosperm trees. Both have the same amount of cellulose (40-50%), in conifers the lignin content is higher (25-35% vs 20-25%), while that of the hemicellulose is lower (20-25% vs 20-35 %) compared to angiosperms (these values are calculated on the dry weight of wood) (Blanchette, 2000; Braadbaart and Poole, 2008; Nardi Berti, 2006; Jostrom, 1993). Different factors, like mechanical or ecological (e.g., arid habitat) stress, etc. also contributes to modify these proportions, both within and between species and different parts of the same wood species (Braadbaart and Poole, 2008). Leaning stems of the same species of trees show a different anatomy and chemical composition than “normal” wood. In softwoods, the wood formed on the lower underside of leaning stems or branches is called compression wood. Its cells have a higher lignin content and a lower cellulose content (Blanchette et al., 1994; Conners, 2001). In angiosperms, the area affected by this alteration is the upper side and is called tension wood. It contains more cellulose and less lignin. This type of wood appears to be as more susceptible to decay (Blanchette et al., 1994; Blanchette, 2000; Conners, 2001). Chemical composition of wood is also different within woods from different geographic areas, ages, climates, and soil conditions (Blanchette et al., 1994). Archaeological wood may have the physical characteristics of sound wood or have some physical changes. This wood can be entirely cellulosic or lignitic, or any combination of these. For instance,

the waterlogged wood is mainly constituted of lignin residues (Fengel and Wegener, 1989; Hoffmann and Jones, 1990; Tamburini et al., 2015) in mineralised wood the inorganic chemicals often replace the organic compounds (Florian, 1990).

1. 4. 1. 4. Wood conservation

The conservation of archaeological and historical wood is related to its chemical composition and biodegradability. Wood as a natural organic material, may be subjected to deterioration processes by different agents. The alteration of its structure can produce physical and mechanical modification, over time. Wood degradation can be induced by abiotic or biotic factors and begins when the tree is alive and is still part of the living biomass. Upon its death, with the exposure to atmospheric agents, the tangible process of degradation initiates. Within this process, lignivorous organisms, such as insects, fungi, and bacteria, accelerate wood deterioration. The conservation of archaeological wood in the archaeological sites is related to its burial conditions. Factors such as humidity, temperature, relative chemical composition, and soil type affect the conservation of wood, which is often missing from the archaeological record. Wood remains can be preserved in dry archaeological deposit (dry woods) and in wet archaeological sites (waterlogged woods). Other types of conservation occurs when wooden remains are subject to carbonization or mineralization processes. Different types of conservation can be present in the same archaeological site (Caramiello and Arobba, 2008; Cartwright, 2015). Footprints of wood carved on clay materials or on parts of building structures with which wood came into contact, are examples of how these materials can even be preserved indirectly. The state of archaeological wood conservation is related to the original state of wood remains and to the transformation it undergoes during its deposition. Sometimes wooden material may appear as a dark coloured layer of humus in the archaeological context, but in several cases the slow decomposition rate allows wood to remain in the soil for a decade to millennia (Castelletti, 1990).

1. 4. 1. 4. 1. Preservation of wood remains from dry contexts

Desiccated woods are specific to desertic and sub arid regions. This particular context impedes life and, therefore, the degradation activity of bacteria and/or fungi, supporting a good state of wood conservation (Blanchette et al., 1994; Cartwright and Parkington, 1997; Cartwright and Taylor, 2008; Cartwright et al., 2011; Gale and Cluter, 2000). Exceptionally, dried wood remains can be preserved similarly in cold and dry environments (Cartwright et al., 2009; Heiss and Oeggel, 2009). Woods stored in these particular environments may have different texture, they can be very hard if they are impregnated by mineral crystals, and powdery, but they can also be in a sort of “intermediate state” (Blanchette, 2000; Castelletti, 1990; Nardi Berti, 2006; Macchioni et al., 2012; Nilsson and Daniel,

1990). In some cases, the rapid deterioration of these remains can occur during their excavation, during which the shape, size, orientation of vessels, of the ray and axial parenchyma, and of fibres (or tracheids and rays for softwoods) look altered. These remains look very similar of some waterlogged wood remains, which have been dried too rapidly (Cartwright, 2014). In other circumstances, desiccated woods may have been so altered as to become dust (Dimbleby, 1978). It should be stressed that in most of these situations, the taxonomical identification of these type of remains (with required techniques) is often still possible.

1. 4. 1. 4. 2. Preservation of wood remains from underwater contexts

Waterlogged woods are, after charcoals, the most recurrent type of wooden remains in the archaeological records of temperate zones.

The retrieval of these remains can take place in archaeological sites located under the water table or in underwater contexts, such as the seabed, lakes, or rivers. It can also occur in artificial waterlogged contexts such as pits, wells and ditches of otherwise 'dry' sites (Abbate Edlmann et al., 1989; Björdal et al., 1999; Brennand, M., Taylor, 2003; Brunning, 2007; Capretti et al., 2008; Cartwright, 1996; Clark, 2020; Costa Vaz et al.,



Figure 3: Fragments of a waterlogged wooden pulley from the Roman Cryptoporticus of Lisbon.

2016; Giachi et al., 2010; Green, 1990; Purdy, 1988; Spriggs, 1981; Steffy, 2006) (Figure 3).

Such environments, due to the low temperature and amount of oxygen, promote the reduction of the activity of degrading agents, and therefore, the restriction of the wood deterioration processes. These types of woods generally appear to be in a good condition, despite the fact that in underwater contexts, strong activity of anaerobic bacteria can be observed which compromises the structural stability of these remains. Generally, the result is the advanced decomposition of the main polysaccharide wood components (cellulose and hemicellulose) and lignin oxidation (Blanchette, 2000; Björdal and Nilsson, 2002; Florian, 1990; Tiano, 2001). The physical, chemical, and mechanical properties of these woods are deteriorated, resulting in a decrease in their density and an increase of their micro porosity and permeability (Brunning, 2007). The colours of these type of woods change, and their structure becomes a spongy substance filled with water, making these

remains easily destructible (Brunning, 2007; Pizzo et al., 2010). The drying process of waterlogged woods leads to a considerable alteration of their dimensions, their cell walls can break down and collapse, causing numerous cracks. In some cases, waterlogged woods lose any diagnostic features useful for their identification (Bugani et al., 2009). These processes can be irreversible, consequently a large part of waterlogged wood cultural heritage is easily lost without any artificial preservation (Schweingruber and Börner, 2018).

1. 4. 1. 4. 3. Wood charcoal

Wood charcoal is the result of a slow and partial combustion of wood, due to insufficient oxygen supply (Castelletti, 1990; Deldicque et al., 2016; Raffi and Serpagli, 1993) (Figure 4).



Figure 4: Transversal section of *Quercus faginea* – charcoal reference sample. Image captured using a Hirox 3D Digital Microscope (Hercules Laboratory).

The carbonization process causes the loss of most of the wood components, and wood charcoal is essentially lignitic and very fragile (Braadbaart and Poole, 2008; Tamburini, 2003). However, despite some partial deformations, the wood micro anatomy remains substantially unchanged (Barone Lumaga et al., 2006; Schweingruber and Börner, 2018). Ordinarily, during combustion the wood cell walls lose their fibrillose structure, suffering a reduction. This reduction can reach 7 to 13% longitudinally and radially, and from 12 to 25% tangentially. Therefore, wood charcoal cell walls are 1/5 - 1/4 of their original size (Chabal, 1997; Schweingruber and Börner, 2018). The most influential aspects affecting wood charcoal features are the combustion phase, the subsequent pre-deposition phase, and finally the burial conditions.

1. 4. 1. 4. 3. 1. Preservation of wood remains by carbonization

This type of conservation is the most common among macro-remains of plants (Chabal et al., 1999; Zohary et al., 2012). Charred wood can be preserved in both wet and dry sites. Normally they are small fragments coming from anthropic deposition as a result of a long or shorter combustion, for instance remains from domestic hearths and furnaces, but also surviving objects or structural elements from fires (Grau Almero, 2011; Celma, M. Baños, 2011; Coradeschi, 2012; Jansen and Nelle, 2011; Moreno-Larrazabal et al., 2011; Mols, 1999; Moser et al., 2013; Moser et al., 2018; O'Donnell, 2011).

Wood charcoal is a chemically inert material and therefore is not affected by chemical and physical decomposition after being buried (Braadbaart and Poole, 2008; Braadbaart et al., 2004; Braadbaart et al., 2007; Schweingruber and Börner, 2018). However, low-temperature combustion produces wood charcoal remains in a somewhat "precarious state", where the changing of burial conditions can lead to the loss of its external coating, and thus to the start of the wood degradation process. On the other hand, wood which burned at high temperatures loses its volatile substances and it can therefore be considered a harder material, but consequently more porous and fragile (Castelletti, 1990).

As the anatomy of these remains is generally altered, the type and extent of their conservation is intrinsically linked to their fragility and to post depositional processes. The prolonged exposure of wood charcoals to the elements and their eventual transport by natural or anthropogenic agents can lead to the deformation of these remains, for instance rounding, smoothing or even pulverization. Wood charcoal remains therefore do not retain their original size and/or shape. The conservation of charcoal also depends on the original tree's species, for example, during the combustion of a porous wood (elm, oak, ash, fig woods etc.), fractures are often formed in correspondence with its spring pore line (Barone Lumaga et al., 2006; Castelletti, 1990). Additionally, small twigs become very fragile, and their retrieval can be extremely rare (Mannoni and Molinari, 1990).

1. 4. 1. 4. 4. Preservation of wood remains by mineralization

Wood can also be retrieved after a long time when it was subjected to a mineralization process, during which there is a progressive replacement of the cell wall organic components in favour of salts or mineral oxides, like calcium carbonate or silica, silicates, iron oxides (Castelletti, 1985; Castelletti et al., 1986). Some of these wooden remains retain their unaltered structure, while others undergo its complete destruction, giving the way to an amorphous and vitreous section. This type of mineralization makes it impossible to determine the anatomical characteristics of the wood (Rottoli, 1997a).

Wood mineralization due to the corrosion of metals products, especially those from iron and copper, which were part of the remains themselves or were adjacent to them, occurs both in prehistoric than in historical contexts (Cronyn, 1990; Gillard et al., 1994; Haneca et al., 2012; Stelzner and Million, 2015).

Occasionally, there is the formation of a replica within which the organic portion is missing rather than a complete replacement of the cellular structure by minerals. Surprisingly, this replica is often very similar to the original remains and it is therefore still possible to discern some diagnostic features (Keepax, 1975).

1. 4. 1. 5. Identification of archaeological woods and charcoals: main procedures, issues, and limitations

The identification of archaeological woods and charcoals often goes to the genus level, sometimes to the species recognition, or to lower categories.

The main issues regarding the identification of these plant macro-remains are strictly related to their conservation rather than their age or size. The first and most important criteria to follow in the study of these materials is the adaptation of methods to the wood conservation type. Other identification problems exist for the distinction of species belonging to some genus, for instance the genus *Quercus*, or the distinction of the genus or of the species belonging some families and subfamilies which may include numerous genera, for instance the subfamily of Maloideae (Castelletti, 1990).

Independent of the type of preservation of wood encountered, an initial identification can take place by observing the structure of the wood macroremains at a macroscopic level, i.e., with the naked eye or with the aid of a magnifying glass. This preliminary observation can already take place at the archaeological site and in some cases, it can help in the distinction between the Angiosperm and Gymnosperm wood. Some genera can be distinguished at a macroscopic level by a particularly experienced eye. However, being archaeological wood, this first identification is also particularly reliant on the good condition of the wood.

Concerning the micro-anatomical identification of archaeological woods and charcoals, preparation and observation of the three diagnostic sections is necessary i.e. the transversal section, perpendicular to the long axis of the stem (frequently abbreviated to TS), the radial longitudinal section (RLS), parallel to the long axis, and the tangential longitudinal section, perpendicular to the long axis and tangential to the growth rings TLS) (Nardi Berti, 2006; Wheeler and Baas, 1998) (Figure 5).

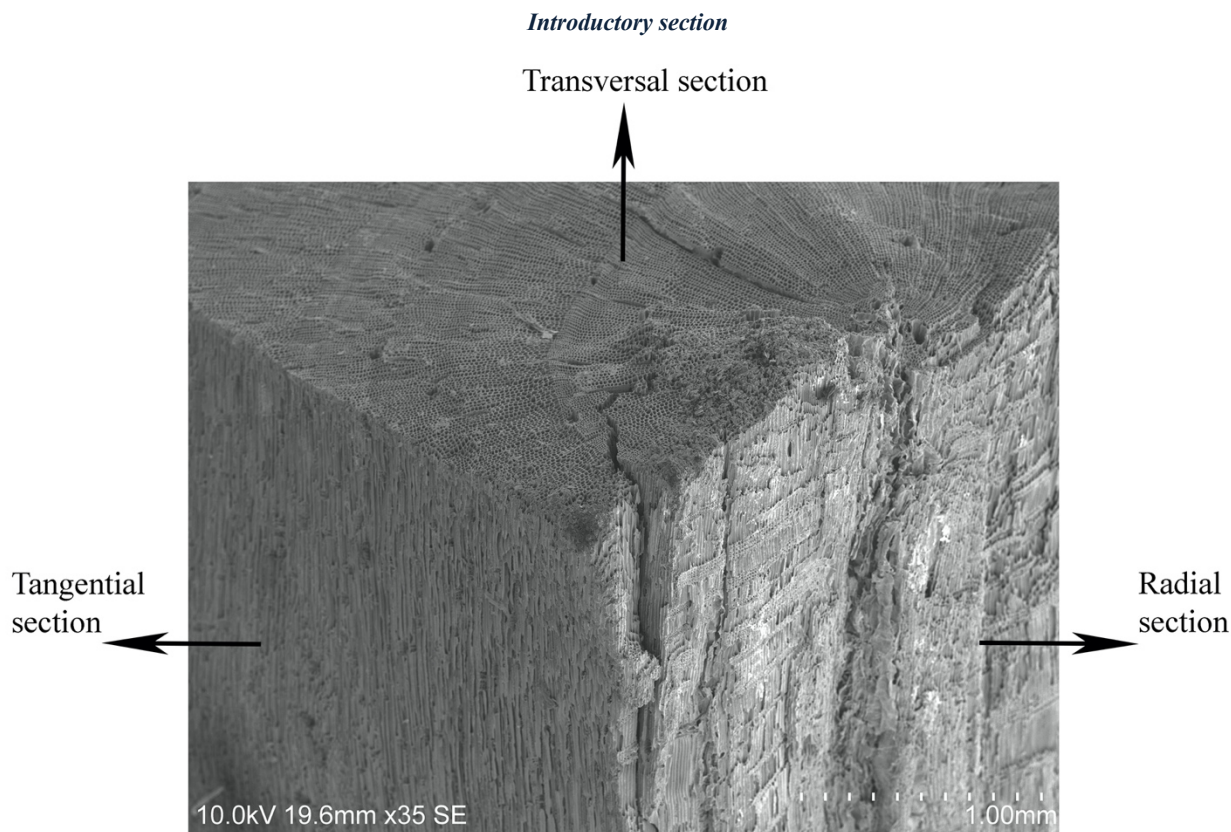


Figure 5: The three principal sections of wood: transverse, tangential and radial. Image captured using a SEM microscope (Hercules Laboratory).

The preparation of the three anatomical sections is the basis for the identification of all types of archaeological wood, whether desiccated, waterlogged, charred or mineral preserved. The preparation techniques of the thin sections are adjusted according to the type of preservation of the wooden remains (Gartner and Schweingruber, 2013). The most common technique is to prepare fresh sections by hand, with the support of a sharp razor blade. Sections can be studied immediately, simply immersed in water, or stained and permanently mounted on slides. If necessary, sections can be obtained with the aid of a sliding microtome, otherwise in the case of very soft materials they can be consolidated or frozen. Often, archaeological wood and charcoal remains are stuck in blocks of sediment or inside clay artifacts and may need to be embedded in resin before being sectioned. In all these cases sections are generally observed under a transmitted light microscope using bright-field or polarized illumination.

Regarding archaeological charcoal identification, the most common technique is to obtain sections by hand or using a sharp razor blade, without any preparation, under a stereomicroscope (Pearsall, 2015). The observation of anatomical features is made using a reflected light microscope. Some authors recommend the use of a liquid corrector or a micro cast (Angeles, 2001) for better visualization of the anatomical components, both in wood and in charcoal remains. The use of a Scanning Electron Microscope (SEM), although not strictly necessary in most cases, can be helpful during the identification of particularly fragile samples, for which sectioning can be difficult if not

impossible, or in the case of particularly small samples whose identification with the optical microscopy would be problematic (Attenbrow and Cartwright, 2014).

Moreover, the utilization of SEM and of later generation electron microscopes, for instance Variable Pressure Scanning Electron Microscope (VP SEM) and Emission Scanning Electron Microscope (FE SEM) is essential for the obtaining of high resolution pictures (Carlquist, 2012; Cartwright et al., 2012; Leme et al., 2010). The use of VP SEM also allows for the observation of wooden samples without being coated, which could be essential for the study of some wooden artifacts (Cartwright, 2015; Ostapkowicz et al., 2013).

In all these cases, the use of reference atlases is recommended for the micro anatomical identification of wooden remains (Crivellaro and Schweingruber, 2013; Nardi Berti, 2006; Parsa Pajouh and Schweingruber, 1985; Schweingruber, 1990; Vernet et al., 2001). Atlases of microscopic wood anatomy are increasingly numerous nowadays, both regarding European species and the so-called “exotic” wood species. However, the use of these type of literature references has two limitations, firstly they generally only describe the micro-anatomy of sound wood samples and secondly, they usually do not contain all the species present in a specific geographical area.

For this reason, for a specific identification (genus or species) the use of an in-lab reference, containing wood species belonging the original area of samples under study, is considered necessary (Pearsall, 2015).

Often, the identification of wood remains does not only take into account the wood *taxon* but also some other important data, for instance the maximum dimensions of fragments, their weight, their shape, the possible conservation of the last ring and of the bark (to determine the season of death), the diameter from the last curvature ring, the possible presence of secondary fillings in the wooden cavities or fungal hyphae etc. (Marguerie and Hunot, 2007). Archaeological wood and charcoal that have suffered reduction, distortion, alteration, or fungal/insect attack, require caution and discretion regarding quantitative descriptors.

The quantitative, the qualitative analysis and the elaboration data it must be established based on the wooden remains' typology and on the issue to be addressed, which vary from different contexts and case studies. Among useful text are: (Baxter, 1994; Drennan, 2010; Pearsall, 2019; Pielou, 1984; Shennan, 1997; VanPool and Robert, 2011).

1. 4. 2. Archaeocarpology

Archeocarpology (from the Greek archaios, ancient, Karpós, fruit and lógos, speech) is a archaeobotany sub-discipline which studies seeds, fruits, and floral annexes (i.e., gramine spikelet residues) retrieved from archaeological sites. These type of remains represent an essential witness to

the past as a vehicle for information about the human activities of collection, storage, transformation, and consumption of plant resources, both wild and cultivated (Mateus et al., 2003). Moreover, archaeological seeds and fruits can give data for the reconstruction of the plant domestication history and of the ancient flora of a specific area (Buxó and Capdevila, 1999; Kislev and Melaned, 2000; Pearsall, 2015).

The beginning of the investigation of archaeological seeds and fruits dates to the 19th century (Kunth, 1826; Pearsall, 2015). The introduction of the Flotation Method for macroremains recovering (which is based on the organic and inorganic material density difference) (Struever, 1968), began by the end of the 1960s. The method underwent many variations over time, including the adoption of different kinds of machines (Figure 6) and even the use of high weight liquids, to promote the flotation of organic material (Pearsall, 2015).

Thanks to this new sampling method, the partial selection of macroremains by the naked eye, and based on the selective criteria of the sampler, has been exceeded. From this moment, the recovery of archaeological seeds and fruits becomes systematic, and their analysis becomes both qualitative and quantitative. Therefore, it has become possible to compare carpological assemblages from different contexts of the same site or from different sites.

Later, the evolution of these studies was improved by the evolution of microscopic techniques, culminating with the introduction of the Scanning Electron Microscopy. This new type of microscopy allowed one to reach a very high level of detail concerning the observation and the identification of these botanical remains (Dykstra and Reuss, 2003; Keshri, 2014; Robards, 1991;).



Figure 6: Flotation machine assembled by the Hercules Laboratory for the treatment and the study of the archaeological sediments.

Since the end of the 1990s, the study of archaeological seeds and fruits has become more common and the diversity of the approach between the European and American schools in this field became less marked. All of these factors contributed to research advances in this area. Nowadays, Archeocarpology is a multidisciplinary research sector, capable of answering different questions, including archaeological, botanical and environmental in a broad sense. To exploit the potential information of archaeological seeds and fruits, Archaeocarpology currently employs different analytical techniques (Pearsall, 2019).

1. 4. 2. 1. Plant seeds and fruits

A seed is the reproductive and propagation unit, which contains the embryo (nutrient reserve tissue, generally the endosperm) that develops from the ovule after its fecundation. It is crucial for the propagation and survival of Spermatophyte plants (Gymnosperms and Angiosperms). In Angiosperms seeds are enclosed by the fruit, whereas in Gymnosperms they are enclosed by cones. A fruit and its adhering parts can be defined as the seed-bearing organ of the plant, grown from the ovary (the female organ of the flower)(Cappers and Bekker, 2013; Esau, 2006).

Seeds and fruits are characterized by great variation in terms of size, shape, and surface appearance. These three morphological characteristics are essential for the identification of these macro remains. In Angiosperms, the seed and the fruit connate in many cases. In these cases, the fruit is not much larger than the seed, and is often wrongly defined as a seed (Cappers and Bekker, 2013). The morphology, the classification and the nomenclature of seeds and fruits (not described in this text by the author's will) can be found in specialist texts, necessary for the study of these remains (Cappers and Bekker, 2013; Renfrew, 1973; Greig, 1989).

1. 4. 2. 2. Carpological assemblages within archaeological site: evidence and interdisciplinary research lines

Seeds and fruits included in the same archaeological deposit, with the same chronology, are part of the same "carpological assemblage". In nature, seeds and fruits form the so-called "seed rain", which settles in soils and are considered part (in more or less abundant quantities) of the local vegetation of a certain area (Wasylikowa, 1986).

Human activities which took place in a certain archaeological context could replace the natural deposition and dispersion actions of seeds and fruits. Carpological assemblages can be produced by plants which grow in a specific area (natural deposit) or by outdoor or indoor human activities (anthropic deposit). Archaeological sites generally include both type of carpological deposit (Bandini Mazzanti et al., 2005; Caramiello and Arobba, 2008)

Archaeological seeds and fruits are generally retrieved in particular archaeological contexts, depending on their conservation. The most common contexts in literature are: humid areas and wet artificial environments; garbage pits; sewers and pit latrines; food storage structures; outdoor and indoor anthropic surfaces; warehouses and silos; furnaces and hearths, necropolises; brick material with footprints/casts (Figure 7) (Chabal, 1997; Nisbet, 1990; Pearsall, 2015; Renfrew, 1973; Zohary et al., 2012).



Figure 7: A. Charred grape seeds (*Vitis vinifera*) from the Roman wine cellar of the archaeological site of Torre dos Namorados (Fundão, Portugal); B. Charred indeterminate caryopsis fragment and charred oat caryopsis (*Avena* sp.) from an Islamic septic tank of the archaeological site of Paço dos Lobos da Gama (Évora, Portugal); C-D. Mineralised grape seeds (*Vitis vinifera*), fig achenes (*Ficus carica*) and chickpea seed (*Cicer* cf. *arietinum*) from a combustion area of the Islamic archaeological site of Paço dos Lobos da Gama.

Knowledge concerning the different archaeological contexts and their formation is crucial for the study of these remains (Bandini Mazzanti et al., 2005).

Anthropic deposits of seeds and fruits are commonly analysed to obtain information concerning the subsistence economy and vegetal diet of past human communities. Remains of food plants and spices can be used to learn about broader practices linked to the production, consumption of food and to the origin of aliments in the past (Cappers and Neef, 2012; Lev et al., 2005; Pearsall, 2019). These remains could also shed light concerning the use of plants for their medical properties in dealing with human health, but also on the rise of agriculture, the domestication of plants, the introduction of exotic plants and more on plant-people relationships generally (Buxó and Capdevila, 1999; Pearsall, 2015; Zohary et al., 2012). Besides the taxonomic identification of archaeological

seeds and fruits, other types of analyses can be done on these types of macroremains, being valuable sources of information. The biometrical data collection of archaeological seeds and fruits is particularly useful to distinguish between cultivated and wild forms. For instance, many morphometrical studies were carried out on archaeological grape seeds in order to shed light on the grape seed domestication (Bouby et al., 2013; Orrù et al., 2015; Ucchesu et al., 2015; Ucchesu et al., 2016). Stable isotopes analyses of archaeological grain and pulses are useful to infer natural and anthropogenic effects on their growing conditions. For instance, to explore the water content status of these plants and to identify a possible manuring use (Fiorentino et al., 2015; Fraser et al., 2013; Masi et al., 2014; Messenger et al., 2010; Nitsch et al., 2015). Ancient DNA studies of archaeological seeds and fruits (Schlumbaum et al., 2008) as well as the characterisation of archaeological seeds and fruits by chemical biomarkers, employing gas chromatography with mass spectrometry, and new approaches on absolute dating of archaeobotanical remains (Caracuta et al., 2014; Schlütz and Bittmann, 2016) are worth mentioning.

1. 4. 2. 3. Identification of archaeological seeds and fruits: main procedures, issues, and limitations

Archaeological seeds and fruits can be identified at the genus or species level, often to the lower categories. In some cases, the identification of these remains is possible even if they are in a fragmentary state. The identification of the family of origin is essential for the characterisation of a single seed or fruit. Some families include numerous genera, and the morphology of seeds and fruits varies considerably. The grouping of genera into families is built on generative plant parts but specific adaptations to seed dispersal mechanisms have also taken place within some families (Cappers and Bekker, 2013). The greatest difficulties for the determination of archaeological seeds and fruits are related to their conservation (Messenger et al., 2010). Seeds and fruits preserved by mineralization are subject to deformation, which often leads to a great reduction of their size (Rottoli, 1997b). Carbonization processes can also affect the size and the shape of these remains, for instance a tendency to decrease in size has been observed for charred cereal grain (caryopsis) (Ferrio et al., 2004; Hopf, 1995; Hubbard and Al Azm, 1990; Renfrew, 1973). Within the analysis of carpological remains, knowledge concerning the context of provenience for the archaeological seed or fruit is required to better evaluate the analytical approach to be used. The first step in the identification of archaeological seeds and fruits is their isolation from the remaining archaeobotanical material, and from any other organic and inorganic residues present in the sediment matrix. This operation is generally performed under a stereo microscope at low magnifications (60-200x) and may involve different timing, depending on the type of fraction under study. The use of a fine-tipped brush is

recommended, tweezers can be employed with the less soft carpological remains. For the taxonomic identification the use of a high-resolution microscope (80-100x) with an external lateral illumination (when possible) may be necessary (Bandini Mazzanti et al., 2005; Caramiello and Arobba, 2008; Pearsall, 2015).

The use of a Scanning Electron Microscope is particularly suitable for the micro-anatomical observation of some particular parts of seeds or fruits, for instance to distinguish wild cereal species from proto-domestic forms (Harlan, 1995; Renfrew, 1973; Zohary et al., 2012). Specialised bibliography and an in-lab reference collection are necessary for the identification of carpological remains (Berggren, 1969; Berggren, 1981; Hubbard, 1992; Jacomet, 2006).

The detection of biometric data may be necessary, depending on the size and type of the carpological remains (wild or domesticated plant) (Primavera and Fiorentino, 2013).

Carpological analysis generally includes counts or weights of carpological remains, which can take place during, or after, their identification (Hubbard and Clapham, 1992). The processing of the results is generally expressed in concentrations or percentages. Data elaboration must be established on the basis of the type of carpological remains, the context, and the issue to be addressed (Pearsall, 2015).

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2nd CHAPTER. Research aims

The exploitation of plant resources in the human history: an archaeobotanical insight

2.1. The general goal of the thesis

Archaeobotany is a discipline which should be considered significant for the study of cultural heritage. In this work, archaeobotanical data are regarded as technological, economical, and social indicators of past human communities. These indicators are capable to broaden our knowledge about the human groups which produced such cultural materials. The thesis aims to explore the interdisciplinary nature of Archaeobotany, looking at it as a fundamental tool for the study of material and immaterial cultural heritage.

The goal of the thesis is the characterization of the relationships between past human communities and their surrounding environment, through the analysis of different kinds of archaeobotanical macroremains recovered from various archaeological sites of different periods: Chalcolithic, Etruscan, Roman and Islamic. This type of analysis has shown how past human societies were able to make conscious decisions about the choice and the use of raw materials.

Above all, the thesis aims to get information concerning the human exploitation of plant resources, for instance, the study of building materials, of the plant selection for ritual purpose, of subsistence and consumption strategies of food and drinks.

The selection of the archaeological sites and materials studied in this thesis work was carried out on the basis of the archaeological questions formulated directly by the archaeologist responsible for the excavation. This emphasises the flexibility and the utility of the analyses.

2.2. Case studies objectives

The objectives of each case study are listed and described below. The case studies are considered in a chronological order.

Case study 1 - “A Multi analytical approach of a Chalcolithic funerary deposit from Perdigões (Alentejo, Portugal) to uncover the human exploitation of the natural resources and the wood burning temperature”

The first case study of the thesis is the anthracological analyses of charcoal from the Pit 16 of Perdigões archaeological site. The large, ditched enclosure of Perdigões is located in the Reguengos de Monsaraz municipality (Alentejo, Portugal). Several funerary features have been discovered. The Pit 16 is a secondary deposition of cremated human remains attributable to the middle of 3rd millennium BC.

This study aims to evaluate the human choice and the use of woods employed during the primary funerary ritual, and estimate the temperature of the funerary pit. Two different approaches were used, the wood *taxa* were determined by anthracological analysis to uncover the human

exploitation of the natural resources, and chemometric analyses of FTIR data of charred woods (modern and archaeological) was used to access the temperature of the funerary pit.

Case study 2 - “The wooden roof framing elements, furniture and furnishing of the Etruscan *Domus dei Dolia* (Vetulonia, Southern Tuscany, Italy)”

The second case study of the thesis is the anthracological analysis of charred woods recovered from the *Domus dei Dolia* archaeological site, located in the little village of Vetulonia (Southern Tuscany, Italy). The *Domus* was inhabited since the 3rd century BC and its destruction dates to the 1st century BC.

The objectives of this study are the *taxa* identification of charred wooden remains (employed for the building of the roof and as furnishings elements of the house) and the understanding of the origin of plants selected. The evaluation of the plant's selection and the assessment of the technological knowledge regarding timber characteristics by the Etruscan carpenters is extremely important for this study.

Case study 3 - “Archaeometric study of waterlogged wood from the Roman *criptopórtico da Rua da Prata* (Lisbon, Portugal)- preliminary data”

The third case study of the thesis is the xylological analyses of the waterlogged wood from the Roman *Criptopórtico da Rua da Prata* (Lisbon, Portugal). The Cryptoporticus was built in the middle of the 1st century AD and was partially dismantled between the end of the 3rd century and the beginning of the 5th century AD.

The aims of this study are the *taxa* identification of the waterlogged samples and the evaluation of the wood conservation status by pyrolysis-gas chromatography - mass spectrometry (Py-GC/MS) analysis.

In addition, this study aims to provide further information concerning the archaeological interpretation of the archaeobotanical remains retrieved from the Roman cryptoporticus and to add new information concerning the story of this monument.

Case study 4 - “A glimpse into the viticulture of the Roman Lusitania: morphometric analysis of charred grape seeds from Torre dos Namorados archaeological site (Fundão, Portugal)”

The fourth case study of the thesis is the analysis of charred grape seeds from the Roman *lacus musti* of Torre dos Namorados archaeological site, located in Quintas da Torre (Fundão, Portugal). The objectives of this study are the characterization of the archaeological grape seeds,

2. Research aims

distinguishing whether they are cultivated or wild and the exploration concerning eventual similarities between the archaeological grape seeds and the modern grape seeds (both wild and cultivated) coming from the National Ampelographic Collection of the Instituto Nacional de Investigação Agrária e Veterinária (INIAV) (Dois Portos, Portugal)

Case study 5 – “Exploring archaeobotanical investigation of Paço dos Lobos da Gama archaeological site: an Islamic suburb of Évora, Alentejo, Portugal”

The fifth case study is represented by the carpological and anthracological analysis of the charred and mineralized archaeobotanical materials from the archaeological site of Paço dos Lobos da Gama, located in the center of the city of Évora (Alentejo, Portugal).

This study aims to get insight on the past diet and on the subsistence, consumption, and storage strategies of this Islamic community, as well as identify possible introduction of exotic species.



3rd CHAPTER. Materials and methods

The exploitation of plant resources in the human history: an archaeobotanical insight

3. 1. Materials under study

Different types of archaeobotanical macroremains were analysed within this thesis. Materials came from different and diachronic archaeological contexts, and they presented different states of preservation.

All materials, both archaeological and modern reference materials will be described in detail from a qualitative and quantitative point of view within each of the chapters describing the individual case studies.

3. 2. Introduction to the analytical methodologies chapter

This chapter lists and describes the methodologies employed and developed in the different case studies of the thesis. Each methodology was chosen according to the state of conservation of the material to be analysed, and the objectives to be achieved. In some cases, the analytical methodology was used in, multiple case studies.

To facilitate the reading, the chapter is organized as follows: the methodologies were grouped and described according to the type of the material analysed. For each method described, the case study for which that method was used is stated in the text. At the end of the chapter, an overview table of methods employed in the thesis is reported (Table 1).

3. 3. Quantification methods

For purpose of clarity, the quantification method employed for each case study will be mentioned and described in the relevant case study chapter.

3. 4. General sampling strategies

In most cases, the sampling of the materials took place previously during the archaeological fieldwork, without the direct involvement of the PhD student. For purpose of clarity each sampling strategy will be mentioned and described in the relevant case study chapter.

3. 5. Sediment treatment

3. 5. 1. Manual Flotation plus water sieving (Case study 5)

Sediment samples volume was measured at first (Figure 8A). Subsequently, botanical macroremains were isolated from the heavier artifacts and soils using the manual flotation technique, which works due to the difference in density between the organic and inorganic materials (Figure 8B). The soil was gently broken down in water (for about 5-10 minutes), then swirled around in a bucket and carefully poured out, washing over the light material (seeds, fruits, charcoals) which is, at the end, collected in a sieve of appropriate mesh. After this first treatment, the heavy fraction (that

3. Materials and methods

remained on the bottom of the flask) is washed manually, in order to extract the non-floating macroremains (Greig, 1989; Pearsall, 2015) (Figure 8C). Three different sieve mesh sizes are used: 4 mm, 2 mm, and 0.25 mm. Thus, the light fraction is collected and then dried overnight at a temperature not exceeding 22 °C.

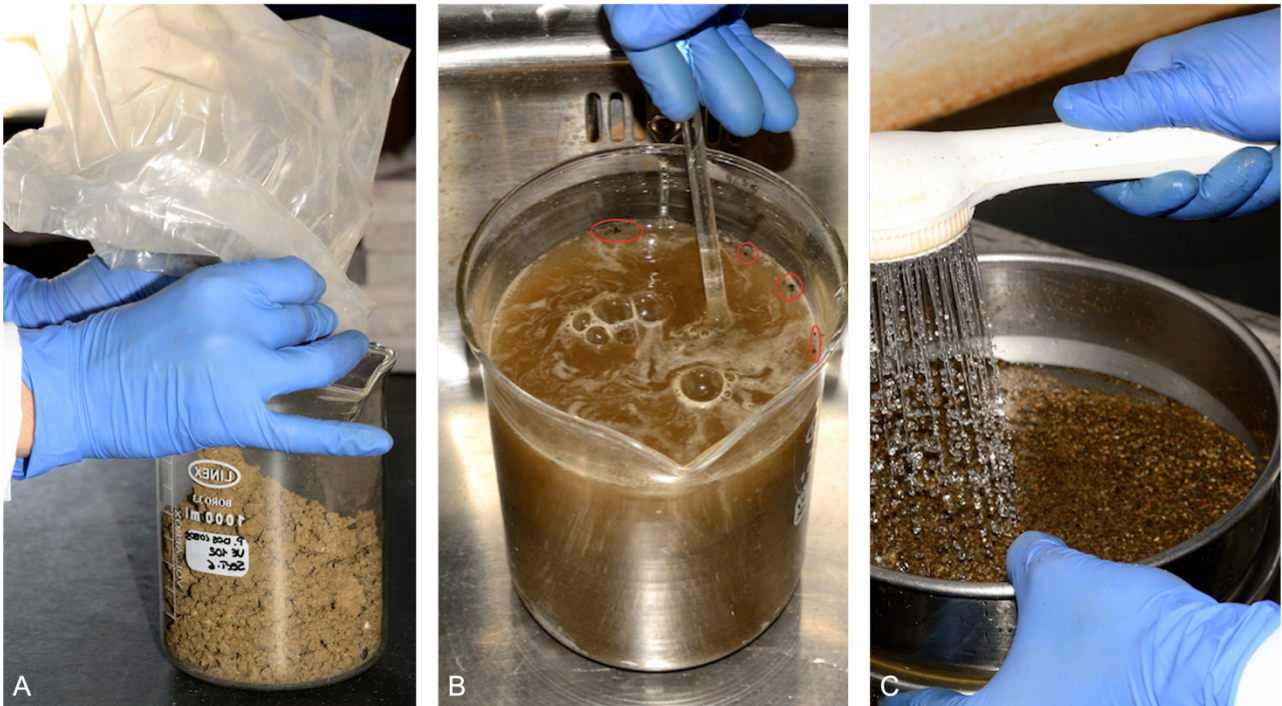


Figure 8: A. Measurement of a sediment sample from Paço dos Lobos da Gama archaeological site (Évora, Alentejo); B. Isolation of botanical macroremains through manual flotation; C. Water sieving of the heavy fraction that remained on the bottom of the flask.

3. 6. Preliminary screening of plant macroremains (Case studies 1, 5)

Seed, fruit, and charcoal samples larger than 3 mm were observed, isolated, and sorted using a Stereo-Zoom Microscope at low magnification (LEICA M205C equipped with a camera).

3. 7. Analysis of charcoals (Case studies 1, 2, 3, 5)

3. 7. 1. Charcoal reference collection

Due to the fragmented and altered nature of archaeological charcoal remains, the building of a comparative reference collection of modern charcoal samples is currently an essential tool for a PhD thesis in Archaeobotany.

3. 7. 1. 1. *The harvesting of species*

The collection of many modern wood species was done in collaboration with Professor Anabela Belo from the Biology Department of the University of Évora.

Most of the species were collected in the vicinity of the Herdade da Mitra (University of Évora), which is located near the village of Valverde, 12 km from Évora. Some other species were collected throughout the Alentejo region and in different parts of Portuguese territory. In particular, the species belonging the genus of *Erica* were collected on the Serra Estrela (Beiras, Portugal), due to the strong presence and variety of this genus in this area of Portugal. Some species belonging the *Quercus* group were collected in Italy, in the vicinity of the *Domus dei Dolia* archaeological site (Vetulonia, Southern Tuscany, Italy). Regarding the species of *Abies alba* and *Fagus sylvatica* they were collected on the Amiata Mountain (Southern Tuscany, Italy).

The following wood species were collected:

Abies alba (Mill.), *Acer campestre* (L.), *Acer negundo* (L.), *Alnus glutinosa* (L.), *Buxus sempervirens* (L.), *Calicotome villosa* (Poir.), *Cistus crispus* (L.), *Cistus ladanifer* (L.), *Cistus monspeliensis* (L.), *Cistus psilosepalus* (L.), *Cistus salvifolius* (L.), *Erica arborea* (L.), *Erica scoparia* (L.), *Erica umbellata* (L.), *Fagus sylvatica* (L.), *Frangula alnus* (L.), *Ficus carica* (L.), *Fraxinus angustifolia* (Vahl.), *Fraxinus excelsior* (L.), *Junglas regia* (L.), *Malus domestica* (Borkh.), *Mirtus communis* (L.), *Olea europaea* (L.), *Olea europaea* (L.) var. *Sylvestris* (Mill.), *Pinus pinea* (L.), *Pinus pinaster* (Aiton), *Populus alba* (L.), *Populus nigra* (L.), *Pirus communis* (L.), *Pistacia lentiscus* (L.), *Phillyrrea latifolia* (L.), *Prunus avium* (L.), *Prunus dulcis* (Mill.), *Prunus pérsica* (L.), *Quercus cerris* (L.), *Quercus coccifera* (L.), *Quercus faginea* (Lam.), *Quercus robur* (L.), *Quercus suber* (L.), *Quercus ilex* (L.) subsp. *Rotundifolia* (Lam.), *Salix atrocinerea* (Brot.), *Taxus baccata* (L.), *Vitis vinifera* (L.).

3. 7. 1. 2. Charring comparative wooden species and creation of a database of Scanning Electron Microscope (SEM) images

After the sampling, wood species samples were charred inside a muffle furnace for 30-60 min at 400 – 500 °C in a reductive atmosphere, depending on wood sample diameter and density. A reductive atmosphere inside the furnace was created by charring the wood samples in sand as some authors have already reported (Pearsall, 2015). Afterwards, samples were stored and catalogued for easy future retrieval and use. They were sorted alphabetically by family, genus, and species names. Scanning Electron Microscope, SEM, (HITACHI S3700N) pictures of some charred modern wooden samples were captured after the gold coating procedure (Figure 9) (for the SEM conditions used see chapter 3- 3.7.2).

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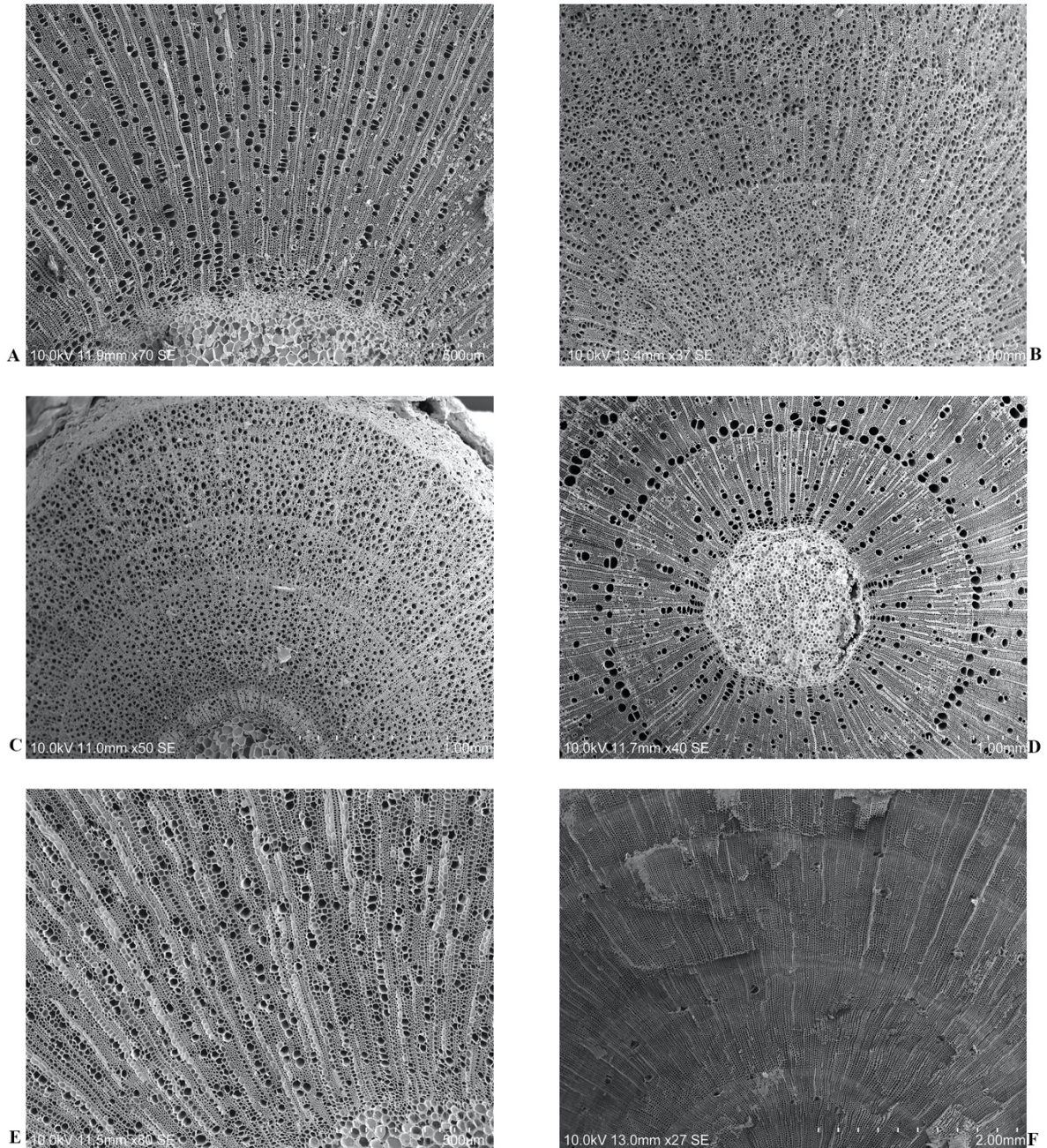


Figure 9 SEM pictures of charcoal reference collection. A. Transversal section of *Acer negundo*; B. Transversal section of *Arbutus unedo*; C. Transversal section of *Cistus salvifolius*; D. Transversal section of *Fraxinus angustifolia*; E. Transversal section of *Olea europaea*; F. Transversal section of *Pinus pinaster*.

3. 7. 2. Taxonomic identification of charcoal samples – Anthracological analysis (Case studies 1, 2, 5)

The charcoal fragments were manually fractured to expose the three diagnostic sections (transversal, tangential and radial) under a stereo-zoom microscope (LEICA M205C equipped with a camera). The charcoal's anatomical features were determined on a reflected light optical microscope (LEICA DM2500 equipped with a camera) at different magnifications (50–1000x) (Figure 10A). A

3. Materials and methods

selection of charcoal fragments was analysed with a SEM to obtain high-resolution images (Figure 10B). Analyses were performed using a variable pressure HITACHI S3700N SEM, and the operating conditions for the analysis were as follows: secondary electron mode (SE), 10 kV accelerating voltage, 10 mm working distance, 65 μ A emission current and <1 Pa pressure in the chamber. All the samples were covered with a gold layer prior to the analysis. Wood atlases (Crivellaro and Schweingruber, 2013; Nardi Berti, 2006; Schweingruber, 1990a; Schweingruber, 1990b) were used as comparative tools for the charcoal identification, together with an in-lab reference collection of wood specimens. The analysis identifies wood charcoal fragments at the highest possible taxonomical level (family/genus/section/species). The level of identification depends, in fact, on the available and visible diagnostic micro-anatomical characters of single wood species/specimens. Some other types of observation were also made, when possible, for instance the growth ring curvature observation and the recording of the presence of xylophagous galleries (Castelletti, 1990; Marguerie and Hunot, 2007; Schweingruber et al., 2008; Théry-Parisot and Henry, 2012).

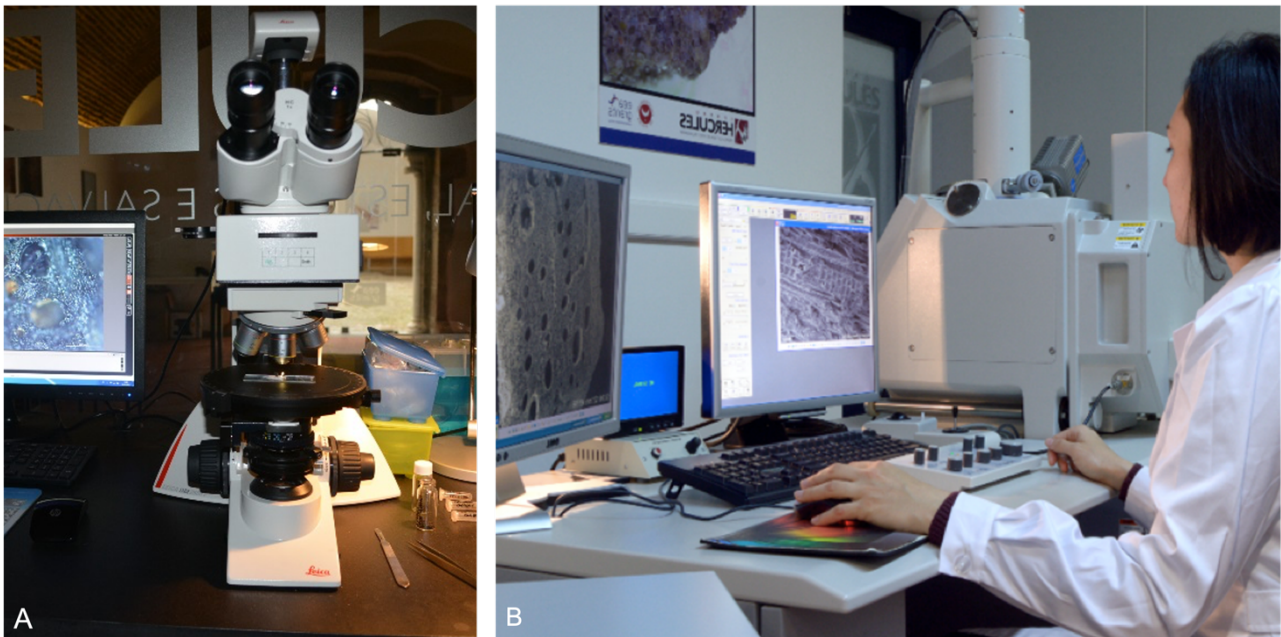


Figure 10: A. Observation of archaeological charcoal sample under a reflected light optical microscope; B. Anthracological analysis using SEM at the HERCULES Laboratory.

3. 7. 3. Preparation of reference material for the statistical model (Case study 1)

In order to make a statistical model able to estimate the absolute combustion temperature of archaeological charcoals an in-lab charcoal reference collection was created, and different experimental combustion tests were carried out. After sampling, sound wood *taxa* samples were cut (dimension: 1 cm³, \approx 3 g) using an automatic saw, then washed with distilled water and dried for 24h (Figure 11A). Samples were then positioned into ceramic crucibles, heated in the absence of oxygen,

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between 350 and 600°C (typical burning temperature of wood in natural fires (Swift et al. 1993) (Figure 11B-C). In total, 6 different heating steps were used, increasing the muffle furnace temperature of 50°C each time.

Samples remained within furnace during 90 min at maximum temperature. For each heating step, two replicates of each *taxon* were burnt. At the end of the heating period, a sample of each laboratory-based combusted charcoal was photographed by SEM after the gold coating procedure (see the instrumental settings described in the previous section). Furthermore, an aliquot from each obtained charred wood samples was combined to create one composite sample for Infrared Spectroscopy Analysis (Figure 3D).

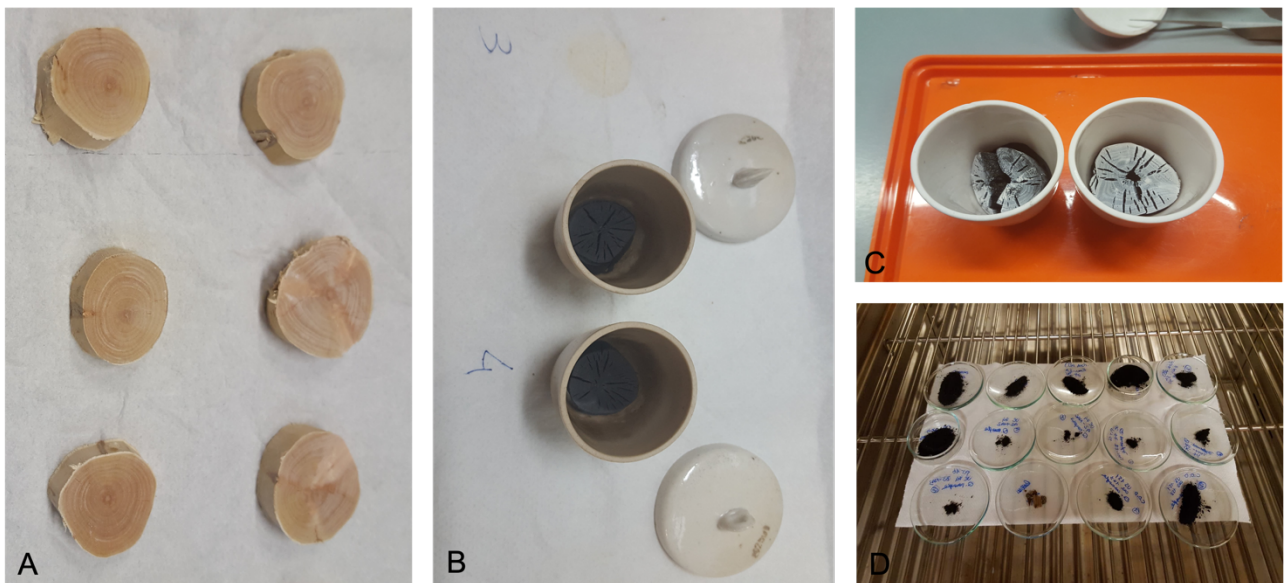


Figure 11: A. Modern sound wood samples of wild olive tree (*Olea europaea* var. *sylyestris* (Mill.)); B. Charred samples of wild olive tree at 350°C ; C. Charred samples of wild olive tree at 550°C; D. Composite charred samples of different wood species analysed for the study of Pit 16 from Perdigoes archaeological site (Alentejo, Portugal).

3. 7. 4. Fourier Transform Mid-Infrared Spectroscopy (FT-MIR) (Case study 1)

Fourier Transform Mid-Infrared spectra were obtained using an Alpha -R™ Spectrometer (Bruker Optics®, Germany), with Attenuated Total Reflectance (ATR) module (Platinum-ATR-sampling module, Bruker, Germany), at a wavelength range of 4000–400 cm^{-1} and a resolution of 2 cm^{-1} (Figure 12A-B). To improve the signal to noise ratio, 60 spectra were co-added and averaged for each recorded spectrum. Spectral data were background corrected to a reference spectrum obtained prior to every measurement, and some spurious absorptions, such as peaks from atmospheric CO_2 , could be removed.

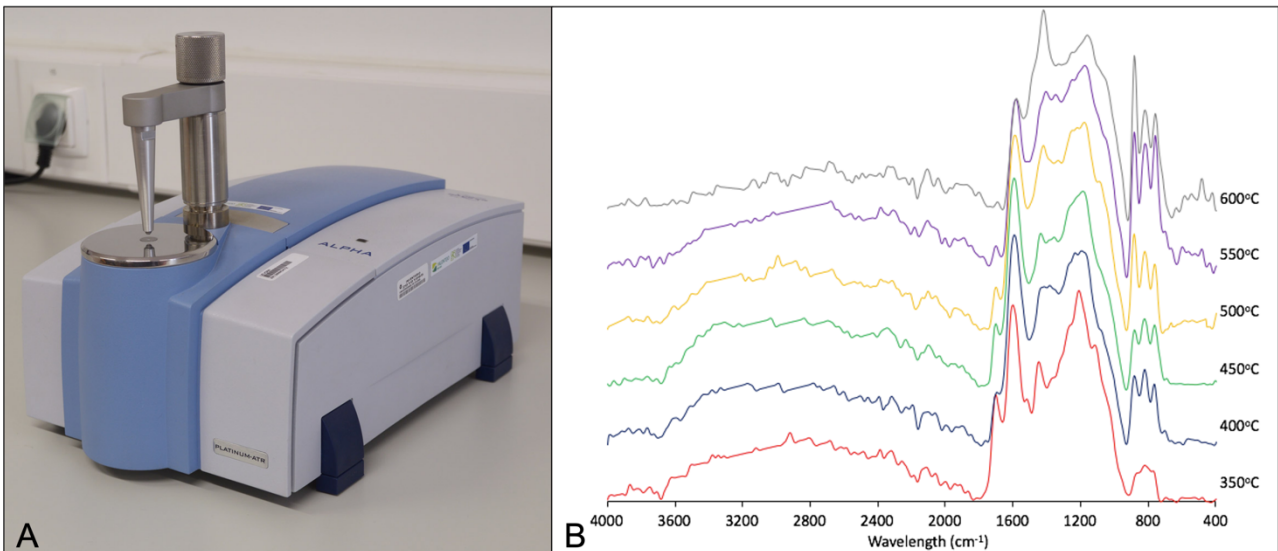


Figure 12: A. Alpha Spectrometer (Bruker Optics®, Germany) of the HERCULES Laboratory employed for the FT-MIR analysis of charred woods from Pit 16 (Perdigões archaeological site); B: Infrared spectra of a sound wild olive sample burnt at different combustion temperature.

3.7.5. Partial Least Squares (PLS) analysis (Case study 1)

Partial Least Squares (PLS) regression models were generated using the ParLeS software (Viscarra Rossel 2008). This method has been used to acquire predictive cremation temperature models from the FT-MIR spectral intensities in the range 1800–400 cm⁻¹ (independent variables, 250 data points). The spectral pre-processing treatments consisted of a light scatter and baseline correction by Standard Noise Variate (SNV), a de-noising by using median filter, and a mean centering (Jiménez-González et al. 2019). The Root Mean Squared Error (RMSE) and the Akaike’s Information Criterion (AIC) were used, in order to prevent overfitting, to determine the best number of factors (latent variables) for each model. Lastly, the diagnostic spectral regions of the FT-MIR spectra were studied by the combination plot of the Variable Importance for Projection (VIP) values in the studied spectral range. The VIP traces can be useful to identify the independent variables (spectral peaks), that may be linked to the temperature of the cremation. Spurious forecast models due to overfitting were discarded after comparing PLS models calculated with the randomized combustion temperature (dependent variable).

3.8. Analysis of waterlogged materials (Case study 3)

3.8.1. Wood thin sections (Case study 3)

Waterlogged wood samples were embedded in PEG 1500 (polyethylene glycol) (Bleicher, 2008) (Figure 13A) and cut using a Rotative Microtome (MicroTec CUT 4055) to obtain transversal, tangential, and radial sections of 10 microns in thickness (Figure 13B-C).

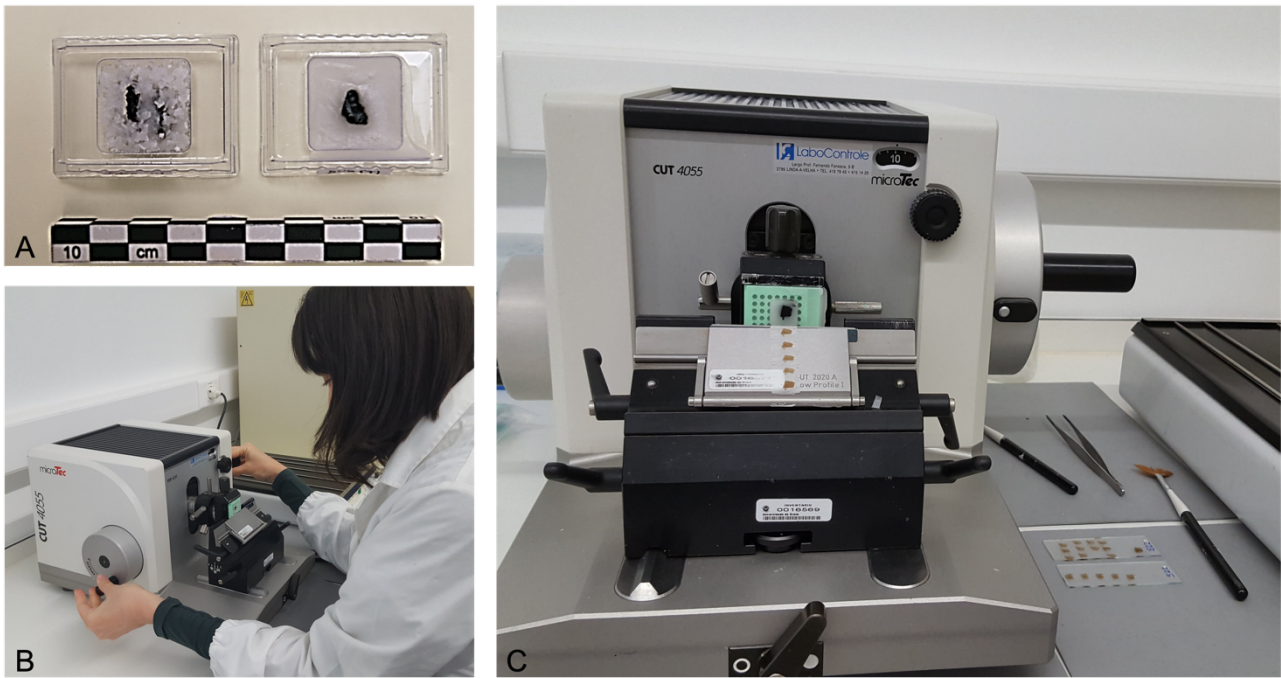


Figure 13: A. Waterlogged wood samples embedded in PEG at the beginning (left) and at the end of the preparation (right); B-C. Sectioning of waterlogged wood samples with the rotative microtome of the Laboratory of Advanced Microscopy and Flow Cytometry.

3. 8. 2. Taxonomic identification

3. 8. 2. 1. Waterlogged wood identification - Xylogological analysis (Case study 3)

Wood taxa were identified by Transmitted Light Microscopy (LEICA DM2500 equipped with a camera) at different magnifications (50–1000x) (Figure 14 A-B). The identification was done based on the diagnostic anatomical features observed in the samples. Specialised bibliography (Crivellaro and Schweingruber, 2013; Nardi Berti, 2006; Schweingruber, 1990a) was used as a comparative tool for the wood *taxa* identification, together with an in-lab reference collection of charred wood specimens and some reference examples from the microscope slide reference collection of wood of the Swiss Federal Institute. The analysis identified wood samples at the highest possible taxonomical level (family/genus/section/species). The level of identification depends, in fact, on the available and visible diagnostic micro-anatomical characters of single wood species/specimens.

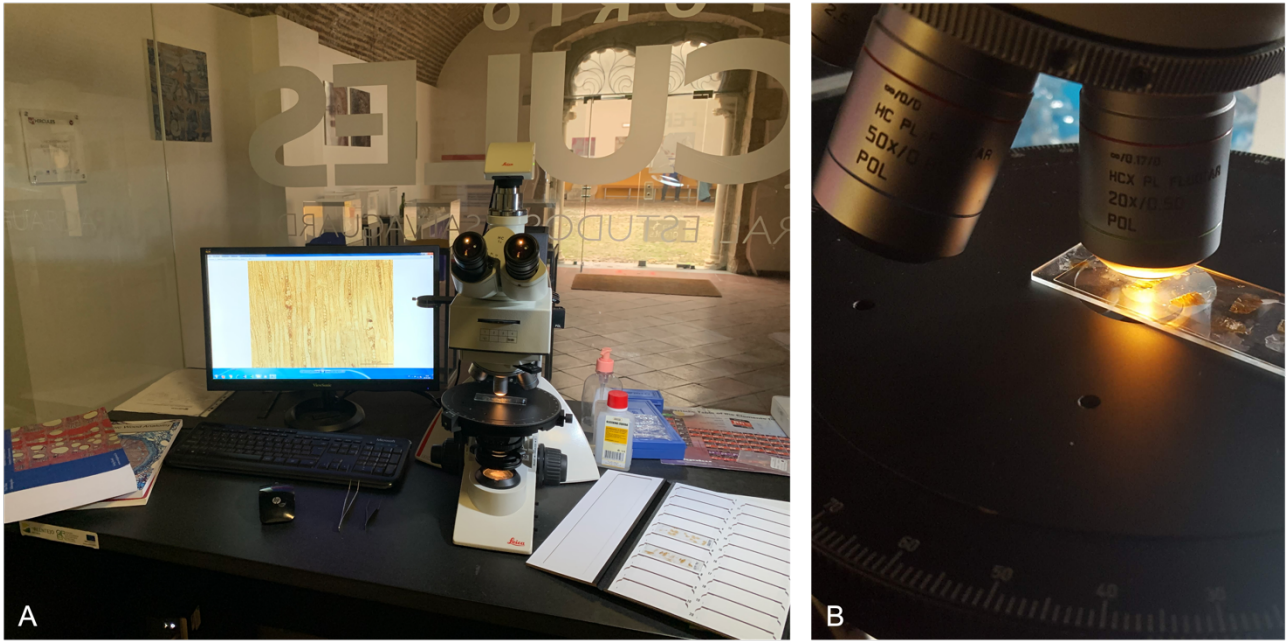


Figure 14: A. Observation of archaeological wood thin section under a transmitted light optical microscope at the HERCULES Laboratory; B. Detail of an archaeological wood thin section observed at 200x.

3. 8. 2. 2. Cork identification (Case study 3)

The cork sample identification was carried out using a SEM (HITACHI S3700N), and the instrumental settings described in section 3.7.2 were used.

3. 8. 3. Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS analysis (Case study 3)

Waterlogged samples were oven-dried for 48 h at 50° C to remove residual water content. For the analysis by Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS), a system with a Frontier Lab PY-3030D double-shot pyrolyzer was used. The interface was maintained at a temperature of 280°C. The pyrolyzer was coupled to a Shimadzu GC2010 gas chromatographer, also coupled to a Shimadzu GCMS-QP2010 Plus mass spectrometer (Figure 15). A capillary column Phenomenex Zebron-ZB-5HT (30-m length, 0,25-mm internal diameter, 0,50- μ m film thickness) was used for separation, with helium as carrier gas, adjusted to a flow rate of 1,5 mL min⁻¹. The split injector (15:1 ratio) operated at a temperature of 250°C. GC temperature programme was as follows: 35°C during 1 min, followed by a series of temperature ramps: until 110°C at 60°C min⁻¹, until 240°C at 14°C min⁻¹, up to 280°C at 6°C min⁻¹, until 320°C at 30°C min⁻¹, and then an isothermal period of 6 min. Source temperature was placed at 240°C, and the interface temperature was maintained at 280°C. The mass spectrometer was programmed to acquire data between 40 and 1090 m/z. Each sample (~200 μ g) was weighed in a 50- μ L Eco-cup capsule and placed in the double-shot pyrolyzer

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using an Eco-stick. The capsule was then placed in the pyrolysis interface and pyrolyzed at 500°C. For each sample, two replicate analyses were performed, and the peak areas of the main identified pyrolysis products were measured. Compound identification was done using the National Institute of Standards and Technology (NIST) and Wiley mass spectra libraries, through AMDIS software. Peak areas were obtained using also the AMDIS software.

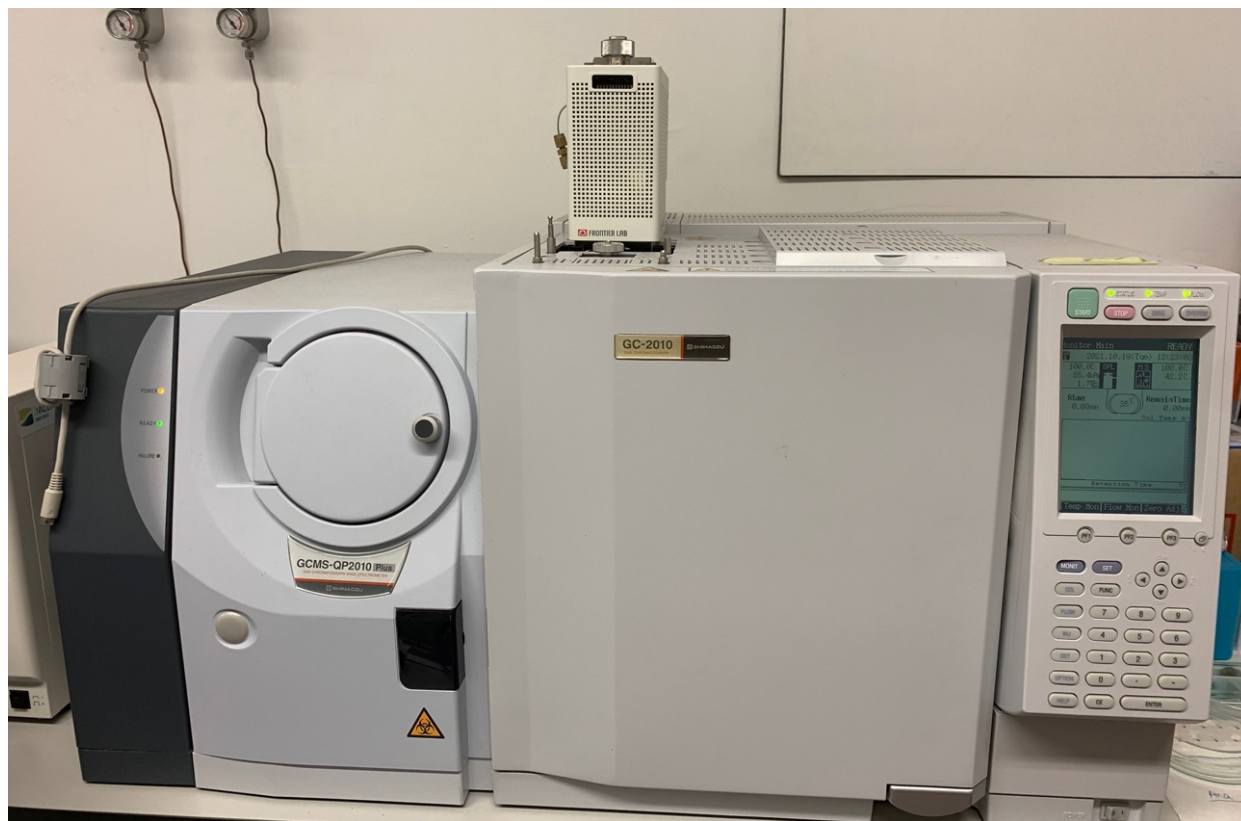


Figure 15: Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS) at the HERCULES Laboratory.

3. 9. Analysis of seeds and fruits (Case studies 4 and 5)

3. 9. 1. Taxonomic identification - Carpological analysis (Case study 5)

The identification of archaeological seeds and fruits were performed through the morphometric and biometric observation of the carpological remains, using a stereo-zoom microscope at low magnification (LEICA M205C equipped with a camera) (Figure 16). The analysis was completed by observing the four visual norms: ventral, dorsal, lateral, and axial. The identification was supported by the use of specialized bibliography (Berggren, 1969; Cappers and Bekker, 2013; Cappers et al., 2012; Jacomet, 2006) together with the carpological reference collection of the Palaeobotany Laboratory of the La Sapienza University of Rome.

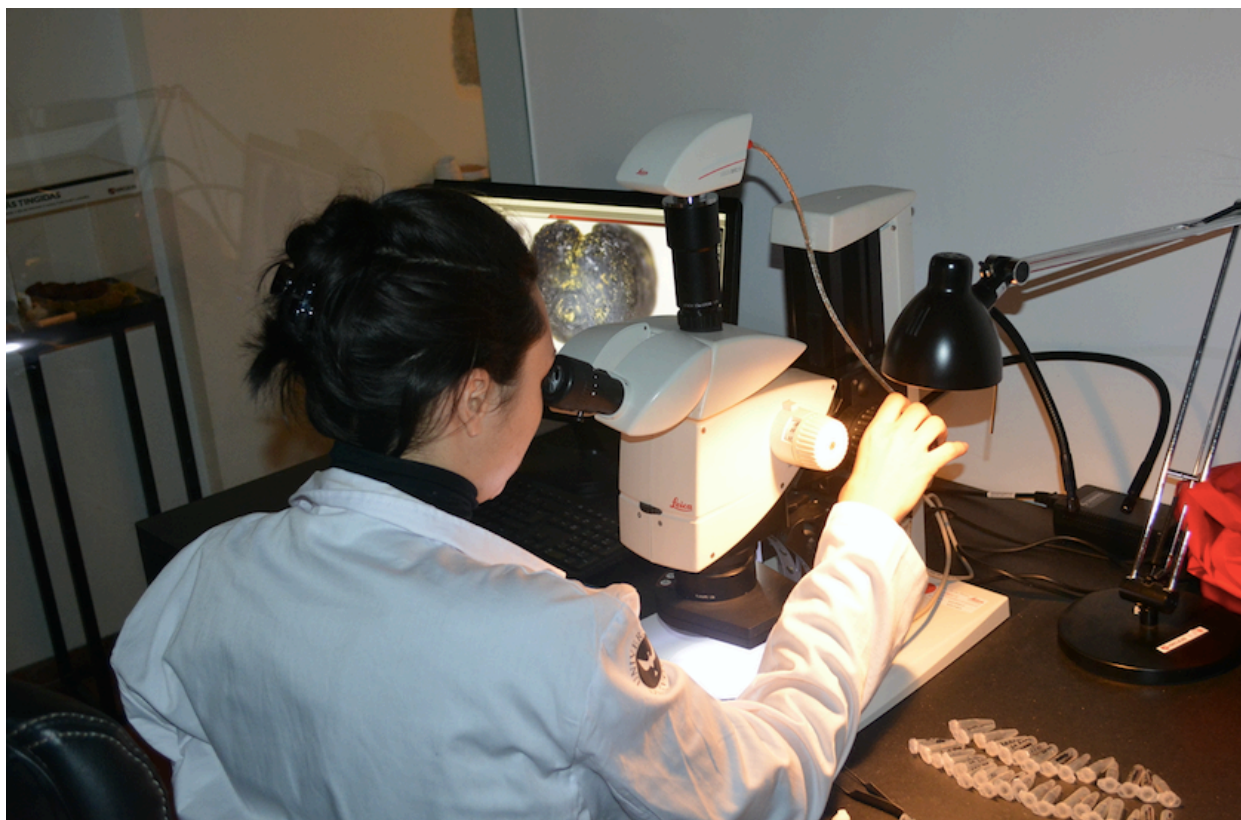


Figure 16: Observation of a charred cereal grain from Paço dos Lobo da Gama archaeological site under a stereo – zoom microscope at the HERCULES Laboratory.

3.9.2. Grape seed database collection (Case study 4)

Grape seeds were collected from native cultivars of *Vitis vinifera* subsp. *vinifera*, coming from the National Ampelographic Collection of the Instituto Nacional de Investigação Agrária e Veterinária (INIAV, Dois Portos, Portugal) (Figure 17A-B). In addition, seed accessions of plants of *Vitis vinifera* subsp. *sylvestris* populations present in Portugal were harvested in the field. Fruits were picked from different plants of the same cultivar variety and from different plants of the same wild population, in order to ensure the greatest morphological variability. Wild fruits were collected from plants isolated from cultivated areas in order to avoid any hybridization. They were sampled between August and September to ensure the complete morphological development of the fruits. Afterwards, seeds (both from cultivars and wild plants) were extracted from fruits, cleaned, dried, and stored at 20°C and 40% RH (Figure 18A-D).

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Figure 17 : A-B. Grape harvesting pictures from native cultivars of *Vitis vinifera* L. of INIAV

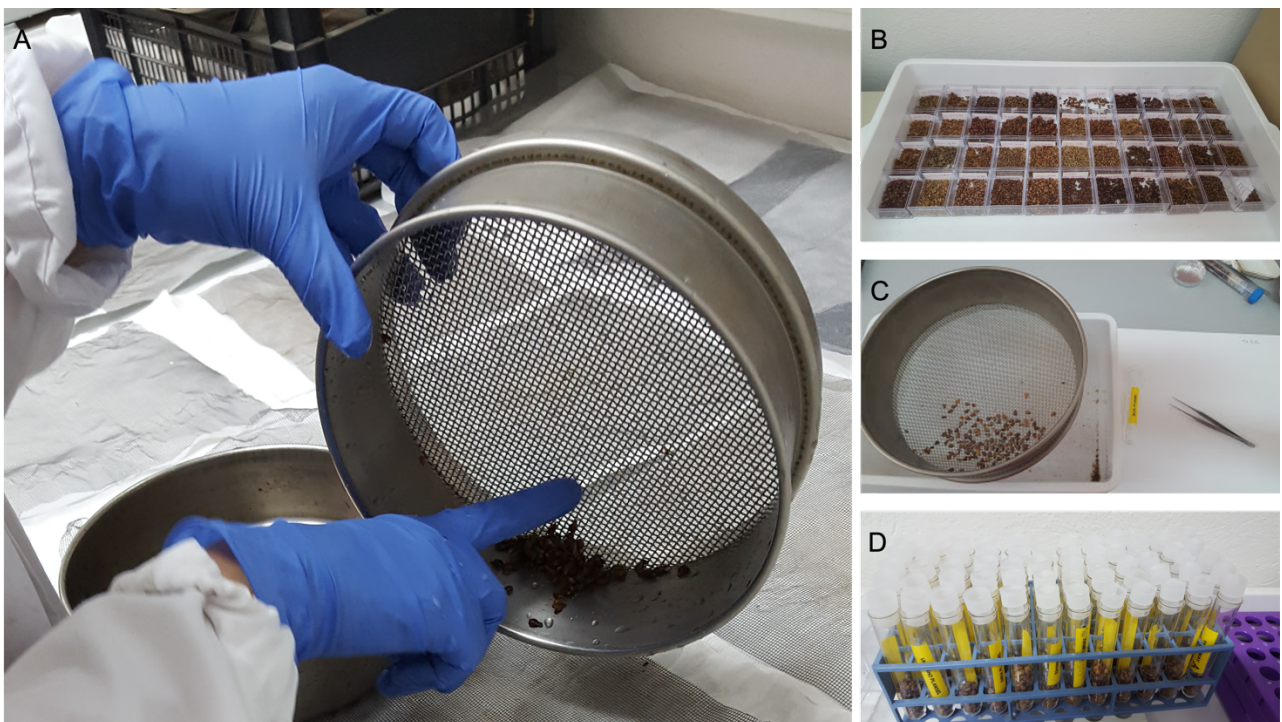


Figure 18: A. Modern grape seeds used for the preparation of a database collection for the study of the archaeological seeds of Torre dos Namorados archaeological site (Fundão, Portugal); B. First storage of washed and dried grape seeds; C. Removing of grape skin residues; D. Final storage of grape seeds at Archaeobotany laboratory of the Hortus Botanicus Karalitanus of the University of Cagliari.

3.9.2.1. Carbonization of grape seed reference samples (Case study 4)

To achieve correct comparison, modern seeds were subjected to carbonization. This procedure has been successfully applied by other authors (Ucchesu et al., 2016) to allow comparison with charred seeds.

The carbonization was carried out on a bonfire, simulating natural carbonisation conditions. The bonfire was made on a pit in the ground, 30cm deep and 1m wide, and the grape seeds were covered with 2cm of topsoil. In total, the bonfire experiment lasted for 10 h (Figure 19A-F).



Figure 19: Representation of the modern grape seed carbonization operating sequence. A. Samples deposit; B-C. reconstruction of the hearth; D. Lit bonfire; E. Samples collection.

3.9.3. Digital Image analysis (Case study 4)

Digital images of archaeological and modern charred grape seeds were acquired utilising a flatbed scanner with 400 dpi of digital resolution (Epson V550) (Figure 20A). The scanning area did not exceed 1024×1024 pixels (Bacchetta et al., 2008). In order to represent the entire variability of the grape seed lots, the samples were scanned twice randomly placing the seeds on the scanner (Figure 20B-C). The software package employed for binarizing the scanned seed images was the ImageJ v. 1.49 (<http://rsb.info.nih.gov/ij>). A particular plugin, Particles 8 (Landini, 2006), freely available online (<http://www.mecourse.com/landinig/software/software.html>) has been used to get 26 morphometric features (Figure 20D). In addition, 80 Elliptic Fourier Descriptors (EFDs) of the seed contour shapes were added using the software package ImageJ v. 1.49 utilising a specific plugin (Diaz, 2017). A total of 106 morphometric parameters have been taken into consideration.

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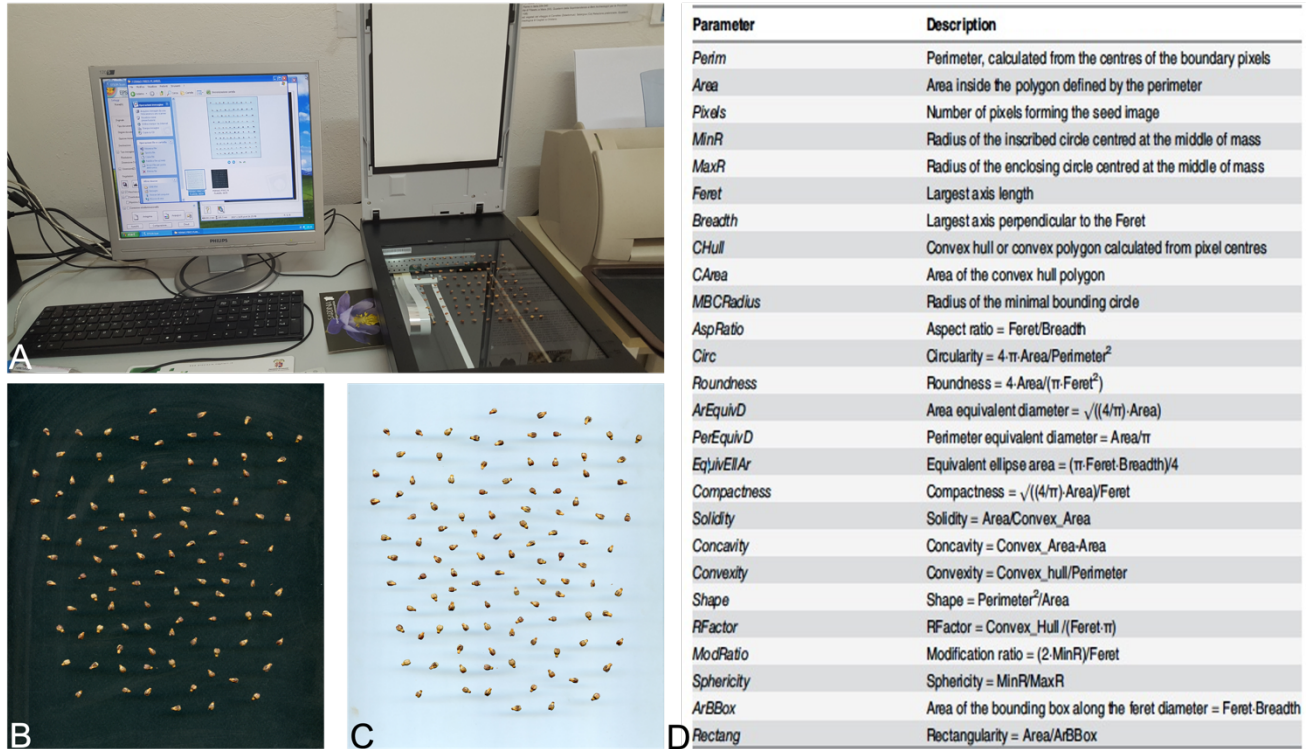


Figure 20: A. The flatbed scanner (Epson Perfection V550) of the Archaeobotany Laboratory of the Hortus Botanicus Karalitanus of the University of Cagliari; B-C. Digital grape seed images with a black and a white background; D. List of the 26 morphometric seed features measured for the analysis of the archaeological grape seeds from Torre dos Namorados archaeological site.

3.9.4. Linear Discriminant analysis (LDA) (Case study 4)

The parameters obtained from the analysis of digital seed images were employed to establish a database of morphometric grape seed features. The data were statistically elaborated by stepwise Linear Discriminant analysis (LDA), using the SPSS software (Statistical Package for the Social Sciences) release 16.0 (SPSS Inc. for Windows, Chicago, Illinois), in order to compare the modern with the archaeological seeds, which were considered as an unidentified group. This approach is commonly used to classify and identify unknown groups that are characterized by quantitative variables. The LDA method compares different known groups, looking for similarities and differences between them, using morphometric parameters. The similarity of the unknown group with the known groups is evaluated according to morphometric parameters as well. The LDA identifies and selects the best and the most statistically significant parameters among the 106 measured, using them to characterise seed samples (Bacchetta et al., 2010). Three statistical variables, the tolerance value, F-to-enter and F-to-remove values are used to define the power of each parameter and their role in the model. All variables with a value below 3.84 have been not used for the analysis (Venora

et al., 2009). Lastly, a cross-validation procedure has been applied to verify the identification system performance, applying the leave one out cross validation (LOOCV) procedure (SPSS 2006)

3. 10. Overview of the analytical methodologies employed in the different case studies

Table 1: Overview table of the analytical methodologies employed in the different case studies of the thesis.

	Methods	Case studies				
		Case study 1	Case study 2	Case study 3	Case study 4	Case study 5
Sediment treatment	Manual flotation - plus water sieving					*
Collection of plant macroremains	Screening	*				*
Charcoals	Charcoal reference collection	*	*	*		*
	Taxonomic identification - Anthracological analysis	*	*			*
	Preparation of reference material for the statistical model	*				
	FT-MIR Spectroscopy	*				
	PLS analysis	*				
Waterlogged materials	Wood thin sections			*		
	Waterlogged wood identification - Xylological analysis			*		
	Cork identification			*		
	Py GC/MS analysis			*		
Seeds and Fruits	Taxonomic identification - Carpological analysis					*
	Grape seed database collection				*	
	Carbonization of grape seed reference samples				*	
	Digital Image Analyses				*	
	LDA analysis				*	

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4th CHAPTER. Case studies

The exploitation of plant resources in the human history: an archaeobotanical insight

4. 1. Introduction to the cases studies of the thesis

The case studies of the thesis are presented in a chronological way within the chapter. Each case study consists of an introduction with the study goals, a description of the archaeological context/s, results obtained, materials and methods employed, and a discussion of the results and conclusions.

4. 2. Case study 1: A Multi analytical approach of a Chalcolithic funerary deposit from Perdigões (Alentejo, Portugal) to uncover the human exploitation of the natural resources and the wood burning temperature

4. 2. 1. Introduction and research aims

Perdigões is a large set of ditched enclosures located in Reguengos de Monsaraz municipality, Évora district, in inner Alentejo region, centre-southern Portugal (Figure 21A). It has at least 16 ditches and a megalithic cromlech, occupying an area of ca. 18 ha. The archaeological site had a long and complex diachrony, extending from 3400 BCE (late Middle Neolithic) to 2000 BCE (Early Bronze Age). Interpreted as a ceremonial and aggregation centre, these ditched enclosures have several funerary structures already excavated, revealing diversified funerary practices in terms of architecture, location within the site, body treatment and votive materials (Evangelista and Valera, 2019; Valera et al., 2014). The archaeological material (i.e., charcoals) under study was recovered in Pit 16 (middle 3rd BC), located in the central area of the enclosures (Valera et al., 2014). Several stratigraphic units (SU) were identified during the archaeological excavation, and those that contain human remains were SU 72 and 74 (Figure 21C).

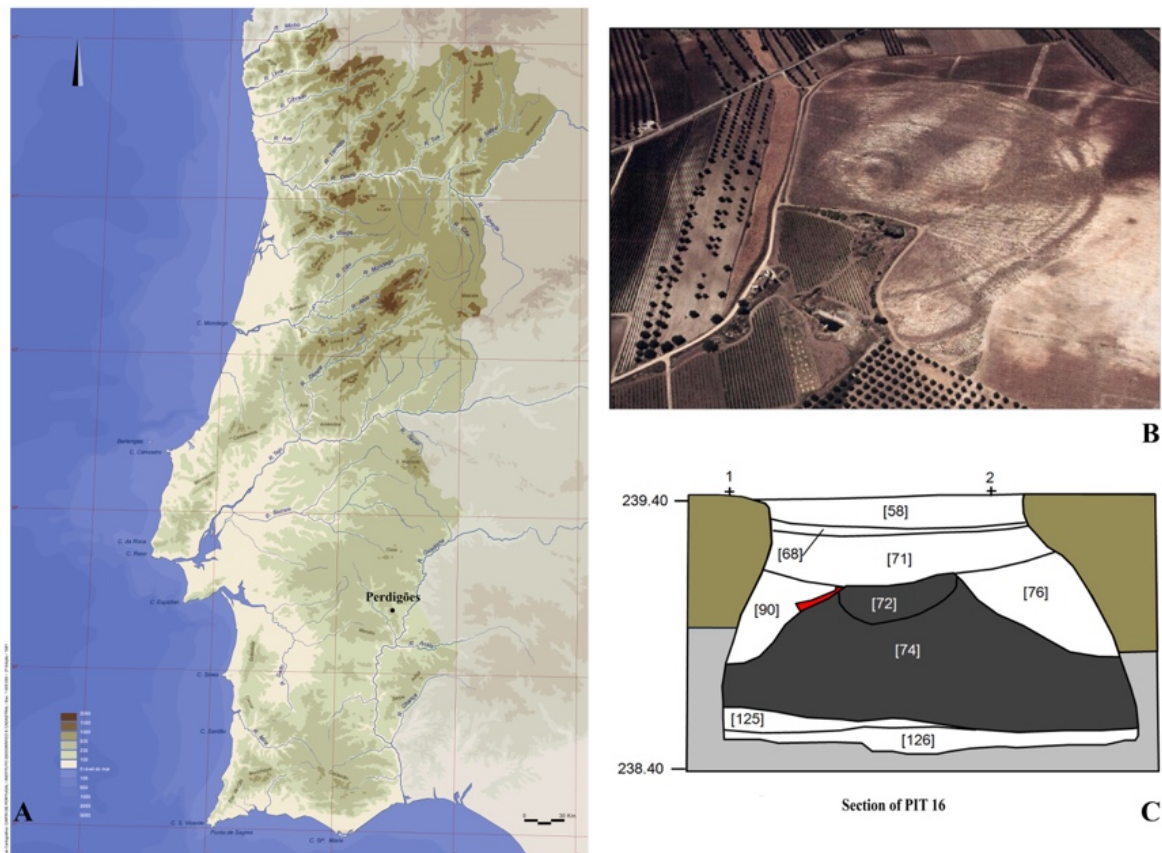


Figure 21: A. Location map of Perdigões archaeological site in Portugal; B. Aerial photo of Perdigões archaeological site; C. Section of Pit 16 with the stratigraphic units (SUs) identified.

Perdigões, along with other recent archaeological discoveries, led to a revision of the Megalithic literature (Evangelista, 2019; Evangelista and Valera, 2019; Valera, 2012a; Valera, 2012b; Valera and Godinho, 2010; Valera et al., 2014; Valera et al., 2017; Weiss-Krejci, 2011) and, at present, a more dynamic picture of the Iberian Chalcolithic funerary world is available if compared with portrayals produced in the past. The reinterpretation of the world of death within this particular chronology has fostered the production of a number of studies on anthropological and archaeozoological remains recovered in these contexts, and the cultural materials associated with them (Boaventura et al., 2014; Cabaço, 2011; Cabrero García et al., 2005; Cámara Serrano et al., 2012; Costa Caramé et al., 2011; Cruz et al., 2016; Díaz-Zorita et al., 2012; Duarte et al., 2004; Evangelista, 2019; Evangelista and Valera, 2019; García Rivero and J.O'Brien, 2014; Silva, 2003; Silva, 2012; Silva and Ferreira, 2008; Valera and Evangelista, 2014; Valera and Godinho, 2010; Weiss-Krejci, 2006).

In the literature, a charcoal assemblage deriving from a delimited archaeological structure (or single SU), either a settlement or a funerary context, is described in archaeobotany as a “single episode deposit”. In this case a single or a short time span episode leads to the formation of a concentrated charcoal deposit (Figueiral and Mosbrugger, 2000), and the evaluation of this type of charcoal assemblage is linked to the interpretation of the archaeological context where they were found (Moskal-del Hoyo, 2012). Indirectly, the main goal of the concentrated charcoal studies is to get palaeoethnographic information, where the relationship between humans and the surrounding nature is investigated.

Moreover, whether different deposits are identified (i.e., SUs) during the archaeological excavation, it might also be possible to consider if different depositions took place or not.

For this purpose, two different but complementary approaches have been used. The first one includes the anthracological analysis of archaeological charcoals to identify the wood *taxa* utilized during the primary cremation ritual. The second one comprises the estimation of the archaeological charcoals absolute burning temperature by the comparison with sound wood samples combusted at different temperature (i.e., formed under controlled temperature and atmosphere using a muffle furnace in the laboratory). The tree main *taxa* identified by the anthracological analyses were employed. In this case thermal transformations of wood must be evaluated and estimated.

The partial combustion process of wood either in a forest fire, in a bone fire or in a funerary pyre is well known. It normally produces charred material which contains a significant amount of refractory carbon (C). This is commonly known as “black carbon” or charcoal (De la Rosa et al., 2008). This material is defined as a continuum of thermally altered products, ranging from slightly charred still degradable biomass, to highly condensed, refractory soot (Goldberg, 1985). The charcoal

particles are chemically heterogeneous, but typically they are made up of a conspicuous amount of unspecific chemical aromatic structures (Hedges et al., 2000).

The concentration of aromatic compounds within the chemical structure of the charcoal particles is significantly associated to direct and indirect factors. Direct factors are linked to combustion characteristics, including temperature, fire permanence and heating rate ($^{\circ}\text{C}/\text{min}$). On the other hand, indirect factors are connected to the fuel (wood) properties, which comprise the type of wood, size, wood resilience, fuel moisture, etc. (Asouti, 2007; Braadbaart et al., 2007; Braadbaart and Poole 2008). Thus, chemical changes in the continuum charcoal structure can be identified, which are influenced by the combustion temperature and the molecular composition of the pyrogenic material (Antal and Gronli, 2003; Hall et al., 2008; Kim and Hanna, 2006).

Currently, different techniques are available for the charred organic matter evaluation of complex matrices such as soils and sediments, as well as to assess their burning temperatures. One of these techniques is the reflectance, which is considered a valuable tool to estimate the burn temperatures of modern and archaeological charcoal using a Reflected Light Microscope (Braadbaart and Poole, 2008; McParland et al., 2010; Veal et al., 2016). Another analytical technique is the Fourier Transform Mid-Infrared (FT-MIR) spectroscopy, which is micro-destructive and widely employed for the chemical characterization of complex matrices in archaeological samples, like wood charcoal particles and bone collagen (Lebon et al., 2008; Lebon et al., 2016; Pizzo et al., 2015).

The combination of FT-MIR spectroscopy and multivariate analytical techniques, such as Partial Least Squared (PLS) regression (Fernández-Getino et al., 2013), has demonstrated to be a powerful tool for the evaluation and prediction of combustion temperature of organic matter and the composition of pyrogenic carbon in soils (Jiménez-González et al., 2019; De la Rosa et al., 2019). In addition, some researchers have studied wood burned samples in terms of the Variable Importance Projection (VIP), which has allowed assessing the MIR bands significantly linked with dependent variables (Jiménez-González et al., 2019; De la Rosa et al., 2019).

Thus, this research wishes to underline the importance of the analysis of charcoal for the study of archaeological cremations. On the other hand, as O'Donnell et al. (2016) have already written about, charcoal represents the real essence of the pyre, being the most direct way to shed light on the importance of the funeral pyre within the ritual of body cremation. This study presents charcoal analyses from Pit 16 of the Perdigões archaeological site and provides new data about the context and the related ritual. One of the main questions under analysis was if the wood selection was either culturally determined or based on the territorial availability. Do the two represent different, coexistent solutions or are they two solutions conditioned by each other?

We consider that the combination of anthracology/experimental burning tests/FT-MIR/chemometric analyses is a good option to successfully evaluate the charcoal assemblage from Pit 16. Results were useful to a better understanding of the archaeological context and the related ritual/s.

4. 2. 2. Materials

4. 2. 2. 1. Archaeological context of the charcoal samples

Pit 16 is interpreted as a secondary funerary collective deposition, a singular funerary context amongst Iberian Chalcolithic. The pit contained human remains of at least 9 individuals (6 adults and 3 sub-adults), mixed with other burnt artefacts (faunal remains, fragments of pottery, ivory idols, and arrowheads) as well as a large number of charcoals. The pit is circular, with a profile shrinking in the top, presenting a diameter of 0.92 m in the top and 1.50 m in the base, being 0.90 m deep (Figure 21C). In total 9 different stratigraphic units (SU) were identified, corresponding to three phases of deposition. The first phase corresponded to the initial filling of the pit, where 2 thin layers (SU126 and SU125) were formed by some residual materials, namely pottery sherds. The second phase corresponded to the secondary deposition of the cremated remains. A conic shaped deposit was formed, indicating the dumping of the remains into the pit (probably from containers). This deposition was subdivided in 2 stratigraphic units (SU72 and SU74), being the later less compact and dustier. Both sediments were dark grey, with a lot of charcoal and ashes, involving abundant cremated human remains (closer to 5000 fragments), some cremated faunal remains and burned archaeological materials (pottery sherds, ivory schematic figurines, a copper awl, stone arrowheads) (Silva et al. 2015; Valera et al. 2014; Valera and Evangelista 2014). Nevertheless, whether the two stratigraphic units represent a single deposition moment, or not, is not clear. The third phase corresponds to the final filling of the pit, with de depositions of several layers with no human remains, incorporating mainly faunal bones and pottery shards (SU76, SU71, SU90, SU68, SU58).

4. 2. 3. Methods employed

4. 2. 3. 1. Sampling

Pit 16 sampling was performed based on the excavation pursued, which divided the pit into several stratigraphic units. For this work sediments from the SU72 and the SU74 (Figure 1C), corresponding to the secondary deposition of cremated human remains, were entirely sampled.

In the field, archaeologist dry sieved the sediments from SU72 and SU74 using a 2 mm mesh size to isolate human remains (Silva et al., 2015) and charcoals. After bone recovery, the remaining sediments were preserved for the anthracological study.

4. 2. 3. 2. Anthracological analysis

Sediments from SU72 and SU74 were additionally treated and observed in the laboratory using a stereo-zoom microscope (model LEICA M205C equipped with a camera) to remove sand grains, small ceramic, and animal bones remains and to isolate charcoal fragments (i.e., larger than 2 mm) to proceed with the anthracological analysis. A total of 754 charcoals were recovered.

Taxa identification was performed following the method previously described (chapter 3 – 3. 7. 2. Taxonomic identification of charcoal samples – Anthracological analysis).

Some other types of observation were also made. When present, fractures in radial directions (radial cracks), vitrification, consisting in a variable fusion of some anatomical parts of the wood (which make the wood structure homogeneous), and radial groves in the inner part of tracheids cells (known as reaction wood) were recorded (Marguerie and Hunot, 2007).

With respect of the evergreen oak's wood samples identification, it is significant to reiterate that it was not possible to make any distinction between the evergreen oak tree species. In Portugal, nowadays, as well as in the Mediterranean region, this group includes holm oak (*Quercus ilex* L.), kermes oak (*Quercus coccifera* L.) and cork oak (*Quercus suber* L.) (Humphries et al. 2005; Schweingruber 1990).

Regarding the identification of the olive wood samples, the cultivated species (*Olea europaea* var. *europaea*) is not differentiable at the anatomical level from the wild one (*Olea europaea* var. *sylvestris*). However, studies concerning the beginning of the cultivation of this plant in the south of the Iberian Peninsula suggests that it started only in the Roman period (Rodríguez-Ariza and Moya, 2005). Considering the investigation period, charcoal fragments of *Olea* from Pit 16 could probably be ascribed to the wild variety, *O. europaea* var. *sylvestris*.

The identification of Fabaceae wood is considered particularly difficult due the large intraspecific structural variability present in this group. Few species of this group were anatomically described (Queiroz et al. 2006; Queiroz 2010; Schweingruber 1990a). In the present study charcoal samples belonging to this *taxon* were identified only at the family level.

4. 2. 3. 3. Data quantification

For data interpretation *taxa* values are interpreted using the relative frequency (%) and the mean values (%U) obtained using the “ubiquity correction” method (Moskal-del Hoyo, 2011). The mean value (%U) of a specific *taxon* (A, for example) on *n* different stratigraphic units (SU) is calculated as: $\%U = \%A_{(SU1)} + \%A_{(SU2)} + \%A_{(SUn)} / n$. This method performs a correction for ubiquity of the relative frequencies of each *taxon*. The values of charcoals size classes for each wood *taxon* identified were also collected.

4. 2. 3. 4. Estimation of the absolute burn temperature of archaeological charcoals

4. 2. 3. 4. 1. Experimental combustion tests on modern reference material

In order to make a statistical model able to estimate the absolute combustion temperature of archaeological charcoals an in-lab charcoal reference collection was created, and different experimental combustion tests were carried out. Sound wood samples of the three main *taxa* identified from Pit 16 with the anthracological analysis, namely olive, evergreen oak, and maritime pine woods have been sampled in the proximity of the archaeological site (Figure 22). Regarding the olive, considering the investigation period (as already reported in the previous section), wooden samples belonging to the wild variety, *Olea europaea* var. *sylvestris* have been chosen as reference material.

About the evergreen oaks, samples belonging to *Quercus suber* species have been chosen as reference material, considering the abundance of this species in the Alentejo region (Blanco et al., 1997). The experimental combustion tests methodology has been previously described (chapter 3 - 3. 7. 3. Preparation of reference material for the statistical model).

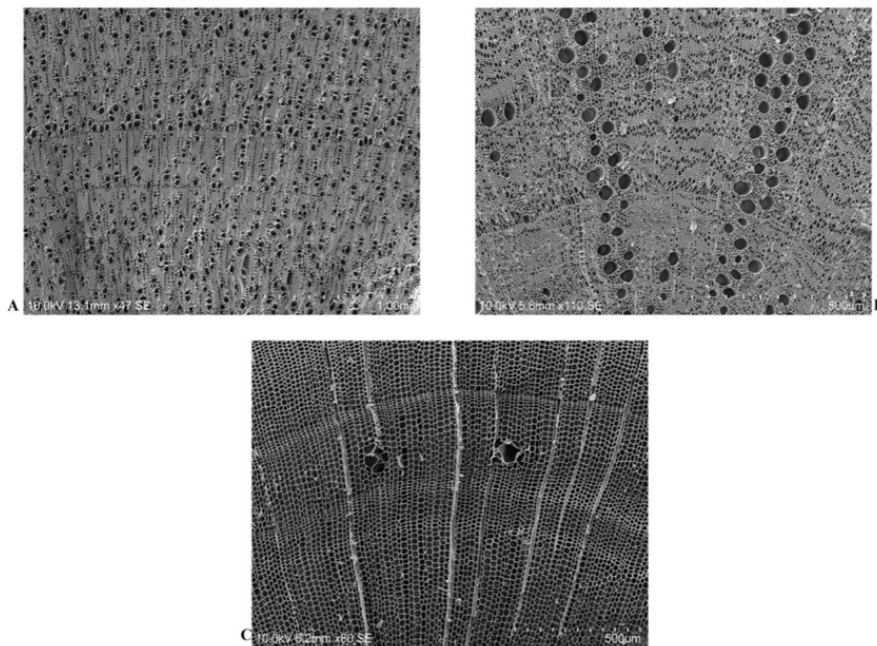


Figure 22: Sound woods reference samples heated at 350 °C. A. *Olea europaea* var. *sylvestris*; B. *Quercus suber*; C. *Pinus pinaster*. Samples were photographed with SEM.

4. 2. 3. 4. 2. FT-MIR Spectroscopy analysis of modern and archaeological charcoals

An aliquot from the two charred replicas of each modern wood sample was detached for FT-MIR analysis. The two charred aliquots of each *taxon* were powdered using an agata mortar and combined to create one composite sample for each different wood *taxon*.

Two aliquots of each archaeological sample of *Olea europaea*, *Quercus* spp. (evergreen) and *Pinus pinaster* were detached for FT-MIR analysis. As for modern samples, the two aliquots of each archaeological *taxon* were powdered and then combined to create one composite samples in order to be analysed.

FT-MIR spectroscopy methodology has been previously described (chapter 3 – 3. 7. 4. Fourier Transform Mid-Infrared Spectroscopy (FT-MIR)).

4. 2. 3. 4. 3. Statistical analysis

The methodology has been previously described (chapter 3 - 3. 7. 5. Partial Least Squares (PLS) analysis).

4. 2. 4. Results

4. 2. 4. 1. Anthracological analysis

After the analysis of 754 charcoal fragments from Pit 16, 7 *taxa* were determined at the species level, *Olea europaea*, *Pinus pinaster*, *Fraxinus* cf. *angustifolia*, *Arbutus unedo*, at the genus level *Quercus* spp. (evergreen), *Cistus* sp., at the family level, Fabaceae, and at the division level, Angiosperm dicotyledon (Table 2, Figure 23-24).

Table 2: Table 1 Absolute number (N), relative frequency (%) and relative frequency corrected for ubiquity (%U) of taxa found in Pit 16.

		SU		72		74		TOTAL		
		TAXA		N	%	N	%	N	%	%U
		Botanical name	Common name							
Trees		<i>Olea europaea</i>	Olive	33	15.9	215	39.4	248	32.9	27.6
		<i>Quercus</i> spp. (evergreen)	Evergreen oak	66	31.7	162	29.7	228	30.2	30.7
		<i>Pinus pinaster</i>	Maritime pine	99	47.6	57	10.4	156	20.7	29
		<i>Fraxinus</i> cf. <i>angustifolia</i>	Narrow-leafed ash	0	0.0	10	1.8	10	1.3	0.9
Shrubs		<i>Cistus</i> sp.	Rock rose	8	3.8	33	6	41	5.4	4.9
		Fabaceae	Legume family	2	1.0	14	2.6	16	2.1	1.8
		<i>Arbutus unedo</i>	Strawberry tree	0	0.0	7	1.3	7	0.9	0.7
	Angiosperm dicotyledon	Dicotyledon	0	0.0	48	8.8	48	6.4	4.4	
Total				208	100	546	100	754	100	100
Minimum taxa number				5		7				

4. Case studies

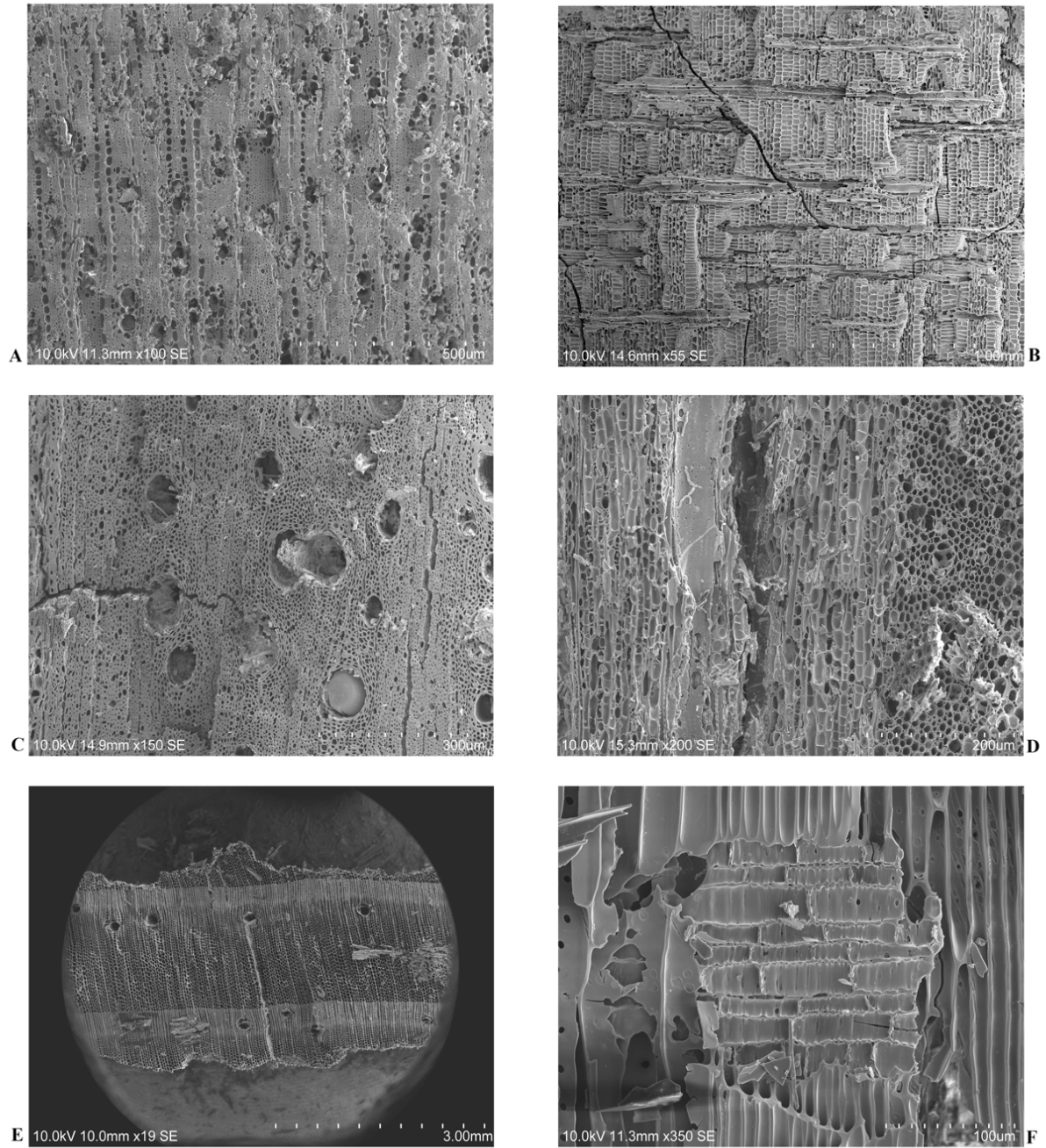


Figure 23: Archaeological charcoal samples observed and photographed with SEM. A-B. Transversal and radial sections of an *Olea europaea* sample; C-D. Transversal and tangential sections of a *Quercus* spp. (evergreen) sample; E-F. Transversal and radial sections of a *Pinus pinaster* sample.

4. Case studies

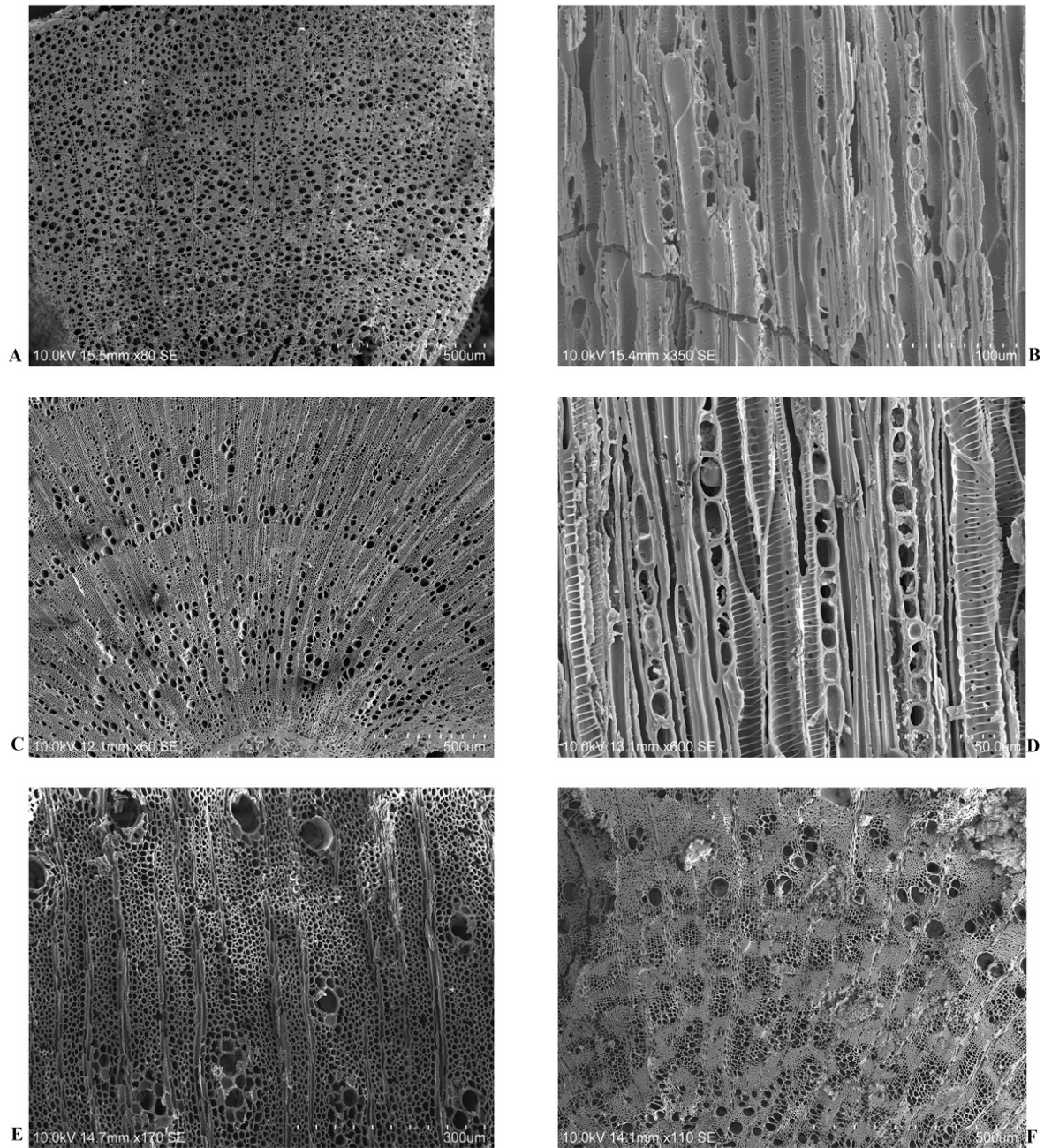


Figure 24: Charcoal samples observed and photographed with SEM. A-B. Transversal and tangential sections of a *Cistus* sp. sample; C-D. Transversal and tangential sections of an *Arbutus unedo* sample; E. Transversal section of a *Fraxinus* cf. *angustifolia* sample; F. Transversal section of a Fabaceae sample.

Three *taxa* are dominant in the anthracological record of Pit 16, featuring the 83.8% and the 87,3%U of total of identified samples: olive tree (*O. europaea*), evergreen oak tree (*Quercus* spp. (evergreen)), and maritime pine tree (*P. pinaster*) (Figure 23). The most abundant *taxa* are olive (32.9%, 27.6%U) and evergreen oak (30.2%, 30.7%U). Finally, the maritime pine is the third *taxon* in abundance, accounting the 20.7% and the 29%U of the totality (Table 2, Figure 25A). From the SU72, 208 charcoal fragments were recovered, and 5 different *taxa* were identified, while from the

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SU74, a total of 546 charcoal fragments were recovered and 7 different *taxa* were identified. Moreover, the amount of the 3 main *taxa* is not similar between the two stratigraphic units, with the maritime pine tree being the main *taxon* of the SU72, while in SU74, olive tree is predominant (Table 2, Figure 25B).

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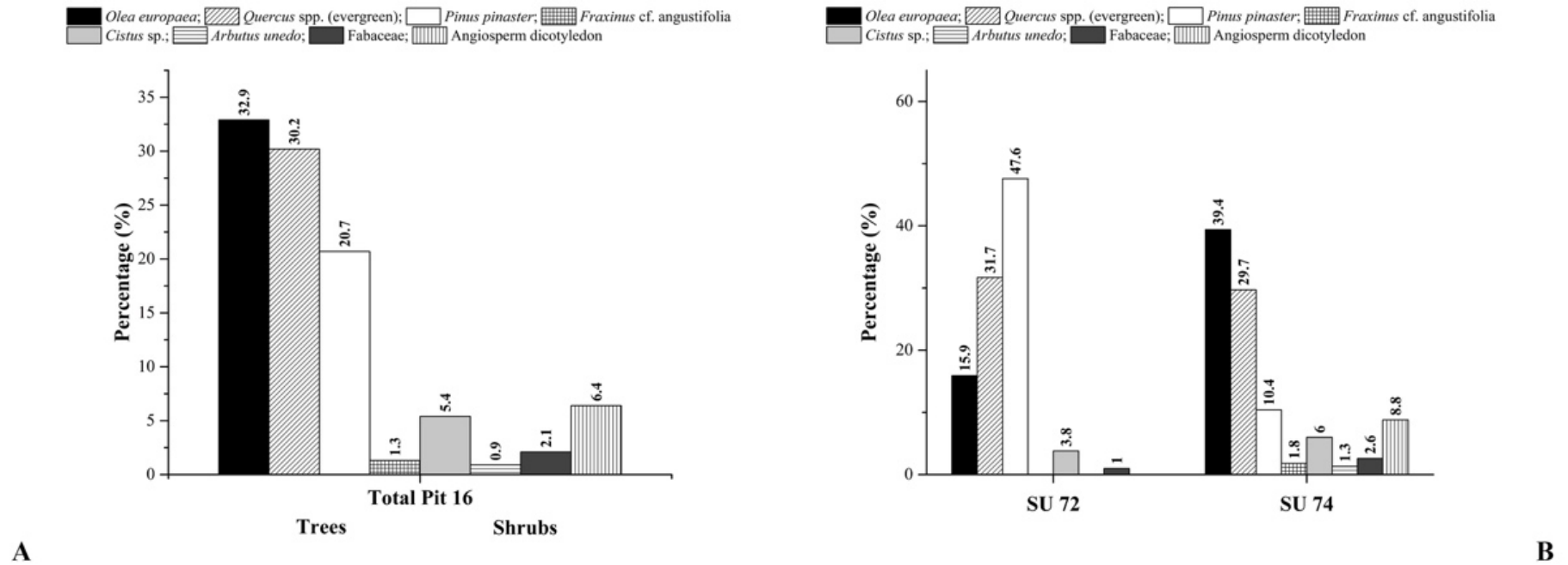


Figure 25: A. Relative frequency of taxa found in Pit 16; B. Relative frequency of taxa found in the SU72 and in the SU74.

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These differences become evident by the application of the “ubiquity correction” method, which accounts not only for the absolute number of charcoal fragments, but also for the *taxa* distribution within the contexts (Table 2).

Regarding charcoals fragments of *Quercus* spp. (evergreen), samples from both SUs 74 and 72 showed the presence of the vitrification. This feature was observed in 85 fragments of this *taxon*. Concerning *P. pinaster* samples from SU 72, fractures in radial directions (radial cracks) were recorded for a total of 90 charcoal fragments. Moreover, for 27 charcoals fragments of this species radial grooves in the inner part of tracheids were recorded.

With reference to charcoal size, the olive tree is the species with the relatively high quantity of the largest charcoal, with 71 fragments with values between 1 and 1.99 cm and 9 fragments between 2 and 2.99 cm., followed by the maritime pine and by the evergreen oak, for which the largest charcoals collected are represented by only 14 fragments (Figure 26).

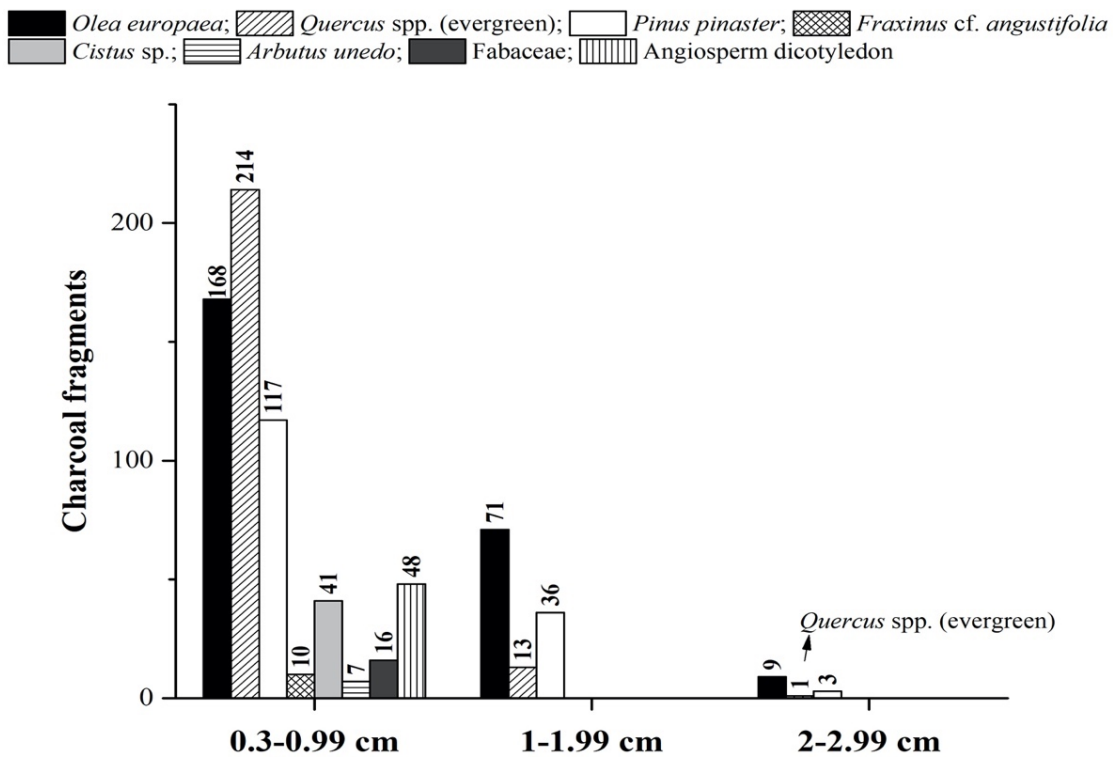


Figure 26: Charcoal size classes identified in Pit 16.

The remainder of arboreal *taxa* it is represented by few fragments of narrow-leaved ash wood (*F. cf. angustifolia*). This *taxon* was recorded only in the SU74, and its presence do not exceed the 1.3% and the 0,9%U (Table 2, Figure 24E). Shrubs were also identified in the anthracological record

of Pit 16: rock rose wood (*Cistus* sp.) legume wood (Fabaceae) and strawberry tree wood (*A. unedo*), accounting the 8.4% and the 7,4%U of the total (Figure 24A-D, F). The most present *taxon* is the rock rose (5.4%, 4,9%U), identified on the genus level as *Cistus* sp. (Table 2, Figure 24A-D). Between shrubs, 2 fragments of *A. unedo* and 3 of Fabaceae have been identified as twigs, having retained their shape. The values of charcoal size, showed that all charcoal fragments belonging these species, were basically smaller, with values between 0.3 and 0.99 cm if compared with the three main *taxa* (Figure 26). Finally, a fava bean seed (*Vicia faba*) was identified in the SU74 (data not shown). Regarding charcoals fragments identified as Angiosperm dicotyledon, it was not possible to identify them beyond the division level due to their state of conservation.

4. 2. 4. 2. Temperature determination

4. 2. 4. 2. 1. FT-MIR Spectroscopy of archaeological samples

Figure 27 displays the infrared spectra, in the region of 1800–400 cm^{-1} , of the archaeological charcoal samples from SU74 (Figure 27A) and SU72 (Figure 27B) belonging to each of the 3 main *taxa* identified after anthracological analysis of archaeological charcoals. In all spectra, different bands were observed namely the 1570 cm^{-1} band related to the amide groups, the 1380 cm^{-1} band linked to C-O stretching of phenolic OH, the 1730 cm^{-1} band related to stretch of groups C = O, and the bands 1270 and 1040 cm^{-1} belonging lignin. These results were in line with those observed by Sharma et al. (2001) and Cai et al. (1996) for burnt organic matter samples. The band at 1570 cm^{-1} does not have an unequivocal assignation (Moreno-Castilla et al., 2000), nevertheless, it may be referred to the C=C stretching in aromatic and quinone structures, that is highly conjugated to carbonyl group (Fuente et al., 2003).

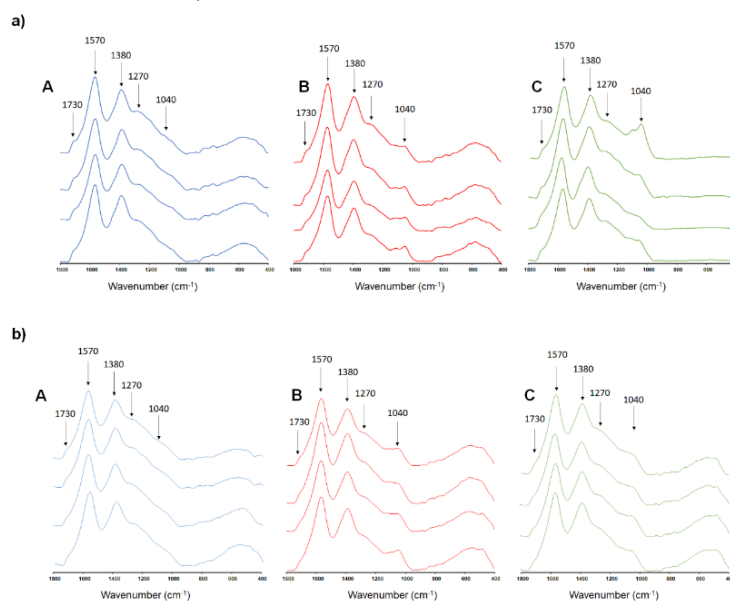


Figure 27: Medium infrared spectrum (MIR) of archaeological charcoal retrieved from a) SU74 and b) SU72. A. *Olea europaea*; B. *Quercus* spp. (evergreen); C. *Pinus pinaster*.

This band is typical of carbonized, fossilized or highly humified organic matter (Oren and Chefetz, 2012). With respect to the band at 1380 cm^{-1} it may be related to unspecific aliphatic structures, as well as hydroaromatic compounds.

4.2.4.2.2. FT-MIR Spectroscopy and PLS analysis of modern charcoals

The cross-validation plots (predicted vs observed values) for the burning temperature of each set of modern wood charred *taxa* are displayed in figure 28. The PLS models, using exclusively the information contained in a discrete infrared spectra range ($1800\text{--}400\text{ cm}^{-1}$), successfully predicted the crematory temperature of each *taxon*. The PLS model of *Olea* showed the highest significant prediction ($R^2=0.998$, $P=0.0000$), following by *Quercus* and *Pinus* ($R^2=0.987$, $P=0.001$ and $R^2=0.957$, $P=0.007$, respectively). In all cases, the RMSE and AIC values suggested the use of 4 factors for all *taxa* (data not shown). The figure 29 shows the VIP values for the different *taxa* used to obtain the different PLS models. The superposition of the average infrared FT-MIR spectrum of each *taxon* with the VIP trace indicates the best diagnostic bands to predict the combustion temperature of each *taxon*. The most significant FT-MIR bands to predict the burning temperature in the case of *Olea* model were those positioned at 1730 , 1580 , 1410 , 800 and 740 cm^{-1} , while for *Quercus* model, the predominant bands were located at 1580 , 1410 , 1370 , 800 and 740 cm^{-1} . In the case of *Pinus* model, the main bands were identified at 1730 , 1410 , 1370 , 1245 and 1040 cm^{-1} .

Each forecasting model was made up of characteristic infrared bands (De la Rosa et al., 2019). In the case of the *Olea* samples, the statistic model used the bands at 1730 and 1580 cm^{-1} corresponding to vibrating $\text{C}=\text{C}$ and $\text{C}=\text{O}$ bonds, respectively, in the aromatic rings (Fuente et al., 2003). In addition, the bands at 1410 cm^{-1} , related to $\text{C}-\text{C}$ stretch (aromatic ring) of lignin compounds (Michell and Higgins, 2002) and 1370 cm^{-1} , linked to $\text{C}-\text{H}$ rock, methyl in alkene compounds (Nakamoto, 2009) were also used by the forecasting model of *Olea*. This *taxon* also used the bands at 800 and 740 cm^{-1} which are typical of polysaccharide compounds (Esteves et al., 2013). Concerning to *Quercus* PLS model, it used the same FT-MIR bands than for the model of *Olea*. However, in this case, the band at 1730 cm^{-1} was not determinant. On the other hand, the model of *Pinus* employed the bands at 1245 cm^{-1} which corresponds to compounds of acid resin (Știrbescu et al., 2017). This assignment may be confirmed for the existence of two characteristic bands at 800 and 740 cm^{-1} , which indicate the presence of resin compounds (Martín-Ramos et al., 2018). In addition, in this model, the band at 1040 cm^{-1} , that is related to the $\text{C}-\text{O}$ stretch, displayed a noticeable high VIP value.

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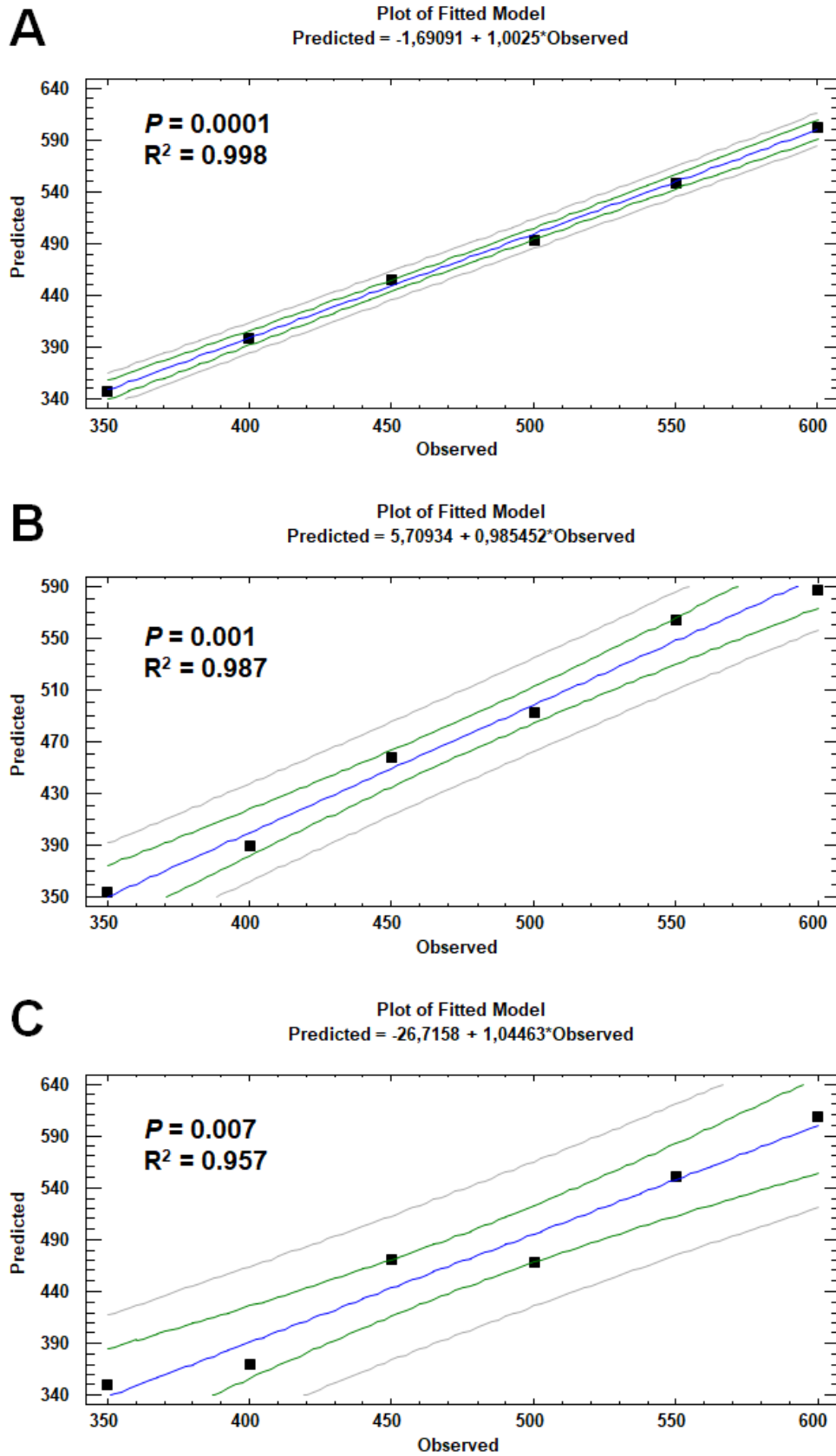


Figure 28: Cross-validation plots for crematory temperature of modern charred woods *taxa*, generated by partial least squares (PLS) regression using infrared spectral as predictors. A. *Olea europaea* var. *sylvestris*; B. *Quercus suber* (evergreen type); C. *Pinus pinaster*.

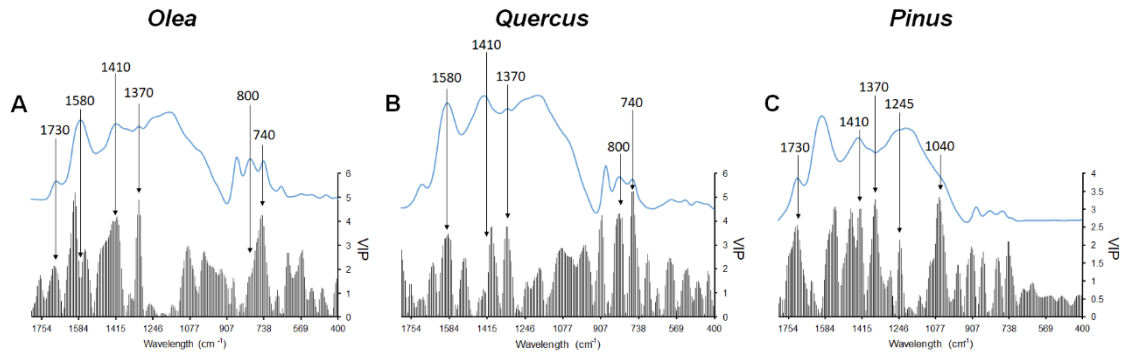


Figure 29: Superposition of Variable Importance for Projection (VIP) of spectral points and average infrared spectra of modern char particles. A. *Olea europaea* var. *sylvestris*; B. *Quercus suber*; C. *Pinus pinaster*.

4. 2. 4. 2. 3. Predicted absolute burn temperature of archaeological charcoals

The predicted absolute burn temperature for the archaeological charcoal samples identified in SU74 and SU72 is displayed in Figure 30 (A, B), respectively. The temperature value is obtained from the models developed using charred modern wood *taxa*. In SU74, the *P. pinaster* showed the highest combustion temperature ($576 \pm 24^{\circ}\text{C}$) in comparison to *O. europaea* ($544 \pm 20^{\circ}\text{C}$) and *Quercus* spp. (evergreen) ($509 \pm 6^{\circ}\text{C}$). The one-way ANOVA analysis showed that temperatures were significantly at 95% different among different wood *taxa* (Figure 30A). However, in SU72 (Figure 30B), the significant ($P < 0.05$) highest crematory temperature is registered by *O. europaea* ($506 \pm 12^{\circ}\text{C}$), while *Quercus* spp. (evergreen) and *P. pinaster* ($492 \pm 3^{\circ}\text{C}$ and $487 \pm 3^{\circ}\text{C}$, respectively) showed no significant ($P > 0.05$) differences among them.

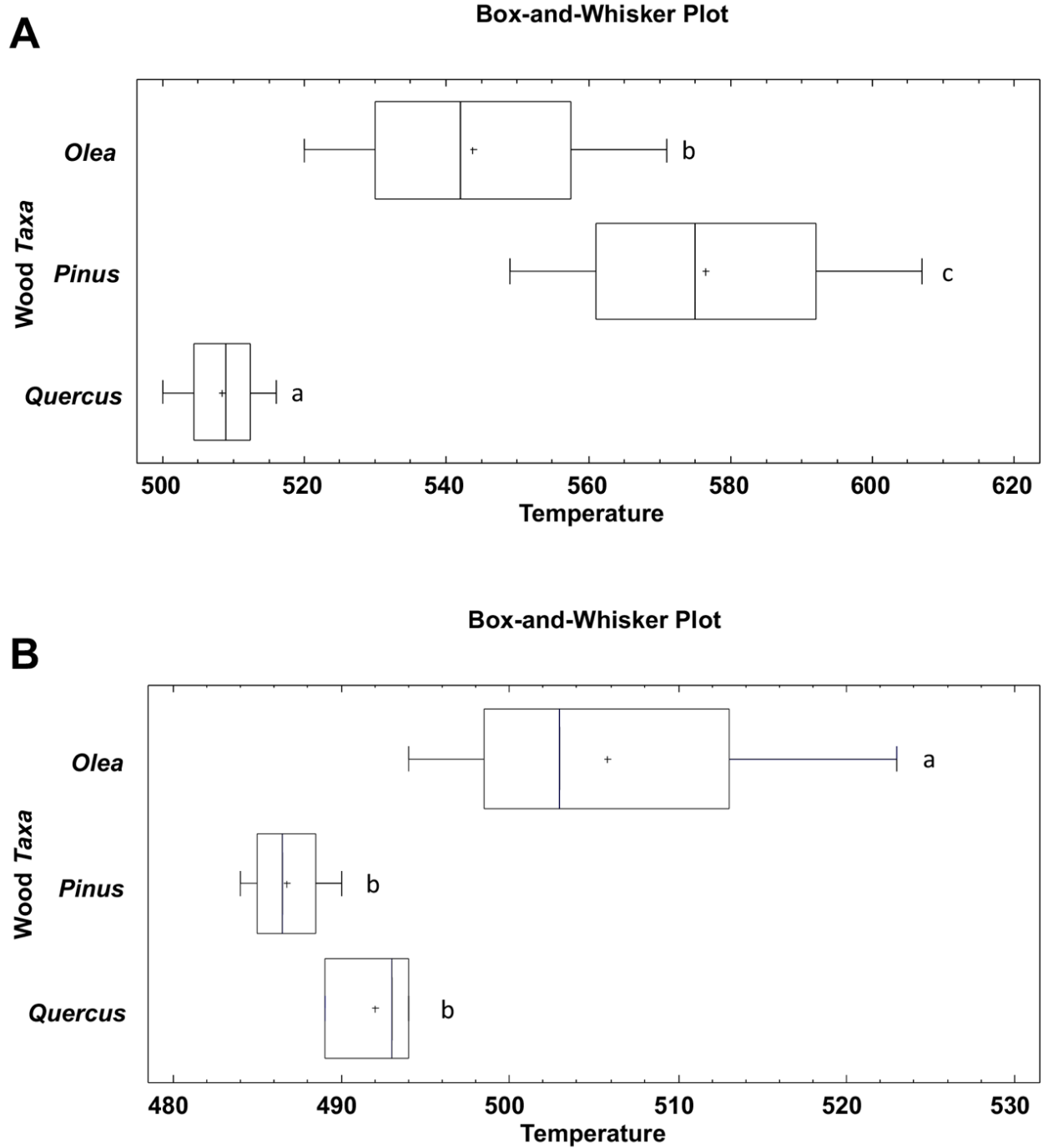


Figure 30: Boxplots of the predicted absolute burning temperature of *Olea europaea*, *Quercus* spp. (evergreen), and *Pinus pinaster* in A) SU74 and B) SU72. Boxplots display the ranges, lower and upper quartiles (Q1, Q3), and the median (Q2). Box plots with different letters indicate significant difference. (ANOVA; Means compared using Tukey test, $P < 0.05$).

4. 2. 5. Discussion

4. 2. 5. 1. Different wood taxa

All *taxa* identified are characteristic of the Mediterranean deciduous and evergreen vegetation.

Starting from the relative frequencies of each species identified and their corresponding mean value, obtained by the application of the “ubiquity correction” method, it was possible to identify three main *taxa* in the anthracological record of Pit 16: *O. europaea*, *Quercus* spp. (evergreen) and *P. pinaster* (Figure 3). Namely, the olive and the evergreen oak woods are the two main *taxa* (Table 1, Figure 2). The presence of the olive wood in the studied context is remarkable, because there are only a few cases where its usage in archaeological human cremations is reported (Coubray 2013; Charlier et al. 2009; Figueiral et al. 2010). The lack of these kind of studies for southern Europe might be the explanation for the scarcity of this species in other funerary archaeological contexts.

On the contrary, regarding oak and pine woods, their presence in different human cremations and burials of both prehistoric (Diogo Monteiro et al. 2015; Gutiérrez-Cuenca et al. 2017; Moskal-del Hoyo 2012; O’Donnell 2011; O’Donnell 2016; Vidal Matutano 2013), and historic sites (Deforce and Haneca 2011; Deforce and Haneca 2012; Figueiral et al. 2010; Hansson and Heiss 2014; Martín-Seijo and César Vila 2018) are numerous. In all these studies the combustion properties of these two types of wood are mentioned. Moreover, the presence of oak and pine woods in the same funerary context is attested since prehistory, suggesting a specific human choice, given their properties and characteristics (Diogo Monteiro et al., 2015). Regarding the identification of *F. cf. angustifolia* it must be emphasized that this species is numerically limited to a small number of charcoal fragments, and it is only present in the SU74 (Table 1, Figure 2). The presence of ash wood within the Pit, despite being in small amount, is an interesting datum, because it is the only arboreal *taxon* of the anthracological record in addition to the three main *taxa* *O. europaea*, *Quercus* spp. (evergreen) and *P. pinaster*. There are plenty of parallels regarding the use of this type of wood for human archaeological cremations, although its use seems to become recurrent only during historical time (Deforce and Haneca 2012; Figueiral et al. 2010; Gutiérrez-Cuenca et al. 2017; Martín-Seijo and César Vila 2018). Regarding the presence of the evergreen *taxa* of *Cistus* sp., *A. unedo* and Fabaceae, numerous studies have already widely attested the use of shrubs and small trees within the archaeological human cremation rituals. The rock rose is a small perennial shrub that grows in dry and rocky soils of the Mediterranean area and its presence is attested in other archaeological human cremations, especially during the Roman period (Figueiral et al. 2010; Martín-Seijo and César Vila 2018). There are also records about the use of Fabaceae woods and of Strawberry tree wood in human cremations (Gutiérrez-Cuenca et al. 2017; Martín-Seijo and César Vila 2018). About *A. unedo*, whose presence in the pit is limited only to the SU 74 (Table 1, Figure 2), there is a parallel of its use for

funerary purposes in the Portuguese chalcolithic archaeological site of Cabeço da Amoreira (Muge, Portugal) (Diogo Monteiro et al., 2015).

4. 2. 5. 2. *Woods properties and their possible origin*

The characteristics and properties of the identified woods provide information regarding the selection, intentional or not, and use of wood by the Chalcolithic community which prepared the pyre/s deposited in Pit 16.

Regarding the tree main *taxa*, nowadays the burning properties of olive wood are well appreciated, in particular the long duration of its fire and the pleasant aroma which derives from its burning. Furthermore, its sacredness is emphasized in the Bible (Stavropoulou, 2009) and in the ethnographic literature (Hansson and Heiss, 2014). It should also be emphasized that, to be one of the main *taxa* of the anthracological record, this wood might have been intentionally selected by the community who made the ritual, and the selection could have been made for cultural and/or social reasons. Concerning oak wood, the literature underlines how it provides an excellent fuel, long lasting and with high calorific power (Gale and Cluter 2000; Peña chocarro et al. 2000). On the contrary, pine wood burns very quickly, reaching high temperatures rapidly, being usually used for fire lighting (Zapata Peña and Peña Chocarro, 2003). The use of pine wood within funerary rituals for symbolic reasons cannot be excluded, especially given its high resin content, which would produce a pleasant smell during the incineration (Figueiral et al. 2010; Martín-Seijo and César Vila 2018; Moskal-del Hoyo 2012).

Regarding the use of ash wood, it is well known that it produces an excellent fuel, but its most interesting characteristic in this context seems to be the property of burning, even when it is not seasoned. Concerning the properties of shrubs in the anthracological record of Pit 16, we underline how small-calibre woods would seem particularly useful in lighting fires. Moreover, they emanate a pleasant smell during their combustion, and they seem to have been much appreciated for their decorative presence. Their use in these contexts may represent an anthropic symbolic choice (Segura Munguía and Torres Ripa, 2009). About rock rose, its presence in funerary context for symbolic reasons has also been contemplated (Figueiral et al., 2010).

Assemblages of concentrated charcoal, especially those ones coming from a single context, cannot be considered reliable for the reconstruction of the vegetation surrounding the archaeological site. In spite of that, the anthracological record of this type of assemblages reflects at least part of the vegetation of the site, representing a very important source of information. In the case of Pit 16 the identified *taxa*, if appropriately compared with the results of pollen analysis at Perdigões (Danielson and Marques Mendes 2013; Wheeler 2010) and with the current vegetation of the region, may offer

insights on the possible supply area of woods. These data provide indirect information on the possible area where the primary ritual could have taken place. Pollen analysis carried out on Late Neolithic layers of Perdigões identified the main three genera observed in the anthracological record of Pit 16, *i.e.*, *Olea*, *Pinus* and *Quercus*. Inside the pollen records, the presence of various species belonging to the genus of *Cistus* and the presence of Fabaceae species has also been verified (Danielson and Marques Mendes, 2013). On the Chalcolithic layers of Perdigões, *Quercus* and Oleaceae, the olive family, were identified (Wheeler, 2010). Comparing the anthracological data with current vegetation data, we observe that all species present in Pit 16 are characteristic of the typically Mediterranean native vegetation, which surrounds the Perdigões archaeological complex (Carrión and Leroy 2010; Costa et al. 2012; Loidi 2017). According to these data, the origin of woods identified could have either been the surroundings of Perdigões or a related area of the same region. Nevertheless, the discussion of the results of this study cannot, however, disregard the comparison with other archaeological data, which highlights how the action area of the communities of Perdigões was, actually greater than previously supposed (Valera and Evangelista, 2014). These data are corroborated by some studies on the mobility of the communities (Žalaitė et al., 2018), which suggests that the presence of exogenous individuals in the archaeological site may have been large. Reflecting on these evidences it is therefore necessary to report that wood species identified in the anthracological record of Pit 16 are typical of most of the Mediterranean basin (Carrión and Leroy 2010; Pignatti 1978; Thompson 2005). In the case of the maritime pine (*P. pinaster*), whose nativity in the hinterland part of Portugal has been disputed for decades, charcoal remains found in different archaeological sites have confirmed their naturalness within the Iberian Peninsula (Carrión and Leroy 2010; Figueiral et al. 2010). Based on this data, it is challenging to formulate a single hypothesis concerning the origin of the wood *taxa* collected in the secondary cremation of Pit 16. At this stage it is not possible to exclude that the primary cremation occurred neither in the vicinity of the archaeological site nor in more distant areas.

4. 2. 5. 3. *The context and the ritual/s?*

Results obtained from the identification of charcoals from Pit 16 shed light on their original context and on the ritual associated therewith. Differences in the number of charcoals and in the number of *taxa* within the two stratigraphic units (SU72 and SU74) (Table 1, Figure 2), seems to suggest that woods were intentionally selected to build the pyre/s during the primary ritual of cremation/s. This consideration strengthens the archaeological data. Moreover, these data allow to hypothesize that the two different stratigraphic units might be two different and subsequent depositional moments, likely as deriving from two different cremations. This hypothesis seems even

more reasonable by looking at the differences in the relative frequencies and in the means values of each identified *taxon* (Table 1, Figure 2). These values confirm that the distribution of *taxa* through the two stratigraphic units is not homogeneous.

Regarding the three main *taxa*, the more significant values differences are related to the species of *O. europaea* and of *P. pinaster* (Table 1, Figure 3). Regarding the rest of the *taxa* *F. cf. angustifolia* and *Arbutus unedo* are only present in SU74 (Table 1, Figure 4). This latter data seems to corroborate the hypothesis of the existence of two different depositional moments (coming from two different cremations), one related to the SU 72 and another one related to the SU 74.

Regarding to the charcoal size classes recorded, results show that larger charcoals belong to the three main arboreal *taxa* identified (Figure 6). This result was predictable, considering that the rest of the anthracological record is mainly formed by shrubs, usually smaller in size than a tree. Some anatomical charcoal alterations were also identified and recorded, and the most significant observations have been reported, but they should not be considered conclusive. With regard to the radial cracks observed in some charcoals of *P. pinaster*, these were probably the result of a conspicuous loss of humidity on burned fresh wood (Tereso, 2014). In our opinion, considering the absence of other signs of degradation, caused by fungi and/or insects on charcoals under study, radial cracks might be the result of the use of this type of wood when it was still green. In this case, the use of *P. pinaster* would have taken place shortly after the cutting of the parts of the tree involved in the ritual. There was no evidence of a strong curvature of the ring for any charcoal fragment of *O. europaea*, leading to the hypothesis that large branches or parts of the trunk were used for the cremation ritual. The vitrification features recorded for some charcoals of *Quercus* spp. (evergreen) have been always observed in association with a strong curvature of the ring, which seem to suggest that branches and twigs were used. However, this strong curvature has not been recorded for charcoals not vitrified, suggesting the possible use of other parts of this wood, as limbs, or the trunk.

The reaction wood observed on some charcoal fragments of *P. pinaster*, namely grooves inside tracheids visible on the longitudinal sections of charcoals, suggests a concentric growth in this part of the tree that would have been developed during its life to prevent fall under its own weight (Marguerie and Hunot, 2007). The finding of reaction wood in archaeological charcoal and woods, is often considered an indicator of the fragment's origin from branches or twigs when it is associated with a strong curvature of the ring. However, in the charcoal fragments of *P. pinaster* of the anthracological record of Pit 16, a strong curvature of the ring was never registered. A strong curvature of the ring was also not recorded in the few *F. cf. angustifolia* charcoals observed. Thus, in both cases large branches and/or part of the trunk have been used.

Concerning the identification of few twigs or small branches of *A. unedo* and Fabaceae, the observation of a strong ring curvature for all charcoal fragments identified seem to indicate the use of twigs or small branches for the cremation/s. The same observation is attributable to all charcoal fragments of *Cistus* sp., for which a strong ring curvature was also observed. The information concerning the possible use of different parts of the tree (larger and small branches, twigs, parts of the trunk), seems to corroborate the hypothesis according to which the charcoals from Pit 16 would have formed the pyre/s used for the primary cremation/s.

4. 2. 5. 4. Forecasted combustion temperature of charcoals from Pit 16

The clear relationship between FT-MIR data on the test samples and the combustion temperature, in the range tested here, had proved the power of this technique to correctly recreate burnt temperatures for wood charcoal produced during the primary cremation ceremony.

All archaeological charcoals retrieved in SU74 showed a relative high burn temperature (> 500°C). This result was in line with data reported from other authors (Veal et al., 2016), who determined the absolute combustion temperature of archaeological charcoal samples from human cremations using reflectance. Nevertheless, in SU72 only the *Olea* charcoal sample particles reached the temperature of 500°C.

The burn temperature calculated by the combination of FT-MIR and chemometrics analysis may suggest to authors the existence of a single cremation for SU 74 i.e., the human cremation was made using a mixture of different types of woods in a single pyre. On the other hand, the statistical analysis of the combustion temperature of charcoal sample particles found in SU74 determined the existence of a significant ($P<0.05$) difference among the temperature reached by the different types of wood. This difference may be due to different factors, such as: i) the chemical composition of each type of wood (inflammable compounds like resins, epicuticular waxes, etc.); ii) the wood moisture content (Dzurenda and Banski, 2017) and/or iii) the resistance to burning (protective cover like suberin) (Jiménez-Morillo *et al.*, 2016). A single deposition coming from a single pyre, employed for the primary cremation regarding the SU74 could be explained by the significant different combustion temperature of each *taxon*.

In the case of SU72, there were not significant differences between the burning temperature of *Quercus* and *Pinus* archaeological charcoal samples. The highest temperature registered by *Olea* may be due to the low wood moisture content in this kind of wood *taxa*. However, the burn temperature of *Olea* samples from SU72 was significantly different from the rest of the main *taxa* (*Quercus* and *Pinus* samples). In the case of *Pinus* samples, it was observed a significant temperature depletion of 89°C (Figure 10) between the two stratigraphic units SUs 72 (486°C) and 74 (575°C).

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This fact may reflect the use of relatively green *Pinus* individuals in SU72, which usually displayed a relative low concentration of resins and waxes compounds (inflammable), burning at lower temperatures. This data could be linked with the radial cracks, observed with the anthracological analysis, in some charcoal fragments of this species, and discussed in the previous section. The possible use of some green woods for the cremation would explain the low combustion temperature of *Pinus* samples from SU 72. In contrast, in SU 74, the highest temperature reached by *Pinus* may reflect the use of seasoned individuals with a relatively noticeable amount of resinous material (high degree of flammability). Therefore, in both cases, the wood seasoning of Pine samples could be considered an important *actor* to determine the temperature of combustion.

From the combustion temperature differences of the tree main *taxa* of Pit 16, especially regarding charcoals fragments of *P. pinaster*, observed between the two stratigraphic units SUs 72 and 74, we can put forward the hypothesis of the existence of 2 different depositional moments, inside the Pit and consequently coming from two different primary rituals (cremations). This hypothesis was already raised with the anthracological analysis and would corroborate the archaeological data regarding the distinction of the two different stratigraphic units

The absolute relative temperature reached by *Quercus* charcoals in SU72 is comparable to that one reached in SU74. This may be due to the use of *Quercus suber* wood in both crematory events. The existence of a burning protective cover, like suberin, on trunk would be able to limit and “average” the combustion temperature of the *Quercus* wood in both crematory events. Moreover, *Quercus suber* tree display a relatively high abundance of this species in the Alentejo region (Blanco et al., 1997). This hypothesis is supportive of the anthracological identification of samples belonging evergreen oak type group, previously displayed in this work.

4. 2. 5. 5. *Culturally selection VS availability selection?*

The anthracological study of Pit 16 has proved to be particularly relevant for the understanding of human choices of timber for funerary purposes, demonstrating its importance in discerning the complexity of the human cremation process: do the culturally determined wood selection and the wood selection based on the territorial availability represent two different solutions, two coexistent solutions or are they intrinsic solutions?

If we consider the so-called least possible effort (Kingsley Zipf, 1950) as a parameter which has often been linked with prehistoric communities that produced concentrated charcoals, we must take into account that this parameter could also have been applied by the Perdigões community during the collection of woods for cremation purposes. Furthermore, the identification of different types of woods can be interpreted as a decision of the community not to choose one or a few species for

specific ritual reasons, but rather indicating a collection based on the easy availability of woods. On the other hand, it is necessary to consider that in the anthracological record of the Pit 16 three woods are dominant: the wild olive, the evergreen oak, and the maritime pine. We know that the first species gains a symbolic significance during historical times, while there are also numerous evidence of the use of the second and the third species in other prehistoric cremations. This is the first time that these kinds of evidence are studied in Perdigões and these results will be essential for the interpretation of the funerary practices in the archaeological site during the Chalcolithic. The data collected so far, and its comparison with other case studies from different prehistoric cremations and burials seems to indicate that cultural choices and choices dictated by the environment go hand in hand in all contexts investigated, emphasizing the intrinsic nature of the two variables.

To answer such a wide question by looking for a singular solution, when human choices seem to be dictated by multiple factors in all periods of communal human existence, past and present, is an unworthy effort.

4.2.6. Conclusion

The charcoal record of Pit 16 is a mixture of different trees species. The most abundant tree *taxa* are *O. europaea*, *Quercus* spp. (evergreen) and *P. pinaster*. All the species identified in the study are characteristic of both deciduous and evergreen Mediterranean vegetation. The origin of plants selected seems to indicate the occurring of the human cremation/s either on site, in the vicinity or in an area with access to a similar vegetation. Anyway, according to the large distribution of identified *taxa* and considering data about human mobility, it is not possible to rule out that the burnt wood could come from cremation places far from the site. Differences in the relative frequencies and in the means values of each identified *taxon* seems to suggest that the two different stratigraphic units might be two or more different and subsequent depositional moments, likely as deriving from different cremation rituals.

Regarding the investigation of the charcoals burn temperature, this work has demonstrated the validity of the combination of FT-MIR and chemometric analyses to establish the absolute combustion temperature of archaeological charcoals from a human cremation. It has been shown that is possible to establish the burn temperature of archaeological charcoals from predictive models generates from sound charcoal reference samples. Considering both the data from the FT-MIR analysis and that from the chemometric analysis, has been possible to hypothesize the existence of different cremation pyres and consequently of different secondary depositional moments, as already raised with the anthracological analysis. It was possible to speculate the existence of 2 different depositional moments inside Pit 16.

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As for the understanding of human choices of wood species employed for funerary purposes, results from this study seem to indicate that both cultural choices and the ones dictated by the environment might have driven the chalcolithic community of Pit 16 in the wood selection for the funerary ritual/s.

This study demonstrated the validity of the use of different approaches, the archaeobotanical, the chemical and the statistical one to shed light concerning the role of woods within the ritual of cremation within the funerary practices present in Perdigões and, in general, in the western part of the Iberian Peninsula during the Chalcolithic.

4. 2. 7. Bibliography

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4.3. Case study 2: The wooden roof framing elements, furniture and furnishing of the Etruscan *Domus dei Dolia* (Vetulonia, Southern Tuscany, Italy)

4.3.1. Introduction and research aims

The Etruscans, known as Tyrsenoi or Thyrranoi by the ancient Greeks, flourished in central Italy between the 9th and the 1st century BC. They mainly settled and ruled in central Italy (in part of the present-day territory of the Tuscany, Lazio and Umbria regions), with important presences in Campania, in a large part of the Po valley (Emilia Romagna, Lombardy, Veneto) and on Corsica Island. Etruscans were one of the most important civilizations of ancient Italy, and the funerary equipment recovered on several monumental burial mounds is widely known, being evidence of the degree of their civilization. Organized through a confederation of 12 city states (Dodecapoli), they are quite famous for their deep metallurgical skills, for the ability of their artisans in gold processing, for their capability of trading, and for their cult of the deaths. Moreover, they were also skilled sailors and traders, having contact with the most important civilizations of the Mediterranean Area. From the 3rd century BC, the Etruscan civilization was slowly adsorbed by the Roman Empire (three of the legendary kings at the dawn of ancient Rome were Etruscans)(Cristofani, 2000; Cristofani and Martelli, 1983; Nestler and Formigli, 2013; Camporeale, 1969).

Even if much is known about their cult of the death, quite little is known, on the contrary, on Etruscan daily life and settlements (Miller, 2015). In particular, the exploitation of natural resources for construction purposes has rarely been addressed (Amicone et al., 2020; Ceccarelli et al., 2020), and few data are available regarding the exploitation and selection of wood (Bound, 1991; Coccolini and Follieri, 1980; Marchesini et al., 2010; Mariotti Lippi et al., 2002; Mariotti Lippi et al., 2020).

Different scholars ascribed to Etruscans, through pollen analysis, the responsibility of forest clearance that heavily altered the surrounding environment (Drescher Shneider et al., 2007; Magny et al., 2007; Negri, 1927; Sadori et al., 2010), but systematic archaeobotanical studies from Etruscan settlements are occasional, and a clear connection between the opening of forests and the wood used for building, cooking and smelting has never been established.

Given the rarity of this type of study, the analysis of the charred woods of the *Domus dei Dolia* proves to be important, in particular for the deepening of knowledge about the Etruscan world of the living, which still today remains largely unknown.

The discovery of the *Domus dei Dolia*, located in the ancient town of Vetulonia (Castiglione della Pescaia, Southern Tuscany, Italy), and the study of the materials retrieved, made it possible to create an accurate reconstruction of the entire Etruscan house, and to describe the life and the activities of the wealthy inhabitants of the *Domus* between the 3rd and the 1st century BC (Beltrame

and Coradeschi, 2016; Rafanelli et al., 2017). Moreover, the extraordinary nature of the architectural data unearthed by the archaeological excavation allowed for the acquisition of information concerning the city of Vetulonia in the last centuries of its Etruscan history. Among the materials retrieved from the excavation of the *Domus*, there were bricks, mortars, roof tiles, different ceramic wares, votive bronze statues, coins, nails, and numerous charred woods, with these being the last the object of this study. The analysis of wood charcoals belonging to several structural and furnishing elements coming from different rooms of the *Domus dei Dolia* represents the first study regarding the choice and the use of wood by the Etruscans of Vetulonia.

Thus, the objectives of this study are:

1. To hypothesize the probable origin of the plants used through the identification of the timber at the family/genus/species level.
2. To understand the reasoning behind the wood species selection for construction purposes through the evaluation of the wood-related technological knowledge of the Etruscan carpenters.

Overall, this study aims to shed light on the technological, economic, and social aspects of the inhabitants of the *Domus dei Dolia*, and, in a sense, of the Etruscan community of Vetulonia.

4.3.2. Archaeological settings

4.3.2.1. *The Domus dei Dolia in the Hellenistic district of Poggiarello Renzetti*

Vetulonia is located in Southern Tuscany, and it was an important city during Etruscan times. It ruled in a vast territory, extremely rich in natural resources such as metals, mined in the so-called area of the Colline Metallifere. During the first centuries of its Etruscan history (the 9th–11th centuries BC) it was a vital centre, full of artisan shops, well known for its bronze workers and goldsmiths (Gregori, 1991). Between the 8th and the 6th centuries BC, Vetulonia was the most important of the 12 Etruscan city states, and it survived to the Roman expansion of the 2nd century BC. Nevertheless, it was forced to become an ally of Rome, maintaining his own identity. This was a period of prosperity for the city. The beginning of the Etruscan Vetulonia decline was the result of its involvement in the Roman civil war, which led to the destruction of the city in the 1st century BC (Semplici, 2015).

The name of the ancient town of Vetulonia had disappeared from official documents in 1201. The first archaeological works, which lasted from 1882 to the early stage of the 1900s, were conducted by Dr. Isidor Falchi (Bruni, 1995). Falchi discovered the funerary area of the city, discovering both the tombe a pozzetto (pit tombs) from the Iron Age, the Villanovan phase of Etruscan Culture (end of the 9th and the beginning of the 8th centuries BC) and the majestic tumulus tombs, attributable to the so-called Etruscan Orientalizing Period (from the 8th century to 580 BC)

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(Cygielman, 2002; Falchi, 1891). Moreover, the excavations led also to the "discovery" of the ancient Etruscan city which, with the royal decree of 22 July 1887, regained the ancient Etruscan name of Vetulonia (Bruni, 1995). The excavations conducted by Falchi were concentrated in the area known as Poggiarello Renzetti, where the Etruscan–Roman district of the city was brought to light (Figure 31). The excavations conducted by Falchi were concentrated in the area known as Poggiarello Renzetti, where the Etruscan–Roman district of the city was brought to light.

The area was subject of new archaeological excavations in the late 1980 by the superintendent *Mario Cygielman*, who discovered the so-called *Domus of Medea* (Cygielman, 2015). Other archaeological evidence of the urban Etruscan area was excavated by Anna Talocchini between 1960 and 1970, referring to the sanctuary part of the city (i.e., the Acropolis). These are the areas of Costia Lippi and Costa Murata, both chronologically attributable to the Hellenistic (4th–3rd centuries BC) and to the Late Republican periods (2nd–1st centuries BC), respectively (Rondini and Zamboni, 2015; Talocchini, 1981). In addition, some evidence (i.e., pottery) suggested that the area of Costa Murata was already occupied (in the second half of the 6th and the middle of the 5th centuries BC) during the archaic period (Rafanelli, 2015). Furthermore, the well-known Mura dell'Arce, for first discovered by Isidoro Falchi at the end of 1800, which according to new research refer to a chronological period between the 3rd and the 2nd centuries BC, are worth mentioning (Cygielman, 2002; Rafanelli, 2015). Finally in 2009, under the direction of *Simona Rafanelli* (current director of the Etruscan Museum of Vetulonia) a new archaeological excavation took place in the *Poggiarello Renzetti* area (the Etruscan–Roman neighbourhood discovered by Falchi), and the *Domus dei Dolia* was brought to light (Agricoli et al., 2016).

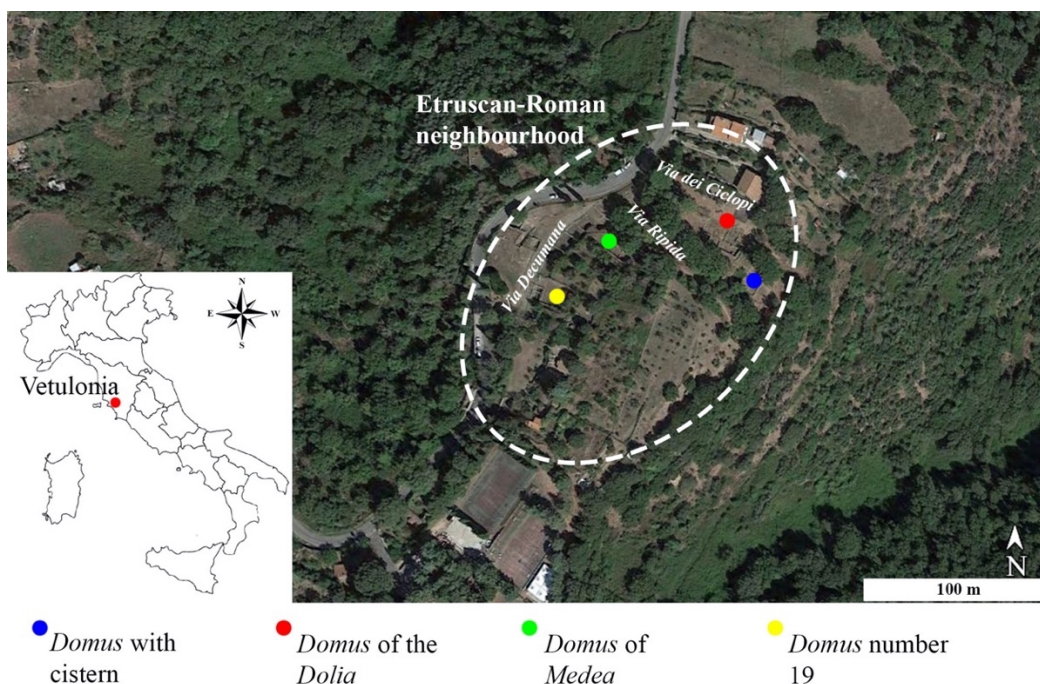


Figure 31: The Etruscan district of Poggiarello Renzetti. Source: Google Earth Pro.

It is within the insula defined by these three paths that the *Domus dei Dolia* was discovered. The *Domus* supports other residential Etruscan structures of Hellenistic period (4th-1st centuries BC) such as the *Domus* n. 19, the *Domus* of Medea and the *Domus* characterized by the presence of a vast atrium with *impluvium* and of the adjacent large cistern carved into the rock (Falchi, 1898; Cygielman et al., 2011) (Figure 31). The Hellenistic quarter of *Poggiarello Renzetti* represents, therefore, with its important housing structures, the most striking archaeological evidence of the revival of Vetulonia to new splendour with a building and economic renaissance, started from the 3rd century BC.

4.3.2.2. Archaeological evidence of *Domus dei Dolia*

The *Domus dei Dolia* was destroyed by a fire. The classification of the recovered archaeological materials, such as black painted ceramic and Greek-Italic amphorae, suggest a chronology comprised between the 3rd and the 1st centuries BC. It is known that the Etruscan city was destroyed by the troops of Lucio Cornelio Silla in the 1st century BC, in the aftermath of the victory over Gaius Mario's army during the Roman civil war (Rafanelli and Spiganti, 2019).

This *Domus* represents an exceptional discovery for the archaeological area of Vetulonia and for Etruscan archaeology, as well-preserved dwellings with high rises (over 1.60 m and about 6 cm in thickness) have rarely been found. Moreover, because of the fire, the roof collapsed, sealing, and preserving an old context with a wide variety of materials.

The building is divided into about 12 rooms (the excavation is still underway) (Figure 32), and from the excavation data (ongoing study) it seems to have had three construction phases. The I phase (beginning of the 3rd century BC) seems to include 5 different rooms (A, C, E, G and D), arranged to the south. Rooms E and G seem to be interconnected and Room D probably served as house entrance at this stage. From the II phase on (2nd century BC), Room E was separated from Room G, becoming one of the most important rooms of the house. From this phase, the *Domus* seemed to be arranged around a big semi-open courtyard, Room D, which is being refurbished, becoming the first atrium of the house, possibly with an *impluvium*. Furthermore, in this phase, Rooms F and H (courtyard with portico) and room B (processing products area) were possibly built. In the III phase (between the 2nd and the beginning of the 1st century BC) the *Domus* will be expanded to the north with the addition of other rooms (e.g., P, S) and a large peristyle (K). During this phase of the house's life, Room E seems to have replaced the function of Room C, becoming the most important room of the house. The *Domus* was configured like that until its destruction (Rafanelli and Spiganti, 2019). Many precious remains were recovered inside the *Domus* revealing the richness of this building and its inhabitants. From one of the most significant rooms of the house, the formal dining room, the *Triclinium* (Room C), archaeologists recovered a small white limestone column, a

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precious bowl decorated with an animal head, and a few bronze coins, possibly associated to a foundation ritual, related to the new life phase of the house (Agricoli et al., 2016).



Figure 32: *Domus dei Dolia* divisions, pictures by Paolo Nannini and Stefano Spiganti.

This room was also characterized by painted walls with red and blue frescos, belonging to the first Pompeian style (Rafanelli and Grassigli, 2018). The *Domus* also included a representative guest reception room, the *Tablinum* (Room E), with a beautiful floor decorated with a meander motif,

formed by white and grey limestone tiles and plaster (*Opus Signinum* style). The walls of this room were also decorated with frescoes (Agricoli et al., 2016; Rafanelli and Grassigli, 2018). Inside the two storage rooms (Rooms A and G) many amphoras and big earthen pots (*Dolia*) were discovered, one of which is about 1, 20 meters high and is still almost intact. The uniqueness of these discoveries justifies the name of the *Domus*. Moreover, in the storage G a small treasure was also discovered consisting of some votive bronzes. The house also included a room perhaps associated with grape or olive processing (Room B) (Rafanelli and Spiganti, 2019). The architecture of the *Domus* seems to refer to a specific type of rural aristocratic housing with peristyle court, diffuse in the Attic region from the V century BC and called “*Pastas Style*”. This type of dwelling is well documented in the southern part of the Italian peninsula during the Hellenistic age (Fentress, 1999, Graham, 1966).

The Material employed for the construction of the *Domus dei Dolia* is sandstone that had been extracted for the construction of the perimeter walls.. In some rooms, the walls were probably finished by using raw clay material. Tiles and brick tiles have been used to cover the roof and raw bricks have been utilized for the structure of some internal divisions of the house. Wood was utilised for the construction of the room framing elements, for the doors and maybe for the internal support of the raw clay parts of the walls which were probably built with a different kind of technique (Agricoli et al., 2016). It is important to report that no wooden remains associated with the building process of the raw clay walls have been retrieved. Moreover, the few raw clay remains bring back the negative of small stem reeds.

From the archaeological data and from the *Domus* typology we can assume that the house was supplied with light from the central courtyard, therefore the presence of windows in the investigated area of the house is not expected. The huge *Domus dei Dolia*, containing several compartments with various well preserved and precious materials uncovered within the wood charcoal remains object of this study, represents a unique case for Etruscan archaeology.

4.3.3. Materials

The materials analysed comprise wood charcoal remains belonging the first archaeological campaigns (2011–2016) of the *Domus dei Dolia*. These archaeological campaigns were conducted within the better-preserved part of the house, the southern part, which was the most relevant investigated area due to the presence of collected wood charcoal evidence. The northern part of the house (excavation ongoing) was largely destroyed by agricultural works, and the archaeobotanical evidence is scarce or absent. The wood charcoal remains under study are representative of the II and III rebuilding phases of the *Domus*, which was ultimately ruined and buried under the fire. The charred wood samples come from seven different compartments of the house, namely Room A

(storage), Room C (*Triclinium*), Room D (semi-open courtyard–first atrium), Room E (*Tablinum*), Room F (court), Room G (storage) and Room H (semi-open room with portico) (Table 3, Figure 32).

4.3.4. Methods employed

4.3.4.1. Sampling

Anthracological analyses were not included in the initial planning of the archaeological excavation. For this reason, most of the wooden material under study was identified visually, collected by hand (in small and restricted areas), drawn and documented. This material consisted of large charred wooden elements, generally broken/fragmented (the largest fragments were 5 cm in diameter), that were interpreted by the archaeologists as wooden roof framing elements based on their size, position and context during the excavation (Figure 33, Table 3).



Figure 33: Drawing and a picture of the different wooden roof framing elements identified in Room G. Picture and drawing by Stefano Spiganti.

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Table 3: The provenance and number of the charcoals analysed, and their archaeological ascription (SU: stratigraphic unit).

Room	SUs	Sample excavation data			N° of wood charcoal fragments
		Classification/Information	Charred wooden elements	Wood charcoal from dark/black areas	
A	61	Wooden roof framing elements	2		20
	62	Wood charcoals		x	60
C	77	Wood charcoals close to nails		x	9
	98	Wood charcoals		x	48
	102	Wood charcoals close to nails		x	80
D	111	Wooden roof framing element	1		10
E	128/129	Wood charcoals		x	71
	129	Wood charcoals close to nails		x	55
F	166	Wood charcoals		x	34
G	143	Wooden roof framing elements	3		30
	143	Wood charcoals		x	50
	143	Wooden roof framing element	1		10
	143	Wood charcoals with plaster - near the collapse		x	65
	143	Wood charcoals - corner of the room		x	27
	145	Wood charcoals from the interior of a <i>Dolium</i>		x	26
	145	Wood charcoals		x	25
	146	Wooden roof framing element - corner of the room	1		10
	146	Wooden roof framing elements - close to a <i>Dolium</i>	2		20
	147	Wood charcoals - close to nails		x	50
	151	Wooden roof framing element	1		10
	151	Wood charcoals - close to amphorae and nails		x	315
	152	Wooden roof framing element	1		10
152	Wood charcoals – centre of the room/near votive bronzes		x	150	
H	159	Large and elongated wooden piece	1		10
Total					1195

However, it must be considered that most of the wooden components of the *Domus* were destroyed by the fire and by the collapse of the roof; the attribution of the material, during the excavation, to a single original wooden element was not always possible. These uninterpreted wood charcoal remains were generally dispersed in wider dark/black areas of the archaeological layers. Attempts were made to collect the entirety of the remains in these darkened areas, but some smaller charcoal fragments may have been lost. Within the category of wood charcoal from the dark/black areas, one should also consider the possibility that remains from furniture and different furnishings/objects of the *Domus* could also be present in the samples recovered for analysis.

From the material retrieved from the excavation, and given its fragmentary nature, the sampling of the wood charcoal remains was organised in the following way: for each charred wooden element identified during the archaeological excavation, 10 of the largest fragments were selected in order to confirm their provenance from the same tree species; the samples not identified during the excavation phase (samples from the dark/black areas) were studied in their totality.

4.3.4.2. Anthracological analysis

The description of the anthracological analysis method has been previously described (Chapter 3 - 3.7.2. Taxonomic identification of charcoal samples – Anthracological analysis).

In addition to the taxonomic identification some other types of observation have also been made. For samples identified as building elements, in fact, when possible, the growth ring curvature have been observed (Castelletti, 1990; Marguerie and Hunot, 2007; Peña chocarro et al., 2000; Schweingruber et al., 2008). When present, galleries of xylophagous insects were also recorded (Marguerie and Hunot, 2007; Théry-Parisot and Henry, 2012).

For most of the charcoal samples the identification of the species was possible, although in some cases the degradation of the wood only allowed for the identification of the genus or the family. In this case the name of the genus is followed by the abbreviation of species (sp.), i.e., *Acer*. sp. When species attribution was highly probable the abbreviation (cf.) is placed between the name of the genus and the name of the species, i.e., *Prunus* cf. *avium*.

Regarding the identification of the wood belonging to the genera *Abies* (fir wood) and *Fagus* (beech wood), their anatomy does not allow any distinction between species (Schweingruber, 1990; Quézel and Médail, 2003), but, considering the investigating period and the present-day distribution, they can be ascribed to *Abies alba* and *Fagus sylvatica* respectively.

For the distinction in *Quercus* (deciduous, semideciduous, and evergreen sections) we followed the indication of Cambini (1967). These guidelines distinguish the deciduous oaks (*Quercus* sect. *robur*) from the semideciduous (*Quercus* sect. *cerris*) and the evergreen ones (*Quercus* sect.

suber). In Italy the deciduous oak group includes *Quercus robur* L., *Quercus pubescens* Wild., *Quercus frainetto* Ten., and *Quercus petraea* (Matt) Liebl., the semideciduous one includes *Quercus cerris* L., *Quercus trojana* Webb, and *Quercus aegilops* L. and the evergreen one includes *Quercus suber* L., *Quercus ilex* L. and *Quercus coccifera* L.

4.3.4.3. Quantification

Regarding quantification analysis, it is generally advisable to previously determine which may be the most useful and relevant method to be used in a particular occurrence (Pearsall, 2019; Pielou, 1984; VanPool and Robert, 2011). In this case-study two different quantitative methods were employed, the frequency and the ubiquity correction (Hubbard, 1980; Moskal-del Hoyo, 2011). The frequency (%), based on the absolute number of charcoal fragments, was employed to evaluate the different categories of wood charcoals which provenience was attributed over the context of the *Domus* - wood roof framing elements, furnishing, court tree/s. The ubiquity correction (%U) was used to show the occurrence of a tree species across the *Domus dei Dolia* contexts.

These methods considered only wood charcoal fragments coming from the dark/black areas. An assessment based only on the quantification of the *taxa* identified as single wood elements during the excavation would potentially obscure the importance of some *taxa* as building material.

4.3.5. Results

In total, 1195 wood charcoal fragments coming from seven different compartments of the house rooms A, C, D, E, F, G, H were analysed and identified (Figure 34).

A total of nine *taxa* were identified. Amongst them were *Abies* cf. *alba*, *Acer* sp., *Buxus sempervirens*, *Fagus* cf. *sylvatica*, *Prunus* cf. *avium*, *Quercus* sect. *cerris*, *Quercus* sect. *robur*, *Quercus* sect. *suber* and Rosaceae (Table 4, Figures 34, 35, 37).

The most exploited woods for the construction of the roof of the *Domus dei Dolia* were deciduous and semideciduous oak and silver fir wood (Table 4).

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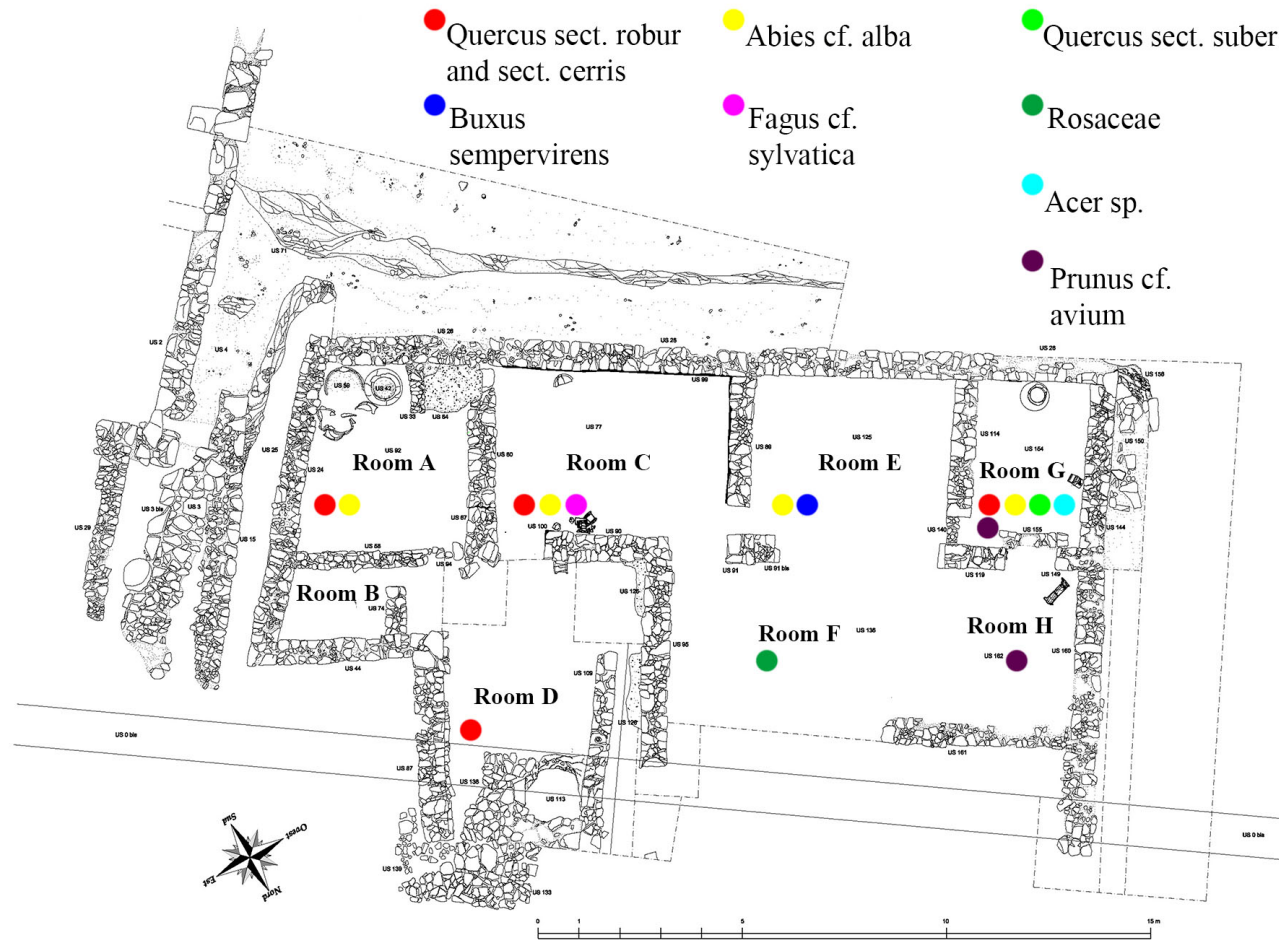


Figure 34: Plant source distribution in the different rooms of the *Domus dei Dolia*.

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Table 4: Plant source and possible interpretation (SU: stratigraphic unit).

Room	SU	Classification based on the archaeological and archaeobotanical data	Plant source	N° of wood charcoals fragments
A	61	Roof beams or rafters	<i>Quercus</i> sect. <i>robur</i>	20
	62	Fragments of wooden roof beam/s or rafters/s	<i>Quercus</i> sect. <i>robur</i>	15
	62	Fragments of wooden roof beam/s or rafter/s	<i>Abies</i> cf. <i>alba</i>	45
C	77	Fragments of wooden roof beam/s or rafter/s	<i>Quercus</i> sect. <i>cerris</i>	9
	98	Fragments of wooden furniture	<i>Fagus</i> cf. <i>sylvatica</i>	48
	102	Fragments of wooden roof beam/s or rafter/s	<i>Quercus</i> sect. <i>cerris</i>	30
	102	Fragments of wooden roof beam/s or rafter/s	<i>Abies</i> cf. <i>alba</i>	50
D	111	Roof beam or rafter	<i>Quercus</i> sect. <i>cerris</i>	10
E	128/129	Fragments of a kline bed foot	<i>Buxus sempervirens</i>	71
	129	Fragments of wooden roof beam/s or rafter/s	<i>Abies</i> cf. <i>alba</i>	55
F	166	Fragments of fruit tree/s of the courtyard	Rosaceae	34
G	143	Roof beams or rafters	<i>Quercus</i> sect. <i>robur</i>	30
	143	Fragments of wooden roof beam/s or rafter/s	<i>Quercus</i> sect. <i>robur</i>	50
	143	Roof beam or rafter	<i>Abies</i> cf. <i>alba</i>	10
	143	Fragments of wooden roof beam/s or rafter/s	<i>Abies</i> cf. <i>alba</i>	65
	143	Fragments of wooden tool	<i>Acer</i> sp.	27
	145	Fragments of wooden roof beam/s	<i>Quercus</i> sect. <i>robur</i>	25
	145	Fragments of a jar lid of a Dolium	<i>Prunus</i> cf. <i>avium</i>	26
	146	Roof beams or rafters	<i>Quercus</i> sect. <i>robur</i>	30
	147	Fragments of wooden roof beam/s or rafter/s	<i>Quercus</i> sect. <i>robur</i>	50
	151	Roof beam or rafter	<i>Quercus</i> sect. <i>robur</i>	10
	151	Fragments of wooden roof beam/s or rafter/s	<i>Quercus</i> sect. <i>robur</i>	315
	152	Roof beam or rafters	<i>Quercus</i> sect. <i>robur</i>	10
	152	Components of a container/support of votive bronzes	<i>Quercus</i> sect. <i>suber</i>	150
H	159	Dividing door of rooms G and H	<i>Prunus</i> cf. <i>avium</i>	10
Total				1195

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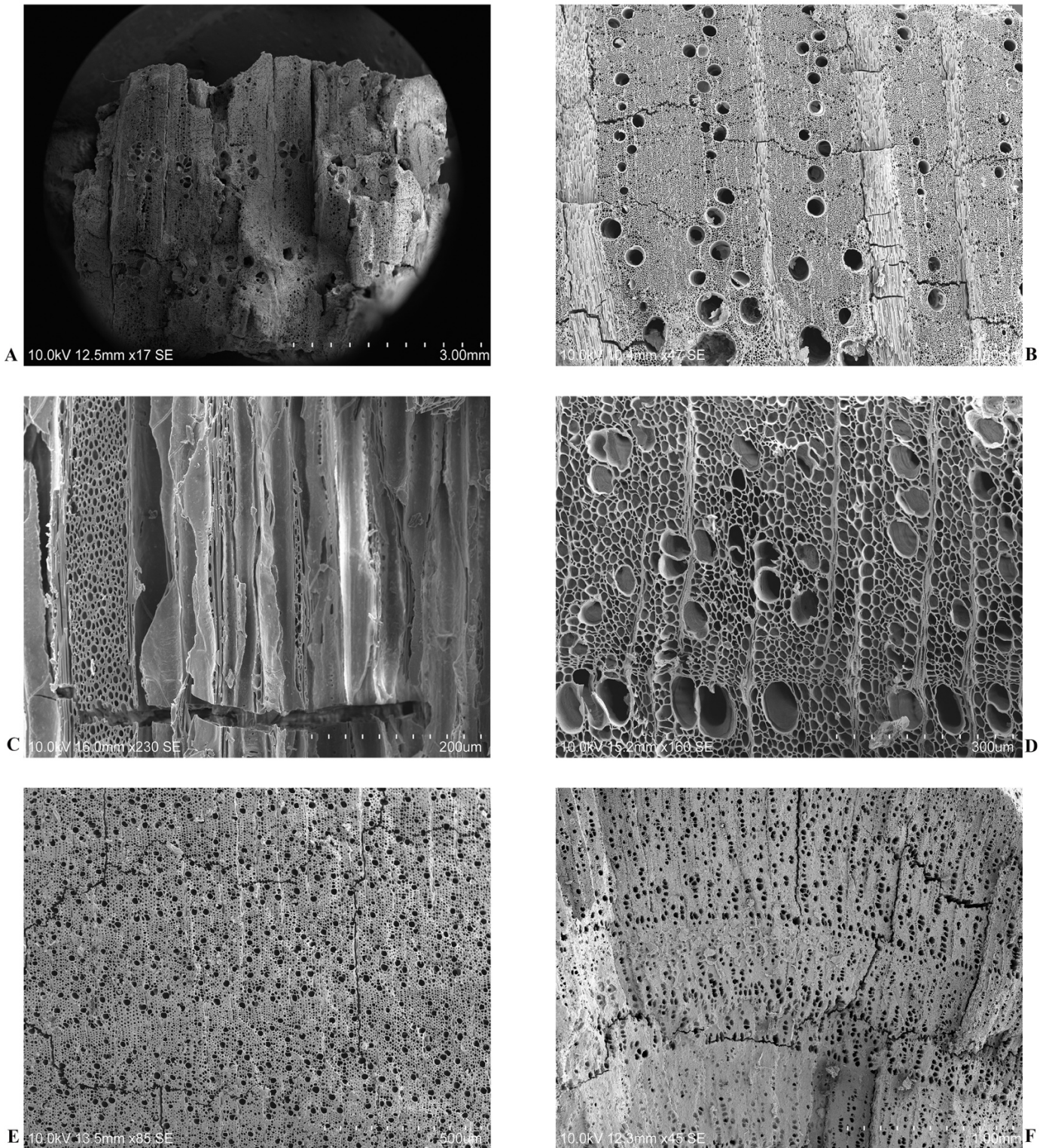


Figure 35: A. Transversal section of *Quercus sect. robur*; B. Transversal section of *Quercus sect. cerris*; C. Tangential section of *Fagus cf. sylvatica*; D. Transversal section of *Prunus cf. avium*; E. Transversal section of *Buxus sempervirens*; F. Transversal section of Rosaceae.

Deciduous and semideciduous oak wood accounts for the highest number of wood charcoal fragments identified; silver fir wood is the most recurrent taxon across the different rooms of the *Domus* (Table 5).

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Table 5: Plant source occurrences in the different divisions of the *Domus dei Dolia* (samples from the dark/black areas).

Plant source										
	<i>Quercus</i> sect. <i>robur</i> and <i>Quercus</i> sect. <i>cerris</i>	<i>Abies</i> cf. <i>alba</i>	<i>Quercus</i> sect. <i>suber</i>	<i>Buxus</i> <i>sempervirens</i>	<i>Fagus</i> cf. <i>sylvatica</i>	Rosaceae sp.	<i>Acer</i> sp.	<i>Prunus</i> cf. <i>avium</i>		
Room	N° of wood of charcoals fragments									Total
A	15	45								60
C	39	50			48					137
E		55		71						126
F						34				34
G	440	65	150				27	26		708
Total	494	215	150	71	48	34	27	26		1065
%	46.38	20.19	14.08	6.67	4.51	3.19	2.54	2.44		100.0
%Ut	23.12	32.87	4.24	11.27	7.01	20	0.76	0.73		100.0

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Wood roof framing elements account for 66.57% of the total of the charcoal fragments analysed. Evergreen oak wood, boxwood, beech wood, maple wood and cherry wood were used for the furniture of the house and the furnishing objects of the house. Furnishing woods account for 30.23% of the total. Charcoal fragments belonging the Rosaceae family were probably part of a tree/s of the garden of the court of the house, accounting for 3.19% of the total (Figure 36).

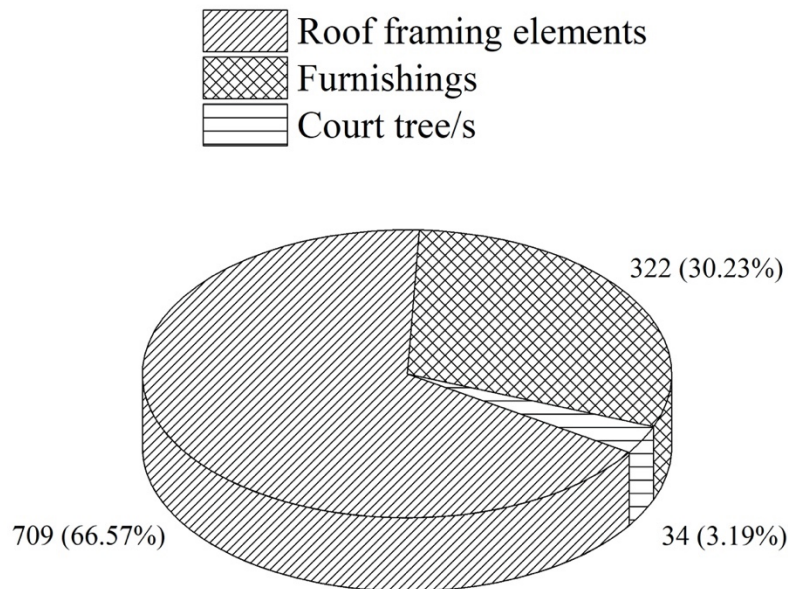


Figure 36: Percentages of the roof framing elements, furnishings and court tree/s identified.

Regarding the tree ring observation, fragments interpreted as wooden roof framing elements (including those identified during the excavation and those from the dark/black areas) it was not possible to identify a curvilinear trend of the tree growth rings. Tyloses were observed in the lumen of the spring wood vessels of 350 samples identified as *Q. sect. robur*. Galleries formed by xylophagous insects were observed in 85 samples of *Q. sect. robur*, seven samples of *Q. sect. cerris*, 174 samples of *A. cf. alba* and 23 samples of *F. cf. sylvatica* (Figure 37). The presence of bark was not detected for any of the samples under study.

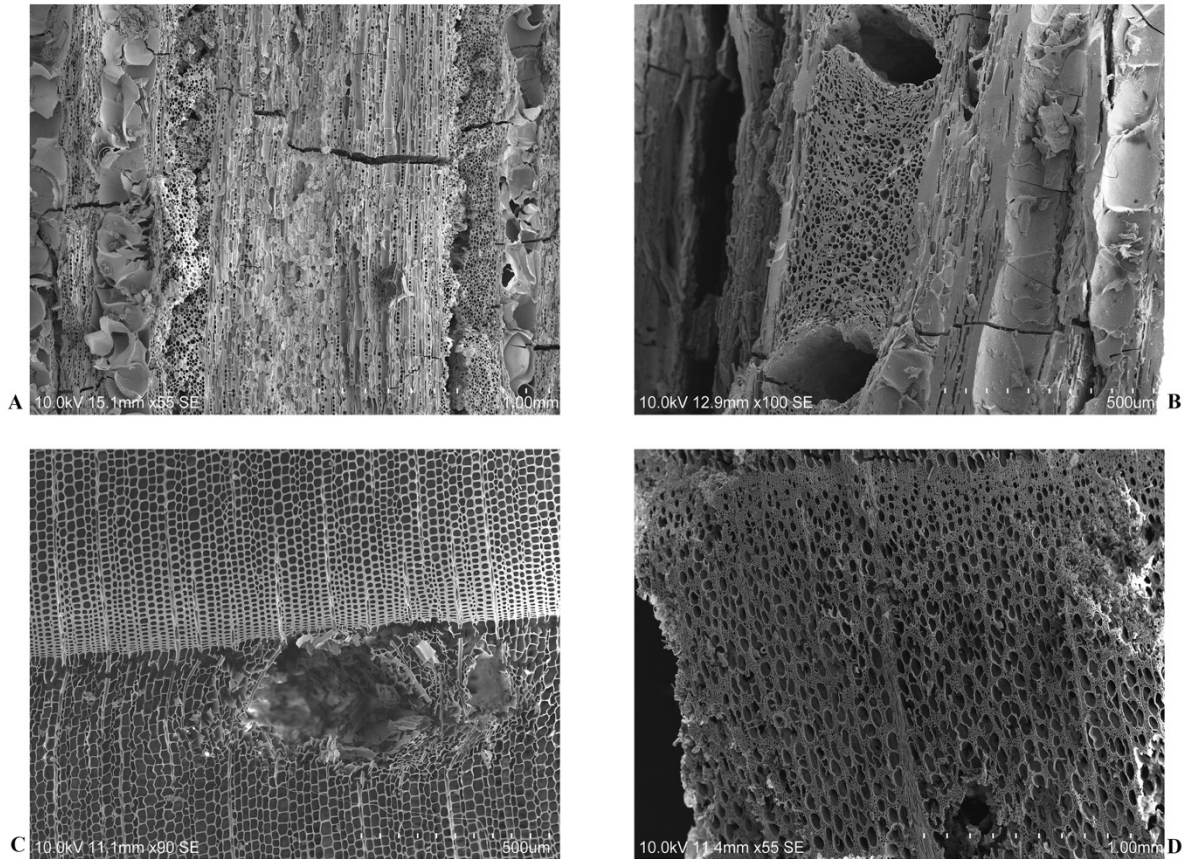


Figure 37: A. Tyloses in the lumen of the spring vessels of a *Quercus sect. robur* fragment (tangential section); B-D. xylophagous' galleries affecting a *Quercus sect. cerris* fragment (tangential section), *Abies cf. alba* fragment (transversal section) and *Fagus cf. sylvatica* fragment (transversal section).

4. 3. 6. Discussion

4. 3. 6. 1. Different wood species for different uses

4. 3. 6. 1. 1. Wooden roof framing elements

Relating to charred woods identified, those employed for the construction of *Domus dei Dolia* are *Q. sect. robur* (deciduous type), *Q. sect. cerris* (semideciduous type) and *A. cf. alba* (silver fir wood) (Table 4). Wood charcoals samples belonging to these species were interpreted as beams or rafters which made up the roof of the *Domus*. As previously reported, some of these wooden elements were identified during the archaeological excavation. In these cases, *taxa* identification has supported the archaeological interpretation. The archaeological data, along with the state of conservation of these wooden remains, did not allow any distinction between beams and/or smallest rafters. In any case, the traces and the position in the archaeological layers of some of these elements made it possible to understand the type of roof covering some parts of the house. Concerning the 4 rooms aligned in the southern part of the house (A, C, E, G) the covering was a single pitch roof. The roof pitch was angled from above to below, where the compluviate rooms were located. In the case of the

wood charcoals found in dark/black areas, the preservation state of the samples and /or their burial conditions did not allow any interpretation during the excavation phase. In these circumstances, the interpretation was possible due a careful study of the excavation data and of the physical and mechanical characteristics of the identified woods. The comparison with other archaeological realities (e.g., where the same species of woods had been also employed for the same purposes), was also useful for their interpretation. Numerous charcoal samples were retrieved in association with many nails bent at right angle (Table 3). The discovery of the nails used to fasten the beams/rafters was a further clue which allowed for the interpretation of these wooden elements.

The deciduous and semideciduous oak wood (i.e., *Q. sect. robur* and *Q. sect. cerris*) was exploited for the manufacturing of the roof beams and or of the rafters of the rooms A, C, D and G. Eleven roof beams/rafters made of this wood were identified during the archaeological excavation as single elements, whilst other wood remains of this species were retrieved in a fragmentary state (sampling of charred woods coming from dark/black areas) (Table 3).

This type of oak wood is well appreciated for its properties, especially for the mechanical strength and the durability of its heartwood as the high content of tannins preserves it from biological attacks (Giachi et al., 2010). Vitruvius and Pliny praised the qualities of this wood (Yegul and Favro, 2018). Its use for the manufacture of structural elements has been well attested since prehistoric times, and oak forests cover large geographical areas included in the Mediterranean basin (Gale and Cluter, 2000). Numerous studies have attested to the use of oak wood for building during the Italian prehistory (Bernabò Brea et al., 1997; Coradeschi, 2012; Coradeschi, 2013; Celant, 1998; Celant, 2002; Corazza et al., 1996; Follieri et al., 1977; Nisbet and Rottoli, 1997), as well as for the historical periods (Caneva, 2005; Signorini et al., 2014). The widespread use of deciduous and semideciduous oak wood for beams, rafters, columns and boards of many Roman buildings, as well as for the construction of naval frames, demonstrate the extent that Romans appreciated this wood for construction purpose (Bernabei et al., 2019; Capretti et al., 2008; Ferrone and Meucci, 1989; Giachi et al., 2003; Moser et al., 2018). Concerning the Etruscan world, the most interesting parallel is the one of the Etruscan Farm of Pian d'Alma (Tuscany) (Mariotti Lippi et al., 2002).

The identification of one roof beam/rafter of turkey wood from the room D (the first atrium of the house with a possible impluvium certifies the existence of a roof cover (at least partial) in the last phase of the life of the *Domus*.

It is likely that fir wood (i.e., *A. cf. alba*) was also used for the roof beams and/or for the rafters of several rooms of *Domus dei Dolia*, namely the rooms A, C, E and G. It was possible to identify only one individual wooden element of this species during the archaeological excavation, while many remains of this species were retrieved only in a fragmentary state (sampling of charred woods coming

from dark/black areas) (Table 3). In this study, samples were identified as likely *Abies alba*, given the wide distribution of this tree in the Italian forests, and in the nearby *Monte Amiata*. Silver fir wood is the tallest native tree of the Italian peninsula, reaching 45 meters in height. The tall, slender stem makes it the ideal wood for the construction of poles, boards and beams. The large stem diameter of this species makes it suitable for the creation of large wooden products. Furthermore, this type of wood can be easily split and saw, making it very suitable for the production of boards (Giordano, 1981). The qualities of the silver fir wood were widely appreciated among the Roman and Greek carpenters (Gagarin, 2010; Ulrich, 2007). Classical authors have described its qualities and prestige with respect to other tree species, especially for its exploitation as building elements (Meiggs, 1982). Theophrastus wrote about its incredible resistance to deformation when tilted, as in the case of roof beams (Ulrich, 2007). Vitruvius describes it as the perfect wood for construction, due to its resistance, lightness, workability and for the length of its stem (Stellacci and Rato, 2019). Livy revealed its essential role for the construction of the Roman naval fleet (Ulrich, 2007).

Numerous fragments of woods and charcoals of fir wood were retrieved in several Italian archaeological sites, largely identified as building elements, to confirm the widespread use of this wood as indicated by ancient sources. Many vertical poles of some Italian prehistoric pile dwelling were made of fir wood (Albore Livadie et al., 2012; Coradeschi, 2012; Coradeschi, 2013; Heussner, 2008; Marzatico, 1997). Several roof beams and rafters of Romans basilicas, temples and private buildings were made of fir wood, as charcoals and woods remains testify (Allevato and Di Pasquale, 2009; Allevato et al., 2012; Caramiello et al., 1992; Di Pasquale and Terzani, 2006; Fioravanti and Gallotta, 2005; Follieri, 1975; Moser et al., 2011; Moser et al., 2013; Moser et al., 2018; Nardi Berti, 2006; Sadori et al., 2007; Ulrich, 2007; Yegul and Favro, 2018). A large amount of wooden remains of this species related to shipbuilding were also retrieved, testifying to the widespread use of this wood for construction frames (Allevato et al., 2010; Begliuomini et al., 2003; Bertacchi et al., 2008; Giachi et al., 2003). Finally, wood remains of fir wood were retrieved from the Etruscan Sanctuary of Pyrgi (Coccolini and Follieri, 1980).

The large amount of charred wood fragments of *A. cf. alba* recovered inside of the *Domus dei Dolia*, and interpreted as roof framing elements, reinforce the hypothesis that the Etruscans were great exploiters of this wood, as were the ancient Romans and Greeks. The final phase of *Domus* occupation coincides with increasing Roman control over the Etruscan cities. Roman choices in wood selection become more culturally influential at this time, which is apparent in the evidence of wood exploitation at this site.

Regarding the compartment E (*Tablinum*), the peculiar use of silver fir wood for the manufacture of roof beams/rafters could be a deliberate choice made by the constructors, perhaps at

the behest of the family of the *Domus dei Dolia*. The use of a more valuable wood species would have made the environment of this important room more beloved; this type of room was normally reserved for the reception of guests.

Considering the number of fragments, the frequency, and the ubiquity we can observe that the roof framing elements are the most abundant wood charcoals identified at the *Domus dei Dolia*. This data should not be surprising considering that woods employed for the construction of the roof of the house were used for all the rooms of the *Domus*, being present in greater quantities even when the *Domus* was still alive. Between the wooden roof framing components, deciduous and semideciduous oak wood has been identified in a greater quantity compared with silver fir wood (46.38 vs 20.19%), in reverse silver fir wood is the most recurrent *taxon* across the different rooms of the *Domus* (32.87%U) (Table 3). The abundance of deciduous oak wood is intelligible since oak wood is considered, in this context, a local plant source. The higher ubiquity value of silver fir wood could be explained by considering the quality and the beauty of this wood.

4.3.6.1.2. *Furnishings and objects*

Among the wood charcoals identified, those employed for the *Domus* furniture, furnishings, and other objects (i.e., tools) are: *Q. sect. suber* (evergreen oak group), *B. sempervirens* (boxwood), *F. cf. sylvatica* (beech wood), *Acer* sp. (maple wood) and *P. cf. avium* (likely cherry wood). Wood charcoal remains of these species were retrieved only in a fragmentary state (sampling of charred woods coming from dark/black areas). The study of the excavation data, of the physical and mechanical characteristics of this wood and the comparison with and other archaeological realities made the interpretation of these wood charcoal samples possible.

Regarding evergreen oak wood remains (i.e., *Q. sect. suber*), they were recovered from Room G of *Domus dei Dolia*. Evergreen oak wood employed for the construction of the *Domus* may belong to one of the following tree species: the cork oak (*Q. suber*), the holm oak (*Q. ilex*) or the Kermes oak (*Q. coccifera*), however it was not possible to distinguish between the various species of this group at the micro-anatomical level.

Evergreen oak wood was well known since the antiquity (Bernabei et al., 2019), especially by Romans who largely used it for the manufacture of objects and furniture. This type of wood has also been used for the covering of sophisticated furniture, given its qualities (Caneva, 2005; Fioravanti and Gallotta, 2005; Signorini et al., 2014). Pliny in his *Naturalis Historia* praises its pleasant nature and reports various uses, in particular its employment for the manufacture of small-sized objects, tools and handles (Pliny, n.d.). He describes its use for the manufacture of the famous Citrus Venus table, owned by the emperor Tiberius (Mols, 1999) and also wrote about its high resistance to friction

and considered this wood very suitable for the construction of wheels (Ulrich, 2007). Cato, recommends the adoption of this wood for the manufacture of agriculture tools handles (Sherwood et al., 2020).

Wood remains of evergreen oak have been retrieved in several archaeological sites both Roman and Etruscan (Mariotti Lippi et al., 2002; Moser et al., 2013). Wooden remains of this species related to shipbuilding were also retrieved (Capretti et al., 2008; Giachi et al., 2003).

Wood charcoal fragment of evergreen oak were retrieved from the centre of Room G of the *Domus dei Dolia* in connection with votive bronzes (Figure 38).

Wood charcoal remains belonging this species were only retrieved in a fragmentary state. However, the characteristics of this wood and their sampling position seems to suggest their possible use for the bronze's storage, as they could have been parts of a cabinet or a box, containing the bronzes.



Figure 38: Votive bronzes surrounded by charcoals at the time of the discovery.

4. 3. 6. 2. Court trees

Concerning the charred wood fragments of Rosaceae recovered from Room F of the *Domus dei Dolia*, it was not possible to distinguish between the various species of this family at the micro-

anatomical level. This family comprises many fruit tree species of the Mediterranean area, for instance apple, pear and plum trees. Considering the archaeological context, wood charcoal remains of Rosaceae are likely to belong to a fruit tree/s. Room F was an open courtyard, and the retrieved wood charcoal remains of fruit tree/s allows us to hypothesize about the existence of a garden inside the *Domus*. Rosaceae wood charcoal fragments were the only taxa present in this room, accounting for 3.19% of the total wood charcoals identified (Table 5, Figure 36). This data seems to confirm the open-space nature of this room, in agreement with the archaeological data.

Regarding the small percentage of woods identified as possible court trees, this data can be explained by the burial conditions of these samples. Wooden remains of trees originally present in a garden—and therefore in an open-air space—have less chance of being preserved over time compared to wooden samples coming from a closed context.

4.3.6.3. *Exploitation of different local and non-local tree species and their possible supply areas*

Everything seems to suggest that the wood choice was based on the characteristics of the woods, in order to employ them for different uses. The comparison of the identified tree species with the local vegetation (present and ancient) gave information regarding the employment of local and/or non-local species for the construction and furnishing of the *Domus dei Dolia*. The study shows primarily the employment of local tree species. The current vegetation of the Tuscan Maremma and its coastal hills, as well as the one where Vetulonia is located, is represented by sclerophyll and broad-leaved forests, there are holm oak woods with scrub and broad-leaved woods (Regione Toscana, 2015). Regarding the species identified in this study, the three types of oak, *Q. sect. robur*, *Q. sect. cerris* and *Q. sect. suber* are considered local species, together with the tree species of *B. sempervirens*, *Acer* sp. and *P. cf. avium* (Regione Toscana, 2015; Selvi, 2010). This is confirmed by the results of pollen and charcoal analysis carried out in nearby areas of Vetulonia (Bellotti et al., 2001; Bellotti et al., 2004; Biserni and Van Geel, 2005; Buonincontri et al., 2011; Drescher Shneider et al., 2007; Giachi et al., 2009; Giorgi et al., 2008; Magny et al., 2007; Mariotti Lippi et al., 2000; Mariotti Lippi et al., 2002; Negri, 1998; Sadori et al., 2010; Vanni re et al., 2008). Many of these studies also revealed the strong impact made by the Etruscans on the Maremma environment, evidenced by the strong exploitation of some local species, including oak wood, as indicated by the decrease in its pollen in the historical record (Drescher Shneider et al., 2007; Mariotti Lippi et al., 2000; Mariotti Lippi et al., 2002; Negri, 1998; Sadori et al., 2010).

Concerning *A. alba* and *F. sylvatica*, indigenous populations of these two species currently naturally grow on the nearby Mount Amiata (approximately 50 kilometers from Vetulonia) (Gabellini

and Angiolini, 2007; Papalini and Miozzo, 2007; Pecoraro, 2007). In this area, native fir populations are found at Pigellato (Piancastagnaio), at Vivo d'Orcia and at the Franciscan Convent of the SS. Trinity of Santa Fiora (Pecoraro, 2007). The presence of silver fir wood on Mount Amiata in the past is proven by palynological data (Bertolani-Marchetti and Jacopi, 1962; Bertolani Marchetti and Soletti, 2007; Clerici, 1903; Tongiorgi, 1938).

The possible supply of both species from Mount Amiata would therefore be supported by ecological data. There is also some historical evidence to support this hypothesis; Pope Pius II (1405-1464) employed the Amiata silver fir trees for the construction of his buildings in Pienza, and in his commentaries he wrote that fir trees of Mount Amiata had also been used for the construction of the ancient Roman buildings (Vanni, 2014; Repetti, 1833). The supply of timber from this mountain appears to be confirmed by the ancient literary sources, which describe how the Romans usually obtained wood from this area of Etruria. The timber travelled on barges along the Ombrone and Albegna rivers and, at the mouths of these rivers, port docks were placed; from there the timber was transported to Rome (Ciampoltrini, 1997; Vanni, 2014). Data from pollen and charcoal analyses (Di Pasquale et al., 2014; Tinner et al., 2013) seems to attest that silver fir would have grown at lower altitudes than the current ones throughout the Italian peninsula in the early Holocene. It would grow at low and medium altitude in forest communities associated with deciduous species, mainly *Quercus cerris*, at least until the Middle Ages. This data was associated with a more extensive presence of this species in Italy. The decline of this species seems to have mainly been caused by climate change and human impact (Di Pasquale et al., 2014). According to this data, some historical literary sources attested to the presence of fir trees at Mount Amiata during the Etruscan and Roman period at lower altitude than they are currently found (Vanni, 2014).

4.3.6.4. Consideration of the “technological data”

Regarding the tree ring observations and the anatomical alterations recorded, it was possible to reconstruct, at least in part, the technological data. In this sense, only a few possible considerations are reported, and they should not be considered conclusive.

Concerning the fragments interpreted as wooden roof framing elements, it was not possible to identify a curvilinear trend in the tree growth rings, and therefore the fragments analysed come from trunks. This observation corroborates the archaeological interpretation of these samples. With regard to the samples identified as deciduous oak wood, tyloses were almost always observed in the lumen of the spring wood vessels. This means that the innermost part of the trunk (the heartwood) was used.

Given the fragmentary nature of many of the wooden roof framing elements identified, we don't know the thickness of the sapwood, and therefore it is not possible to know the thickness of the

trunks from which the wood samples were obtained. The presence of bark was not detected for any of the samples under study, and it was not possible to detect the diameter of the complete stem, nor the season of tree felling.

Concerning the conservation state of the wood, the analysis enabled the identification of galleries formed by xylophagous insects in numerous charred wood fragments of the silver fir wood, and a few in the deciduous and semideciduous oak wood. These channels indicate a partial degradation of some structural elements of the *Domus* before the starting of the fire.

Regarding the samples identified as furniture and furnishing elements, the analysis made it possible to verify their good conditions at the time of the fire, as for most of them no signs of deterioration by wood-decomposing organisms were detected. Some beech wood fragments, in which channels formed by lignivorous insects were detected, are an exception, which indicates their partial degradation before the advent of the fire, but beech wood is not considered to be a very durable wood (Ruffinatto et al., 2019).

4.3.7. Conclusion

The study of *Domus dei Dolia* is the first study concerning construction and furniture woods retrieved from an Etruscan residence of this type. The information obtained from the anthraological analyses provided knowledge about the type of residence and occupants of the *Domus*.

Tree species employed for constructing the roof of the building were deciduous and semi-deciduous oak wood (*Quercus* sect. *robur*, *Quercus* sect. *cerris*) and silver fir wood (*Abies* cf. *alba*). Evergreen oak wood (*Quercus* sect. *suber*), boxwood (*Buxus sempervirens*), beech wood (*Fagus* cf. *sylvatica*), maple wood (*Acer* sp.) and cherry wood (*Prunus* cf. *avium*) were adopted for the furniture and furnishings of the house. Moreover, wood charcoal fragments of fruit trees belonging to the family of Rosaceae were identified, documenting a possible garden inside the court of the house.

Results also allowed us to establish the utilization of local and non-local woods. Furthermore, the observation of the anatomical characteristics provided evidence of the conservation state of the woods before the advent of the fire

4.3.8. Bibliography

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4.4. Archaeometric study of waterlogged wood from the Roman criptopórtico da Rua da Prata (Lisbon, Portugal) - preliminary data

4.4.1. Introduction and research aims

Archaeological wooden artefacts can be easily degraded by fungi, bacteria, or insects. Nevertheless, in certain conditions their activity is limited. In underwater environment, microbial and fungal activity is reduced, but anaerobic bacteria can still promote the depletion of wood carbohydrate leaving an unstable structure mainly constituted of lignin, which can also suffer degradation (Björdal and Nilsson, 2002; Colombini et al., 2007; Tiano, 2002). This type of wood usually looks relatively unaltered, but its physical, chemical, and mechanical properties are deteriorated. The structure of the wood is altered thus, these remains are strongly susceptible to destruction during their excavation and study (Bugani et al., 2009; Pizzo et al., 2010;). For being extremely rare and valuable, waterlogged woods require careful attention, conservation, and study.

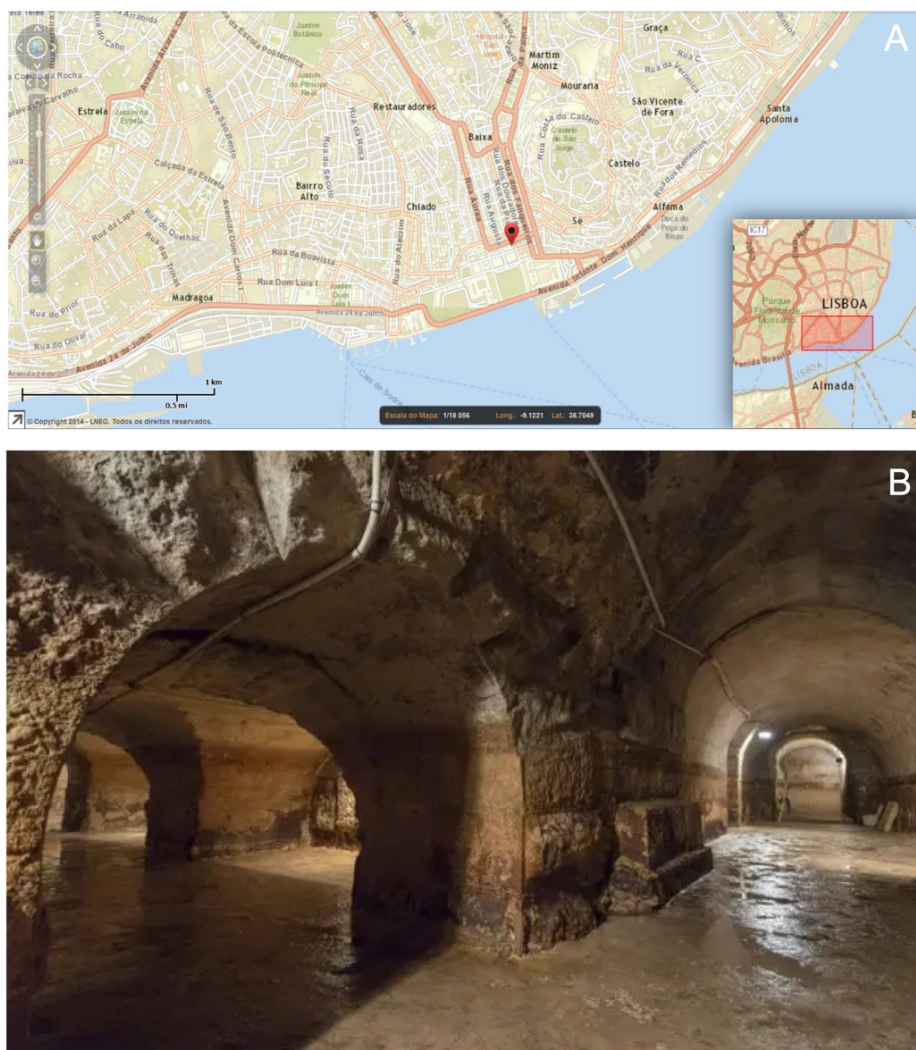


Figure 39: A. Localization of the Roman Criptopórtico da Rua da Prata; B. The Cryptoporticus nowadays.

In the Iberian Peninsula, there are only a few studies concerning Roman archaeological waterlogged wood (Carrión and Rosser, 2010; Costa Vaz et al., 2016; Machado, 2013).

In this chapter, we present results of preliminary archaeobotanical, and Py-GC-MS analysis carried out on waterlogged wood samples, recovered from the Roman criptopórtico da Rua da Prata. This architectural structure is located in the historical centre of the city of Lisbon, below the current Rua da Prata, Rua da Conceição and Rua de São Julião, in the area called “*Baixa Pombalina*” (Figure 39, 40).

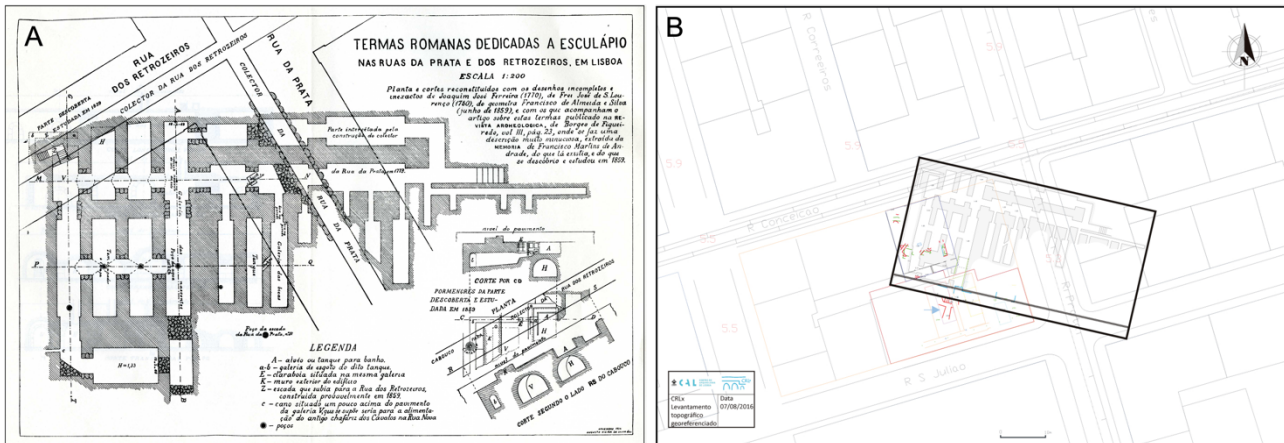


Figure 40: A. Drawing of Augusto Vieira da Silva based on the survey of José Valentim de Freitas (1934); B. The structures raised in the nineteenth century (gray) and those identified between 2015 and 2016 (red and green) with the cloaca indicated by a blue narrow.

This monument was first discovered in 1773, during the reconstruction of the city after the great Lisbon earthquake in 1755. Most likely, the original function of the monument was to support the public building of Baths associated to the old port of the ancient Roman city of *Felicitas Iulia Olisipo* (Nowadays Lisbon) (Figure 40). The vaulted complex was constructed in the middle of the 1st century AD and was partially dismantled in a period between the end of the 3rd century and the beginning of the 5th century AD. Everything seems to indicate that the monument was already flooded during that century.

Since 2015, the Centro de Arqueologia of Lisbon (CAL) has been excavating the site and recovered several archaeobotanical macro-remains, like the waterlogged woods, object of this work.

In order to appropriately preserve the waterlogged wood remains under study, it is necessary to obtain accurate information about them, namely, to identify the wood *taxon* and their state of preservation.

The aim of the analysis was the taxonomic identification of different wooden objects and undefined samples, together with the evaluation of the conservation degree of some of these remains. This case study, embodying an exploratory approach, and future analysis concerning the evaluation of the state of preservation and the identification of all waterlogged wood materials coming from this particular context, are expected.

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4. 4. 2. Archaeological setting

4. 4. 2. 1 The Roman Cloaca

The wood samples under study were recovered from a *cloaca* (Figure 41).

This structure seems to have been added later to the original project, as it was built externally in one of the internal galleries of the cryptoporticus. Its function was not related to an urban sewage in the narrow sense, but rather to a smart solution derived from a specific problem: the entry of the water into the galleries.

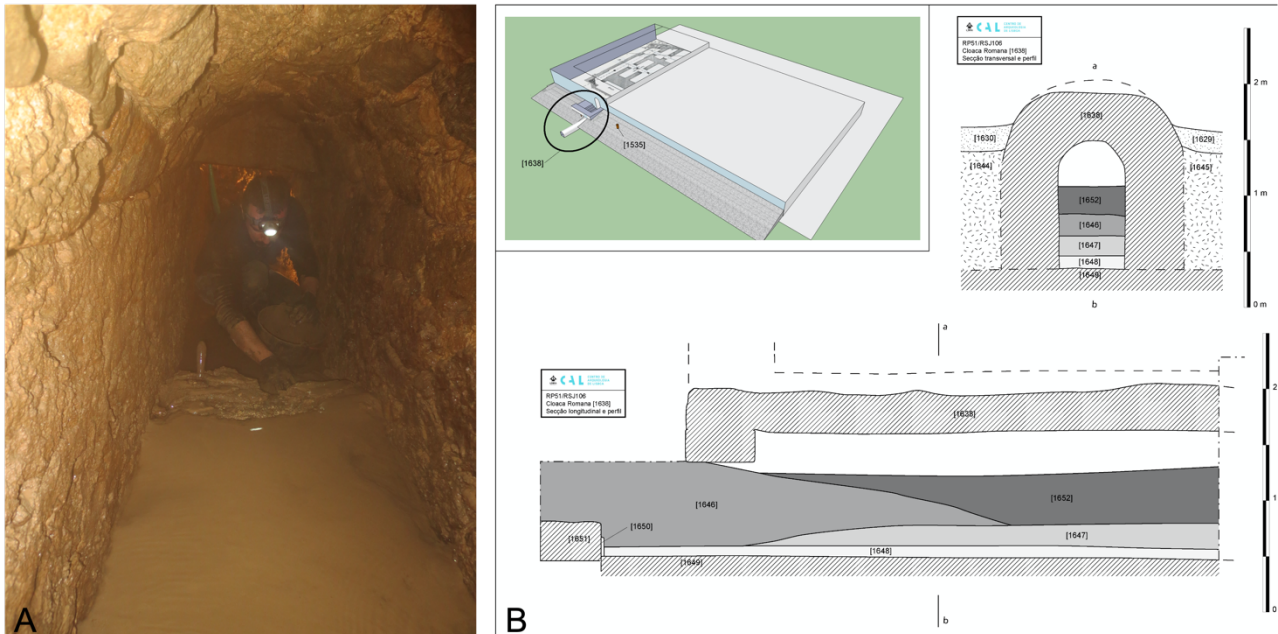


Figure 41: A. Excavation inside the sewer (source CAL); B. Transversal and tangential profile of the sewer with stratigraphy.

Although the cryptoporticus continued to be used, the cessation or the deactivation of this *cloaca* occurred in the 3rd century, when the rise of the average water level prevented an effective drainage, leaving the wood preserved in the muddy sediments that accumulated on the inside.

4. 4. 3. Materials

4. 4. 3. 1. Waterlogged remains under study

Woods were recovered during archaeological campaigns and include structural elements (i.e., parts of window frames and boards), utilitarian objects (i.e., a pulley), and several unidentified wooden fragments. Additionally, a set of four samples interpreted as cork were also analysed (Table 6, Figure 42).



Figure 42: A. Wood artifact interpreted as part of window frames; B. Wood artifact interpreted as a pulley; C. Cork fragments; D. Waterlogged wood interpreted as a foundation board.

4. 4. 4. Methods employed

4. 4. 4. 1. Sampling

Waterlogged remains were stored in their sediments, then transported to the laboratory facilities for analysis, and maintained at a temperature not exceeding 15C°. Regarding the taxonomic identification, and to minimize the damage, samples of about 5 mm were removed for the interpreted materials, and up to 10 mm for the undefined remains.

4. 4. 4. 2. Taxonomic identification

4. 4. 4. 2. 1. Preparing the wood for microscopic examination

Due their bad state of preservation in order to stabilize the waterlogged wood samples under study, a method based on the use of a water-soluble compound was employed. The use of this method allowed the sectioning of the samples. The methodology has been previously described (chapter 3 - 3 .8. 1. Wood thin sections).

4.4.4.2.2. Waterlogged wood identification

The methodology has been previously described (chapter 3 - 3.8.2.1. Waterlogged wood identification - Xylological analysis).

4.4.4.2.3. Cork identification

The methodology has been previously described (chapter 3 - 3.8.2.2. Cork identification).

4.4.4.3. Py-GC/MS analysis

Two wooden artifacts, interpreted as wooden frame fragments from a window/s (named for the study purposes window A and B) were analysed with Py-GC/MS (Table 1). Moreover, a sound wood sample of maritime pine wood (*Pinus pinaster*) was also analysed with the same technique, in order to make a comparison with the archaeological samples and to evaluate their conservation degree. The methodology employed has been previously described (chapter 3 - 3.8.3. Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS analysis)).

4.4.5. Results

4.4.5.1. Waterlogged material identification

All the studied wooden samples were identified as pine wood, probably maritime pine (*Pinus cf. pinaster*), except for the pulley which was made of evergreen oak wood (*Quercus* spp. (evergreen)) (Table 6, Figure. 43-44).

Table 6: Waterlogged remains analysed in the study and their possible interpretation.

N. Sample	SU	Archaeological interpretation	Micro anatomical identification	PY-GC/MS
502	1647	Window frame fragment	<i>Pinus cf. pinaster</i>	
503	1647	Window frame fragment	<i>Pinus cf. pinaster</i>	*
504	1647	Window frame fragment	<i>Pinus cf. pinaster</i>	*
505	1647	Window frame fragment	<i>Pinus cf. pinaster</i>	
509	1647	Board	<i>Pinus cf. pinaster</i>	
510	1647	unindefied wood artifact	<i>Pinus cf. pinaster</i>	
514	1647	Cork (4 fragments)	<i>Quercus suber</i>	
522	1647	Pulley	<i>Quercus</i> spp (evergreen)	
523	1647	Window frame fragment	<i>Pinus cf. pinaster</i>	
530	1650	Foundation board	<i>Pinus cf. pinaster</i>	

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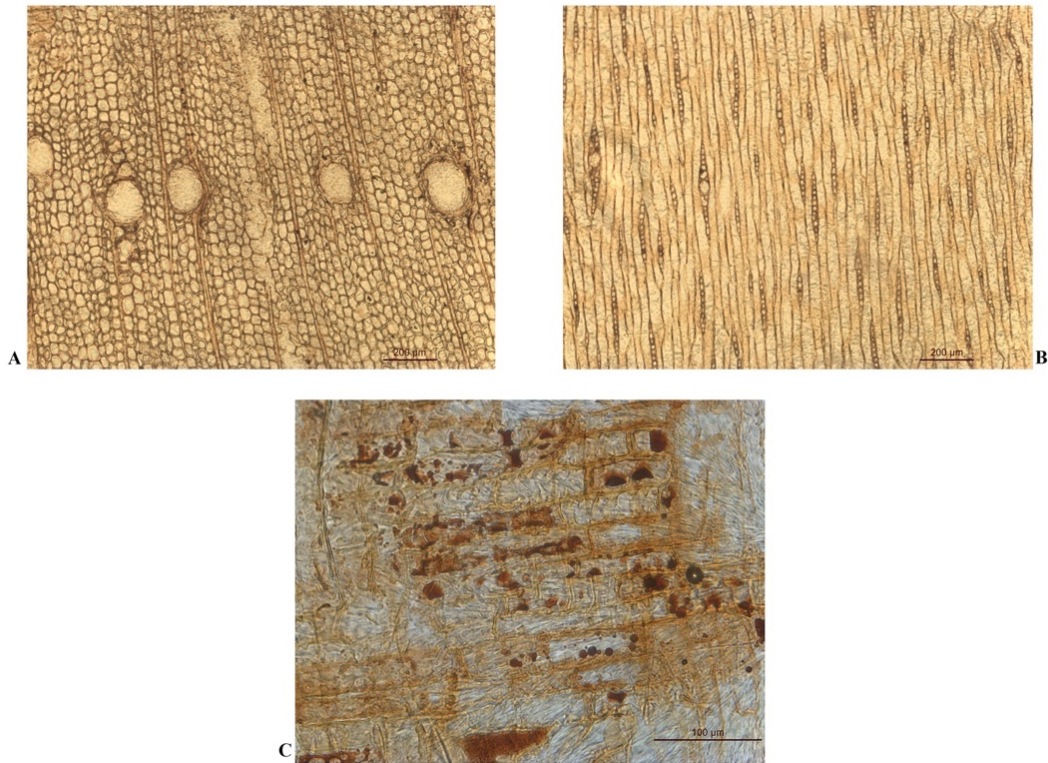


Figure 43: Transversal, tangential and radial sections of a *Pinus cf. pinaster* waterlogged wood sample

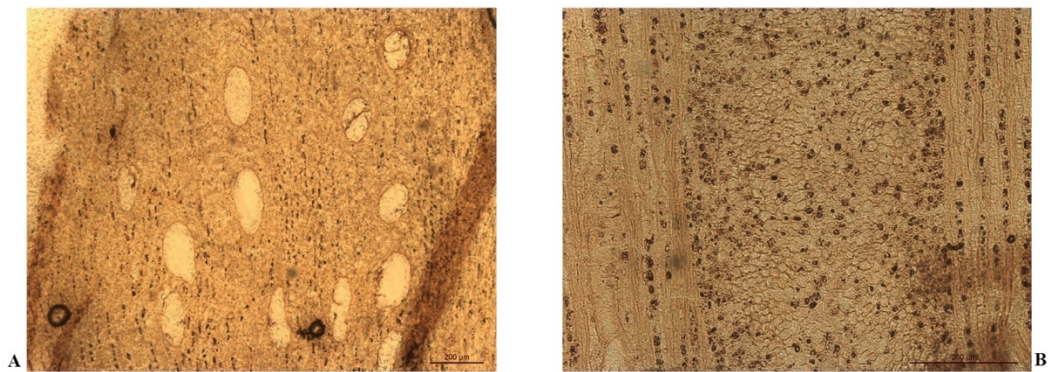


Figure 44: Transversal and tangential and sections of a *Quercus* spp. (evergreen) waterlogged wood sample.

SEM analysis carried out on transversal sections of cork samples allowed to identify the rectangular and honeycomb cells, typical of cork oak (*Quercus suber* cork), confirming the visual interpretation of the samples (Figure 45).

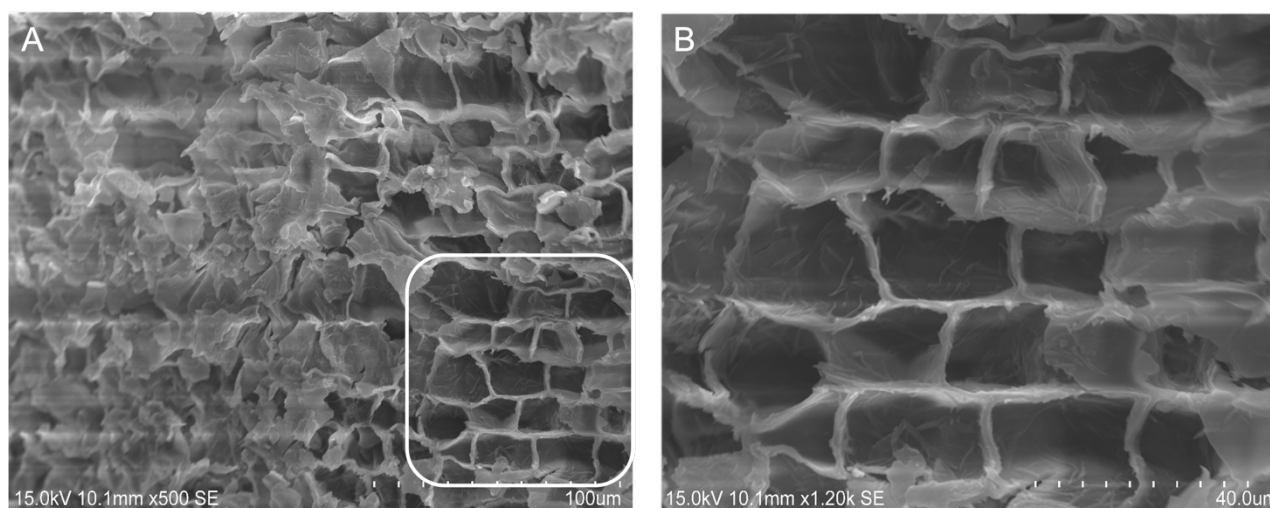


Figure 45: A. Transversal section of an archaeological *Quercus suber* cork sample from the Cryptoporticus of Rua da Prata archaeological site, image captured with a SEM; B. Detail of the rectangular and honeycomb cells of the archaeological *Quercus suber* cork sample.

4. 4. 5. 2. Waterlogged wood conservation degree

Wood is a very complex material from the chemical point of view, and its complexity increases when degradation occurs. Chemical degradation involves alteration of the wood components (cellulose, hemicellulose and lignin), and the assessment of the degree of degradation can be done by Py-GC-MS of wood samples (Tamburini et al., 2015). Pyrolysis enables the analysis of macromolecules by the controlled formation of smaller and simple molecules. The primary reactions of cellulose and hemicellulose pyrolysis involves the scission of the polymeric chains and water elimination, yielding anhydrosugars, which latter rearrange to form smaller molecules such as furans, pyrans and cyclopentenones. During pyrolysis of the lignin the first pyrolytic reaction is the formation of the cinnamic alcohols monomers, followed by cleavage of the alcohol side chain and introduction of different functionalities (Sabato et al., 2015).

Anaerobic bacteria tend to degrade the cellulose and hemicellulose in waterlogged wood, leaving a porous fragile structure poor in polysaccharides and mainly composed of residual lignin, which also tends to be partially oxidized (Colombini et al., 2009). Knowing the type and extent of degradation is important because during conservation treatment of waterlogged wood the consolidants should be chosen according to the degree of degradation of the wood structure, as their main function is to fill the porous left by the leaching of the degradation products of the cellulose and hemicellulose (Colombini et al., 2009).

In this preliminary study, two samples were chosen for pyrolysis, together with a sample of sound wood for comparison purposes. The chromatograms of the archaeological samples analysed (window A and B) presents high amounts of oxidized compounds, derived from coumaryl and

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coniferyl alcohols, and the small amounts of cellulose and hemicellulose derivatives, when compared with the chromatograms of the *Pinus pinaster* sound wood (Figure 46).

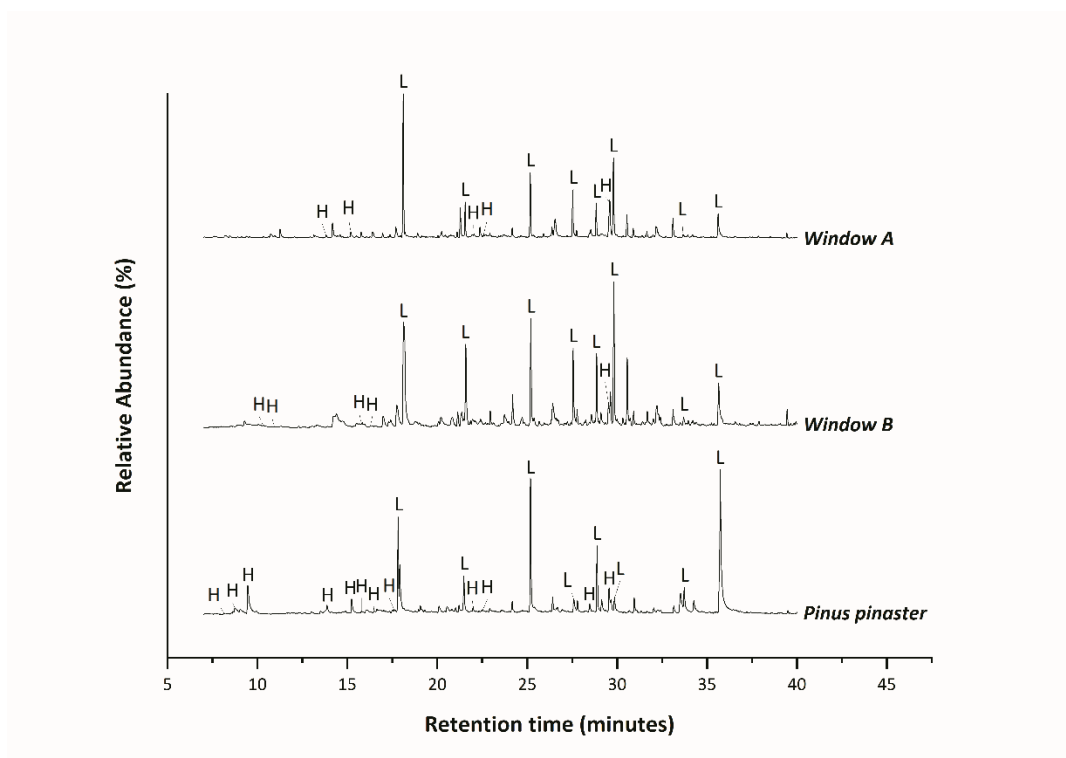


Figure 46: Chromatograms of Window A and B samples and of *Pinus pinaster* sound wood sample.

L: Lignin, H: Holocelulose.

These results provide strong evidence that the structure of the archaeological woods is highly degraded.

In Table 7 there is a list of the compounds identified in the chromatograms, but not all compounds were detected in all the chromatograms. As expected, there is a conspicuous absence of carbohydrate derivatives in the waterlogged woods when compared with the sound wood, and the peaks areas of the oxidized lignin derivatives (e.g., vanillin) is also larger in the archaeological samples (Figure 46 and Table 7).

Table 7 List of compounds identified in the chromatograms. L: Lignin, H: Holocelulose.

Rt (min)	Compound name	Sound wood	Window A	Window B	Source
8.12	2-Methyl-2-cyclopent-1-one	x	x	x	H
8.74	2(5H)-Furanone	x	x	x	H
9.46	1,2-Cyclopentanedione	x	x	x	H
13.789	2H-Pyran-2,6(3H)-dione	x	x	x	H
15.238	2-Hydroxy-3-methyl-2-cyclopent-1-one	x	x	x	H
15.787	2,3-Dimethyl-2-cyclopent-1-one	x			H

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Rt (min)	Compound name	Sound wood	Window A	Window B	Source
16.519	2-Hydroxy-3,5-dimethylcyclopent-2-en-1-one	x			H
17.615	Methyl 2-furoate	x			H
17.823	Guaiacol	x	x	x	L
21.480	Methylguaiacol	x	x	x	L
21.993	1,4:3,6-Dianhydro- α -D-glucopyranose	x			H
22.528	2,3-Anhydro-D-mannosan	x			H
25.178	Vinylguaiacol	x	x	x	L
27.584	Vanilina	x	x	x	L
28.468	1,6-Anhydro- β -D-talopyranose	x			H
28.879	Isoeugenol	x	x	x	L
29.545	1,6-Anhydro- β -D-glucopyranose	x			H
29.815	Acetovanillone	x	x	x	L
33.725	Homovanillic acid	x	x	x	L
33.733	Coniferol	x			L
35.650	Coniferol aldehyde		x	x	L

The chromatographic areas of the compounds listed in Table 7 were used to evaluate the wood conservation status based in the following parameters:

- H/L coefficient: the ratio between the sum of the chromatographic peak areas of the carbohydrate pyrolysis products and the lignin pyrolysis products; this coefficient is a parameter that has been demonstrated to be correlated with the conservation state of wood (Colombini et al., 2009; Lucejko et al., 2012).
- O/L coefficient: the sum of the the chromatographic peak areas of the main oxidation lignin products (vanillin, acetovanillone, vanillic acid and coniferyl-aldehyde) divided by the sum of the areas of all the lignin pyrolysis products; this parameter is an indicator of the degree of oxidation of lignin (Tamburini et al., 2014).

Results presented in Figure 47 show that the degradation is not uniform in the two samples analysed.

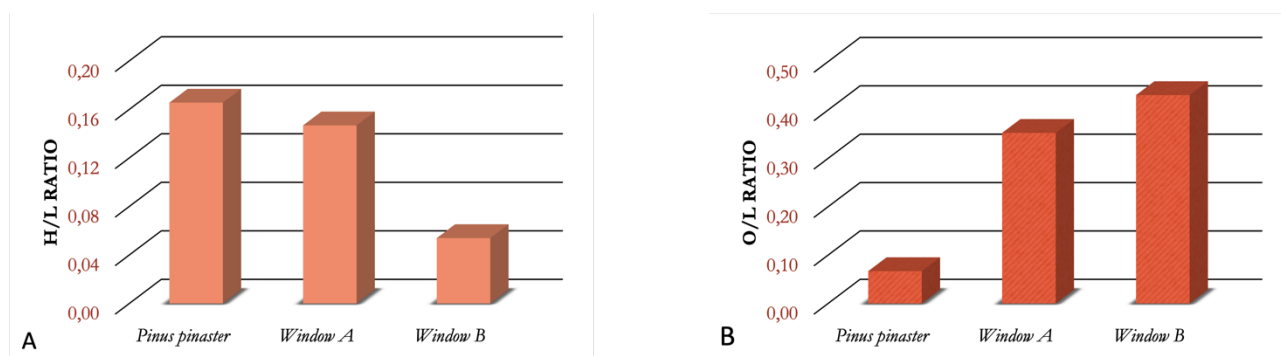


Figure 47: A. H/L ratio of wooden samples analysed; B. O/L ratio of wooden sample analysed.

The window, sample B, is the most degraded wood sample, presenting lower amounts of residual carbohydrates and higher amounts of oxidized lignin products in the chromatograms.

4. 4. 6. Discussion

Pine wood, probably the Maritime species (*Pinus cf. pinaster*) has been used for the manufacturing of all the wooden fragments identified as window frames, as well as the construction boards and all the identified samples. On the contrary, evergreen oak wood (*Quercus* spp. evergreen) was employed only for the manufacturing of the pulley.

There are parallels for the use of both types of wood in the Roman world (Capretti et al., 2008; Costa Vaz et al., 2016). Pine wood was used by ancient communities since the prehistory and is well known as construction material due to its resistance to shrinking and swelling (Nardi Berti, 2006; Marston, 2009). The qualities of evergreen oak wood were well known by classical historians, in particular its high resistance to friction (Ulrich, 2007). The use of this type of wood for the manufacturing of objects, small tools, agricultural implements, and furniture was well known in the Roman world (Bernabei et al., 2019; Mols, 1999; Pliny, n.d.; Sherwood et al., 2020; Ulrich, 2007). Moreover, this type of wood has been also used for the upholstery of sophisticated furniture (Caneva, 2005; Fioravanti and Gallotta, 2005; Signorini et al., 2014).

SEM analysis confirmed the identification of the *Quercus suber* cork samples. The presence of this type of organic material was already attested in other Roman archaeological contexts in Portugal (Costa Vaz et al., 2016). Cork fragments could be interpreted as parts of stoppers for amphorae or other containers, for example, vessels or even glass bottles.

Regarding the study done with Py-GC/MS, this type of analysis promotes the depolymerisation of carbohydrates and lignin polymers, yielding information about the conservation status of the waterlogged wood artefacts (Colombini et al., 2007). Analytical pyrolysis analysis of waterlogged wood remains from the Roman Cryptoporticus of Lisbon confirmed the validity of this technique for the study of waterlogged wood decay, even when samples are in a bad state of

preservation. Py-GC/MS analysis of samples named as window A and B showed the high degradation of these samples, evident by the detection of a large amount of oxidized lignin compounds, and by the lower amounts of cellulose and hemicellulose based-compounds. Results also showed a not-uniform degradation of the waterlogged wood samples, suggesting that different degradations mechanisms/agents were responsible for the damages in the wood structure. Future analysis of other wooden artefacts from this context will complete and clarify the current analytical picture.

4.4.7. Conclusion

Preliminary results from the study of waterlogged remains from the Roman Cryptoporticus of Lisbon stress the large use of pine wood for the manufacturing of the wooden artifacts. Evergreen oak wood was employed for the manufacture of the pulley. Moreover, SEM analysis confirmed the identification of few fragments of cork. Results obtained from the Py-GC/MS analysis of window A and Window B samples underline how the degradation of the samples is evident, by the detection of a large amount of oxidized lignin compounds, and by the lower amounts of cellulose and hemicellulose based-compounds. Moreover, they also displayed how the samples degradation is not uniform. Future analyses with both approaches regarding other waterlogged artifacts coming from the cryptoporticus are expected, to add more information regarding the waterlogged remains from this context and Roman contexts in Portugal in general, as these studies are very rare.

4.4.8. Bibliography

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4.5. Case study 4: A glimpse into the viticulture of the Roman Lusitania: morphometric analysis of charred grape seeds from Torre dos Namorados archaeological site (Fundão, Portugal)

4.5.1. Introduction and research aims

The grape family (Vitaceae Juss.) belongs to the Vitales Juss. ex Bercht. & J.Presl order, it comprises 17 genera and about 950 species (most of them are tendril-bearing vines) (Jun Wen et al., 2018), among which is the grapevine (*Vitis vinifera* L.). Grapevine is the most planted crop in the world, with approximately height million vineyards (<http://faostat.fao.org/>). The economic, social, and cultural value of the grapevine has been known since ancient times, and it is related to the harvesting and the consumption of its fruits, especially when used for wine production. The majority cultivars comes from wild populations of *Vitis vinifera* subsp. *sylvestris* (C.C.Gmel.) Hegi (Cunha et al., 2007; Sefc et al., 2003; This et al., 2007). The wild grapevine is an heliophilous liana which since the Mid-Holocene is domesticated and cultivated in the South-West of Asia, the North-East of Africa and Southern Europe. It grows in riparian and lowland deciduous and semi-deciduous forests (Levadoux, 1956; Mariotti Lippi et al., 2020; Miller, 2008; Orrù et al., 2012a; Orrù et al., 2012b; Uccesu et al., 2015).

According to the most accepted theory, grape domestication took place in the Caucasus and in the Middle Eastern regions, between the 7th and the 4th millennium BC (Negrul A.M., 1946)(Olmo, 1996). However, genetic analysis suggests the existence of secondary centres of domestication (Aradhya et al., 2003; Arroyo-García et al., 2006; Grassi et al., 2003; Imazio et al., 2006; Sabato et al., 2015). Many questions are still under debate, especially concerning the times and the frequency of the grapevine domestication process. In this regard, the study of archaeological grape seeds, both wild and domesticated, sheds light on these issues and can be particularly helpful in order to get information on the history of the viticulture. A large number of grape pips have been retrieved in different Neolithic archaeological sites in the Middle East. The most ancient archaeobotanical evidence of grapevine domestication comes from the Jordan Valley and dates back to the Chalcolithic (Barnard et al., 2011; McGovern, 2003; Zohary et al., 2012). Regarding the Eastern Mediterranean, archaeobotanical evidence of early viticulture have been retrieved in Greece, Cyprus and Egypt from the Ancient Bronze Age (McGovern et al., 1997; Kroll, 1991; Pagnoux et al., 2014; Valamoti et al., 2007). Archaeobotanical remains of grapevine domestication have also been recovered in the Western Mediterranean area. In Sardinia, the study of grape seeds from a Nuragic settlement (Middle Bronze Age) provides the first data of primitive cultivated *V. vinifera* in this area, representing one of the oldest disclosures of the western Mediterranean (Uccesu et al., 2015). In the Italian peninsula, the

increase of grape seeds in the archaeological levels of the Iron Age could be an indicator of the beginning of the grapevine domestication (Marvelli et al., 2013). In Spain, archaeobotanical evidence related to winemaking have been found in different Phoenician archaeological sites from the 7th century BC (Jane Evans, 2018). In Southern France, archaeobotanical remains link the beginning of the grapevine cultivation with the foundation of the Greek city of Massalia, 600 BC (Brun, 2011). Moreover, the study of grape seeds from different archaeological sites prove that the grapevine domestication process was still in progress during the Roman period (Bouby et al., 2013).

In Portugal, the discovery of a few grape seeds from different prehistorical sites (Almeida, 1996; Rego P.R, 1993; Godinho Pamplona Reis, 2018; Rego P.R, 1993; Tereso, 2012) confirm the collection and the consumption of fruits from the wild plants by prehistorical communities. Indeed, the wild grapevine is a native species of Portugal (Flora on, Portuguese Botanical Society. <http://www.flora-on.pt/#wVitis+vinifera+subsp.+sylvestris>, consulted on 24/09/2021), with pollen and anthracological analysis confirming its presence in this area since the Prehistory (Leeuwaarden and Janseen, 1985; Sanches and Figueiral, 1998). Regarding the Iron Age, a small number of grape seeds have been recovered in various archaeological sites located in the northwest (Seabra, 2015; Tereso J.P., Rego, P.R., Almeida da Silva, 2011; Tereso, 2012; Tereso et al., 2013b; Tereso and Cruz, 2014), but the existing bibliography suggests little evidence to support the presence of *V. vinifera* in the rest of the territory during this period (Barros, 2001; Queiroz et al, 2006). Finally, during Roman times the presence of grape seeds in the archaeological layers becomes more frequent and quantitatively more considerable, both in rural and urban sites (Almeida, 1996; Almeida and Veiga Ferreira, 1967; Peña-Chocarro et al., 2019; Pinto da Silva, 1988; Queiroga, 1992; Tereso and Cruz, 2014; Tereso et al., 2013a). This data suggests the beginning of the viticulture along this period. This hypothesis seems to be confirmed also from the ancient literature (Almeida, 1996). In some cases, archaeological grape seeds have been retrieved in correlation with remains of exotic wine amphorae, dated back between the second half of the 1st century BC and the first half of the 1st century AD (Morais, 1997). This evidence points out the relevance of wine imports in the early stages of the Roman occupation. This would be confirmed by the decrease of the same type of amphorae from the 2nd century AD (Morais, 1997). The discovery of several archaeological wine press structures (*lagares*) related to the wine production dated from the 2nd century AD in the area, known as the province of Lusitania Romana (which nowadays broadly correspond to the southern and central Portuguese territory plus the Spanish Estremadura and a small part of the province of Salamanca) seems to be the first expression of a local viticulture, definitively intensive and widespread in Portugal. However, in many cases the exact chronology of these *lagares* is difficult to assess. Actually, similar wineries have been utilized up to the 20th century (Almeida, 1996; Pereira, 2017).

The most recent studies have reported chronologies, related to the 1th century (Pereira and Silvino, 2018).

In addition to the scarcity of archaeobotanical and archaeological evidence, in Portugal there is also a lack of studies concerning the discrimination of big assemblages of archaeological grape seeds coming from dated contexts. Differences in size and in shape of seeds allowed the discrimination between wild and cultivated grapes. Some of these morphometric features have been considered useful for the discrimination of the two subspecies (Di Vora and Castelletti, 1995)(Mangafa and Kotsakis, 1996) (Stummer, 1991). However, according to some authors the deformation in the shape and size induced by the carbonization would produce an overlap between wild and domesticated seed shapes, making this methodology ineffective in discriminating charred archaeological seeds. Some researchers argued that the main problem arises from the use of a small reference collection (Bouby et al., 2018). In recent years, morphometric studies based on wide reference collections have shown that it is possible to distinguish archaeological charred grape seeds when assemblages are numerous and well preserved (Bouby et al., 2018; Ucchesu et al., 2016). Additionally, the application of the image analysis technologies on archaeobotanical materials, such as seeds and fruits, has proved to be a reproducible and non-destructive method to characterise and distinguish wild forms from cultivated ones (Bouby et al., 2013; Pagnoux et al., 2014; Sabato et al., 2015; Terral et al., 2010; Ucchesu et al., 2017). At the same time, several studies have attested the importance of Linear Discriminant Analysis (hereafter LDA) applied to the digital image techniques in order to characterise *V. vinifera* seeds, both modern and archaeological (Orrù et al., 2012a; Orrù et al., 2012b; Orrù et al., 2013; Ucchesu et al., 2015; Ucchesu et al., 2016).

The discovery of thousands of charred grape seeds from the Roman winery of Torres dos Namorados (Fundão, Portugal) is unique, considering the rarity of these evidence in Portugal. The morphometric study of the grape seeds presented here is a first step towards a broader understanding of the viticulture during Roman times in Portugal.

This aims of this study are:

- 1 To characterise the archaeological grape seeds of Torre dos Namorados, distinguishing whether they are cultivated or wild.
- 2 To investigate similarities with modern Portuguese native grapevine cultivars.
- 3 To give a contribution to the understanding of the viticulture during Roman time.

4. 5. 2. Archaeological setting

4. 5. 2. 1. Torre dos Namorados archaeological site and its surroundings

Torre dos Namorados (Fundão, Castelo Branco district) is an archaeological site classified as a *vicus*, located in the southern part of Beiras region (centre of Portugal) (Figure 48).

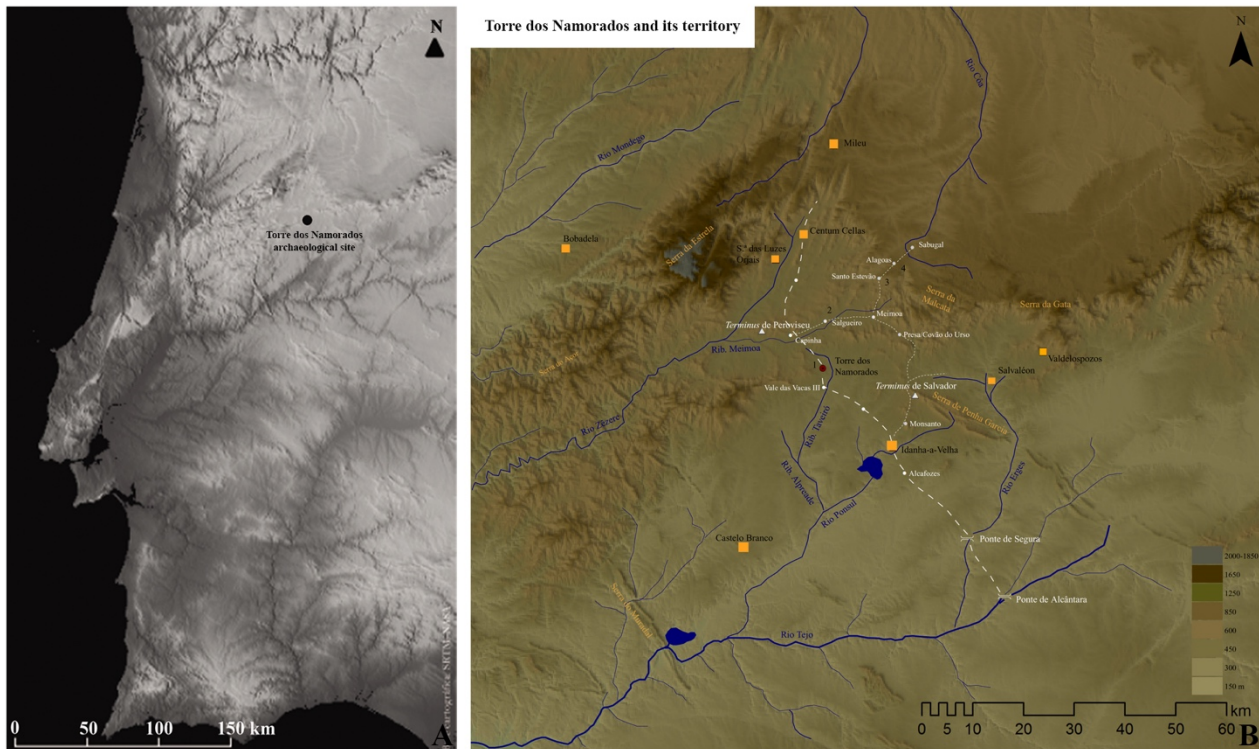


Figure 48: A. Torre dos Namorados archaeological site; B. Torre dos Namorados and its territory.

From a geographic point of view, the archaeological site is at southeast of the Serra Estrela and southwest of the Serra of Malcata (Figure 48). It sits in an interface area opening into the plateau of Beira Baixa, the Castelo Branco Platform, at the bottom of the Cruzinhas/Casinhas hill. On top of that hill there is a proto historical walled settlement (Covilhã Velha), being close to the layout of the main Roman road that links *Augusta Emerita* (Mérida) to *Bracara Augusta* (Braga).

Superficial and excavation surveys were carried out between 2003 and 2007 in the archaeological area of Torre dos Namorados. The investigation revealed it to be a unique case in the Roman territory of this region, not only because of the high and wide dispersion of archaeological materials at the surface (approximately 53 ha), but also because of its functionality. The latter is revealed by the presence of archaeological structures and mill elements for wine production, spread in four clustering areas of the Roman site – Tapada da Torre, Fonte Velha, Vale Cortiço and Coitos (Figure 49).

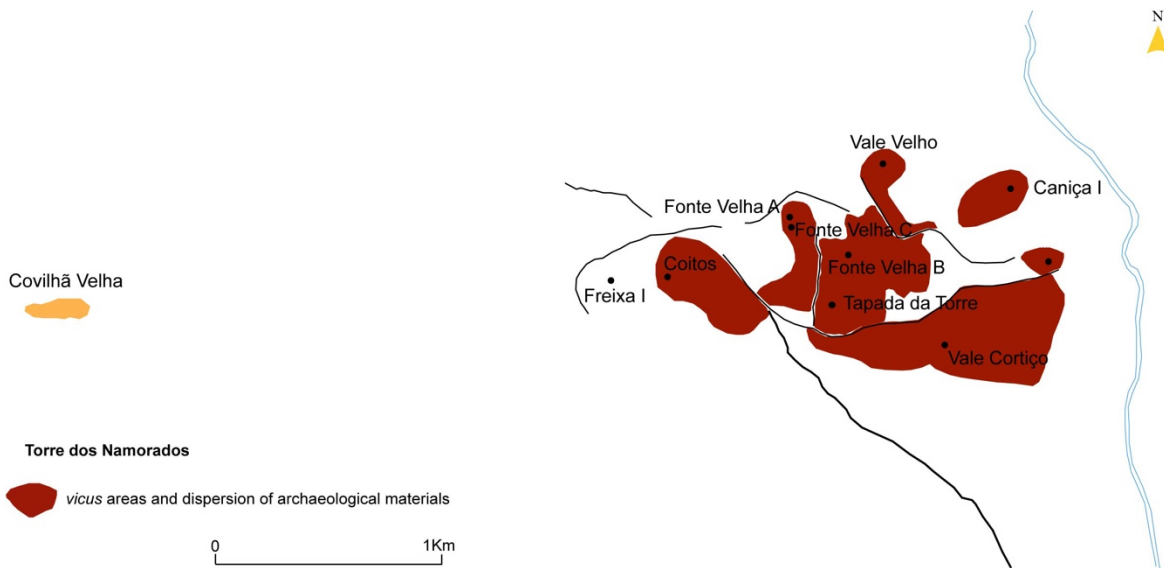


Figure 49: Torre dos Namorados vicus and dispersion of archaeological remains.

4. 5. 2. 1. 1. *The Lacus musti: archeological grape seeds context*

The 2006-2007 archaeological excavations at the Coitos cluster revealed a structure interpreted as a *cella vinaria* (or wine press) and another one related to its use. Amongst them, a *lacus musti* (or settling vat) stands out. It is a box built with *tegulae* and bricks which contained macro-remains of charred grape seeds and grape skins (Figure 50A-C). This is an exceptional and rare archaeological finding in Portugal.



Figure 50: A. The *lacus musti* of Torre dos Namorados; B-C. The discovered archaeological charred grape seeds.

The uncovered rooms, called Compartment 1 and Compartment 2, revealed sealed layers preserved by the collapse of the roof, which was made of *tegulae* and *imbrices*. The Compartment 1 is the press room (*area*), a square or rectangular room, about 7 m wide. It is paved with quartzite pebbles, covered in *opus signinum* which is particularly preserved along the walls. The Compartment

2 is the counterweight room, showed two large resting cylindrical counterweights and a circular structure around counterweight II (Figure 51 A-C).

The material assemblage recovered in the excavation of Compartment 2 comprises ceramic building materials (*tegulae*, *imbrices* and bricks), storage pottery (*dolia*) and common ware. Also noteworthy is the recovery of a Late Hispanic *sigillata* fragment, a bowl decorated with *guilloché* (Drag. 37 type), whose production chronology is established between 350 to 500 AD. The fragment was found in the abandonment level of the circular structure, around counterweight II, thus helping to date it.

A *lacus musti* was attached to the wall, bordering the room. It is a box made of *tegulae* and brick fragments, lined with clay mortar, where plant macro remains - grape seeds and skins - were preserved (Figure 51B).

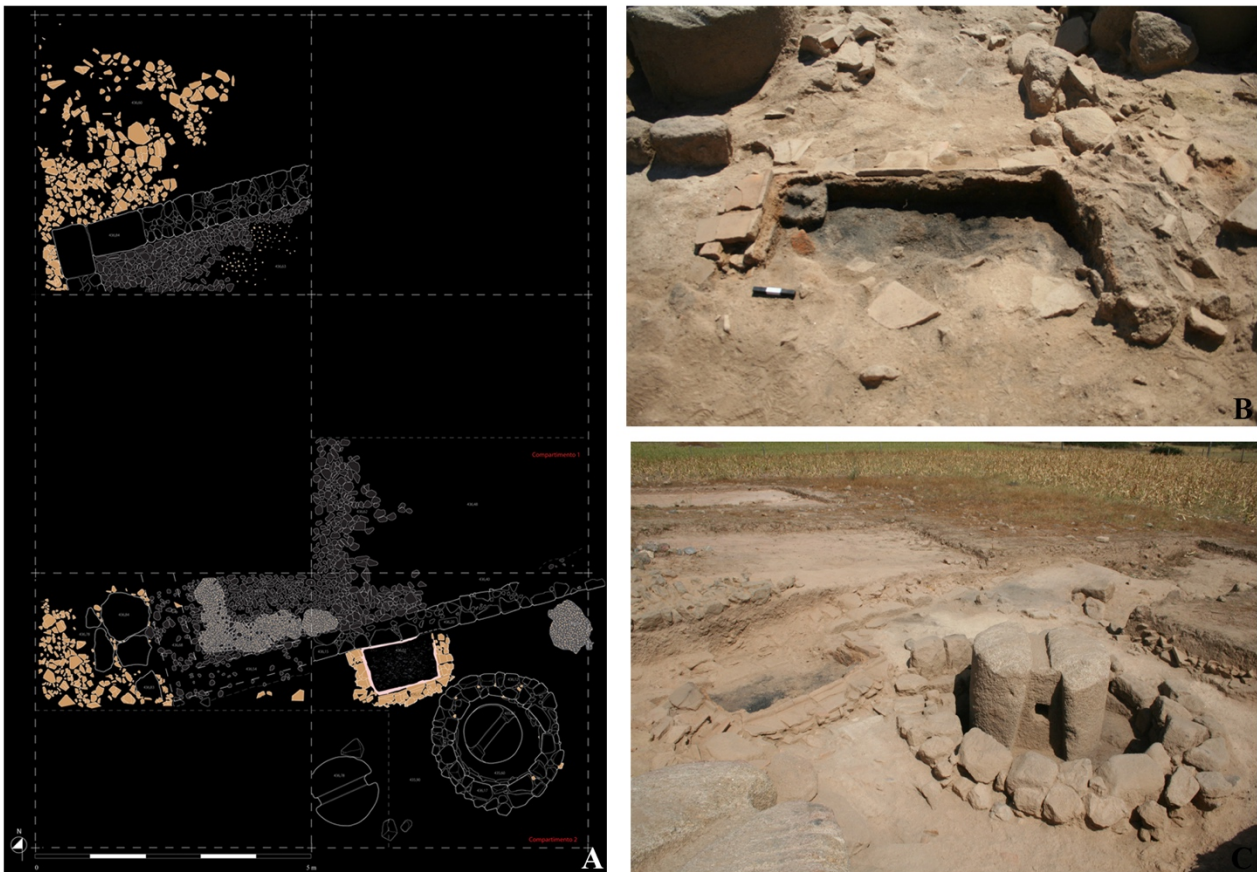


Figure 51: A. The map of the compartment 2 of Torre dos Namorados; B-C. The *lacus musti* and a counterweight.

The Coitos wine press would work with a screw pressing system. Both counterweights (having the upper grooves extending laterally in swallow tail) show a central hole, where wooden fittings (*stipites*) and iron hooks would be fixed to support a wooden beam (*prelum*). In order to operate the press, large beams would be used. Those would also be fitted in the holes found in the north wall of Compartment 1 or anchored in an *arbol* that would cross the entire press room (*area*). The structures were complemented by the *lacus musti*, where the pressed wine flowed.

4. 5. 3. Materials

4. 5. 3. 1. Archaeological grape seeds samples

Charred grape seeds were isolated from sediments collected from the *lacus musti* using a sieves column of different mesh size (0.5 mm, 2 mm, 4 mm). A total of 1122 archaeological grape seeds were analysed in this study. The grape seeds have been radiocarbon dated and their chronology, corresponding to the last use of the vat, coincides with the dating proposed by the pottery's circulation. The dating of SUERC - 81645 (GU48796) grape seed sample is 95,4% reliable between the 2nd third of the 4th century AD (339-435 CAL AD [76.4%]; 451-471 CAL AD [2.9%]) and the beginning of the 2nd third of the 6th century AD (487-534 CAL AD [16.1%]). Considering the percentages value and the archaeological context, the chronology between the end of the 4th and the beginning of the 5th century AD is considered the reference value for this work by authors (Figure 52).

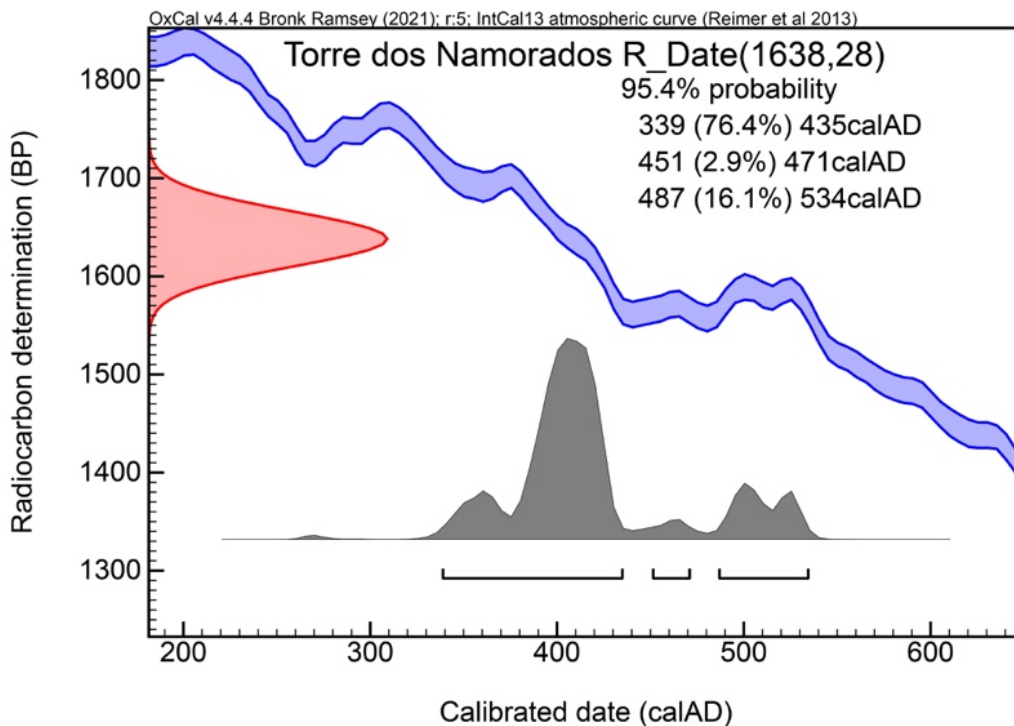


Figure 52: The radiocarbon dating of a grape seed sample -SUERC - 81645 (GU48796) - from Torre dos Namorados (the calibration was performed by the SUERC Radiocarbon Laboratory of Scotland).

4. 5. 3. 2. Cultivated and wild grape seeds reference samples

Grape seeds were collected from 35 native cultivars of *V. vinifera* subsp. *vinifera*, coming from the National Ampelographic Collection of the Instituto Nacional de Investigação Agrária e Veterinária (INIAV) (Dois Portos, Portugal) (Table 8).

4. Case studies

In addition, seeds accessions of nine plants of *V. vinifera* subsp. *sylvestris* populations present in Portugal (Table 9) were harvested in the field. See the description of the modern grape seed database at the chapter 3- 3. 9. 2. Grape seed database collection.

Table 8: Ancient grape cultivars of *Vitis vinifera* L. *vinifera* provided by the Instituto Nacional de Investigação Agrária e Veterinária (INIAV, Portugal), used in the study.

Code	Origin	Cultivar name	Nº seeds	Code	Origin	Cultivar name	Nº seeds
White varieties							
50711	Portugal	Alicante Branco	100	52014	Portugal	Rabigato	100
51117	Portugal	Bastardo Branca	100	52112	Portugal	Gouveio	100
51211	Portugal	Uva Cavaco	100	52307	Portugal	Donzelinho Branco	100
51316	Portugal	Sarigo	100	52309	Portugal	Boal Ratinho	100
51410	Portugal	Douradinha	100	52310	Portugal	Avesso	100
51411	Portugal	Dorinto	100	52314	Portugal	Fonte Cal	100
51412	Portugal	Arinto do Interior	100	52410	Portugal	Cerceal Branco	100
51514	Portugal	Folha de Figueira	100	52507	Portugal	Batoca	100
51516	Spain	Samarrinho	100	52513	Portugal	Diagalves	100
51910	Portugal	Tamarez	100	52709	Portugal	Folgasão	100
Black varieties							
41209	Portugal	Alvarelhão Ceitão	100	52102	Portugal	Tinta Carvalha	100
41302	Portugal	Barreto	100	52206	Portugal	Touriga Nacional	100
42303	Portugal	Castelõa	100	52403	Portugal	Camarate	100
50607	Portugal	Tinta Gorda	100	52603	Spain	Aragonez	100
50705	Portugal	Touriga Brasileira	100	53006	Portugal	Trincadeira Preta	100
51002	Portugal	Castelã	100	53205	Portugal	Malvasia Preta	100
52002	Portugal	Marufo	100	53307	Portugal	Tinto Cão	100
52004	Portugal	Cornifesto	100				

4. Case studies

Table 9: *Vitis vinifera* L. *sylvestris* sampling in the field.

Population	N° Plants sampled	N° seeds	Elevation	PopRisk	Type of Substrates	Habitat	Location
Montemor (Alentejo interior)	2	35	306	3	Soil originated from sandstones	<i>Viti viniferae-Salicetum atrocinnereae</i> Rivas-Martínez & Costa (Rivas-Martínez et al., 1980)	Riparian forest
Castelo Branco (Beira baixa)	3	194	119	7	Soil originated from sandstones	<i>Salicetum salviifoliae</i> Oberdorfer & Tüxen (Tüxen & Oberdorfer, 1958)	Riparian forest
Alcácer do Sal (Alentejo litoral)	2	155	49	7	Soil originated from sandstones	<i>Viti viniferae-Salicetum atrocinnereae</i> Rivas-Martínez & Costa (Rivas-Martínez et al. 1980).	Riparian forest
Portél (Alentejo interior)	2	215	197	3	Schist lithosols	<i>Viti viniferae-Salicetum atrocinnereae</i> Rivas-Martínez & Costa (Rivas-Martínez et al., 1980)	Riparian forest
Total	9						

4.5.4. Methods employed

4.5.4.1. Carbonization of grape seeds reference samples

To achieve correct comparison, modern seeds from cultivated varieties as well as from wild grape populations were subjected to carbonization. In this study an experimental bonfire was employed. This method was favoured over a carbonization by a laboratory muffle. The experimental bonfire allows us to replicate all the variables naturally present during the carbonization process of the organic material, also allowing the carbonization of a large reference collection. A large database is necessary to improve the identification system and minimising misclassifications. The methodology has been previously described (chapter 3 - 3.9.2.1. Carbonization of grape seed reference samples).

4.5.4.2. Digital image analysis

The methodology has been previously described (chapter 3 - 3.9.3. Digital Image analysis).

4.5.4.3. Statistical analysis

The methodology has been previously described (chapter 3 - 3.9.4. Linear Discriminant analysis).

4.5.5. Results

Before the statistical processing, a standardization of all raw data was executed detecting a total of 330 outliers in modern material both cultivar and wild. Thus, these outliers were not considered in the discriminant analyses, resulting in a total of 6834 seed images for the cultivars and 1032 seed images for the wild samples.

With the aim of classifying the archaeological pips found in Torre dos Namorados the sample of archaeological pips was compared with 35 ancient grape cultivars and four populations of wild grape seeds. From the comparison between the archaeological grape seeds, which were considered individually and added to the classifier as unknown groups, and the modern *V. vinifera* subsp. *vinifera* and *V. vinifera* subsp. *sylvestris* pips, an overall percentage of 99.1 % of the pips were correctly classified (Table 10). The archaeological grape seeds from Torre dos Namorados were identified as *V. vinifera* subsp. *vinifera* in 38.0 % and *V. vinifera* subsp. *sylvestris* in 62.0% of the cases (Table 10).

4. Case studies

Table 10: Classification percentage among the archaeological grape seeds (considered as unknown groups) from Torre dos Namorados and modern charred *Vitis vinifera* subsp. *vinifera* and *Vitis vinifera* subsp. *sylvestris*. The numbers of grape seeds that were analysed are in brackets.

	<i>V. vinifera</i> subsp. <i>vinifera</i>	<i>V. vinifera</i> subsp. <i>sylvestris</i>	Total
<i>V. vinifera</i> subsp. <i>vinifera</i>	99.8 (6820)	0.2 (14)	100 (6834)
<i>V. vinifera</i> subsp. <i>sylvestris</i>	5.6 (58)	94.4 (974)	100 (1032)
Torre dos Namorados	38.0 (426)	62.0 (696)	100 (1122)
Cross-validated			99.1 % (8,988)

The archaeological seeds that showed similarities to the wild populations (62% of the total, or 696 pips) were compared with the grape seeds of wild populations collected from the different geographical locations in the Centre and South of Portugal (Table 11). The archaeological samples were added individually to the classifier as an unknown group.

Table 11: Correct classification percentages among populations of *Vitis vinifera* subsp. *sylvestris* and archaeological seeds from Torre dos Namorados. The numbers of grape seeds that were analysed are in brackets.

	Alcaçer do Sal	Castelo Branco	Montemor o Novo	Portel	Total
Alcaçer do Sal	71.9 (217)	5.6 (17)	5.0 (15)	17.5 (53)	100 (302)
Castelo Branco	7.4 (20)	74.9 (203)	10.7 (29)	7.0 (19)	100 (271)
Montemor-o-Novo	8.8 (5)	17.5 (10)	59.6 (34)	14.0 (8)	100 (57)
Portel	12.2 (49)	5.5 (22)	4.0 (16)	78.4 (315)	100 (402)
Torre dos Namorados	15.2 (106)	31.6 (220)	1.7 (12)	51.4 (358)	100 (696)
Cross-validated					74.5% (1,728)

Similarly, considering the percentage (38%) of archaeological seeds that showed similarities to *Vitis vinifera* subsp. *vinifera*, the archaeological seeds, which were now considered individually as unknown groups, were compared with 35 ancient grape cultivars. In fact, the archaeological grape seeds showed similarities with 18 ancient grape cultivars (Figure 53). In particular, the archaeological grape seeds showed a high percentage of similarity with two cultivars, Tamarez and Aragonez in 44.8% and 15.7% of the cases, respectively (Figure 53). Moreover, considering the grape skin colour of the ancient cultivars, the archaeological seeds from Torre dos Namorados classified as cultivars (426 pips) were now considered individually as unknown groups and were mostly classified as white grapes (72.8 %), and only 27.2 % of the cases were classified as black grapes (Table 12).

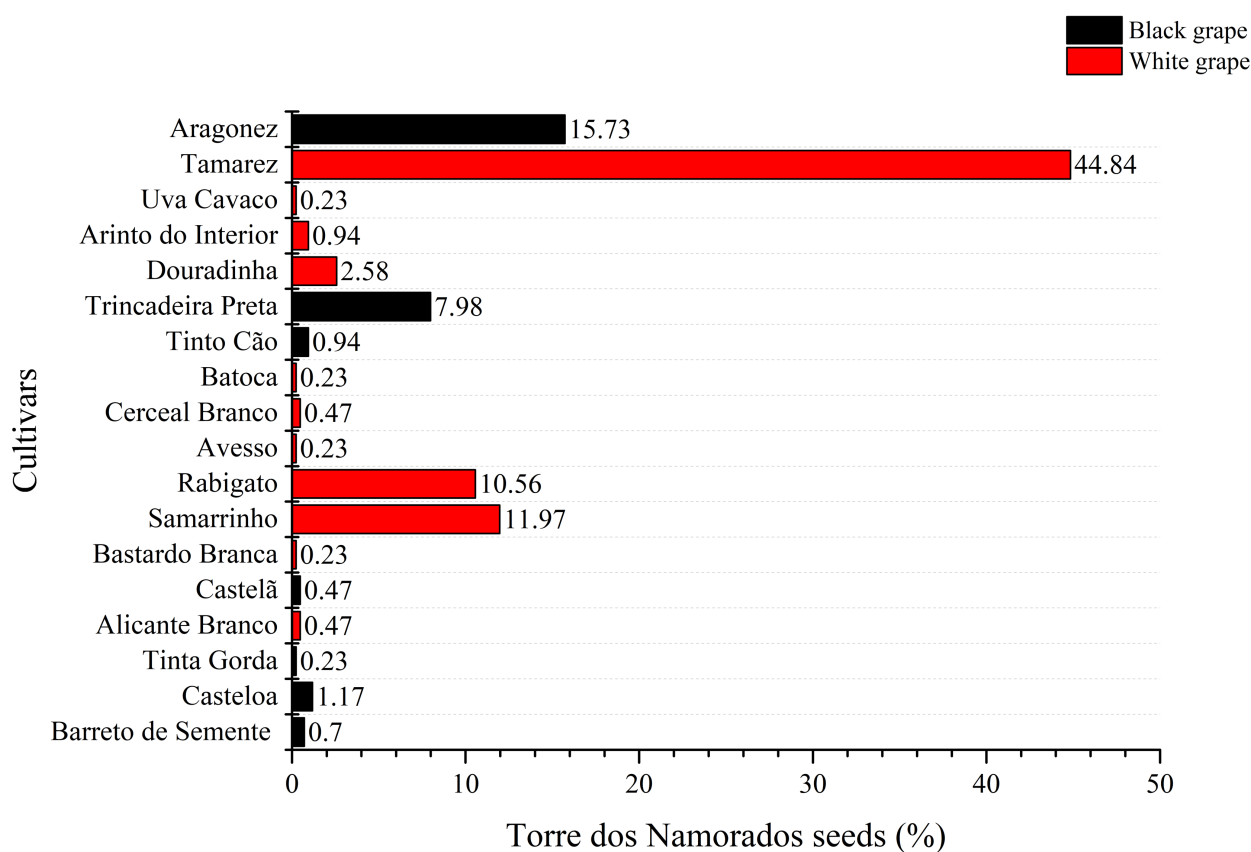


Figure 53: Graphical representation of the percentages of correct classification of the archaeological grape seeds of Torre dos Namorados with the ancient grape seed's cultivars of *Vitis vinifera* subsp. *vinifera*.

Table 12: Correct classification percentages among white and black grape cultivars and archaeological seeds from Torre dos Namorados (considered unknown groups). The numbers of grape seeds that were analysed are in brackets.

	White grape	Black grape	Total
White grape	65.4 (2555)	34.6 (1352)	100 (3907)
Black grape	35.5 (1040)	64.5 (1887)	100 (2927)
Torre dos Namorados	72.8 (310)	27.2 (116)	100 (426)
Cross-validated			64.6% (7260)

4. 5. 6. Discussion

4. 5. 6. 1. *The relevance of the disclosure*

The archaeological site of Torre dos Namorados is located in the centre of Portugal in the southern part of the Beiras region, a territory bordering with Spain. The Beiras region is particularly rich in archaeological findings, the Côa Paleolithic rock art being perhaps the most popular, but there are several other archaeological interests including Castros and places from the Copper and Bronze Age, Roman sites, and Medieval Castles (Vilaça, 2008; Carvalho, 2006).

The 2006-2007 archaeological excavations at the Roman Coitos cluster of the archaeological site of Torre dos Namorados (Fundão) revealed the presence of wine cellar elements (*cella vinaria* or wine press) with an amazing box (*Lacus musti* or settling vat) built with *tegulae* and bricks and sealed by the collapse of the roof, which was full of exceptionally well-preserved macro-remains of grape seeds. This finding is very significant due of the excellent state of conservation of the materials retrieved, and especially for the rare preservation of this type of remains in Portuguese wine production archaeological contexts.

Image analysis techniques done on charred grape seeds from Torre dos Namorados is the first study of this kind which allowed to investigate the viticulture in the Roman Lusitania. Moreover, grape pips have been radiocarbon dated for this research, showing a chronology (with a 76,4% of reliability) between the end of the 4th and the beginning of the 5th century AD (Figure 52).

4. 5. 6. 2. *Did the Romans make wine from wild grapevines?*

The approach followed in this study allowed the comparison of archaeological grape seeds with ancient cultivars from the Portuguese National Ampelographic Collection, as well as seeds of *Vitis vinifera* subsp. *sylvestris* collected from different wild populations in Portugal. The results showed a clear association between archaeological and wild plant seeds reinforcing the idea that in the Roman period wild plants, predecessors of the cultivated varieties, would dominate in this region. Wild grapevine is autochthonous in Portugal and few populations are still existent nowadays (Cunha et al., 2007; Sanches and Figueiral, 1998; Leeuwaarden and Janseen, 1985). The human impact and American pathogens have led to the loss of numerous populations of *Vitis vinifera* sub. *sylvestris*, existing in the Portuguese territory (Arnold et al., 1998; Arnold et al., 2005).

Previous morphometric analyses, carried out on grape seeds from different archaeological sites of southern France, showed the use of wild grape morphotypes by the Romans, associated to the winemaking (Bouby et al., 2013; Figueiral et al., 2010).

Our results did not allow us to verify whether the wild morphotypes employed at Torre dos Namorados belonged to completely wild individuals or to weakly domesticated specimens. The

higher similarity showed between the archaeological seeds from Torre dos Namorados with the *V. vinifera* subsp. *sylvestris* populations of Castelo Branco (31,6%) (Beiras region, in the vicinity of the site) and of Portel (51.4%) (Alentejo region, roughly 200 km from the site) (Table 11) seems to indicate the use of both local wild morphotypes and the weakly domesticated forms coming from other areas, even far from the site. To wholly understand these results, it is necessary to refer to the presence of several Roman residences, typologically defined as *Villae*. These residences are considered as an expression of the intensive Roman agricultural exploitation. They typically had annexed structures connected to wine production. Many *Villae* were established throughout the Roman Province of Lusitania (Pereira, 2017), including the Southern regions of modern-day Alentejo and the Algarve. Some of the remains of these structures have been located in archaeological sites in the vicinity of Portel (Alentejo) where the wild grape population was sampled (Table 11).

Unfortunately, there is a lack of published studies and absolute dating regarding these sites. Most of the residences seem to have been established from the I or the II century AD, while the agricultural functionality may have only intensified in the following centuries (Pereira, 2017). Moreover, regarding the few and rare carpological remains retrieved associated to this type of archaeological sites there is, also, an absence of studies. This information together with the results obtained from this study allowed us to raise the hypothesis concerning the existence of an intensive production network of the Lusitania Roman wine. This network would take into consideration production facilities located in different areas of the Roman Lusitania, using local wild populations and/or wild populations from different areas, possibly weakly domesticated.

The use of weakly domesticated wild morphotypes could be linked, according to some authors (Bouby et al., 2013), to the reproductive biology system of these ancient grapevines. Most modern grape varieties have hermaphroditic and self-compatible flowers, while wild grape is dioecious and female plants need to be pollinated by male plants in order to produce fruits (Levadoux, 1956; Olmo, 1996; Coito et al., 2019). A great diversification of primitive forms would have characterized Roman's vineyards. Accordingly, this factor would have decelerated the generalization of the hermaphroditism (Bouby et al., 2013).

These data allowed us to deduce that the viniculture process in the Roman Lusitania was still ongoing during this period. Data previously mentioned regarding other archaeological realities seems to suggest that the viniculture process by the Romans was still in progress throughout much of Western Europe. Coming studies of this kind, concerning other archaeological sites, both in Portugal and in Europe, will allow to add further information in this regard.

4. 5. 6. 3. *Different cultivated grapevines*

The comparison between the archaeological grape seeds of Torre dos Namorados and modern seeds from the native cultivars of *Vitis vinifera* subsp. *vinifera*, coming from the National Ampelographic Collection, considered the warranted main varieties nowadays accepted for the wine production of Portugal (Portaria n° 380, 2012), showed a higher similarity with the varieties of Tamarez, Aragonez, Samarrinho, Rabigato and Trincadeira Preta, in descending order respectively (Figure 53). Tamarez, Aragonez and Trincadeira Preta varieties are still associated to the Beiras Wine Region up to present days. According to some authors, the Tamarez variety is nowadays mainly mixed with other varieties, and its use was more frequent during the past (Martins, 2009). Aragonez is considered the Iberian grape variety par excellence (with about 20,000 hectares planted nowadays in Portugal), one of the varieties to be valued on both sides of the border between Portugal and Spain (Institute of wine and vine, Ministry of Agriculture. <https://www.ivv.gov.pt/np4/112/np4/318.html>, consulted on 24/09/2021). Particularly, the origins of Trincadeira Preta are associated to this region (Lobo, 1790). Little is known about the origin of the white varieties of Samarrinho and Rabigato. The latter was distributed throughout the country during the 19th century (Cincinnato da Costa, 1900).

Furthermore, the results of this study suggested a greater use of white grape varieties compared to black varieties at Torre dos Namorados (Table 12). Many white grapevines are nowadays still cultivated and employed for winemaking in this area of Portugal (Institute of wine and vine, Ministry of Agriculture. <https://www.ivv.gov.pt/np4/112/np4/318.html>, consulted on 24/09/2021). This would validate the hypothesis for which white grapes were already used by the Romans for the Lusitania's viticulture.

It is reasonable to think that the Romans intentionally proceeded to the selection of different populations of wild grapes (using both local populations and wild populations from other territories for which a process of domestication had possibly already begun) together with some domesticated grape

4. 5. 6. 4. *Morphometric analysis- a valuable method to analyse of charred material*

This study further confirms the Image analysis techniques applying the Stepwise Linear Discriminant analysis as a valuable and non-destructive method to discriminate archaeological grape seeds, and to investigate grape domestication (Bouby et al., 2013; Bouby et al., 2018; Orrù et al., 2012a; Uccesu et al., 2015; Terral et al., 2010; Pagnoux et al., 2014). The reliability of this method for the characterisation of archaeological grape seeds, even when preserved by carbonization, is confirmed by the results of this study. Results and information obtained from this study must be taken into account, considering the few valuable methods existing to investigate this type of materials and

the viticulture process. Indeed, all difficulties, still present nowadays, for the extraction of genetic material from charred archaeological seeds must be considered (Bacilleri et al., 2017). For all the reasons mentioned here, the Image Analysis Techniques has proved to be a valid method for studying the history of grape domestication in Portugal during Roman times.

4.5.7. Conclusion

Even if we are still far from a full understanding of the Roman viniculture process in the Lusitania province, the morphological comparison of archaeological charred grape seeds from Torre dos Namorados with modern reference, both wild and cultivated, made possible to show a clear association between the archaeological and the wild plant seeds, reinforcing the idea that in the Roman period wild plants would dominate in this region.

Results also showed a clear association between the archaeological grape seeds and some cultivated modern varieties, mostly belonging to white grape plants. These data allowed us to infer about the evolution of the varieties of *Vitis vinifera* in the region.

The use of both wild and cultivated grape reenforced the hypothesis for which the viniculture process in the Roman Lusitania was still ongoing during this period. The hypothesis of the existence of an intensive production network of the Lusitania Roman wine, with production facilities located in different parts of this area was expectable.

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4. 6. Case study 5: Exploring archaeobotanical investigation of Paço dos Lobos da Gama archaeological site: an Islamic suburb of Yabura (Évora, Alentejo, Portugal)

4. 6. 1. Introduction and research aims

Treatises on agriculture and geography provide fundamental data about agricultural practices and population diets in the Islamic period in the Iberian Peninsula. A large amount of information is available regarding the *al-Āndalus* region in general (incorporating modern day Spain and Portugal with the exception of some Christian territories that were maintained in the north/northwest of the Peninsula), despite sparse data on the westernmost part of this territory, properly known as the *Gharb-al-Āndalus* (the Portuguese part of the territory) (Gomez, 2013). However, although the written sources are an important testimony for the investigation of the ancient societies, they mainly reflect the habits of the upper class. Hence, the importance of a study from an archaeobotanical perspective, which, through the study of plant macro-remains, allows to gather information about the consumption of different plants, their economic value, and the processes involved in their production, without the social bias (Buxó and Piqué, 2008).

The study of plants macro-remains from Paço dos Lobos da Gama – situated in Évora – involves the analysis of charcoals, seeds and fruits found in the site, which is an Islamic context from the late 9th/ early 12th century.

This work aims at collecting information about the plant food consumed by the inhabitants of what nowadays is the site of Paço dos Lobos da Gama, trying to define the connection between the different types of food and their production processes, storage, and consumption. Another objective is to provide a better interpretation of the structures from where the sediments – containing the plant macro-remains – have been collected. Lastly, the importance of providing new data to the pre-existing archaeobotanical studies of the area is stressed.

4. 6. 2. Archaeological setting

4. 6. 2. 1. Paço do Lobos da Gama - The archaeological site

Paço dos Lobos da Gama was a manor house built by the Lobo da Gama family in Évora at the beginning of the 17th century. It was located next to the Santa Clara monastery along rua Serpa Pinto, one of the main roads of the city connecting the ancient Porta de Alconchel and the north-west angle of Praça do Giraldo (Figure 54).

In 2008, in view of the restoration works to convert the building in a private apartment complex, the site was the target of a rescue archaeological excavation which was carried out in three phases according to the work pace.



Figure 54: The white rectangle indicates the localization of Paço do lobos da Gama archaeological site. The internal drawn line defines the Roman-Islamic walls and the external one the so called Ferninandas walls, built in the XIV century AD. Image by Gonzalo Lopes.

The archaeological intervention – conducted by the company ARKEOHABILIS - *Arqueologia e Paisagem Lda* – mainly focused on the backyard of the building, which has been excavated in six distinct sectors. Sectors 1 to 3 have been excavated during the first phase, while sectors n. 4 and n. 5 have been excavated during the second phase. Lastly, sector n. 6 – whose excavation has been coordinated by the archaeologist Conceição Roque – has been dug during the third phase.

Sector 6 occupied the northern side of the backyard and revealed a significant assemblage of archaeological remains, dating back to the period between the 1st- 2nd century AD and the end of the Early Modern Period, with a particular focus on the Islamic period. Seeds, fruits, and charcoals of small size are the object of this study.

4. 6. 3. Materials

4. 6. 3. 1. *Archaeological contexts of macroremains*

The plants macro-remains under study are all coming from Islamic contexts, and they are preserved by partial combustion and mineralisation. They have all been taken from sediment samples collected from sector. 6. Except for one structure – specifically, the one corresponding to the stratigraphic unit 72 – the other structures are negative stratigraphic units. More precisely, they were structures used for storage, food-processing, and garbage collection purposes; namely, a septic tank, a sewage pipe, two silos and a well (Figure 55).

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Figure 55: A. The septic tank SU [57]; B. The well SU [101]; C. The sewage pipe SU [73].

The two stratigraphic units 65 and 66 were contained in the septic tank that corresponds to the SU [57]. Two sediment samples of 6 and 4 litres – corresponding respectively to SU [65] and SU [66] – were collected from the tank. The stratigraphic unit 65 is an intermediate layer, while SU [66] is a small clay deposit found at the bottom of the tank; it was probably resulting from the use of the tank itself. Within this unit, an almost intact ceramic lamp (“candil de bico” in Portuguese) was found. From a chronological point of view, its finding sets the beginning of the use of this structure around the middle/end of the 9th century AD.

The sewage pipe – named SU [73] – was filled by SU [74], from which another sediment sample (6 litres) has been collected. The pipe was located by the north-western corner of sector n. 6; a small silver coin – which appears to be a *Quirate* minted in the name of *al-Mutawakkil*, king of the Taifa de Badajoz between 1073/79 and 1094 – has been found inside the pipe.

Two sediment samples of 6 litres each – corresponding to the units 117 and 92 – were collected from the two silos, named respectively SU [126] and SU [93]. The latter was almost completely torn down. The silo [126] was instead quite well preserved, and contained ceramic materials dating back to a period between the end of the 11th century AD and the beginning of the 12th century AD.

The only stratigraphic unit excavated was the n. 117, since the level of the ground water made it impossible to proceed in the excavation.

Unit 72 was a layer extending on the eastern side of sector n. 6. A 4-litre sediment sample has been collected from a darker area that could probably correspond to the remains of a combustion structure. Some leftover ashes were found, together with some ceramic fragments belonging to a vessel that can be identified approximately as a pot from the 9th century AD.

The well – SU [101] – in the north-western corner of sector n. 6 has not been entirely excavated for logistical issues and because of the level of the ground water. For these reasons, only the upper layer of its filling material – SU [102] – has been investigated; 6 litres of sediment have been collected from this stratigraphic unit, together with the extremity of a spindle that is probably dating back between the end of the 9th century AD and the beginning of the 12th century AD.

4. 6. 4. Methods employed

4. 6. 4. 1. Sampling

A large part of the collected sediment was previously treated during the archaeological excavation through wet sieving, by means of sieves with 1 mm and 0,5 mm sieve openings. The goal of this treatment was to retrieve archaeological materials of small dimensions, with particular attention to faunal bones (Costa and Lopes, 2012).

On a total of 38 sediment litres, 28 litres have been treated during the excavation, and only the remaining 10 litres have been entirely analysed in the laboratory.

4. 6. 4. 2. Plant remains recovering

4. 6. 4. 2. 1 Manual Flotation plus water sieving

This methodology has been previously described (chapter 3 - 3. 5. 1. Manual Flotation plus water sieving).

4. 6. 4. 2. 2. Preliminary screening of macroremains

This methodology has been previously described (chapter 3 - 3. 6. Preliminary screening of plant macroremains).

4. 6. 4. 3. Taxonomic identification

4. 6. 4. 3. 1. Carpological analysis

This methodology has been previously described (3. 9. 1. Taxonomic identification - Carpological analysis).

4. 6. 4. 3. 2. Anthracological analysis

This methodology has been previously described (chapter 3 - 3. 7. 2. Taxonomic identification of charcoal samples – Anthracological analysis).

4. 6. 5. Results

The analysis allowed to the identification of different types of archaeological seeds, fruits and charcoals. The results are summarised in table 13 below. To allow a better understanding of table 13, fruits and seeds are clustered as follows: cultivated plants (cereals, fragments of spikelets, leguminous plants, fruit trees and bushes) and wild plants.

Figure 56 shows that the archaeobotanical material from the site is mainly composed of macro-remains preserved by mineralization. It should be noted that the entirety of the mineralized remains comes from the filling material of the septic tank (SU [65]); this detail could provide more information on this structure once the analyses will be concluded.

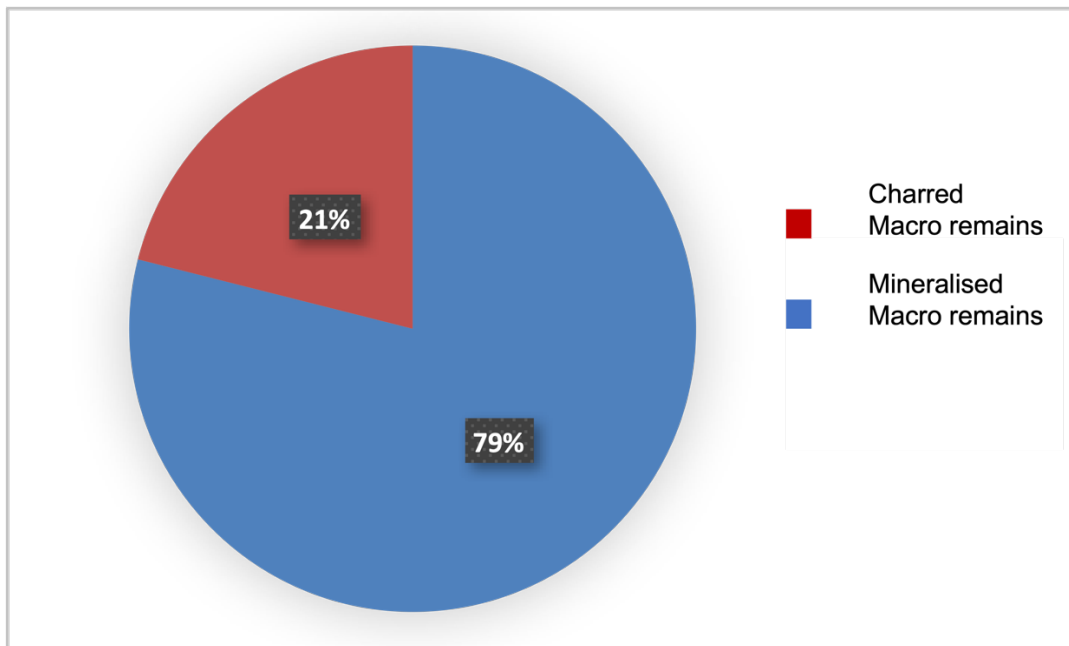


Figure 56: Percentages of mineralized and charred macro-remains identified within the carpological assemblage from Paço dos Lobos da Gama.

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Table 13: Summary of the plants macro-remains identified in Paço dos Lobos da Gama.

	STRATIGRAPHIC UNITS (SUs)							TOTAL
	SU 65	SU 66	SU 72	SU 74	SU 92	SU 102	SU 117	
FRUITS AND SEEDS								
CULTIVATED PLANTS								
(Cereals: entire grains)								
<i>Hordeum vulgare</i>			1				1	2
<i>Triticum cf. aestivum/durum</i>						1		1
<i>Avena sp.</i>			1					1
(Cereals: Fragments of spikelets)								
Rachis fragment			1					1
Culm fragment			1					1
(Leguminous plants)								
<i>Cicer cf. arietinum</i>	1							1
(Fruit trees and shrubs)								
<i>Vitis vinifera</i>	467							467
<i>Ficus carica</i>	73		2			1		76
<i>Morus sp.</i>	8							8
<i>Prunus cf. dulcis</i> (fragment)	1							1
<i>Cucumis sativus/melo</i>	1							1
WILD PLANTS								
<i>Silene sp.</i>			1	1				2
<i>Silene gallica</i>						1		1
TOTAL	551	0	7	1	0	3	1	563
CHARCOAL FRAGMENTS								
<i>Quercus spp.</i> (evergreen)					75			75
<i>Erica cf. arborea</i>					35			35
<i>Arbutus unedo</i>					25			25
TOTAL								135
								698

4. 6. 6. Discussion

Keeping in mind that for each stratigraphic unit the sediment samples have only been partially collected – due to the urgent nature of the intervention – and that the analyses are still ongoing, only few observations can be made.

A very important aspect to be underlined is the methodological approach. In this study, we have treated samples that had undergone a pre-treatment during the excavation, where sieves with sieve openings larger than 0,25 mm have been used. This has led to a loss of significant information, that would otherwise have been preserved.

The collected material is quite heterogeneous and mainly comes from structures used for storage and waste materials collection. Besides the assemblage being composed of a small amount of macro-remains (especially for what concerns charcoals), it is representative for the plant resources used between the end of the 9th century and the beginning of the 12th century in the region of Évora, located in the south of Portugal. Namely, for what refers to food consumption, construction timber, and/or firewood.

Regarding the carpological remains that have been analysed so far, the following cereals are represented: barley (*Hordeum vulgare*) (Figure 57D); wheat (*Triticum aestivum/durum*) and oat (*Avena* sp.) (Fig 57E).

The importance of cereals in the diet of Islamic population of *al-Āndalus* is nowadays accepted by the scientific community. They were stored in silos (Catarina, 1998), or communal silos in case of lower classes (García Baena, 2008) in several places. The silos were frequently covered by millstones; their use proves once more how significant cereals were in the diet of these populations (Arruda et al., 2002).

The amount of information in the Arabic sources (Ibn, 1981; Ibn, 1988) is complemented by other archaeobotanical studies performed on the territory of *al-Āndalus* (Queiroz, 1999; Queiroz, 2001; Queiroz, 2009a; Queiroz, 2009b; Queiroz, 2009c; Queiroz and Mateus, 2014; Queiroz et al., 2004; Van Leeuwen et al., 2000; Van Leeuwen and Queiroz, 2001; Van Leeuwen and Queiroz, 2003).

Currently, the only leguminous plant found in the collection of macro-remains from Paço dos Lobos de Gama is the chickpea (*Cicer* cf. *arietinum*), represented by one well preserved specimen. References on the use and cultivation of leguminous plants in *al-Āndalus* are noticeable as well (Ibn, 1981; Ibn, 1988). From a nutritional point of view, cereals and leguminous plants are complementary; the latter have indeed a higher protein content and make the soil where they are cultivated more productive.

The chickpea (*himmis*) was particularly appreciated by the communities of *andalusīs*; it could have been cooked with the addition of cumin and cinnamon or, more simply, eaten raw with the addition of salt, pepper, and thyme (García Baena, 2008).

The consumption of fruits is well documented by the several fruits and seeds mineralized remains from the septic tank (SUs 65 and 66). This deposit turns out to be particularly rich in fig and grape seeds remains.

The following fruits have been identified so far: grape (*Vitis vinifera*) (Figure 57A); fig (*Ficus carica*) (Figure 57B-C), blackberry (*Morus* sp.) (Figure 57H), almond (*Prunus* cf. *dulcis*), possibly cucumber or melon (*Cucumis sativus/melo*).

The role of the fruit consumption in the diet of the population from *al-Āndalus* is reported in many Arabic sources, namely agriculture and cookery treatises.

It is known that fig was one of the most appreciated fruit and, just as nowadays, it was consumed both fresh and dry (Leandro Martins, 2013). It can be said that the cultivation of vineyard could either be for grape consumption or for wine production – which was fairly consumed, despite the clear prohibition given by the Koran. The grapes could also be dried and transformed into raisins, a method that was largely used for their preservation (Gomez, 2013). Literature on the cultivation of blackberry in the Islamic world is quite broad, dating back to the 9th century AD (San José, 1996). As for almonds, they were used for confectionery together with honey and walnuts (Gomez, 2013).

The possible presence of melon is very interesting. On one side, indeed, there are many references in the Islamic sources about the cultivation of different typologies of this fruit in *al-Āndalus* (San José, 1996). On the other side, a comparison could be made between this work and the other archaeobotanical studies in which some remains of melon have been found (Pais, 1996). References on the cultivation of cucumber also exist, with different varieties cultivated in the medieval Islamic world (San José, 1996).

It is important to underline that the above-mentioned fruits are still consumed nowadays by the population of the Mediterranean basin, either fresh or transformed in conserves (Bugalhão and Queiroz, 2005). It is also important to note that food surpluses could have a relevant role in the feed of domestic animals. In addition to the different sources (such as treatises) reporting the importance of fruits in the *andalusīs* diet, it is pivotal to mention the data obtained from the few archaeobotanical studies already performed in this area of the Iberic peninsula (Queiroz, 2001; Pais, 1996; (Van Leeuwaarden et al., 2000; Van Leeuwaarden and Queiroz, 2001). They allowed to improve the knowledge about this aspect of the diet of these populations and represent an essential term of comparison for present and future studies. More specifically, these archaeobotanical studies are the

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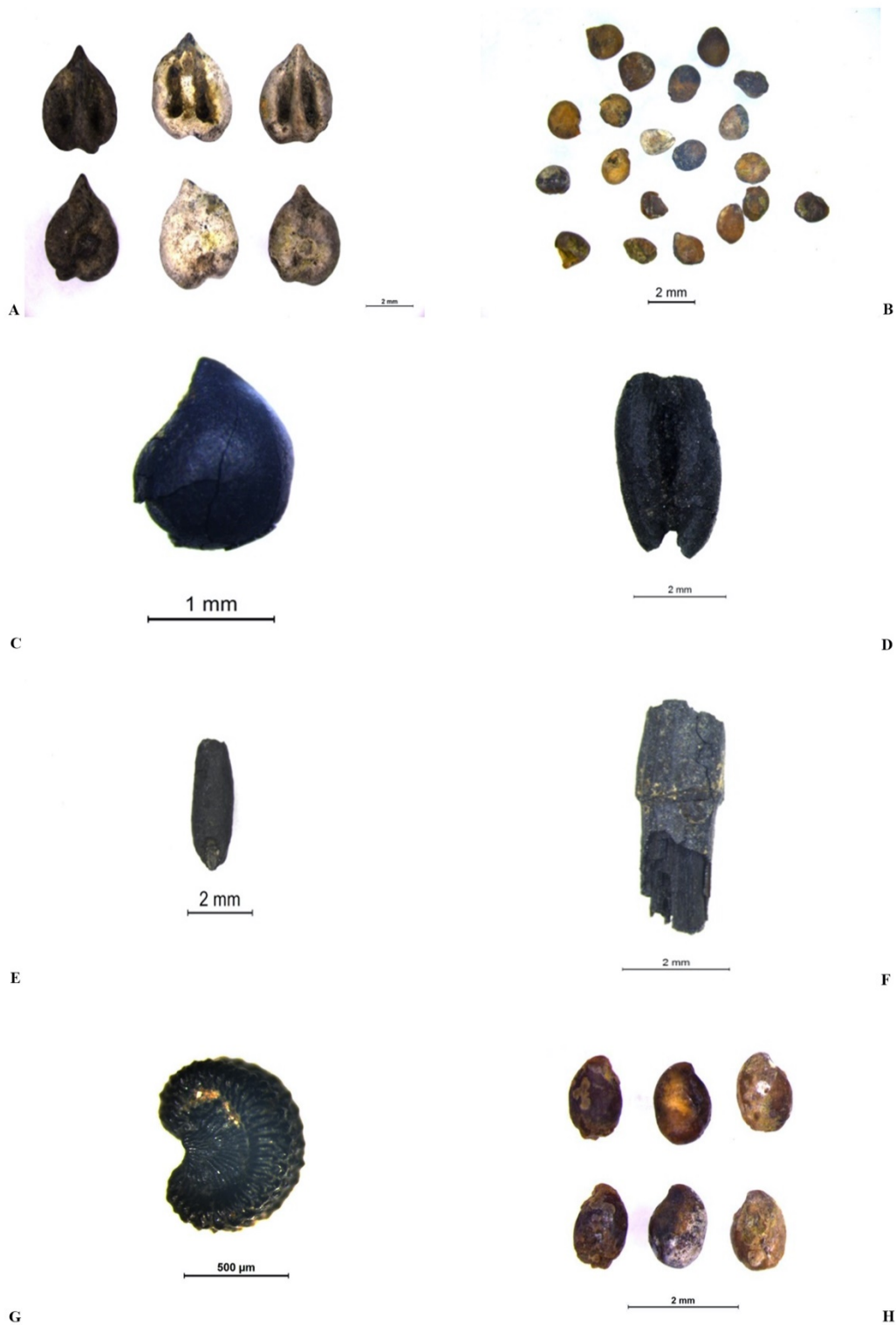


Figure 57: A. *Vitis vinifera* (Grapes); B-C. *Ficus carica* (Figs-mineralised); D. *Hordeum vulgare* (Barley); E. *Avena* sp. (Oat); F. Culm fragment; G. *Silene gallica* (windmill pink); H. *Morus* sp. (Mulberries).

one of the Núcleo Arqueológico dos Correiros, in Lisbon (Bugalhão and Queiroz, 2005; Queiroz, 1999), and the one of the Convento de São Francisco de Santarém (Van Leeuwaarden and Queiroz, 2001), which revealed contexts particularly rich in grape seeds and fig achenes remains. The data collected in these two sites present great similarities with the plants remains found in the filling

materials of the septic tank (SUs 65 and 66) from Paço dos Lobos da Gama, allowing for the gathering of crucial data about the context and for future comparisons.

It should be noted that among the investigated carpological remains some seeds of *Silene gallica* (Figure 57G) (common catchfly) have been identified; their presence is an indicator for moderately dry soils. This species develops very well in wastelands, and is generally an invasive species for some cultivations, such as oat and wheat.

Regarding the spikelets, the few remains that have been found come from the SU 72, which is the only context associated with a combustion structure. The finding of a ceramic pot in the same stratigraphic unit is of great interest in view of future analyses on possible organic traces, as they could provide relevant data.

From the few fragments of charcoals that have been analysed, we can confirm the use of wood of cork oak (*Quercus suber*), heath (*Erica cf. arborea*), and strawberry tree (*Arbutus unedo*).

Cork oak wood is hard and durable and has been used since ancient times as building material and as fuel, due to its great calorific value. Many archaeobotanical records can be found about its use by humans (Queiroz et al., 2005; Queiroz, 2008).

Heath wood and strawberry tree wood are both known and employed as firewood since Pre-History, especially for their combustibility; remains of both the species have been found in different archaeological sites from different periods in Portugal (Queiroz, 2009c; Queiroz, 2010; Queiroz, 2012).

Strawberry tree wood was also appreciated for its fruits, as reported in the Arabic sources mentioned above. Whenever some berry remains are found – though it happens rarely – they are generally preserved by carbonization; therefore, we can only hypothesize its consumption in Paço dos Lobos da Gama.

A broader understanding of the subjects and the questions mentioned so far can only be achieved once the archaeobotanical analyses will be completed.

4. 6. 7. Conclusion

Preliminary results obtained from the carpological and anthracological study permitted the identification of different type of seeds, fruits and charcoal fragments. Accordingly, information regarding the plant food consumed by the Islamic inhabitants of Paço dos Lobos da Gama have been collected. Data regarding the exploitation of the wood resources were also obtained. Moreover, the gathered data allowed for a better understanding of the structural contexts in which macroremains have been conserved. Future analyses concerning the identification off all carpological and anthracological remains from this context will add more data regarding the composition and

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consumption of the plant-based diet and the exploitation of wood resources by the Islamic community of Paço dos Lobos da Gama and more in general by the Islamic populations of the Gharb al-Āndalus.

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5th CHAPTER. Synthesis

The exploitation of plant resources in the human history: an archaeobotanical insight

5. 1. Final remarks

Results obtained from this thesis validate the importance of Archaeobotany as a discipline, professing its important role within the study of tangible and intangible Cultural Heritage.

Through the analysis of different types of botanical macroremains coming from different types of diachronic archaeological sites and contexts, the thesis work explored diverse kinds of relationships between past human communities and plant resources. In particular, the thesis addressed several issues i.e., the evaluation of human plant selection and use for ritual purpose; the analysis of charred wooden remains employed as building materials and furnishings of a residence; the identification of waterlogged wooden objects and the evaluation of their degree of conservation; the study of plant-based diets concerning the consumption and processing of vegetal foods and drinks by past human communities.

In order to address different issues and to stress the utility and interdisciplinarity potential of Archaeobotany, the thesis also dealt with the adoption of different types of techniques and analyses. Traditional archaeobotanical methods, namely anthracological, xylological and carpological analysis were adopted, together with the use of analytical techniques (Py-GC/MS and FT-MIR Spectroscopy), image analyses, and two different types of statistical analyses (PLS and LDA analyses). The thesis also consists of an experimental component, where contemporaneous grape seeds and wood were charred to mimic the archaeological samples. The charred grape seeds were used for image analysis and comparison with the archaeological samples, while the wood samples were used to develop a mathematical model, using FT-MIR and chemometrics, to determine the absolute combustion temperatures of archaeological charcoals.

Much information has been collected within this dissertation, highlighting how past human societies were able to make conscious decisions about the selection of vegetable raw materials, mainly using local resources. These data revealed technological, economical, and social indicators of past human communities, capable of broadening our knowledge about the human communities that produced such cultural materials and the contexts from which they came.

The observations stemming from the results achieved for each single case study will be reported below, stressing the contribution that these studies have brought to the discipline. These observations will be reported in chronological order.

5. 1. 1. Case studies observation

Concerning the study of charcoals from Pit 16, a secondary collective human cremation (Case Study 1), the results shed light on the plant exploitation by humans for funerary purposes during the Chalcolithic period at the Perdigões archaeological site (south of Portugal). Moreover, this study

demonstrated the efficacy of using two different approaches, based on different techniques. The first one includes the anthracological analysis of archaeological charcoals to identify the wood *taxa* utilized during the primary cremation ritual. The second one comprises the estimation of the absolute burning temperature of archaeological charcoals, creating a mathematical model developed with sound wood samples combusted at different temperatures (i.e., formed under a controlled temperature and atmosphere using a muffle furnace in the laboratory), using FT-MIR spectroscopy and chemometric analysis.

Anthracological and MF-MIR spectroscopy analyses made the identification of numerous wood species employed for the cremation ritual possible, together with a correct estimation of the absolute burn temperature of the three main wood *taxa* from Pit 16. Furthermore, it was possible to support the hypothesis of the existence of different depositional moments within the pit, consequently resulting from distinct cremation rituals, that had been put forward during the excavation of the pit.

This study permitted the collection of information concerning the role of woods within the ritual of cremation in the funerary practices present at Perdigões and, in general, in the western part of the Iberian Peninsula during the Chalcolithic.

The study also enable the development of a new analytical methodology to study the combustion temperature of archaeological charcoals. Future analysis of this type regarding other human cremations from the Perdigões archaeological site and from other Chalcolithic burial sites located in this part of Iberian Peninsula, will allow for the corroboration of the data obtained from this study, opening up a new perspective into the funerary practices during this time period.

With regards to the study of charred woods from *Domus dei Dolia* (Case Study 2), it is particularly important to reiterate that this work embodies the first study concerning wooden remains retrieved from an Etruscan residence of this type. The analysis provided information regarding which types of woods were employed for the construction and the furnishing of the *Domus* and regarding the timber knowledge of the Etruscan builders. Additionally, information regarding the utilization of local and non-local woods was obtained, together with that of the conservation state of the woods before the advent of the fire that burned the house.

We must consider that studies on this type of material from Etruscan archaeological sites are very rare throughout the Italian peninsula and that most of our knowledge concerning Etruscan society comes from the study of materials from funerary contexts. Accordingly, this study indirectly provided access to the economical, technological, and social aspects of the Etruscan inhabitants of Vetulonia, and more generally of the Etruscan way of living. The excavation of the *Domus dei Dolia* is still underway, and future research lines foresee the study of charred woods retrieved in the other rooms (the northern part) of the *Domus*.

4. Case studies

Finally, it is important to emphasize that this study was part of a larger project titled "WELCOME HOME", the results of which were presented both at international conferences and at local seminars carried out with the purpose of informing the local people. In particular, results were presented at "tourismA" - International Fair of Archaeology (Palazzo dei congressi, Firenze) the 24th of February 2019.

Concerning the study of waterlogged woods coming from a sewer of the Roman Criptopórtico da Rua da Prata (Case Study 3), the greater challenge of this case study was to deal with the bad preservation state of these remains. Wooden samples under study looked relatively unaltered, but the physical, chemical, and mechanical properties of wood had decay, making these samples highly susceptible to destruction. To stabilize the waterlogged wood samples, a method based on the use of a water-soluble compound was employed. The use of this method allowed for the sectioning of the samples. Diagnostic features were observed on wood thin sections, using Transmitted Light Microscopy. All samples were identified at the genus level. Moreover, a sample of a cork oak was also identified with SEM, confirming its archaeological interpretation. Pyrolysis- Gas Chromatography- Mass Spectrometry (Py-GC-MS) analyses were also carried out to evaluate the degree of degradation of some waterlogged wood remains. This latter technique confirmed its validity for the study of waterlogged wood decay, even when samples are in a bad state of preservation.

The data collected by this study facilitated the addition of information about the archaeological interpretation of waterlogged wooden objects as well as some samples whose archaeological interpretation was uncertain.

Analyses of waterlogged woods from Roman contexts in Portugal are very rare, and this study provided some information on a still poorly defined picture. This case study, embodying an exploratory approach, and future analysis concerning the evaluation of the state of preservation and the identification of all waterlogged wood materials coming from this particular context are expected. Moreover, a study regarding the comparison of two different techniques, Py-GC/MS, and Optical Microscopy, employed for the evaluation of the degree of wood decay is already underway. The study takes advantage of both modern reference samples and waterlogged samples coming from the Roman Cryptoporticus of Lisbon and will add further information to the picture already outlined with this thesis work.

Regarding the study of charred grape pips from a Roman wine cellar of the Torre dos Namorados archaeological site (Case Study 4), image analysis techniques were adopted in order to make a comparison between the archaeological grape seeds and modern ones from the native cultivars of *V. vinifera* subsp. *vinifera*, which came from the National Ampelographic Collection, and are considered the warranted main varieties nowadays accepted for wine production in Portugal.

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Archaeological grape seeds were also compared with modern seed accessions of plants of *V. vinifera* subsp. *sylvestris* populations present in Portugal. To achieve correct comparisons, modern seeds from cultivated varieties as well as from wild grape populations were subjected to carbonization, employing an open-air bonfire. Results permitted the understanding of which type of grapes were used for the Roman wine production of Torre dos Namorados. Furthermore, they also allowed for speculation over whether the viticulture process was already ongoing during this period.

Due the rarity of the preservation of this type of remains in archaeological contexts of Roman wine production, this study is the first of its kind to investigate viticulture from a site of the Roman province of Lusitania. Further investigations in this field are necessary to obtain a more comprehensive view of the issue, and to be able to compare the data obtained with other contemporary realities.

This study was possible thanks to collaboration with the Archaeobotany Laboratory of the Hortus Botanicus Karalitanus (University of Cagliari, Sardinia, Italy), where part of the analysis was developed, and with the National Institute for Agricultural and Veterinary Research (INIAV) (Dois Portos, Portugal), which made available the grape reference material necessary for the analyses, both cultivated and wild. These collaborations highlight the interdisciplinary nature and the value of this part of the thesis.

Concerning the study of the Paço dos Lobos da Gama archaeological site (Case Study 5), plant macroremains under study came exclusively from Islamic contexts. This study incorporated the analysis of archaeological seeds, fruits, and charcoals. Regarding the carpological assemblage some seeds and fruits were preserved by mineralization and others by carbonization. One of the main challenges of this case study was the identification of mineralized macroremains, a process which significantly changed both the size and the shape of these remains, making their identification very difficult.

Results obtained with the carpological and anthracological analyses allowed for the characterization of the plant food consumed by the Islamic inhabitants of Paço dos Lobos da Gama. Moreover, data collected also allowed for a better understanding of the structural contexts in which samples have been collected. Data regarding the exploitation of the wood resources were also obtained.

Ancient sources provided much information about agricultural practices and diets of populations in the Islamic period in the Iberian Peninsula. However, as the written sources mainly reflected the habits of the upper class, the importance of a study with an archaeobotanical perspective, aimed to obtain information regarding the cultural material of a whole community, even the lower

classes, is apparent. The importance of providing new data to the pre-existing archaeobotanical studies concerning the *al-Āndalus* should also be stressed.

Furthermore, it must also be highlighted that the carpological and anthracological remains from the Paço dos Lobos da Gama Islamic contexts are considered part of the cultural heritage of Évora, a city listed by UNESCO as World Heritage. In this respect, part of these results was already transposed to the general public during the exhibition “YABURA: uma cidade do *al-Āndalus*” (Convento dos Remedios, Évora - 15th April- 15th August 2015).

This case study represents an exploratory study, and future analyses concerning the identification of all macroremains from this archaeological site will allow for a more complete picture of the food habits of Islamic Évora.

5. 2. Thesis contribution to the objectives of the HERITAS Doctoral Program – Heritage Studies

The multidisciplinary HERITAS Doctoral Program, required the enhancement of scientific knowledge and the development of integrated strategies for the valorisation and the promotion of Cultural Heritage, tangible and intangible.

From a perspective initially developed in the Anglo-Saxon academic world, the intersection of archaeological issues with the methods and concepts of the Natural Sciences field creates the broad field of Archaeological Sciences. Among the disciplines that pursue such integration, there is Archaeobotany, nowadays considered as an independent discipline in almost all the academic world. This thesis dissertation has used traditional archaeobotanical methods, chemical analytical methods, and other types of techniques, in order to achieve the main objective of the HERITAS Doctoral Program, namely the creation of a common vocabulary between the humanities and natural sciences.

The analytical part of the work presented in the thesis was mainly carried out in one of the most well-equipped and advanced laboratories in Europe for the research in Cultural Heritage, the HERCULES Laboratory, which has instrumentation capable of carrying out research which can make an important contribution to the development of the Archaeobotany field. This thesis also made use of the expertise of various universities and international research centres in the sector. Finally, this thesis aimed to be a vehicle for the dissemination of Cultural Heritage, which should not be considered as scientific-academic property but must be accessible to all. In this sense, part of the thesis results has been not only presented in scientific meetings, also in museum exhibitions and cultural events which were open to a wider audience.

