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Tracing the biographies of textiles in the transition of medieval to modern times: Wool fabrics and brigandines from an Iberian castle

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ABSTRACT

Findings of archaeological textiles and fibres in Northern Iberia are extremely rare. The occurrence of a set of textile fragments, dated between the 14th and 16th centuries CE at the Pambre castle (Palas de Rei, Lugo, Spain) is exceptional. The original stone roof of the southeastern tower was intact. The dark, cold and moist conditions inside the tower favoured the preservation of a unique series of waterlogged textile remains. In addition, a set of pseudomorphs preserved by mineral replacement were recovered from the east edge of the north wing in the main hall of the castle. Fibres have been identified using optical and scanning electron microscopy (SEM), and they have been chemically characterised using Energy Dispersive X-Ray Spectroscopy (EDX). We also performed analytical pyrolysis-GC-MS (Py-GC-MS) and thermally assisted hydrolysis and methylation (THM-GC-MS) of the wool fabrics and pseudomorphs to assess their state of degradation and the presence of chemical markers associated to the use of these textile remains. High performance liquid chromatography with diode array detection (HPLC-DAD-MS) and ultra-high performance liquid chromatography coupled to high-resolution mass spectrometry (UHPLC-HRMS/MS) analysis were applied on wool fabrics to identify the chemical markers of dyes but without success. To expand the information related to raw material identification and the technical aspects of the fabrics, further evidence such as adherences identified as opal phytoliths, seeds, and insect remains associated to wool fabrics were examined. These findings offer a unique glimpse into the clothing dated to the end of the Medieval period, and its life-cycle. Wool scraps were probably part of at least two different garments, whereas the mineralised textiles probably formed part of at least two brigandines which were made of bast fibres, flax, or hemp.

1. Introduction

Textiles rapidly perish when buried in the ground. Their organic nature makes them easily degradable in most archaeological contexts, being only preserved when decay processes are truncated under specific conditions (waterlogging, mineral replacement, desiccation, or charring) (Margariti et al., 2023). For proteinaceous and cellulosic fibres, such conditions include a pH value that does neither damage them nor allows colonization of bacteria and fungi -plant fibres are rarely preserved in acidic environments, whilst animal fibres are usually destroyed under alkaline conditions (Good 2001; Gleba and Mannering 2012; Grömer 2016). Waterlogging (or water saturation) and mineral

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replacement are two of the most common ways of preservation of archaeological textile remains and fibres. In moist environments, anaerobic conditions inhibit the growth of microorganisms that cause their decay (Good 2001; Gleba and Mannering 2012). Metal corrosion products form negative or positive casts around fibres in direct contact, retaining their external morphology and size -pseudomorphs– and favouring the formation of easily legible traces, specially on iron and bronze objects (Jakes and Sibley 1984; Chen et al., 1998; Gleba and Mannering 2012). Corrosion processes can lead to a complete replacement of the organic matrix by inorganic matter while maintaining the physical shapes of fibers, yarns, and fabrics (Vollmer 1974; Gillard et al., 1994; Good 2001).

In the Iberian Peninsula, the study of textiles and clothing has been addressed mostly upon the information retrieved from visual sources -manuscript illuminations, effigies of wood and stone or paintings, among others-as well as from written records (*e.g.* Bernis 1957, 1978; Falcón 1995; Sigüenza 1998; Aragonés 1999; Vegas 2013). The study of textiles themselves has focused on fabrics, tapestries and embroideries from regal, noble and ecclesiastical environments (*e.g.* Herrero 1988; Feliciano 2014; Sanz de Bremond 2016, 2018, 2020), whereas those recovered from archaeological contexts dated to the transition between Medieval to Post-Medieval periods are scarce (Borrego 2008; Borrego et al., 2017), and few of them have focused on Medieval textiles (Gómez 1946; Martínez 2000; Martínez and Pastrana 2002; Gutiérrez-Cuenca et al., 2014). Contrary to the development of textile research in other European areas where multiple examples of Medieval textiles have been studied from settlements (e.g. Walton 1989; Crowfoot et al., 1992; Sherman 2014) and funerary sites (e.g. Médard et al., 2007; McCullough et al., 2018), archaeological contexts from Southwestern Europe have provided limited evidence (e.g. Gutiérrez-Cuenca et al., 2014; de Bruijne and Monteiro 2019).

Despite these preservation constraints, archaeological textile studies address relevant issues ranging from aesthetics and style to status and gender; from technological development to production and exchange economics (Good 2001). During the Middle Ages, clothing developed roles beyond cover and body warmth, becoming a social and political symbol, having the power to communicate social status (Le Goff 1994; Pastoreau 2006; Staples 2008; Rosenthal 2009). Complexity in weave, design, and manufacture are, in a very general sense, attributes of wealth and prestige (Good 2001). In the 14th and 15th centuries CE there was a steady flow of sumptuary legislation that attempted to control who wore what, and these regulations illustrate the significance of clothing and its importance in the self-expression of that society's members (Staples 2008). Written sources, religious sculptures and church wall paintings attested the variety of fabrics used during the Late Middle Ages, as well as their quality, colour, prize and provenance (Vegas 2013; Barral Rivadulla 2023).

Aristocrats sought social differentiation through dress codes because social status depended not only on luxurious cloth but also on how cloth was fashioned into garments that followed precise, often individual, guidelines (Rosenthal 2009). Artifacts or worldly possessions and luxurious clothing, however, were separate and distinct from the household items that constituted instead an individual's property and investment for one's heirs (Rosenthal 2009). The life-cycle of clothing was complex and textiles were continually in movement during the Middle Ages. They moved from body to body in the form of gifts and payments, they were separated into discrete parts that circulated and recirculated after the death of a person, they were altered and realtered for individual family members and for individuals in larger networks that extended beyond the family (Rosenthal 2009), and they were even used as material for insulating wooden buildings (Erickson 1982).

The set of archaeological textile remains recovered at the Pambre castle, comprising organic fabrics and pseudomorphs, is an exceptional finding in Southwestern Europe. The first aim of the current study is to inform us about the raw materials, the kind of threads, fabric and textures, as well as their form, their function and the technology used to make them. The second goal is to deepen our understanding on the clothing used in the transition between the Late Middle Ages and the beginning of the Post-Medieval period. Thirdly, processes related to the biography of textiles are addressed to reconstruct both their uses and their processes of discard or abandonment. Finally, we aim to highlight the relevance of interdisciplinary approaches to address the study of perishable material culture recovered from contexts dated from Middle Ages onwards. There is a noticeable gap of research focused on organics dated to these chronologies, which contrasts with their importance in the day-to-day life of these communities.

2. Material and methods

2.1. Site

The Pambre castle (Palas de Rei, Lugo) is placed in a rocky promontory at 430m a.s.l. on the Pambre river bank (Fig. 1). It is one of the best examples of military architecture dated to the Middle Ages in present-day Galicia (Northwestern Spain). Its construction began between the end of the 14th century or the beginning of the 15th century CE, and it has been attributed to Gonzalo Ozores de Ulloa. It was later modified by Lope Sánchez de Ulloa, who ordered the first defensive wall, while his grandson, Sancho Sánchez de Ulloa, ordered a second one (Pardo de Guevara et al., 2012, Rouco 2017). These two constructive phases are clearly visible in the castle structure with a keep and two defensive walls around it (Pardo de Guevara et al., 2012). This castle was used as regular residence by Inés de Castro and her son, Sancho de Ulloa, during the 15th century CE, function that ended after Sancho's death in the year 1505 (Rouco 2017). The archaeological survey of the castle started in 2013 and comprised monitoring of topsoil removal, test pit evaluation and open area excavation. Excavations were conducted in the hall (250 m^2), the northeast (6.35 m^2), southeast (15 m^2) and southwest (4.32 m^2) towers, the keep and the chapel, among other areas.

2.2. Samples

A total of 16 textile fragments from handpicked samples have been analyzed (Tables 1 and 2). Eight of the samples were recovered in the southeast tower of the castle (Fig. 2) from stratigraphic units (SU) ST003-005 during the excavation of one of the profiles inside this structure. The other eight samples were collected from the east edge of the north wing in the main hall in SU NW005 (Fig. 2). No chemical treatments were applied to textiles after their recovery. Water saturated fragments were simply unfolded by rehydrating the pieces with a mixture of distilled water and alcohol to preserve their integrity. All the macroscopic remains of fibres and organic adherences removed during the unfolding process of the organic textiles were gathered and stored. Some of the fibres that were detached during this process were subsequently used for destructive analysis such as radiocarbon dating or the analysis of chemical markers, preserving the integrity of the fabrics.

2.3. Raw material identification

The identification of raw material was performed by combining observations under stereoscopic microscope Olympus SZX7 and Scanning Electron Microscope ZEISS EVO LS 15 (RIAIDT-Universidade de Santiago de Compostela). To achieve an identification, the surface and medulla of fibres were observed (Cybulska et al., 2008, Bender Jørgensen and Grömer 2012; Gleba and Mannering 2012) following the method described by Rast-Eicher (2016). Atlases (Hausman 1920; Rast-Eicher 2016) and the FIBRANET online database (Margariti 2019a) of plant and animal fibres were used for comparison. Beyond fibre identification -animal or plant origin, taxonomic identification- and during the observation in the SEM the wear and damage of individual fibres was also registered (Gleba and Mannering 2012).

Fig. 1. Left, map of Northwest Iberia showing the location of Pambre Castle (Lugo, Galicia, Spain) (Elaborated by Emilio Abad). Right, Pambre Castle and southeast tower in the forefront (Photo: Santiago Vázquez Collazo).

Table 1
Description and raw material identification of the set of organic textile remains.

Code	Preservation	State	Size	Colour	Raw material	Pattern	Yarn	Threads per cm Sys. 1/Sys. 2	Diameter Sys. 1/Sys. 2	Twist direction Sys. 1/Sys. 2
Lab-001-001	Organic	Fragmentary	8.0 × 9.8 cm	Brown reddish	Wool	Tabby	Single	10, 7	0.4–0.6/0.6–0.8 mm	Z, S
Lab-001-002	Organic	Fragmentary	5.9 × 7.1 cm	Brown reddish	Wool	Tabby	Single	10, 6	0.6–0.8/0.6–0.8 mm	Z, S
Lab-002	Organic	Fragmentary	20.9 × 16.7 cm	Brown reddish	Wool	Tabby	Single	6, 6	1.1–1.5/1.0–1.7 mm	Z, Z
Lab-003	Organic	Fragmentary	45.9 × 31.3 cm	Brown reddish	Wool	Tabby	Single	6, 6	0.9–2.0/0.9–1.5 mm	Z, Z
Lab-004-001	Organic	Fragmentary	24.9 × 23.2 cm	Brown reddish	Wool	Tabby	Single	6, 7	0.9–1.3/1.1–1.3 mm	Z, Z
Lab-004-002	Organic	Fragmentary	21.0 × 17.5 cm	Brown reddish	Wool	Tabby	Single	–	–	–
Lab-005-001	Organic	Fragmentary	4.7 × 9.6 cm	Brown reddish	Wool	Tabby	Single	–	–	–
Lab-005-002	Organic	Fragmentary	15.0 × 14.5 cm	Brown reddish	Wool	Tabby	Single	5, 6	1.0–1.3/1.1–1.4 mm	Z, Z

Table 2
Description of the set of textile pseudomorphs.

Code	Preservation	Raw material	State	Yarn	Pattern	Threads per cm Sys. 1/Sys. 2	Diameter Sys. 1/Sys. 2	Twist direction Sys. 1/Sys. 2
007	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	8/10	0.6–1.0/0.5–0.7 mm	Z/Z
1130	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	10/10	0.4–0.7/0.5–0.7 mm	Z/Z
1290	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	8/12	–	Z/Z
1291	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	9/10	0.6–0.9/0.4–0.9 mm	Z/Z
1292	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	–	0.3/0.3 mm	Z/Z
1293	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	9/10	0.7–0.8/0.8–1.0 mm	Z/Z
1294	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	–	0.5–0.6/0.5–0.6 mm	Z/Z
1295	Mineralised	Bast fibre	Fragmentary	Simple	Tabby	8/9	0.6–1.0/0.6–0.9 mm	Z/Z

2.4. Technical analysis of textiles

All the studied textiles were fragmented. Their technical analysis comprises different stages (Bender Jørgensen and Grömer 2012). The first stage includes the description of the condition of the textile (type and state of preservation, dimensions and colour). This is followed by the description of the type and condition of the fibres, as well as evidence of fibre preparation; the number of threads per cm in warp and weft (if it is not possible to identify warp and weft, system 1 and 2 is used). The description of the yarns includes twist direction, diameter of the yarns of both systems, and whether it is single or plied yarn.

2.5. Radiocarbon dating

One sample from the set of waterlogged textiles was selected for AMS radiocarbon dating (Lab-001-001). The direct dating of the textile allowed to adjust the relative chronology assigned to the set of textiles based on archaeological materials, as proposed by Bender Jørgensen and Grömer (2012)

2.6. Analysis of chemical markers

Analytical pyrolysis is a useful technique to screen for

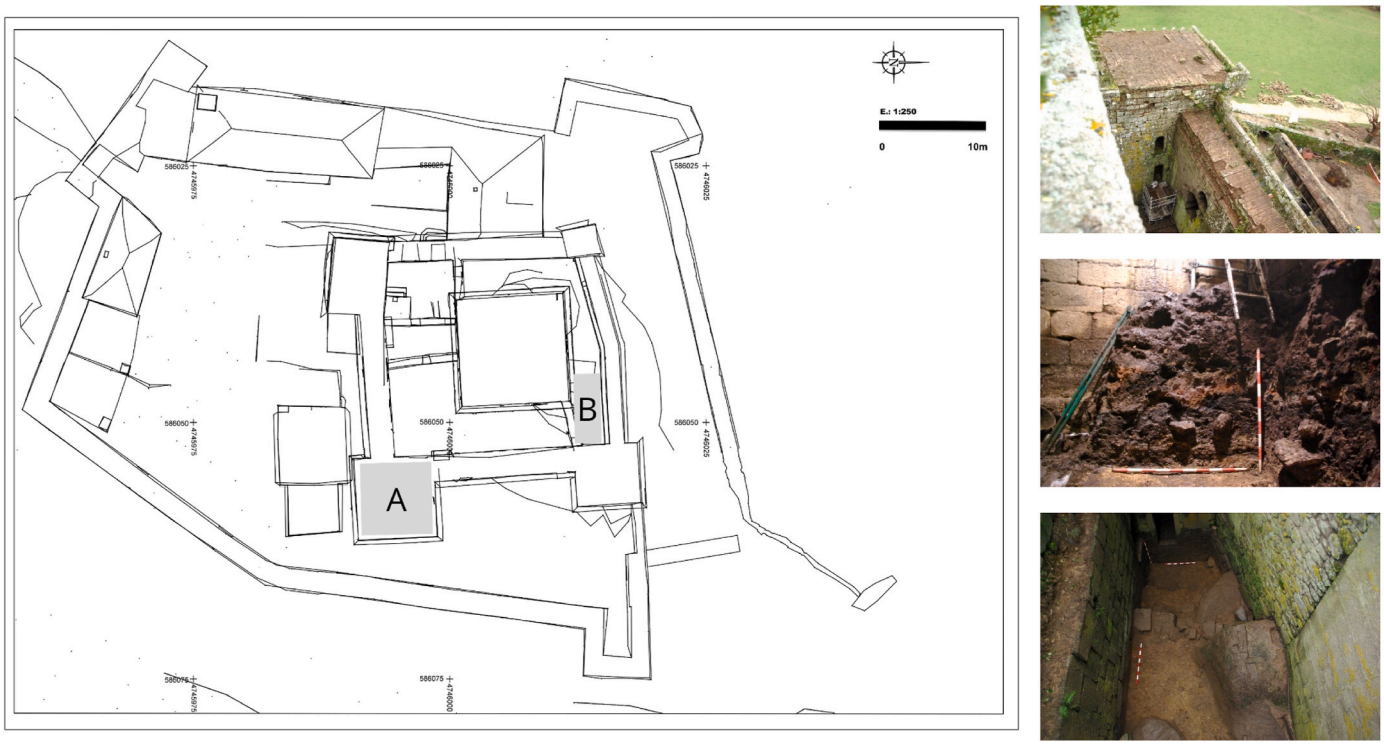

Fig. 2. From left to right and up to down. Spatial layout map of textile samples in the castle: A: organic remains, and B: pseudomorphs (Map: Santiago Vázquez Collazo). View of the southeast tower of the Pambre castle which preserves its original stone roof cover. Stratigraphic units where the organic textiles were recovered. East edge of the north wing in the main hall where the pseudomorphs were recovered. (Photos: Santiago Vázquez Collazo).

macromolecular organic matter. In textile analysis, Pyrolysis-GC-MS has been used to identify differences in flax fiber and yarn quality (Hardin 1996; Morrison and Archibald 1998). Sam (2017) discussed some Py-GC-MS traces of cellulosic cloth, including cotton, hemp, flax and bamboo. Several studies used Py-GC-MS and some thermally assisted hydrolysis and methylation (THM-GC-MS) to identify wool (keratin) and other N-rich biopolymers such as collagen and chitin (e.g Sabatini et al., 2018). This method can also be used to identify dyes, if present in large concentrations, but it is well known that it is far less sensitive to these materials and induces loss of diagnostic features due to the invasive nature of the pyrolytic cleavage.

Six samples of wool textiles (1 mg) were analyzed by Py-GC-MS and THM-GC-MS, whereas two samples of cellulose textile were analyzed by Py-GC-MS only. Conventional Py-GC-MS and THM-GC-MS was performed using a Pyroprobe-Agilent instrument. The pyrolysis and THM reactions were done at 650 °C for 20 s. The difference between Py- and THM-GC-MS is the addition of an aliquot of tetramethylammonium hydroxide (TMAH, 25 % in water) for the latter. Briefly, this reagent causes hydrolysis and methylation reactions, which improves the visibility of organic moieties with polar functional groups, such as the carboxylic moieties in fatty acids and amino acids, and hydroxylic moieties in lignin (Challinor 1989). Further details on instrumentation and analytical parameters are provided in Kaal et al. (2016, 2020). Compounds were identified on the basis of mass spectral features provided by the NIST 14 library and literature of N-rich materials, for Py-GC-MS (Fabbri et al., 2012; Adamiano et al., 2013; Sabatini et al., 2018; Cersoy et al., 2018) and THM-GC-MS (Knicker et al., 2001; Lejay et al., 2019).

The composition of dyes was studied by high performance liquid chromatography with diode array detection (HPLC-DAD-MS) and ultra-high performance liquid chromatography coupled to high-resolution mass spectrometry (UHPLC-HRMS/MS). Three different procedures were tested regarding their extraction efficiency: (i) (HCl) 37%: MeOH: H2O (2:1:1, v/v/v) (Wouters, 1985; Wouters and Verhecken, 1989); (ii) Trifluoroacetic acid 2 M (Valianou et al., 2009), and (iii) Oxalic acid 0.2 M: acetone: MeOH: H2O (0.1:3:3:4, v/v/v/v) (Marques et al., 2009). The last method was chosen and six samples were analyzed. The extracts were evaporated in a vacuum line, the threads (circa 2 mm) were removed and the residues were dissolved in 50 μL of methanol/water, 7:3 (v/v). After a centrifugation step, about 45 μL of each supernatant was removed for HPLC-DAD-MS and UHPLC-HRMS/MS analysis.

The HPLC-DAD-MS analysis was performed on a Dionex Ultimate 3000SD system with a diode array detector coupled online to an LCQ Fleet ion trap mass spectrometer (Thermo Scientific, Waltham, MA, USA). Separations were carried out with a Kinetex C18 100 Å (150 × 2.1 mm, 5 μm, Phe-nomenex) at 40 °C, using a flow rate of 0.3 mL/min. The mobile phase was 0.1% of acid formic in water (v/v, eluent A) and acetonitrile (eluent B), and the elution gradient was as follows: 0–1 min linear gradient to 0% B; 1–5min linear gradient to 7% B, 5–18 min linear gradient to 100 % B, 18–22 min isocratic 100% B, and then the column was re-equilibrated with 0 % B for 7min.The mass spectrometer was operated in the ESI positive and negative ion modes, with the following optimized parameters: ion spray voltage, ±4. 5 kV; capillary voltage, 16/-18 V; tube lens offset, −70/58 V; sheath gas (N2), 40 arbitrary units; auxiliary gas (N2), 20 arbitrary units; capillary temperature, 300 °C. Spectra typically correspond to the average of 20–35 scans, and were recorded in the range between 100 and 1000 Da. The more relevant ions that were identified in the full scan mass spectra were studied by collision induced dissociation (CID) to obtain the MS2 mass spectra of each precursor ion and its fragmentation path. Data acquisition and processing were performed using the software Xcalibur 2.2. Aliquots of 3 μL of the dye extracts were also analyzed on a UHPLC Elute system coupled on-line with a quadrupole time-of-flight Impact II mass spectrometer equipped with an ESI source (Bruker Daltoniks, Bremen, Germany). Chromatographic separation was carried out on a 150 mm × 2.1 mm, 2.6-μm particle size; Phenomenex column. The mobile phase consisted of water (A) and acetonitrile (B), containing 0.1% formic acid, at a flow rate of 350 μL/min, using the elution gradient above mentioned. The

column and the autosampler were maintained at 45 °C and 8 °C, respectively. High resolution mass spectra were acquired in the ESI negative mode. The mass spectrometric parameters were set as follows: end plate offset, 500V; capillary voltage, −2.5 kV; nebulizer, 4 bars; dry gas, 8 L/min; heater temperature, 200 °C. Internal calibration was achieved with an ammonium formate solution introduced to the ion source via a 20 μL loop at the beginning of each analysis, using a six-port valve. Acquisition was performed in full scan mode in the m/z 100–1000 range and in a data-dependent MS/MS mode with an acquisition rate of 5 Hz using a dynamic method with a fixed cycle time of 3, and an m/z dependent isolation window of 0.03 Da. Probable compounds were identified based on their accurate m/z values released as de(protonated) molecules [M-H]-/[MH]+, considering the accuracy and precision of measurement parameters such as error, (ppm) and mSigma. The molecular formula was validated by extracting ionic chromatograms from the raw data, and accurate masses, isotopic patterns and fragmentation paths were evaluated, supporting the respective proposed chemical structures. Data acquisition and processing were performed using the Data Analysis 5.1 software (Silva et al., 2022).

2.7. Analysis of plant remains

Phytoliths (Lab-002) and seed remains (Lab-004) preserved on the textile samples were studied after being observed under the Scanning Electron Microscope ZEISS EVO LS 15. Epidermal material examination focused on multicellular (anatomically connected or articulated) phytoliths from the textile samples described above. In the current study, different fragments of articulated phytoliths were examined per sample, from multiple SEM microphotographs. These mainly represent multicellular elongate dendritic phytoliths, still in their original anatomical origin. The sampling aimed to examine as much morphological variation as possible. Identifications were based on modern plant reference collections (Albert et al., 2008, 2016; Portillo et al., 2014) and standard literature (Twiss et al., 1969; Twiss 1992; Rosen 1992; Piperno 2006). The terms used to describe phytolith morphologies follow the standards of the International Code for Phytolith Nomenclature, ICPN 2.0 (Neumann et al., 2019). The seeds were taxonomically identified to species level by comparing the surface cell pattern of the preserved remains with materials deposited in the reference collections of the Archaeobotany Laboratory of the School of Archaeology of the University of Oxford and the Archaeobiology Laboratory of the Instituto de Historia, Spanish National Research Council (CCHS–CSIC), as well as specialised literature (Nasu et al., 2007; Lu et al., 2009, Zhang et al., 2011).

2.8. Identification of insect remains

During the process of unfolding one of the water saturated samples (Lab-001-001) insect remains were recovered, which were identified from the SEM microphotographs and the photo from the stereomicroscope using entomological keys.

3. Results

3.1. Set of organic textiles

All the organic fabrics made of wool appeared as separated scraps that were wrinkled and folded (Fig. 3, Table 1). The scales in the majority of fibres were clearly visible under the SEM although evidence of degradation was also observed; some of the fibres were brittle, sometimes broken and partly without scales (Fig. 4). The state of preservation

Fig. 3. Set of organic textiles (scale 1 cm).

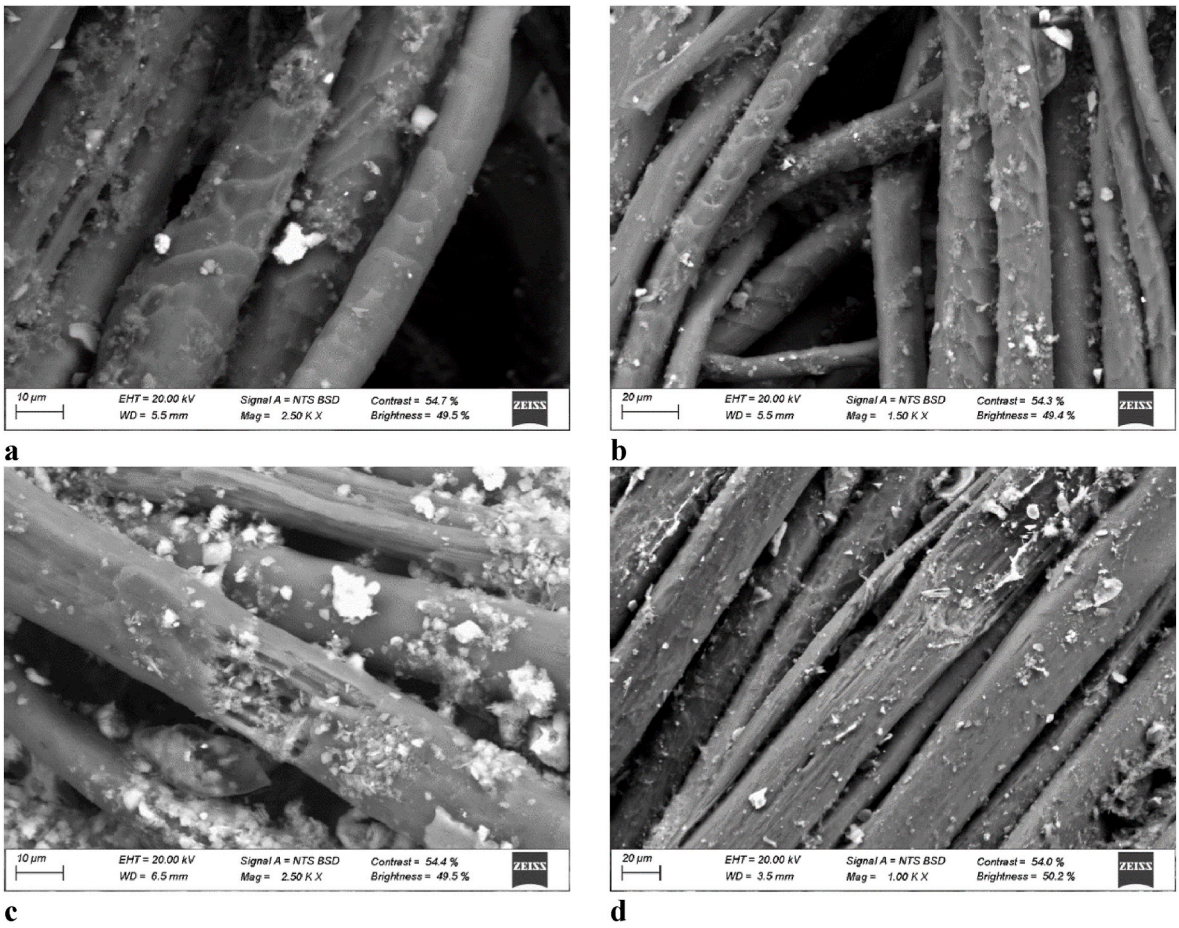

Fig. 4. SEM images of the wool fibres. Detail of the scales: a) and b) (Lab-004-002); evidence of degradation; c) Lab-005-002, and d) Lab-002.

of the fabrics was fragmentary, no edges and selvedges were preserved, so system 1 and 2 was used for describing the technical characteristics of the textiles. Their current colour is brown-reddish due to depositional and post-depositional conditions, althoughthis was not the original colour of the fabric. All of them were woven in tabby (plain weave) with single yarn. The number of threads per cm was measured in six samples. This kind of fabric with 5–10 threads per cm can be regarded as medium-fine. Two groups of clothes could be separated based on the number of threads per square cm, diameter and twist direction of the yarn (Figs. 5 and 6). The first group is comprised by samples Lab-001-001 and Lab-001-002, which appeared together, and the second group includes samples Lab-002, Lab-003, Lab-004-001 and Lab-005-002. The state of preservation of the yarns and the dirt stuck to the fabrics in samples Lab-004-002 (Fig. 5f) and Lab-005-001 (Fig. 5g) prevented us from including them in any group because it was not possible to observe clearly the number of threads per cm, the diameter of the yarns and their twist direction. Combed thread with changing scale direction was identified in Lab-002 and Lab-003, this feature is related to the kind of combing during wool processing (Rast-Eicher 2016: 43, Grömer 2016). Lab-001 was radiocarbon dated in a period ranging between the 15th and 17th centuries CE (Beta-522819, 340 ± 30 BP, 1470–1640 cal CE, 95.4%).

Using Py-GC-MS, the samples of wool generated a wide range of nitrogen-containing pyrolysis products, such as pyrroles, pyridines, acetamide, indoles, cyanobenzenes, piperazinediones and diketopiperazines (dimerization products) such as diketodipyrrole, from the keratin polymer (Fig. 7, Supplementary Material). The sum of N-compounds (average and SD of six samples) accounts for 72.7 ± 1.0 of TQPA, but if N-lacking compounds that can also originate from proteinaceous matter such as toluene, methylthiophene, methylphenols and unidentified products are included, this sum increases to 96.3 ± 1.2 %, clearly showing the prevailing proteinaceous fabric. The methylthiophene may reflect cysteine moieties in the keratin polymer. Acetamide is difficult to explain in these samples. It is a product of chitin, and may therefore be related to the exoskeleton of weevil, but a secondary rearrangement of proteins may also be involved. The "exogenous" compounds detected are fatty acids (0.5 ± 0.4 %), of many possible sources, and three products of lignin (guaiacol, 4-vinylguaiacol and syringol; 3.2 ± 0.9 %), probably from plant materials. These compounds may explain the dark appearance of the textiles. There were no major differences in pyrolysis fingerprints between the samples that can be used to describe differences in sources. Suffice it to mention that 1) the abundance of lignin products (possibly indicating incorporation of plant materials after the textiles were discarded) decreases in the order Lab-001 > Lab-003 > Lab-002 & Lab-004-001 > Lab-004-002 & Lab-005, whereas the ratio of diketopiperazines to other N-compounds decreased in the similar order Lab-001 & Lab-003 > Lab-004-002 > Lab-002 & Lab-004-001 > Lab-005. This latter ratio may indicate the degree of protein preservation (Kaal et al., 2016), but further proof of this relationship is required.

With THM-GC-MS, a large suite of N-containing products were detected, such as the methyl esters (ME) of proline, alanine, leucine and other amino acids, and products such as methylated pyrrolidinediones, indoles and unidentified N-containing products. The lignin is clearly identified by 3,4-di- and 3,4,5-trimethoxybenzenes, even though some of these compounds may originate from proteins as well (such as the THM product of tyrosine, which is the same product as that of *p*-coumaric acid). The products of lignin are again relatively abundant in sample Lab-001. THM-GC-MS showed a wider variety of fatty acids (detected as fatty acid MEs, or FAMEs, with chain length ranges between C_{14} and C_{32}). These FAMEs originate from plant materials such as epicuticular waxes (especially the C_{22}–C_{32} FAMEs) but also bacteria (*iso*-

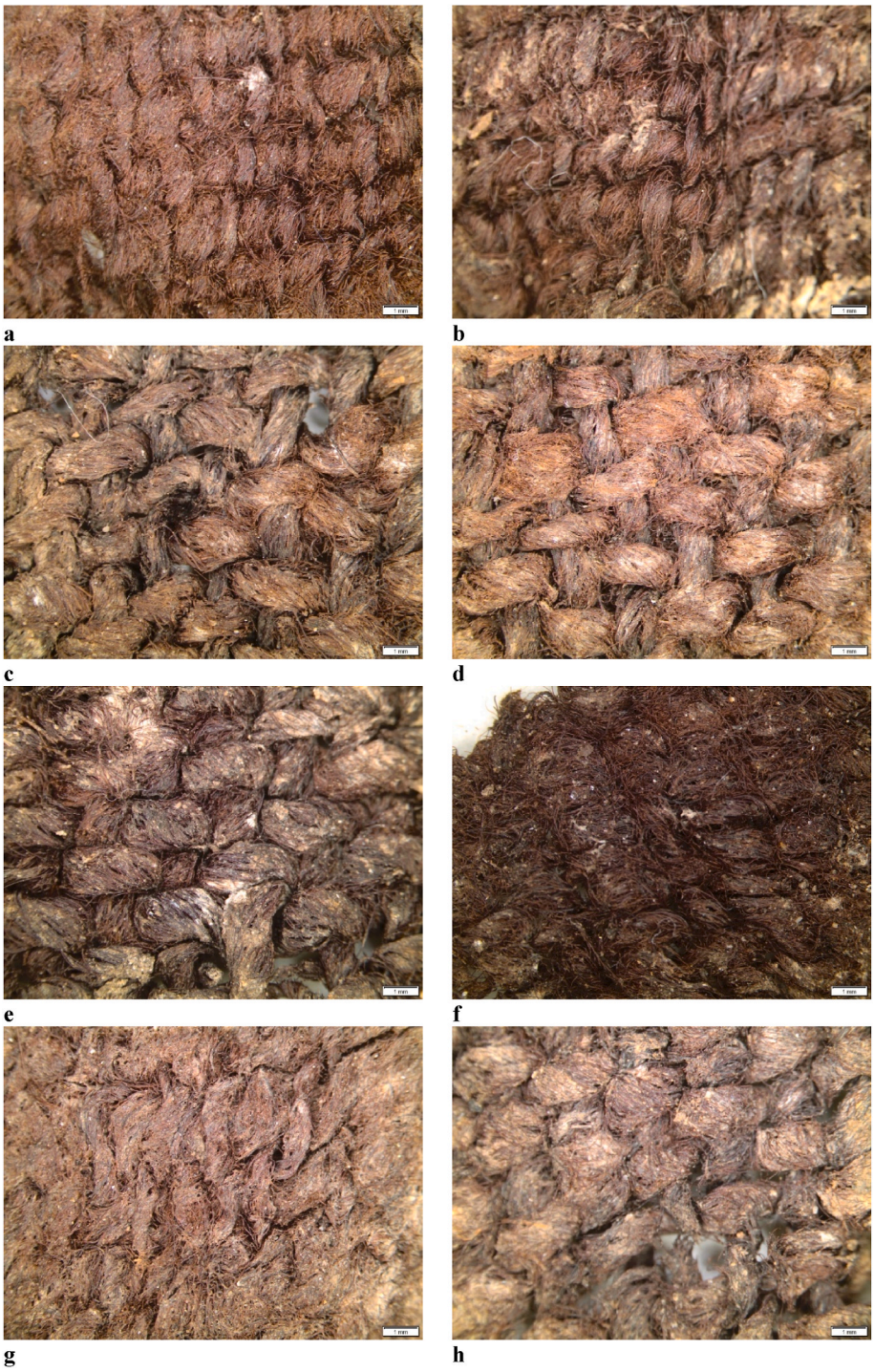

Fig. 5. Photographs of wool fabrics using stereoscopic microscopy: a) Lab-001-001, b) Lab-001-002, c) Lab-002, d) Lab-003, e) Lab-004-001, f) Lab-004-002, g) Lab-005-001, and h) Lab-005-002.

and *anteiso*-C_{15} FAME). Probably the most remarkable finding are the series of compounds with main ion fragments *m/z* 255, 287 and 370, and M^+ 402. These products should probably be ascribed to dimethyl ethers of deoxycholic acid ME, or fragments *m/z* 255, 273 and 370 from deoxycholic acid ME (identification with NIST mass spectral library). These compounds, which may indicate bile acids (excremental/faecal fabric), were most abundant in sample Lab-004-002 (0.9 %), followed by Lab-004-001 (0.5 %), Lab-002 and Lab-003 (0.2 %), and Lab-001 and Lab-005 (<0.1 %). None of the compounds could be ascribed to dyes or pigments.

Unfortunately, the HPLC-DAD-MS and UHPLC-HRMS/MS analyses were not conclusive. The extracts presented a small peak with an absorption band around 260 nm, suggesting that no natural yellow/red chromophores were present. In fact, the main classes of yellow chromophores have two characteristic I and II bands in the UV spectra that were not detected in our samples, ranging from 310 to 390 nm and 240–270 nm, respectively.

The absorption of tannins occurs in the aromatic characteristic range. Considering that the aromatic rings were the main chromophore groups detected and given the brownish hue observed in the sample

Fig. 6. Twist direction of the yarns. From left to right, Lab-001-001 and Lab-004-001.

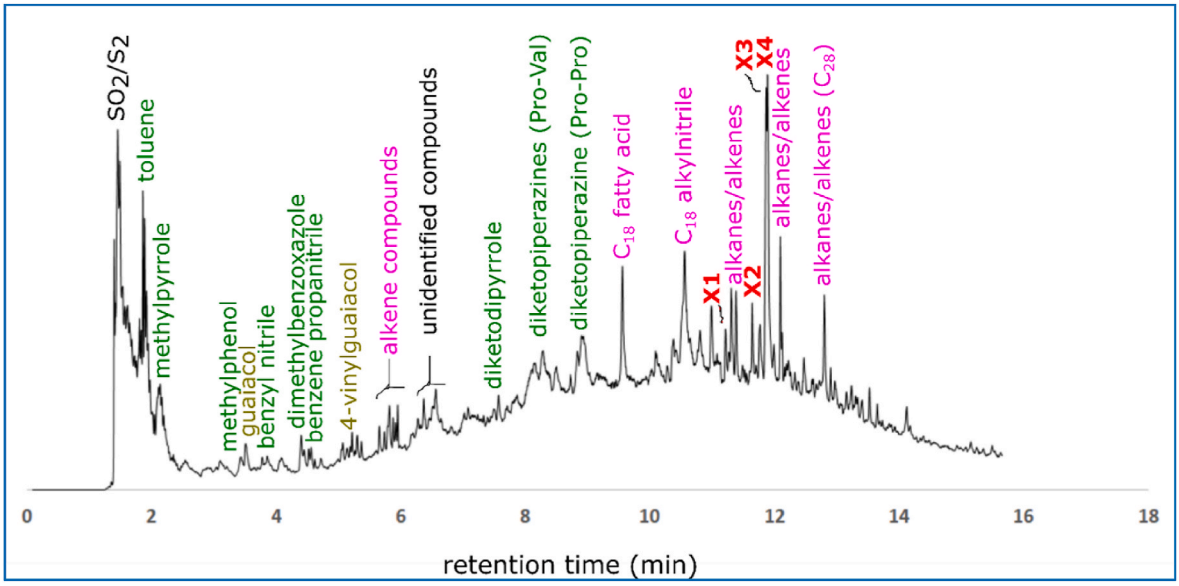

Fig. 7. Plot with the Py-GC-MS results.

yarns, we postulated that the fibers may have been dyed using Sumac species (*Rhus* spp.), oak leaves, or grapes, all of which have elevated tannin levels and are native to Europe. Nonetheless, given the samples' age and storage conditions, it is anticipated that only signals linked to the decomposition products, particularly small organic molecules, would be discerned in the extracts. The LC-DAD-LRMS results provided an overview of the primary signals within the samples, yet the data does not allow the identification of the compounds present nor to propose

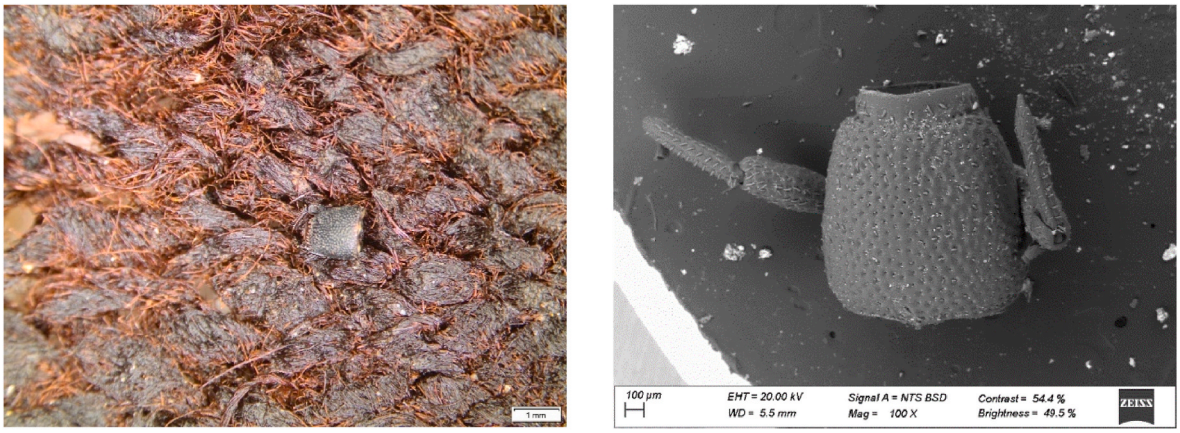

Fig. 8. From left to right. Cossoninae weevil under stereoscopic microscopy and SEM.

molecular formulas. Unfortunately, the UHPLC-HRMS/MS analyses were also not conclusive, not allowing the identification of the dye used in fiber colouring. Given the apparent state of degradation of the fibers, small organic molecules characteristic of the degradation products of flavonoids or tannins should be present. However, we were not able to identify neither benzoic nor gallic acids, which are characteristic of the degradation of flavonoids and tannins, respectively.

During the study of Lab-001-001 a pronotum of a beetle was recovered, with its legs still attached (Fig. 8). The pronotum belonged to a cossonine weevil, *Mesites pallidipennis* Bohe. This species is broadly distributed in the Mediterranean littoral zone from the Iberian peninsula to the Balkans, Egypt and Lebanon (Alonso-Zarazaga and Lyal, 1999). It is a stenotopic species associated to pieces of wood and trunks deposited on the beaches by the waves, driftwood of willows and poplars, and sometimes conifers in the littoral zone (Folwaczny 1973, Hoffmann, 1954). There are additional fossil records from mid-Holocene deposits on Corsica (Poher et al., 2016), Roman Fréjus (Ponel 2011) and Post-Medieval Marseille (Ponel et al., 2014).

Furthermore, in the adherences associated with Lab-002, multicellular grass phytoliths (articulated or anatomically connected) were recorded (Fig. 9). The phytolith morphological observations point towards a general similarity between all the examined assemblages,

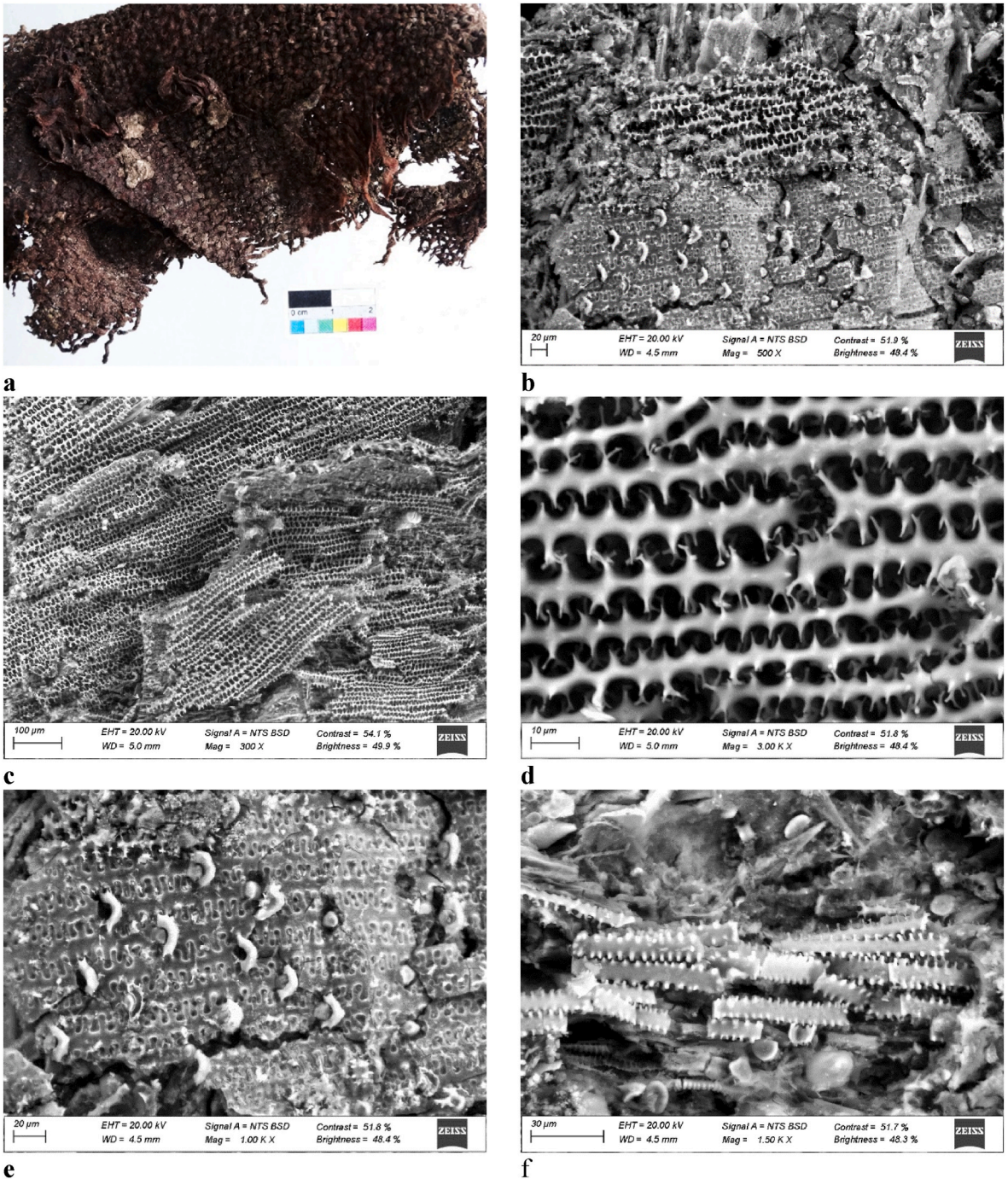

Fig. 9. a) Textile fragment folded with adherences (Lab-002). Photomicrographs under SEM of plant remains adhering to the fabric: b) c) d) and e) articulated multicellular elongate dendritic grass phytoliths, f) multicellular elongate dendritic phytoliths with articulated epidermal appendages (papillate and acute bulbosus) from grass inflorescences.

dominated by grass epidermal material, particularly from the Pooideae subfamily according to the grass silica short cell morphologies (Twiss et al., 1969; Twiss 1992). Grass inflorescences were characterized by articulated elongate phytoliths with decorated margins (mainly dendritics) in addition to epidermal cells such as papillate and acute bulbosus (silicified hairs, Fig. 9f). The latter morphologies which are considered as delicate or fragile cells, in association with the dominance of multicelled or anatomically interconnected dendritics, indicate a general good state of preservation of the phytolith records (Cabanes et al., 2011; Portillo et al., 2021).

In sample Lab-004, a seed of the spikelet was found and the cell decoration pattern allowed to identify the remain as a foxtail millet (*Setaria italica* (L.) P.Beauv.) grain (Fig. 10). Foxtail millet was a widespread crop in the northern regions of the Iberian Peninsula during the Middle and Modern Ages, but its cultivation was less common than other cereals (Peña-Chocarro et al., 2019; Teira-Brión 2022; Teira-Brión et al., 2023). Ethnographic data suggests that foxtail millet could be grown mainly for animal breeding, as fodder for livestock or poultry feed (Teira-Brión 2022).

3.2. Textile pseudomorphs

Physical traces of fabrics were preserved in contact to metal plates (Fig. 11). Based on the clearly visible quite thick nodes, the pseudomorphs were classified as bast fibres (Table 2, Fig. 12), although chemically the EDX analysis indicates that they were almost completely mineral-replaced by Fe corrosion products (Table 3). As the replacement of the organic matter of these fibres was almost complete, we have classified them as pseudomorphs (Margariti 2019b: 14). They were originally fabrics made of flax or hemp, but distinguishing between both fibres is difficult when they have been mineral-replaced, even under the SEM (Grömer 2016). According to the diameter of the fibres -less than 10 up to 20 microns- and their hexagonal and oval cross sections, their identification as flax is more probable than hemp (Rast-Eicher 2016: 90), but there is still a high degree of uncertainty. All the textiles were woven in tabby with single yarn with spinning -Z (Table 2, Fig. 15). The number of threads per cm was measured in seven samples. The yarn diameter and threads per cm are quite similar between the different samples, only in 1292 the yarn diameter is clearly thinner than in the other textiles. Despite this, the measurements might not be the original ones, because metal oxides could cause an increase of volume of the threads (Figs. 13 and 14).

The cellulosic cloth analyses (Py-GC-MS only) were supported by the analysis of reference materials of flax, hemp and nettle, and samples 1130 and 007. As expected, the reference materials and samples produced mainly furans, pyrans and anhydrosugars upon pyrolysis, reflecting the prevailing cellulose component and thus the plant origin of the textiles. Of the reference samples, the flax sample had a stronger lignin fingerprint than hemp and nettle. In terms of the relative intensities of the polysaccharide products (qualitative observations), the resemblance of sample 7 is stronger for flax and hemp than for nettle, and perhaps the signal is slightly more similar to that of hemp than of flax. For sample 1130, the mineral load (degree of substitution of organic for inorganic constituents) is too high to allow for an adequate analysis directly, as shown by the poor resolution of the chromatogram. We intended to intensify the signal by dissolving the inorganic phases in 1M HCl solution (followed by centrifuging-decantation-rinsing cycles with distilled water), but in spite of emergence of some furaldehydes from the polysaccharides, the signals were still poor. We did not identify any compound that could be ascribed to dyes or other sources, including

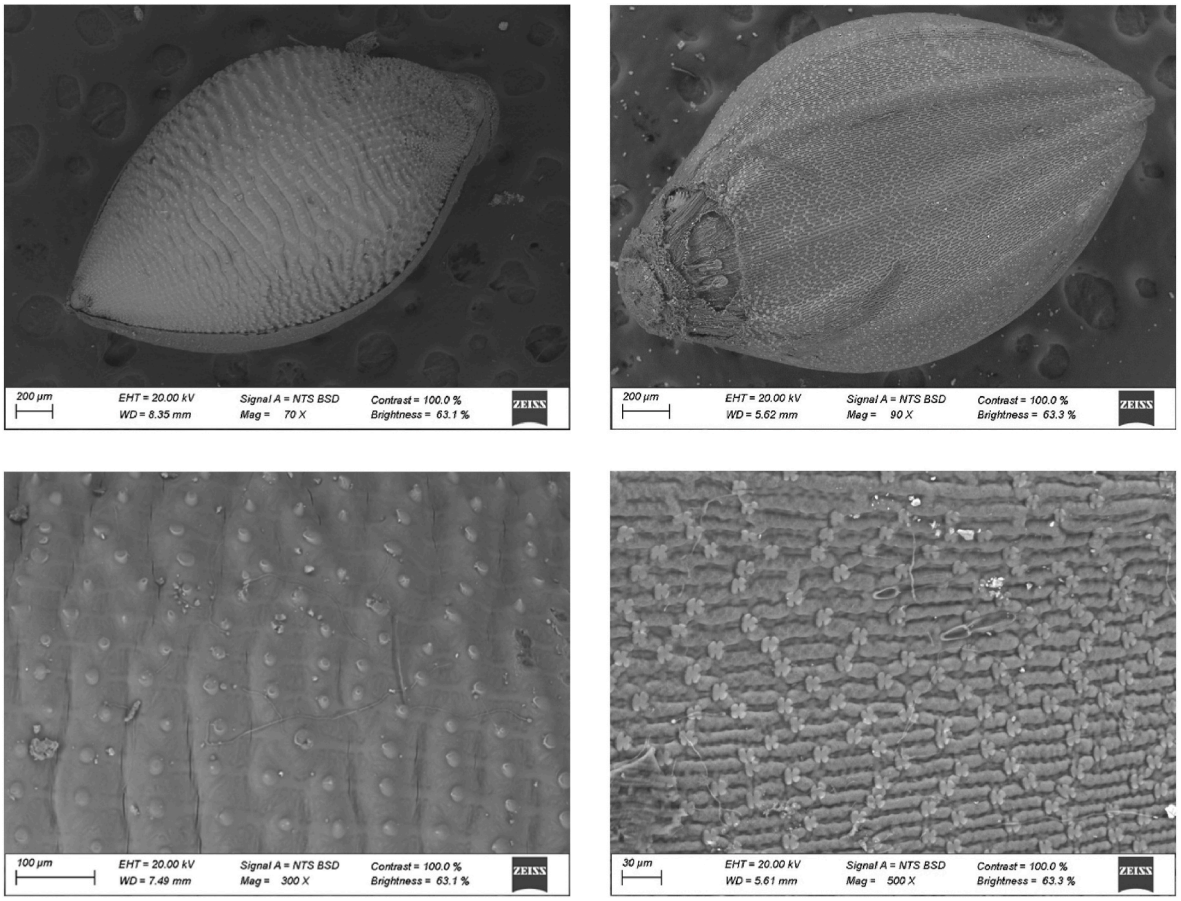

Fig. 10. Photographs under SEM of foxtail millet remains recovered from one of the wool textiles (Lab-004-001).

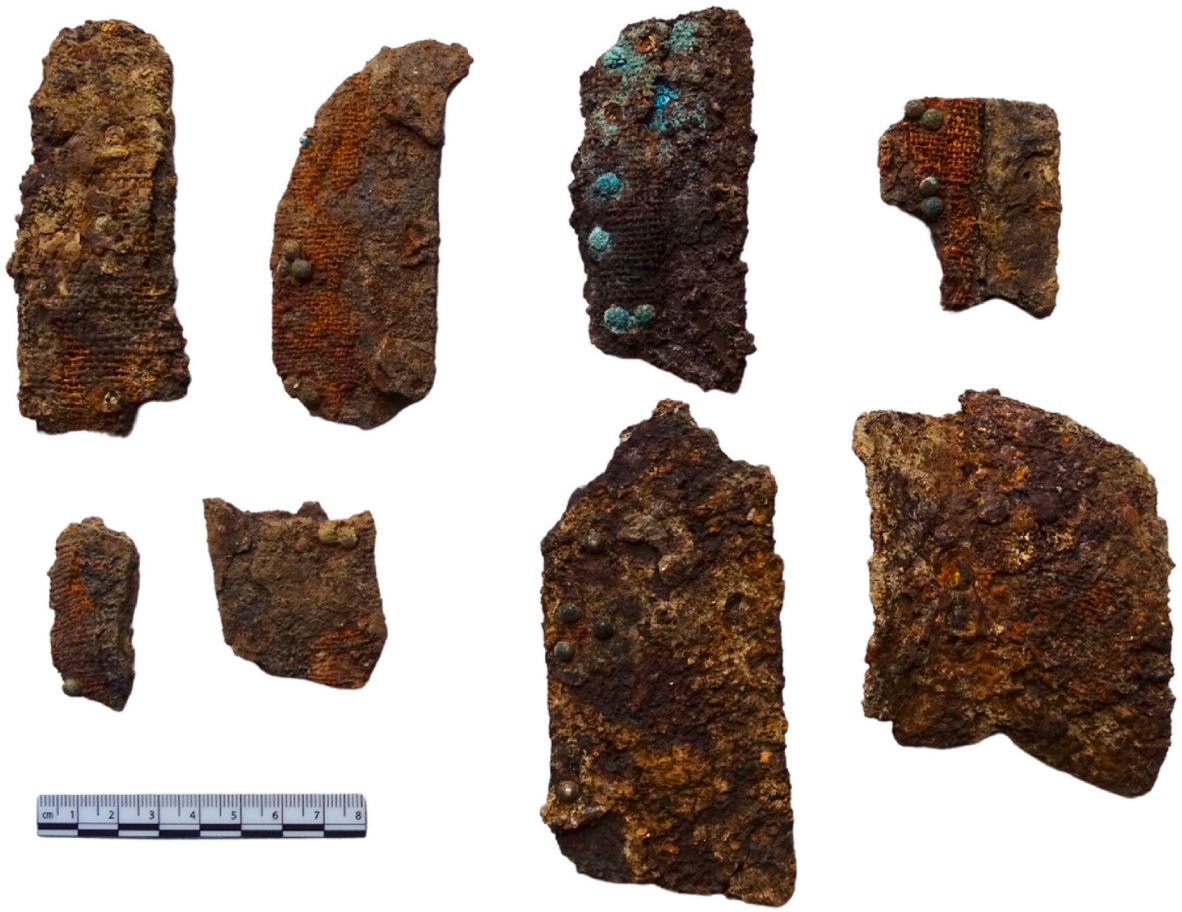

Fig. 11. Set of textile pseudomorphs.

compounds of the soil environment.

4. Discussion

4.1. Wool fabrics

The set of wool fabrics provide a glimpse to clothing in the transition between the Late Middle Ages and the beginning of the Early Modern period in Northwest Iberia. All of them were simplest weave, tabby with single yarn. Despite their fragmentary state of preservation, two groups of fabrics were differentiated according to the number of threads per square cm, diameter and twist direction of the yarn. The samples Lab-001-001 and Lab-001-002 with thinner yarns than the rest of the set, showed mixed spinning -Z-spun system 1, S-spun system 2- where the fibres all lie in the same direction when woven, which gives rise to a firm cloth (Crowfoot et al., 1992). No edges and selvedges were preserved, and no evidence of stitching was identified, making it impossible to identify the loom type, the original size and morphology of the fabrics, and preventing us from assessing their initial function. The brown-reddish appearance of the Pambre textiles was probably caused by the humic acids in the soil that cause cloths to become permanently stained in various shades of brown, losing their original colourfulness; wool textiles are more prone to be affected by this action (Crowfoot et al., 1992; Cybulska and Maik 2007). As no distinctive signs of chemical markers of dyes were identified, it was not possible to determine their original colour.

In the case of the woolen textiles of Pambre it is challenging to ascertain the complete life-cycle that these textiles may have undergone before they were disposed inside the tower. Textiles have a considerable reuse value, beyond their original function and extremely complex life-cycles. They can be handed down (Crowfoot et al., 1992), they might be cut up, patched, darned, shortened or otherwise refashioned for other wearers, and finally thrown away in refuse pits or even cesspits (Coatsworth and Owen-Crocker 2007). The set of wool textiles of Pambre were subjected to successive alterations before ending up inside the tower. They were used, weared, shortened from larger fabrics, and then thrown away inside the bottom of the tower accompanied by different kind of organic debris including different types of plant materials and fecal material identified by the presence of bile acids in variable concentrations in the different wool samples. This may imply that at some time the lower part of the tower was used for defecation. A key question is to assess the defecators, and whether these fecal materials originated from human or animal sources. Further faecal biomarkers, integrating both bile acids and fecal sterols may allow to distinguish between human, porcine, herbivore, or carnivore sources (e.g. Bull et al., 2002; Blong and Shillito, 2021). The presence of small sections of cloth in Medieval cess/rubbish pits has been previously attested (Greig 1981: 275–276) and interpreted as the equivalent to toilet paper (Smith 2013). Microcontextual studies at high-resolution integrating soil micromorphology, plant macroremains, phytoliths and palynological records of deposits from Medieval castles have revealed herbivore dung accumulations, indicative of changes in animal management, whereabouts within the castles the animals were stabled, and livestock alimentation, including millets (e.g. Banerjea et al., 2019, 2020). In the current study, it is not possible to assess the origin of the Poaceae phytoliths and the foxtail millet grain adhering to the textile fabrics as human or animal dung components.

The pronotum of *M. pallidipennis* recovered with wool fabric Lab-001-001 is difficult to explain. It may be purely incidental, having been caught up in someone's clothing on a visit to the shore or brought

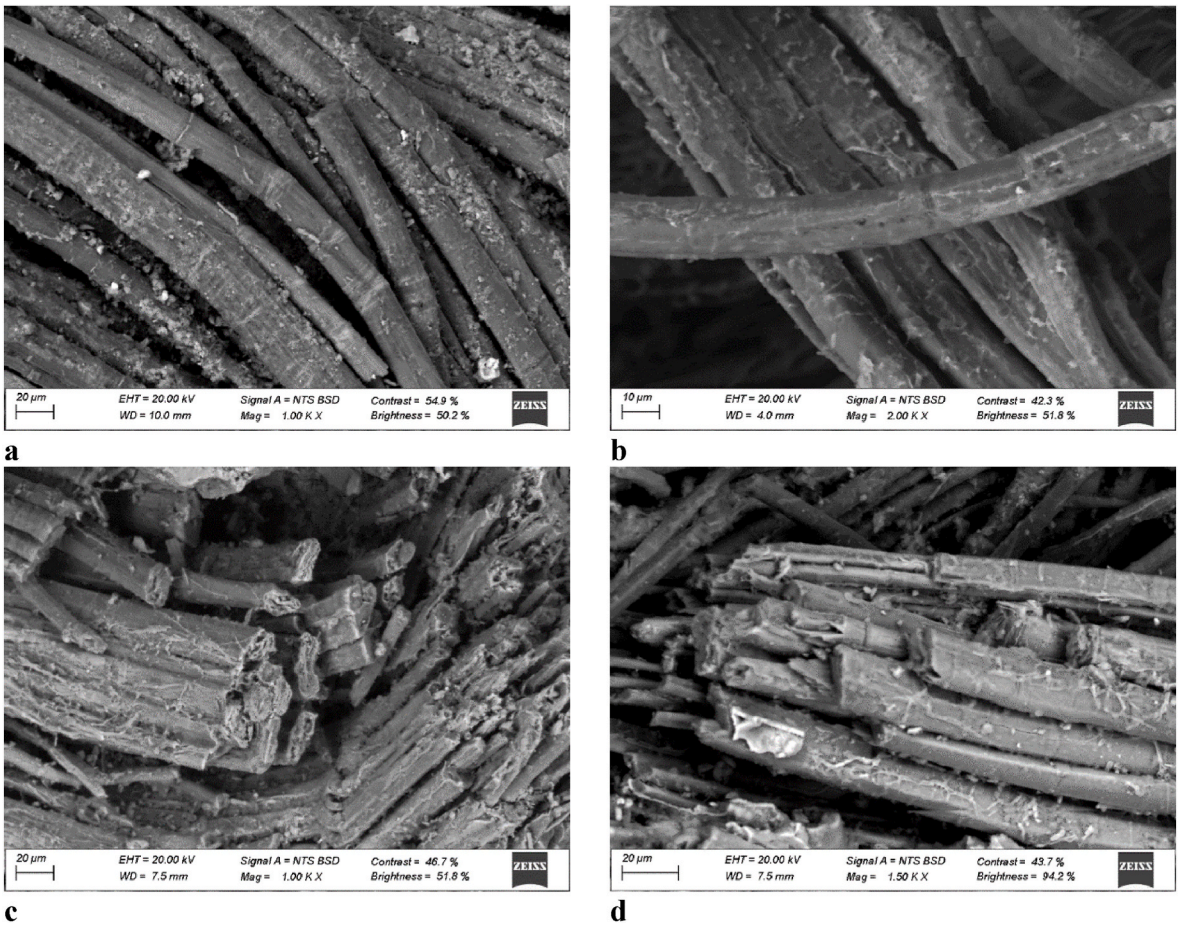

Fig. 12. SEM images of mineral preserved fibres. Surface of fibres: a) Sample 1295, b) Sample 1291. Cross-section of fibres: c) Sample 7, d) Sample 1292.

Table 3
Elemental composition of a pseudomorph from Sample 1130 obtained through EDX analysis.

Element	Weight %	Atomic %
C K	12.41	26.14
O K	29.11	46.00
Al K	0.49	0.46
Si K	0.36	0.32
P K	2.66	2.17
Cl K	0.24	0.17
Fe K	54.01	24.45
Cu K	0.72	0.29
Total	100.00	100.00

by someone or on something arriving from a coastal area to the castle. This fortress was a site where people and commodities arriving from distant places met, as part of the political and social networks established by the Ulloa, but also in relation to the movement of people to pay tributes, merchants or pilgrims, for example (Rouco 2017). Another explanation of the presence of this weevil might be in association to driftwood brought together with marine fish and molluscs transported from the coastal areas to the castle. The presence of marine resources has been attested by zooarchaeological analysis (González-Gómez De Agüero et al., 2021). Despite no clear evidence of wood decay (that might be related to driftwood), *Salix/Populus* was identified in the anthracological record (Martín-Seijo and Vázquez Collazo 2020), and it is possible that wood could have been thrown away with other garbage.

4.2. Brigandines

The set of metallic plaques studied in Pambre has been identified as part of one or, more likely, several brigandines. The brigandine was a form of fully articulated body armour introduced around the 14th century CE which superseded the coats of plates (La Rocca 2017). It was typical of foot soldiers, but its use spread even among the wealthy aristocrats. It was a light, vest-like body defence made of multiple small iron or steel plates attached to a cloth covering (dos Reis 1961; González Castañón, 2014). The overlapping plates remained frequently hidden under leather or cloth, with only the gilded nails being visible (Grancsay 1931, 1950; Barroca et al., 2000; Agostinho 2013). The plates were riveted to a sturdy fabric, such as canvas, usually faced to an outer layer of finer material: leather, silk or velvet (Agostinho 2013; La Rocca 2017). The external fabric was also related to the use of its colour to identify the knight in front of his companions and enemies on the battlefield (Agostinho, 2013). The rivets could be single or triangular clusters of three, and in addition to being decorative, they were also functional in fixing the plates to the fabric layers (La Rocca 2017). The presence of organic remains over brigantine's plaques has been reported sporadically (Amici 1989: 462, Scalini 2003: 382–383). Brigandines appear to have been worn in great numbers, judging from contemporary pictures (Fig. 16), and we can understand that they formed flexible and strong defenses, better in many ways than chain-mail.

There are very few examples of brigantine plaques recovered at archaeological contexts of the Iberian Peninsula (González Castañón, 2014) and these from Pambre are the first of them related to a detailed study of associated organic remains. In the case of the Pambre set of pseudomorphs, the textiles preserved were part of the inner cloth between the plaque and the outer layer. Only the textile in contact with the

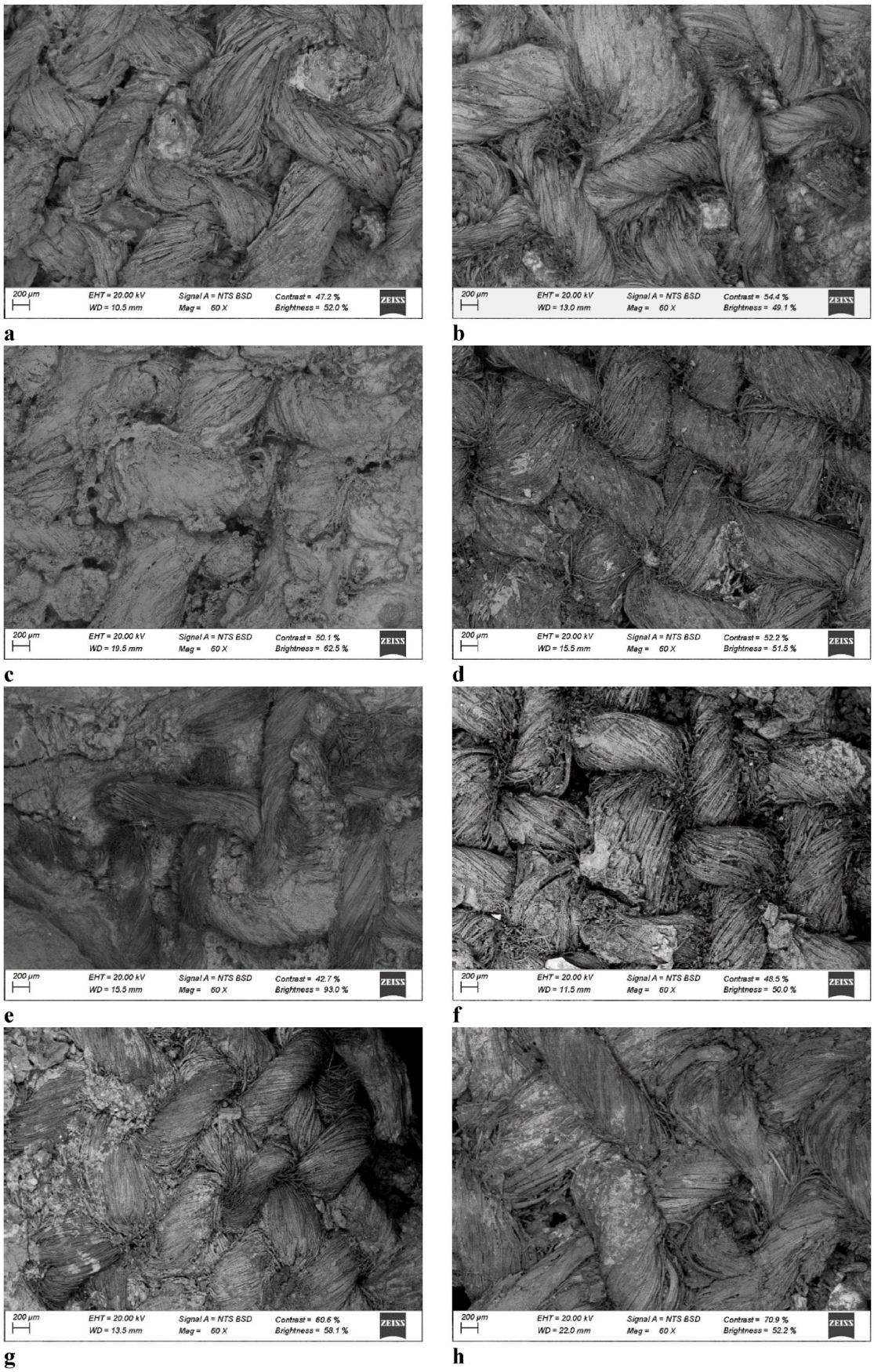

Fig. 13. SEM images of mineral replaced fabrics: a) Sample 7, b) Sample 1130, c) Sample 1290, d) Sample 1291, e) Sample 1292, f) Sample 1293, g) Sample 1294, and h) Sample 1295.

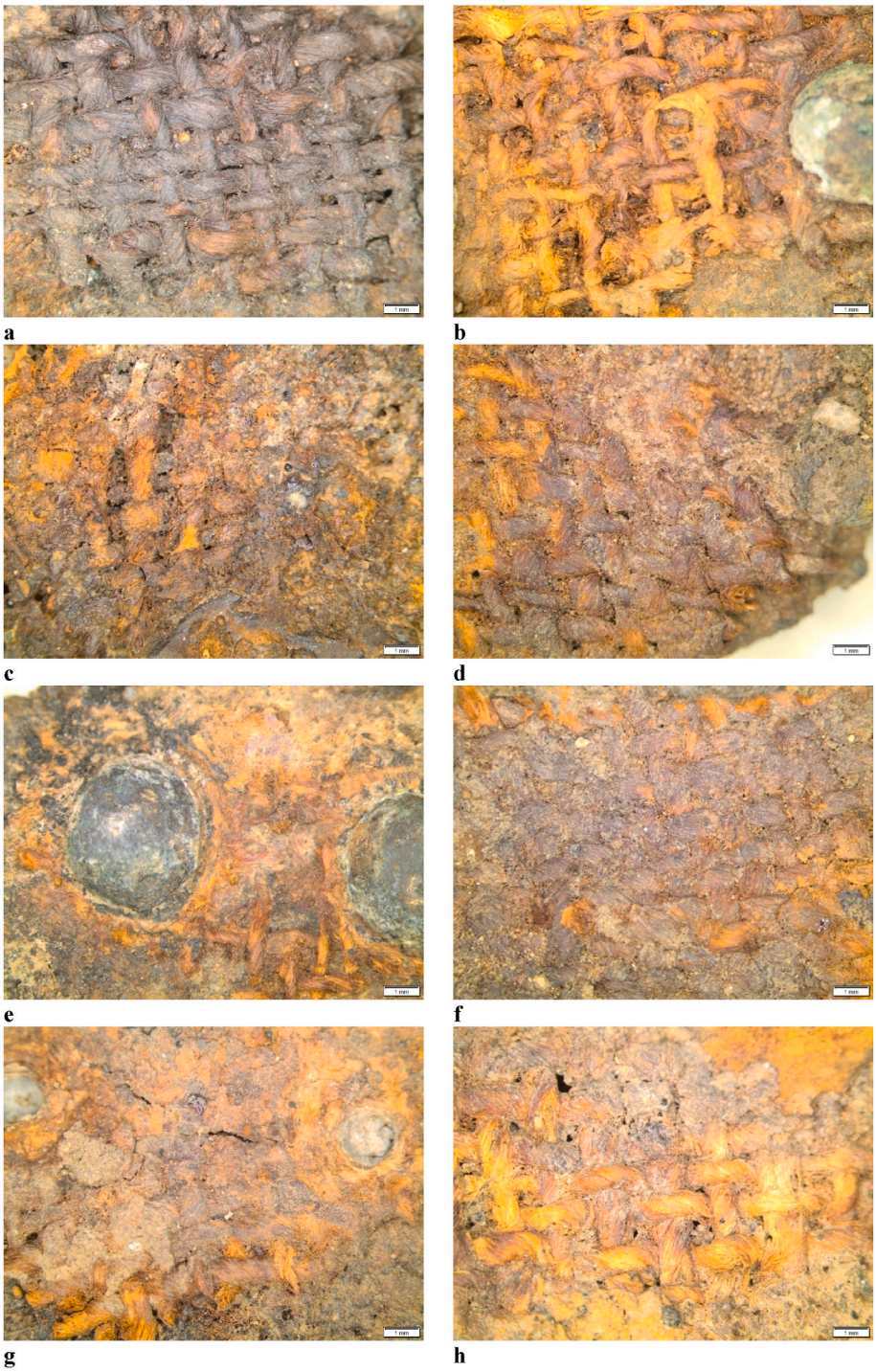

Fig. 14. Stereoscopic microscopy images of pseudomorphs: a) Sample 7, b) Sample 1130, c) Sample 1290, d) Sample 1291, e) Sample 1292, f) Sample 1293, g) Sample 1294, and h) Sample 1295.

metallic plate was preserved. It was probably a canvas made of bast fibres -flax or hemp-woven as tabby tissue. No evidence of the outer textile layer was preserved, even under the metallic rivets. The external appearance of the plaques and the distribution patterns of the rivets is not the same in the different samples. This might be related to different patterns through the brigandine or to different brigandines. Their presence on a restricted area suggests that they were abandoned or discarded probably once this castle lost its military function. This exceptional finding provides a unique glimpse to the body armour.

5. Conclusion

The interdisciplinary study of the exceptional set of textile remains from the Pambre Castle has revealed new data about clothing and the complex biographies of textiles in the transition between the Late Middle Ages and the Early Modern period. In the case of the wool fabrics, despite their fragmentary state, it was possible to propose a reconstruction of their complex life-cycle including successive alterations, shortening, and eventual disposal inside the tower, accompanied by various organic debris, including plant materials and fecal matter. Despite the challenging nature of the analysis, the data provided by the

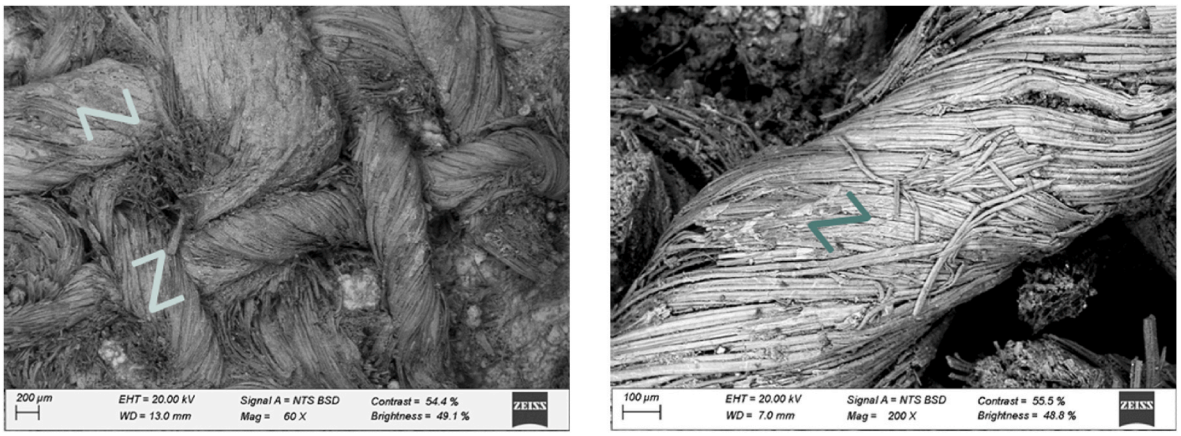

Fig. 15. Twist direction of the yarns in Sample 1295.

Fig. 16. Paintings of the Massacre of the Innocents; Medieval paintings at the Mondoñedo Cathedral (Lugo, Spain) (CC-BY-SA-4.0).

fabrics points to a use of the tower as a cess/rubbish area where human or animal dung was discarded. The unexpected presence of a pronotum of *Mesites pallidipennis* Bohe. adds a layer of complexity, with potential explanations ranging from incidental transportation to associations with goods or people arriving from coastal areas. Concerning the study of the metallic pseudomorphs -interpreted as part of brigandines-the Pambre archaeological assemblage is the most exhaustively studied example of this form of body armor which was common from the 14th century CE onwards. This exceptional finding offers a rare glimpse into this kind of body armor in Southern Europe.

From a methodological point of view this study stresses the relevance of combining the technological analysis of the objects themselves with other methods and integrating multiple proxies to address a biographical approach. Although, some of the methods applied did not provide conclusive results, for example on the molecular characterization of the identification of the dyestuff and subsequently on the original color of the woolen clothes, the methodological program designed might produce results in other organic remains in a less advanced stage of degradation of the chemical compounds. This research has highlighted that embedded in the fabrics themselves is rich evidence of the

extremely complex life-cycles of textiles in the transition between the Medieval and the Modern period that can only be reconstructed using an interdisciplinary methodological approach.

CRediT authorship contribution statement

María Martín Seijo: Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Joeri Kaal:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **César Oliveira:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Marta Portillo:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Eva Panagiotakopulu:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Andrés Teira Brión:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **M. Conceição Oliveira:** Methodology, Investigation. **Santiago Vázquez Collazo:** Writing – review & editing, Resources, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jas.2024.105974.

References

Adamiano, A., Fabbri, D., Falini, G., Belcastro, M.G., 2013. A complementary approach using analytical pyrolysis to evaluate collagen degradation and mineral fossilization in archaeological bones: the case study of Vicenne-Campochiaro necropolis (Italy). J. Anal. Appl. Pyrol. 100, 173–180.

Agostinho, P.J.S., 2013. Vestidos para matar: o armamento de guerra na cronística portuguesa de quatrocentos, vol. 2013. Imprensa da Universidade de Coimbra/Coimbra University Press.

Albert, R.M., Shahack-Gross, R., Cabanes, D., Gilboa, A., Lev-Yadun, S., Portillo, M., Sharon, I., Boaretto, E., Weiner, S., 2008. Phytolith-rich layers from the late bronze and iron ages at tel dor (Israel): mode of formation and archaeological significance. J. Archaeol. Sci. 35, 57–75.

Albert, R.M., Ruiz, J.A., Sans, A., 2016. PhytCore ODB: a new tool to improve efficiency in the management and exchange of information on phytoliths. J. Archaeol. Sci. 68, 98–105.

Alonso-Zarazaga, M.A., Lyal, C.H.C., 1999. A World Catalogue of Families and Genera of Curculionoidea (Insecta: Coleoptera) (Excepting Scolytidae and Platypodidae). Barcelona, Entomopraxis S. C. P.

Amici, S., 1989. I reperti metallici e non metallici delle campagne di scavo 1983-1984. Archeol. Mediev. 16, 460–479.

Aragonés, E., 1999. La moda medieval navarra: siglos XII, XIII y XIV. Cuad. etnol. Etnogr. Navar. 31 (74), 521–562.

Banerjea, R.Y., García-Contreras Ruiz, G., Kalniņš, G., Karczewski, M., Pluskowski, A., Valk, H., Brown, A.D., 2019. Geoarchaeology and castlescapes: heritage management case studies in Spain and the eastern baltic. Landscapes 20 (2), 178–201.

Banerjea, R.Y., Badura, M., Brown, A., Morandi, L.F., Marcinkowski, M., Valk, H., Ismail-Meyer, K.M., Pluskowski, A., 2020. Feeding the crusades: Archaeobotany, animal husbandry and livestock alimentation on the baltic frontier. Environ. Archaeol. 25 (2), 135–150.

Barral Rivadulla, M.D., 2023. Pasarelas Medievales en Galicia. Sobre la moda, ropas y complementos como objeto de estudio. In: Teixeira da Conceição, R.F., Ibáñez Beltrán, L.M., Negri, M., Pérez Valiño, A. (Eds.), *El Camino del Medievalista. Nuevos Trabajos en Estudios Medievales. E fazian fondamentos*. Santiago de Compostela. Universidade de Santiago de Compostela, pp. 189–207.

Barroca, M.J., Gouveia Monteiro, J., Soler del Campo, A., 2000. Pera Guerrejar: Armamento Medieval No Espaço Português, vol. 2000. Câmara Municipal de Palmela.

Bender Jørgensen, L., Grömer, K., 2012. The Archaeology of Textiles–Recent advances and new methods. Portal: godišnjak Hrvatskoga restauratorskog zavoda (3), 45–68.

Bernis, C., 1957. Indumentaria española del siglo XV: la camisa de mujer. Arch. Español Arte 30 (119), 187.

Bernis, C., 1978. Trajes y modas en la España de los Reyes Católicos. Instituto Diego Velázquez.

Blong, J.C., Shillito, L.M., 2021. Coprolite research: archaeological and paleoenvironmental potentials. Archaeological and Anthropological Sciences 13, 15.

Borrego, P., 2008. Indumentaria de Teresa gil. Convento de Sancti spiritus. Toro (zamora). Boletín de Instituto de Patrimonio Cultural de España 2, 9.

Borrego, P., Sanz, E., Pérez, P.P., Albar, A., Partearroyo, C., Arteaga, Á., Marras, S., Roquero, A., 2017. Caracterización de materiales y análisis técnico de tejidos medievales. Ge-conservación 12, 6–30.

Bull, I.D., Lockheart, M., Elhmmali, M., Roberts, D.J., Evershed, R.P., 2002. The origin of faeces by means of biomarker detection. Environ. Int. 27, 647–654.

Cabanes, D., Weiner, S., Shahack-Gross, R., 2011. Stability of phytoliths in the archaeological record: a dissolution study of modern and fossil phytoliths. J. Archaeol. Sci. (9), 2480–2490.

Cersoy, S., Daheur, G., Zazzo, A., Zirah, S., Sablier, M., 2018. Pyrolysis comprehensive gas chromatography and mass spectrometry: a new tool to assess the purity of ancient collagen prior to radiocarbon dating. Anal. Chim. Acta 1041, 131–145.

Challinor, J.M., 1989. A pyrolysis-derivatisation-gas chromatography technique for the structural elucidation of some synthetic polymers. J. Anal. Appl. Pyrol. 16, 323–333.

Chen, H.L., Jakes, K.A., Foreman, D.W., 1998. Preservation of archaeological textiles through fibre mineralization. J. Archaeol. Sci. 25 (10), 1015–1021.

Coatsworth, E., Owen-Crocker, G.R., 2007. Medieval Textiles of the British Isles AD 450-1100: an Annotated Bibliography, vol. 445. British Archaeological Reports Limited, Oxford.

Crowfoot, E., Pritchard, F., Staniland, K., 1992. Medieval Finds from Excavations in London: 4-textiles and Clothing C. 1150-c. 1450. HMSO, London.

Cybulska, M., Jedraszek-Bomba, A., Kuberski, S., Wrzosek, H., 2008. Methods of chemical and physicochemical analysis in the identification of archaeological and historical textiles. Fibres Text. East. Eur. 5 (70), 67–73, 16.

Cybulska, M., Maik, J., 2007. Archaeological textiles–A need for new methods of analysis and reconstruction. Fibres Text. East. Eur. 15 (5–6), 185–189.

de Bruijne, M., Monteiro, P., 2019. An archaeological textile from the Monastery of Pombeiro: between conservation and perception. Conservar Património 31, 203–214.

dos Reis, J.C., 1961. Noções da Arte da Armaria (II). Rev. Hist. 23 (48), 501–528.

Erickson, A., 1982. An overview of the historic use of textiles for residential interior insulation. Journal of Interior Design Education and Research 8 (1), 18–21.

Fabbri, D., Adamiano, A., Falini, G., de Marco, R., Mancini, I., 2012. Analytical pyrolysis of dipeptides containing proline and amino acids with polar side chains. Novel 2,5-diketopiperazine markers in the pyrolysates of proteins. J. Anal. Appl. Pyrol. 95, 145e155.

Falcón, M.I., 1995. Sobre la industria del vestido en Zaragoza en el siglo XV: las ordenanzas de la cofradía de sastres, calceteros y juboneros. Aragón Edad Media (12), 241–266.

Feliciano, M.J., 2014. Medieval textiles in Iberia: studies for a new approach. Envisioning Islamic Art and Architecture. Essays in Honor of Renata Holod 46–65.

Folwaczny, B., 1973. Bestimmungstabelle der palaarktischen Cossoninae (Coleoptera, Curculionidae) ohne die nur in China und Japan vorkommenden Gattungen, nebst Angaben zur Verbreitung. Entomologische Blatter 69 (1), 65–180.

Gillard, R.D., Hardman, S.M., Thomas, R.G., Watkinson, D.E., 1994. The mineralization of fibres in burial environments. Stud. Conserv. 39 (2), 132–140.

Gleba, M., Mannering, U., 2012. Introduction: textile preservation, analysis and technology. Textiles and Textile Production in Europe from Prehistory to AD 400, 1–26.

Gómez, M., 1946. El Panteón Real de las Huelgas de Burgos. Consejo Superior de Investigaciones Científicas. Instituto Diego Velázquez. Madrid.

González Castañón, M., 2014. Cornatel (León): un castillo bajomedieval berciano y los utensilios metálicos recuperados en sus excavaciones. Medievalismo (24), 171–212.
González-Gómez De Agüero, E., Fernández-Rodríguez, C., Bejega-García, V., Fuertes-Prieto, M.N., 2021. El consumo de moluscos marinos en fortalezas medievales de Galicia (Noroeste Ibérico): el registro zooarqueológico. Monogr. Soc. Hist. Nat. les Balears 32, 357–371.
Good, I., 2001. Archaeological textiles: a review of current research. Annu. Rev. Anthropol. 30 (1), 209–226.
Grancsay, S.V., 1931. The mutual influence of costume and armor: a study of specimens in the metropolitan museum of art. Metrop. Mus. Stud. 3 (2), 194–208.
Grancsay, S.V., 1950. The interrelationships of costume and armor. The Metropolitan Museum of Art Bulletin, New Series 8, 176–188.
Greig, J., 1981. The investigation of a medieval barrel-latrine from Worcester. J. Archaeol. Sci. 8 (3), 265–282.
Grömer, K., 2016. The art of prehistoric textile making. The Development of craft traditions and clothing in Central Europe. Vienna, Natural History Museum Vienna.
Gutiérrez-Cuenca, E., Hierro Gárate, J., Alfaro Giner, C., 2014. Restos textiles de la cueva de Riocueva, Hoznayo (Entrambasaguas, Cantabria). In: Production and trade of textiles and dyes in the Roman Empire and neighbouring regions: Actas del IV Symposium Internacional sobre Textiles y Tintes del Mediterráneo en el mundo antiguo (Valencia, 5 al 6 de noviembre, 2010). Universitat de València, Valencia, pp. 73–81.
Hardin, I.R., 1996. Investigations of textiles by analytical pyrolysis. In: Raheel, M. (Ed.), Modern Textile Characterization Methods. CRC Press, New York, pp. 175–206.
Hausman, L.A., 1920. Structural characteristics of the hair of mammals. Am. Nat. 54 (635), 496–523.
Herrero, C., 1988. Museo de Telas medievales. Monasterio de Santa Maria la Real de Huelgas.
Hoffmann, A., 1954. Coleoptérès curculionides 2. Faune Fr. 59, 487–1208. Paris, Lechevalier.
Jakes, K.A., Sibley, L.R., 1984. An examination of the phenomenon of textile fabric pseudomorphism. Archaeological Chemistry- III (20), 403–424.
Kaal, J., López, Costas O., Martínez, Cortizas A., 2016. Diagenetic effects on pyrolysis fingerprints of extracted collagen in archaeological human bones from NW Spain, as determined by pyrolysis-GC-MS. J. Archaeol. Sci. 65, 1–10.
Kaal, J., Martín, Seijo M., Oliveira, C., Wagner-Wysiecka, E., McCoy, V., Kraemer, M.S., Kerner, A., Wenig, P., Mayo, C., Mayo, J.S., 2020. Golden artefacts, resin figurines, body adhesives and tomb sediments from the pre-Columbian burial site El Caño (Gran Coclé, Panamá): tracing organic contents using molecular archaeometry. Journal of Archaeolaeological Science 113, 105045.
Knicker, H., Río, J.C., Hatcher, P., Minard, R., 2001. Identification of Protein remnants in insoluble geopolymers using TMAH thermochemolysis/GC-MS. Org. Geochem. 32, 397–409.
La Rocca, D.J., 2017. How to Read European Armor. Metropolitan Museum of Art, New York.
Le Goff, J., 1994. Lo maravilloso y lo cotidiano en el Occidente medieval. Barcelona, Gedisa.
Lejay, M., Alexis, M., Quénéa, K., Anquetil, C., Bon, F., 2019. The organic signature of an experimental meat-cooking fireplace: the identification of nitrogen compounds and their archaeological potential. Org. Geochem. 138, 103923.
Lu, H., Zhang, J., Wu, N., Liu, K.-b., Xu, D., Li, Q., 2009. Phytoliths analysis for the discrimination of foxtail millet (*Setaria italica*) and common millet (*Panicum miliaceum*). PLoS One 4 (2), e4448.
Margariti, C., 2019a. FIBRANET Database, Centre for Textile Research/University of Copenhagen, Project Funded under the EU Horizon 2020 Marie Skłodowska Curie Programme under Grant Agreement No 745865.
Margariti, C., 2019b. The application of FTIR microspectroscopy in a non-invasive and non-destructive way to the study and conservation of mineralised excavated textiles. Heritage Science 7, 1–14.
Margariti, C., Sava, G., Sava, T., Boudin, M., Nosch, M.L., 2023. Radiocarbon dating of archaeological textiles at different states of preservation. Heritage Science 11 (1), 44.
Marques, R., Sousa, M.M., Oliveira, M.C., Melo, M.J., 2009. Characterization of weld (*Reseda luteola* L.) and spurge flax (*Daphne gnidium* L.) by high-performance liquid chromatography–diode array detection–mass spectrometry in Arraiolos historical textiles. J. Chromatogr. A 1216 (9), 1395–1402.
Martín-Seijo, M., Vázquez Collazo, S., 2020. Ad comburendum. El uso de la leña en el Castillo de Pambre (Palas de Rei, Lugo) durante los siglos XV-XVII. SPAL-Revista de Prehistoria y Arqueología 29 (1), 321–341.
Martínez, A., 2000. Ajuar funerario de doña María, Restauración & Rehabilitación. Revista internacional del Patrimonio Histórico 39, 22–27.
Martínez, A., Pastrana, P., 2002. Ajuar funerario de la Infanta doña María. Panteón real de San isidoro. Tratamiento integral de conservación y restauración, Ars Sacra 21, 89–93.
McCullough, F., Rast-Eicher, A., Nhoem, S., 2018. La nécropole du Haut moyen âge de Courtepin/fin dessus. Cahiers d'Archéologie Fribourgeoise 20, 76–131.
Médard, F., Rohmer, P., Moulherat, C., Guillaume, J., 2007. La nécropole mérovingienne d'Erstein (Bas-Rhin): étude des textiles minéralisés au contact des fibules. Revue archéologique de l'Est, Tome 55, 307–322.
Morrison, W.H., Archibald, D.D., 1998. Analysis of graded flax fiber and yarn by pyrolysis mass spectrometry and pyrolysis gas chromatography mass spectrometry. J. Agric. Food Chem. 46, 1870–1876.
Nasu, H., Momohara, A., Yasuda, Y., He, J., 2007. The occurrence and identification of Setaria italica (L.) P. Beauv.(foxtail millet) grains from the Chengtoushan site (ca. 5800 cal BP) in central China, with reference to the domestication centre in Asia. Veg. Hist. Archaeobotany 16, 481–494.
Neumann, K., Strömberg, A.E.C., Ball, T.B., Albert, R.M., Vrydaghs, L., Scott-Cummings, L., International Committee for Phytolith Taxonomy ICPT), 2019. International code for phytolith nomenclature (ICPN) 2.0. Ann. Bot. 124 (2), 189–199.
Pardo de Guevara, E., Otero, P.S., García, X.A., 2012. La huella de los primeros Ulloa en las torres y fortalezas de la "terra" de Ulloa. Castillos de España164- 165–166, 59–67.
Pastoreau, M., 2006. In: Katz (Ed.), Una historia simbólica de la Edad Media Occidental. Buenos Aires.
Peña-Chocarro, L., Pérez-Jordà, G., Alonso, N., Antolín, F., Teira-Brión, A., Tereso, J.P., Montes, E.M., López-Reyes, D., 2019. Roman and medieval crops in the Iberian Peninsula: a first overview of seeds and fruits from archaeological sites. Quat. Int. 499, 49–66.
Piperno, D.R., 2006. Phytoliths: A Comprehensive Guide for Archaeologists and Paleoecologists. Altamira Press, Lanham.
Poher, Y., Ponel, P., Médail, F., Andrieu-Ponel, V., Guiter, F., 2016. Holocene environmental history of a small Mediterranean island in response to sea-level changes, climate and human impact. Palaeogeogr. Palaeoclimatol. Palaeoecol. 465, 247–263.
Ponel, P., 2011. 5. Analyses entomologiques (coléoptères). In: Excoffon, P. (Ed.), Ville et campagne de Fréjus Romaine. Aix-en-Provence. Publications du Centre Camille Jullian, pp. 245–256.
Ponel, P., Andrieu-Ponel, V., Bouiron, M., 2014. Vegetation and landscape from 14th to 17th century AD in Marseille city centre, reconstructed from insect and pollen assemblages. Quat. Int. 341, 152–171.
Portillo, M., Kadowaki, S., Nishiaki, Y., Albert, R.M., 2014. Early Neolithic household behavior at Tell Seker al-Aheimar (Upper Khabur, Syria): a comparison to ethnoarchaeological study of phytoliths and dung spherulites. J. Archaeol. Sci. 42, 107–118.
Portillo, M., Dudgeon, K., Allistone, G., Raeuf Aziz, K., Matthews, W., 2021. The taphonomy of plant and livestock dung microfossils: an ethnoarchaeological and experimental approach. Environ. Archaeol. 26, 439–454.
Rast-Eicher, A., 2016. Fibres: Microscopy of Archaeological Textiles and Furs. Budapest, Archaeolingua.
Rosen, A.M., 1992. Preliminary identification of silica skeletons from Near Eastern archaeological sites: an anatomical approach. In: Rapp Jr., G., Mulholland, S.C. (Eds.), Phytolith Systematics: Emerging Issues, Advances in Archaeological and Museum Science. Plenum Press, New York, pp. 129–148.
Rosenthal, M.F., 2009. Cultures of clothing in later medieval and early modern Europe. J. Mediev Early Mod. Stud. 39 (3), 459–481.
Rouco, J., 2017. Las funciones de una fortaleza bajomedieval: el caso de Pambre. Cuad. Estud. Gallegos 64 (130), 167–197.
Sabatini, F., Naccinacci, T., Degano, I., Colombini, M.P., 2018. Investigating the composition and degradation of wool through EGA/MS and Py-GC/MS. J. Anal. Appl. Pyrol. 135, 111–121.
Sam, 2017. Pyrolysis GC/MS of plant derived (cellulosic) textiles. Application note #119 of CDS Analytical. www.cdsanalytical.com.
Sanz de Bremond, C., 2016. La indumentaria de los sepulcros de don Raimundo de Zaera y de doña Estefanía de Zarea (1363) del Instituto Valencia de Don Juan, Madrid. Cuad. Estud. Borj. (59), 65–75.
Sanz de Bremond, C., 2018. Juan de Coloma y la Corte: una aportación desde la indumentaria. Cuad. Estud. Borj. (61), 175–190.
Sanz de Bremond, C., 2020. El" Carlista" de Maluenda: estudio de la indumentaria de un personaje de la Baja Edad Media. Cuad. Estud. Borj. 63, 57–62.
Scalini, M., 2003. Corazzine e bacinetti dalla Rocca di Campiglia. Campiglia. In: Bianchi, G. (Ed.), Campiglia. Un castello e il suo territorio, Indagine Archeologica. Florencia Ed, vol. 2. All'Insegna del Giglio, pp. 382–396.
Sherman, H.M., 2014. The flax and linen of medieval novgorod. In: Textiles and the Medieval Economy: Production, Trade, and Consumption of Textiles, 8th_16th Centuries, vol. 16, p. 104, 2014.
Sigüenza, C., 1998. La vida cotidiana en la Edad Media: la moda en el vestir en la pintura gótica. In: La vida cotidiana en la Edad Media: VIII Semana de Estudios Medievales: Nájera, del 4 al 8 de agosto de 1997. Instituto de Estudios Riojanos, pp. 353–368.
Silva, J., Parola, A.J., Oliveira, M.C., Lavédrine, B., Ramos, A.M., 2022. Contributions to the characterization of chromogenic dyes in color slides. Heritage 5, 3946–3969.
Smith, D.N., 2013. Defining an indicator package to allow identification of 'cesspits' in the archaeological record. J. Archaeol. Sci. 40 (1), 526–543.
Staples, K., 2008. Fripperers and the used clothing trade in late medieval london. In: Netherton, R., Owen-Crocker, G.R. (Eds.), Medieval Clothing and Textiles, vol. 4. Boydell & Brewer Ltd, pp. 151–171.
Teira-Brión, A., 2022. Traditional millet cultivation in the Iberian Peninsula: ethnoarchaeological reflections through the lens of social relations and economic concerns. In: Kirleis, W., Dal Corso, M., Filipović, D. (Eds.), Millet and what Else? the Wider Context of the Adoption of Millet Cultivation in Europe. Sidestone Press, Leiden, pp. 263–278.
Teira-Brión, A., Constela Doce, X., Sartal Lorenzo, M., Gil Agra, D., Rúa Carril, V., 2023. The city as dissipative structure: the flow of agricultural production in the medieval Kingdom of Galicia. J. Anthropol. Archaeol. 69, 101482.
Twiss, P.C., 1992. Predicted world distribution of C3 and C4 grass phytoliths. In: Rapp Jr., G., Mulholland, S.C. (Eds.), Phytolith Systematics: Emerging Issues, Advances in Archaeological and Museum Science. Plenum Press, New York, pp. 113–128.
Twiss, P.C., Suess, E., Smith, R.M., 1969. Morphological classification of grass phytoliths. Soil Sci. Soc. Am. Proc. 33, 109–115.

Valianou, L., Karapanagiotis, I., Chryssoulakis, Y., 2009. Comparison of extraction methods for the analysis of natural dyes in historical textiles by high-performance liquid chromatography. Anal. Bioanal. Chem. 395, 2175–2189.
Vegas, L., 2013. Indumentaria masculina en la corte de Castilla a mediados del siglo XV: prendas de ir desnudo en la Cámara Real de Juan II el último año de su reinado. Anales del Historia del Arte 23, 95–103.
Vollmer, J., 1974. Textile pseudomorphs on Chinese bronzes. In: Fiske, P.L. (Ed.), Irene Emory Roundtable on Museum Textiles, 1974 Proceedings, pp. 170–174. Washington, DC: Text. Mus.
Walton, P., 1989. Textiles, Cordage and Raw Fibre from 16-22 Coppergate. York Archaeological Trust, York.
Wouters, J., 1985. High performance liquid chromatography of anthraquinones: analysis of plant and insect extracts and dyed textiles. Stud. Conserv. 30 (3), 119–128.
Wouters, J., Verhecken, A., 1989. The coccid insect dyes: HPLC and computerized diode-array analysis of dyed yarns. Stud. Conserv. 34 (4), 189–200.
Zhang, J., Lu, H., Wu, N., Yang, X., Diao, X., 2011. Phytolith analysis for differentiating between foxtail millet (*Setaria italica*) and green foxtail (*Setaria viridis*). PLoS One 6 (5), e19726. https://doi.org/10.1371/journal.pone.0019726.