

Marble and pitch: Original data on Roman wall revetment, the example of the Saint-Martin-au-Val sanctuary (Chartres, France)

Arnaud Coutelas, Bruno Bazin, Armelle Charrié-Duhaut, Sébastien Cormier,

Christophe Loiseau

▶ To cite this version:

Arnaud Coutelas, Bruno Bazin, Armelle Charrié-Duhaut, Sébastien Cormier, Christophe Loiseau. Marble and pitch: Original data on Roman wall revetment, the example of the Saint-Martin-au-Val sanctuary (Chartres, France). Journal of Archaeological Science: Reports, 2024, 57, pp.104642. 10.1016/j.jasrep.2024.104642. hal-04614208

HAL Id: hal-04614208 https://hal.science/hal-04614208v1

Submitted on 17 Jun2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers. L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution 4.0 International License



Contents lists available at ScienceDirect

Journal of Archaeological Science: Reports



journal homepage: www.elsevier.com/locate/jasrep

Marble and pitch: Original data on Roman wall revetment, the example of the Saint-Martin-au-Val sanctuary (Chartres, France)

Arnaud Coutelas^{a,*}, Bruno Bazin^{a,b}, Armelle Charrié-Duhaut^c, Sébastien Cormier^{a,d}, Christophe Loiseau^{a,e}

^a Laboratoire Archéologie et Philologie d'Orient et d'Occident (UMR 8546), École Normale Supérieure, 75230 Paris, France

^b Directorate of Archaeology, Chartres Métropole, France

^c Laboratory of Mass Spectrometry of Interactions and Systems (LSMIS), UMR 7140 CMC, Strasbourg, France

^d Mission Archéologique Départementale de l'Eure, 27930 Le Vieil-Évreux, France

^e Éveha, Ester Technopole, 87068 Limoges, France

ARTICLE INFO

Keywords: Roman wall revetment Chartres (France) Pitch and beeswax-bearing mortar binder FTIR GC/MS

ABSTRACT

The rising water table has saved some of the marble decorations in the ancient sanctuary of Saint-Martin-au-Val. It has also preserved materials of organic origin. Yet a grey to black material has appeared associated with large stone shims visible on the plaster surface. These shims are known from Roman plasters. They are traditionally understood to be fitting elements, but this remains hypothetical. The material used is unprecedented: a kind of mortar made from pitch, beeswax and marble grains. It was obviously used to glue the shims to the underside of the marble slabs. These shims therefore had a completely different function: they were actually used to hold the marble parietal decoration in place, adding points of contact between the decoration and its plaster.

1. Introduction

The sanctuary of Saint-Martin-au-Val, one of the largest religious complexes in Roman Gaul, lies on the immediate outskirts of the ancient *Autricum* (Chartres). Built between the late 1st and early 2nd centuries AD, it covers an area of over 11 ha. It stands on the western slope of the Eure valley (Fig. 1).

Excavations have been underway at this complex for around fifteen years.¹ In August 2021, a week of study was organised to investigate the architectural layout of one of the buildings,² erected between the years AD 120–150, situated to the north-east of a large quadriporticus temple (Fig. 2). Its wall coverings are in a remarkable state of preservation for northern Gaul. A fire, followed by a rapid rise in the water table after the building was abandoned, helped preserve the marble veneers on the lower part of the wall facings, as well as the basin copings. The state of immersion, without air or light, was also what stopped some of the perishable materials used to construct the building from decomposing. It was therefore possible to collect nearly 2000 pieces of wood, forming

part of a lavishly decorated ceiling, all remarkably preserved. These elements make up a rare, exceptional find. As we shall see here though, this is not the only one.

A study of the wall coverings revealed an unusual substance found in contact with large "shims" on the surface of the veneer bonding mortar (Fig. 2). These shims are mostly made of limestone. They are chips or offcuts of slabs or flagstones positioned in an irregular manner. These shims have also been seen on other sites in the Roman world (Giuliani, 1990), particularly on buildings in Rome and the Bay of Naples. For the most part, they are made of marble or terracotta (amphora fragments). However, ancient authors make no mention of them. The literature presents several possible interpretations, which are important because they condition the overall restitution of the wall revetment technique. Most often, they are interpreted as markers used to keep the decoration vertical. They were either attached to the surface of the plaster after it had been laid (Adam, 1989), or put in place before the plaster on "islands" of mortar stuck to the wall (Bitterer, 2013). For other authors, they were embedded into the plaster to make it easier to adjust the slabs

* Corresponding author.

https://doi.org/10.1016/j.jasrep.2024.104642

Received 12 February 2024; Received in revised form 27 May 2024; Accepted 11 June 2024

2352-409X/© 2024 The Author(s). Published by Elsevier Ltd. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

E-mail address: coutelas@gmail.com (A. Coutelas).

¹ Under the direction of Bruno Bazin, archaeologist at the Chartres Métropole Directorage of Archaeology (Eure-et-Loir, France). See: Bazin, 2013; Bazin et al., 2014.

² The project brought together Arnaud Coutelas for his expertise on lime mortars, Sébastien Cormier for the marble veneers, and Christophe Loiseau for the metal elements. Bruno Bazin contributed his architectural expertise and knowledge about the building with basins (Bazin and Bouilly, 2021).



Fig. 1. Map showing the location of remains associated with the ancient sanctuary of Saint-Martin-au-Val.



Fig. 2. Plan of the building with basins (excavation 2022) and view of the decorative plinth on the north wall.

when they were being laid, to provide greater leverage, and so the hollows could accommodate any excess mortar (Ball, 2002). Finally, other authors maintain that these fragments were glued to the back of

the revetment panels using "mastic" to create additional holding points for the mortar (Guidobaldi and Angelelli, 2005). This hypothesis has unfortunately never been supported by material analyses, except



Fig. 3. Detail of the north wall, two shims with all around the remains of a grey and granular mortar.



Fig. 4. Appearance of the material just exposed to the air, by removing a fragment of marble slab. Its greyer appearance in the lower left-hand corner reflects earlier exposure to air.

perhaps for in the work of Ponti and Sinibaldi (2004), who discovered an organic binder on the reverse side of the *opus sectile* at the Villa dei Quintili (Rome). They interpreted this material as rosin (colophony), a

material obtained by distilling pine gum, but the analyses presented are insufficient to confirm this. More recently, studies have been carried out on elements from the northern necropolis of Antinoopolis (Egypt) dated



Fig. 5. Thin section of the PR1 sample (dimensions 2.40×3.60 cm).

to the 4th-5th century CE (Ribechini et al., 2009). These are shards of glass or splinters of rock that were probably used as an *opus sectile* on the walls or as a floor covering. On the reverse side, an adhesive made from a mixture of pine pitch, beeswax, and *brassicaceae* seed oil has been identified.

There is therefore no consensus on the usefulness of the shims. One hypothesis suggests the presence of an organic material in contact with them, but there is insufficient material analysis, and the few recent studies have focused on *opus sectile*, i.e. small rock or glass elements, but not large marble slabs. Based on the architectural observations undertaken at the sanctuary of Saint-Martin-au-Val and the analysis, carried out in a specialist laboratory, of three samples of the unusual substance found in contact with these shims, we can reassess all these hypotheses and identify the roles played by this material and the shims in the various stages of the installation of ancient wall revetments.

2. Materials and methods

2.1. Sample collection context

In 2016, the dismantlement of several marble coverings on the north wall and the north basin copings revealed a granular material full of little white grains (Fig. 3). This material has been systematically discovered behind decorative plinths still in place, in contact with the limestone shims set up in the fixing mortar. They vary in thickness from 0.5 to 1.5 cm. The texture is sometimes still greasy and vesicular, with a caramel to black hue (Fig. 4) which turns grey and becomes drier if left out in the open air for a certain amount of time. The disappearance of this material is evidently what caused the shrinkages of 0.5–2 cm observed, on several occasions, between the limestone shims and the face of the bedding mortar in contact with the marble slabs.

Samples of this material were collected from the lower part of the north and south walls of the building and under the west coping of the north basin. Collection followed the same protocol for all samples. After removing several marble slabs, the substance in contact with the limestone slabs was scraped off an area of 2 cm². A triangular metal spatula, previously cleaned and rinsed with acetone to remove all traces of organic matter, was used to remove the preserved substance to a thickness of between 5 mm and 1 cm. Thanks to its excellent state of preservation, we were able to take several samples. Once the residues had been sealed in a bag, they were stored in a cold room at an average temperature of 8 $^\circ\text{C},$ protected from air and light. Regular visual checks were carried out approximately every two weeks. No deterioration was observed until they were sent to the laboratory for analysis, no later than two months after on-site sampling. In the end, three samples from the north and south perimeter walls were analysed: sample PR1 (sample from the north wall, inventory no. 10808.1); sample PR2 (sample from the south wall, intermediate plate, inventory no. 11233.1); and sample PR3 (sample from the south wall, west plate, inventory no. 11233.2).

2.2. Analysis methods

2.2.1. Optical transmission microscopy (MOT)

The lime mortars (not presented here) and the grey-hued material (PR1) underwent petrographic study (Elsen, 2006; Pavía and Caro, 2008; Coutelas et al., 2009; Pecchioni et al., 2020). The materials were consolidated by impregnation in a resin under vacuum. Sample PR1 was impregnated avoiding any heating. The samples were cut and then polished to be glued on glass slides. The thickness was reduced and samples were polished by hand to about $30 \,\mu\text{m}$ (Fig. 5). The thin sections were examined under a petrographic microscope, incorporating eye pieces with magnifications of $2\times$, $10\times$, $20\times$ and $40\times$, using both natural



Fig. 6. Microphotographs of the PR1 thin section (in plane polarized light on the left side and crossed polarized light on the right). The honey to orange matrix is opaque in crossed polarized light, with a partially iridescent appearance. The angular grains visible in A and B are calcite crystals rich in macles. The grains visible in C and D are limestone fragments, with miliolus-type foraminifera in D. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

and polarised light.

2.2.2. Sample preparation

The analytical strategy used to study the organic part of these materials involved preparing an organic extract to be analysed by Fourier transform infrared spectroscopy (FTIR) and gas chromatography coupled with mass spectrometry (GC/MS) according to protocols specifically implemented in the laboratory, published in the literature and standardized (Charrié-Duhaut et al., 2009a). All solvents were HPLC grade (Sigma Aldrich, purity: 99.9 %) and were used without further purification. Pyridine and N, O, bis(trimethyl)silyltrifluoroacetamide (BSTFA) were purchased from Sigma Adrich. To avoid any contamination, only dichloromethane cleaned glassware was used and no plastic. The sample – a few mg finely ground in an agate mortar – was ultrasonically extracted three times for 1 min, using the solvent mixture dichloromethane/methanol 60:40 (v/v). The combined extracts formed the total organic extract, which was evaporated under a gentle flow of nitrogen to dryness and purified by filtration on a column of dry silica gel. A third of this purified extract was analysed using FTIR. The remaining two thirds were derivatised using 200 μ L of N,0-bis (trimethylsilyl)trifluoroacetamide (BSTFA) after being dissolved in 50 μ L of pyridine (2 h at 70 °C). The final reaction medium was evaporated



Fig. 7. FTIR-ATR spectra of the extraction residues of the three samples.

to dryness under a flow of nitrogen and dissolved in dichloromethane before being injected in GC–MS.

heterogeneous, ranging from 1:3 to 1:4.

2.2.3. Fourier transformed infrared spectroscopy (FTIR)

The FTIR investigations were carried out both on the purified organic extracts and on the extraction residues. The spectra were obtained in attenuated total reflectance (ATR) mode with a PerkinElmer Spectrum Two instrument at a temperature of 20 °C and recorded in a range of 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

2.2.4. Gas chromatography / mass spectrometry (GC/MS)

GC–MS analyses were performed using an Agilent 8890 chromatographer coupled with an Agilent 5977B MSD. The parameters of the separation by gas chromatography were as follows: HP-MS column (30 m × 0.25 mm × 0.25 µm film thickness) with a He constant flow of 1.5 mL.min⁻¹ and a temperature gradient of 40 °C for 2 min, then 10 °C/min until 100 °C, followed by 4 °C/min up to 320 °C, with a hold time of 60 min. The mass spectrometer was operating in electron impact mode (EI) at 70 eV with a source temperature set at 210 °C and a range of 70–800 Da. Compounds were identified based on their retention time and their mass spectrum through a comparison with data from the literature, the NIST database, and the laboratory's internal data bank.

3. Results

The grey material is indeed a mortar, but not a lime mortar. Under the microscope, its matrix has a caramel hue under crossed polarised light (Fig. 6). Under polarised and analysed light, it has a heterogenous, opaque appearance marked by numerous stains. Segments of binder poorly mixed with the rest can be seen at several points. Its surface is darker and features fewer grains. However, the aggregate content is still high. For the material as a whole, the binder/aggregate ratio is

3.1. Aggregate

The grains in the material are angular; they are rock fragments purposefully crushed for this purpose. Some fairly large grains can also be seen, with a diameter of around 0.5–2 mm, combined with much finer grains the size of silt or very fine sand.

Three quarters of the aggregate consists of calcite crystals. They are monocrystalline or polycrystalline characterised by a large number of twins (Fig. 6). This is marble. We have not carried out any studies to identify this, but these grains are likely scrap materials from cutting up the decorative elements, i.e. a mixture of marble from Carrara (Italy; Prochaska et al., 2018) and Châtelperron (Allier, France; Blanc and Blanc, 2010). Indeed, the Chartres region has no marble quarries, so the nearest source was the construction site itself. Moreover, the remaining quarter of the aggregate is made up of a few quartz crystals, and, most significantly, grains of limestone (Fig. 6). This is a rock characterised by large calcite crystals and foraminifera; the characteristics of a local limestone known as Lutetian Limestone. This Eocene limestone is frequently used in the Paris basin for construction and lime manufacturing purposes. It is also the main stone of Paris (Fröhlich and Schubnel, 2000). It was used in the floors and architectural elements of the sanctuary of Saint-Martin-au-Val and appears to make up the bulk of the large shims used behind the decorative plaques.

FTIR analyses of the extraction residues of the three samples confirm the preponderance of calcium carbonate (with broad and intense bands between 1148 and 1418 cm⁻¹, and fine bands between 873 and 712 cm⁻¹) and a small amount of silicates (with a broad band at around 1029 cm⁻¹) (Fig. 7; Farcas and Touzé, 2001). These extraction residues correspond to the inorganic part of the mortar. Their composition highlighted by FTIR is consistent with the presence of grains of marble,

Sample PR1



Fig. 8. FTIR-ATR spectra of the organic extract of the three samples.

quartz and limestone indicated previously.

3.2. The binding agent

FTIR analyses alone are not sufficient to determine the composition of an organic material, especially if it is composite. However, such analyses do reveal the functional groups present. The spectra obtained confirm the presence of organic matter, although in lower quantities for sample PR3 (Fig. 8). The absorption bands observed are common to many organic substances such as waxes, resins, tars, and fats: 3600 to 3200 cm⁻¹ ν (O–H), 2918 and 2850 cm⁻¹ ν (C–H), and 1462 and 1379 cm^{-1} δ (C–H). A noteworthy characteristic should nevertheless be mentioned. The signal from aromatic C-C bonds at 1603 cm⁻¹ is relatively intense in the spectra of the three samples. They could therefore be products that have undergone extensive heat treatment, leading to the aromatisation of the structures. The spectrum of sample PR1 could correspond to a plant tar, as described by Font et al. (2007). The spectrum of sample PR2 shows a strong contribution of the carbonyl signals (1710 cm⁻¹ ν (C=O)), which make up the majority compared to the carbon chain signals (2918 and 2850 cm^{-1} ν (C–H), 1462 and 1379 cm^{-1} δ (C–H)). This observation could indicate the contribution of a substance rich in ester groups such as beeswax, whose characteristic biomarkers are long-chain palmitic acid esters called wax esters.

GC/MS profiles from samples PR1 and PR3 are largely dominated by dehydroabietic acid (Fig. 9). This acid is also one of the majority compounds detected in sample PR2. Other diagnostic molecules whose skeletons are also linked to abietic acid are also present, namely, abietic acid, 7-oxo-dehydroabietic acid, 7-hydroxy-dehydroabietic acid, retene, and tetrahydroretene. This clearly reflects the presence of a product derived from conifers and, more precisely, from Pinaceae, which is commonly referred to as pitch in the literature (Colombini et al., 2003).

The organic extract from sample PR2 presents a significantly more complex profile (Fig. 9c). Accompanying the family of abietic acidrelated biomarkers, several compounds were detected. These are linear alcohols (predominantly with an even distribution of C_{16} to C_{32}), linear acids (C_{16} , C_{18} , C_{24} , C_{26}), derivatives of plant sterols (β -sitosterol), and wax esters (esters of palmitic acid from C_{40} to C_{46} with a long chain, even predominance). This set of molecules corresponds to the molecular fingerprint of beeswax (Garnier et al., 2002). This beeswax has also been identified in sample PR1 (Fig. 9a) but in smaller proportions compared to the pitch. No conclusion at this level could be made concerning sample PR3 because the quantities of extract are too low.

The binding agent used in samples PR1 and PR2 is therefore essentially made up of pitch and beeswax. No other ingredients were



Fig. 9. TIC of the trimethylsilylated organic extract obtained by GC–MS of the three samples. List of compounds: 7-oxo-dehydroabietic acid 1, 7-hydroxy-dehydroabietic acid 2, abietic acid 3, dehydroabietic acid 4, retene 5, tetrahydroretene 6.

identified using the analytical strategy presented before. Compounds such as triterpenes from the betulin family (no birch bark tar), triglycerides or their degradation products (no fats), hopanes and steranes (bitumen) were not detected. To ensure this, analyzes by detection of the characteristic fragments of each of the biomarkers were carried out. It is, however, not impossible that an other natural product in a very trace amount has not been highlighted but it would not be part of the materials selectively sought thanks to their specific biomarkers, namely birch bark tar, fats, bitumen, pitch and beeswax.

4. Discussion

4.1. A material made from pitch and beeswax

Thanks to these analyses, we were able to clearly identify the organic material contained in the samples, which is pitch, a product derived from the Pinaceae (pine) family, mixed with beeswax.

The definition of pitch is ambiguous in the literature (Bailly, 2015). While it is commonly described as a plant tar obtained through the heat treatment of wood from the pine family under controlled conditions, it could also be heated Pinaceae resin or a mixture of the two. It is still difficult to distinguish between these substances using biomarkers (Bailly et al., 2016; Davara et al., 2023). The significant presence of polyaromatic structures in these substances supports the hypothesis that it is a tar made from wood.

Ancient texts (i.e. Pliny the Elder, *Naturalis Historia* XVI, 21) mention two possible uses for pitch (Acovitsioti-Hameau et al., 1993): as a sealant (for jars, containers, boats, woodwork, etc.); and for preserving wine (pitch mixed with must). From a chemical perspective, it is well recognised in archaeological contexts (Robinson et al., 1987; Loewen, 2005; Colombini et al., 2005; Romanus et al., 2009; Charrié-Duhaut et al., 2009b; Izzo et al., 2013; Charrié-Duhaut, 2019). It was used very early on in Europe as an adhesive, for fixing handles on tools, caulking boats, and sealing or repairing containers at different times (Regert, 2004). However, its use as a mortar binder has not been seen before. At best, Vitruvius mentions that the brick walls of Babylon were built with bitumen (Vitruvius, *De Arch.*, I, 5, 8) and that pitch could be used to coat *tegulae mammatae*, which were used to set up ventilation spaces in the wettest walls (Vitruvius, *De Arch.*, VII, 4, 2).

In Roman Gaul, Lozère and the Atlantic coast of Landes and Gironde were home to several sites where pitch was produced (see Aufan and Thierry, 1990; Trintignac, 2003). These areas were favoured by their wood (pine) resources and their proximity to busy markets: vineyard and shipbuilding regions. Production was not merely opportunistic: there was a real trade at the time. This is borne out by the contents of wrecks discovered in the Mediterranean, from the Adriatic to Spain (Carre, 2021), or in the Atlantic, for example the cargo of the Roman wreck of Guernsey which sank around AD 280 after loading up at least half a tonne of pitch somewhere on the Landes coast (Connan et al., 2002). For these ships, the quantities transported far exceeded their requirements on board. However, none of them had a mixed cargo combining both pitch and blocks or slabs of marble.

The preparation and trade of beeswax, on the other hand, was less a cause for concern. Going by what Pliny the Elder says on the matter (Nat. Hist. XXXIII, 40), however, some specific recipes may have existed. For encaustic painting, he mentions "punic wax", which is obtained through a process geared at purifying yellow beeswax; the wax is bleached, its free acidity removed, and it is sometimes partially saponified (Colinart et al., 1999). The Fayum portraits - Egyptian funeral depictions on wood panels from the Roman period (1st-4th century CE) - were in fact painted using a beeswax-based binder (Colinart, 1998). In addition to this technique - known as "encaustic painting" - beeswax, particularly this punic wax, is said to have been used to secure a chromatic rendering, and to weather-proof statues that were frequently kept outdoors. This Greek technique, known as ganosis, likely entailed mixing wax with other products, particularly oil (Neri et al., 2021). Similarly, wax was used as an adhesive, either on its own or mixed with other organic compounds. It is found, for instance, on the material on the reverse side of the opus sectile elements at Antinoopolis, mixed with pitch and oil. This recipe is quite similar to the one found at Saint-Martin-au-Val. It is not possible to estimate the proportion between pitch and wax, due to the alteration of these raw materials. These proportions were also likely not entirely homogenous. Be that as it may, the addition of wax to pitch was certainly intended to modify the physico-chemical characteristics of the pine pitch since it may have been used to produce a more malleable and mouldable adhesive (Ribechini et al., 2009; Duce et al.,

2015). The aim was also to make the material more resistant to heat and support its preservation.

4.2. A pitch and wax-based mixture used for marble wall revetment

While it may seem surprising that these veneers are not mentioned in ancient texts, the dearth of studies currently being carried out on the techniques used to put them in place is even more remarkable. Middleton (1892) seems to have been the first to consider the methods used to install these wall decorations. It was not until the early 2000s, however, that any real syntheses emerged, based on the decorations discovered in Rome and Pompeii (Ball, 2002; Guidobaldi and Angelelli, 2005; Bitterer, 2013). We now know that, unlike for mural paintings, stone coverings were laid from the bottom up. Before the mortar was laid, metal clamps were inserted into the wall (Loiseau, 2012). These were secured with shims, generally made of marble, in the form of small cubes with a width of several centimetres. These clamps were inserted into the slabs of rock along the sides and used to hold them in place. However, it was the lime mortar that really held everything in place.

The plethora of hypotheses surrounding the function and techniques used to lay these large stone or terracotta shims, as noted in the introduction, may stem from the fact that not all the authors have considered that the mortar could be poured in behind the marble slabs. This was the case for larger panels, at least, which were held in a vertical position using metal clamps before the mortar was poured in behind (Giuliani, 1990; Seigne, 1999; Guidobaldi and Angelelli, 2005; Coutelas et al., 2009; Coutelas et al., 2010).

In Rome, Pompeii, and elsewhere, these large shims can therefore be seen on the surface of the plaster which is poured in behind the slabs. At times, they are embedded in the surface of several cast volumes, with no visible lime mortar on the reverse side and no drips of mortar on the surface. The original mortar from Saint-Martin-au-Val, made from pitch, wax and marble grains, can be seen around the stone wedges, always on the surface of the rendering, and sometimes slightly between the wedges and the slabs. We can therefore conclude that the shims were held in place behind the slabs with this pitch. This is the "mastic" mentioned in Guidobaldi and Angelelli's hypothesis.

We can therefore build a picture of the different stages involved in the installation of wall revetment in the Saint-Martin-au-Val sanctuary. The shims are fastened directly to the back of the decorative marble slabs using mortar made from pitch. The slab is then positioned along the facing and held in place using metal clamps. The space between this plinth and the wall facing is filled with rendering mortar, which is poured in several stages. The marble shims previously fixed to the back of the slabs are then "trapped" in the plaster, which helps to hold the decoration in place more securely. This technique would therefore have presented a solution when the rendering mortar was not sufficiently fluid to mould to all the rough patches on the underside of the slabs, or when the underside of the slabs was too smooth.

5. Conclusion

Thanks to the optimal state of preservation of the organic elements and wall coverings found at the ancient building with basins at Saint-Martin-au-Val, an exceptional discovery was made. The shims visible on the surface of the plaster were originally fastened to the underside of the slabs of rock using a material made of pitch and beeswax. Traditionally understood to be fitting elements, these shims are now considered to be wedges used to hold the marble wall decoration in place, in addition to the lime mortar used to bind everything together.

Except for the two *opus sectile* cases discussed above – the *Villa dei Quintili* and the northern necropolis of Antinoopolis – this specific use of an organic material does not appear to have been observed on any other ancient archaeological site. Might we nonetheless consider it to have been universal in Roman construction? And might we deduce that its identification at Saint-Martin-au-Val was thanks only to the remarkable



Fig. 10. Front façade of the building known as the "Temple of the Genius of Augustus" (Pompeii, VII 9, 2), with the remains of the plaster for the marble veneers. The marble shims are surrounded by a small void (the two shims on the right side of the zoom) or by restoration mortar (the three shims on the left).

state of preservation of the veneers and to the fact that we were specifically focused on understanding the use of these large shims? This is the hypothesis we would put forward. Since this material cannot be preserved, it is logical that empty spaces would have appeared around the shims once they had disappeared. This, we believe, is what can be seen in Rome, Gabies, Cumes, Oplontis, and Pompeii (Fig. 10), where these empty spaces are sometimes filled with modern restoration mortar. The recipe identified at Antinoopolis, moreover, is much the same; there too, the material also contains calcite, which implies that grains of limestone or marble were added, which observations under the microscope would quickly confirm.

This discovery opens up many possible avenues of research. The use of pitch for construction or decoration purposes has not been seen before. We might therefore question how widespread such a use was, the kind of volumes that were required, and how this would have affected the trade of this raw material. Was there an opportunistic local supply based on demand or a specific production, possibly associated with the marble trade? Evidence from wrecks has not revealed pitch and stone being used in combination with one another, but we know that certain places did produce both (Wurch-Kozelj and Kozelj, 1999).

What is more, while a pitch-, beeswax- and marble-based mortar was discovered at both Saint-Martin-au-Val and Antinoopolis, was this always the mixture that was used? Could organic materials have been used on their own, without any aggregates? Is it not possible that other organic materials or mixtures were used, such as birch tar or bitumen? Let's hope that other sites will provide us with some answers to these questions.

CRediT authorship contribution statement

Arnaud Coutelas: Writing – original draft, Supervision, Methodology, Investigation, Formal analysis. **Bruno Bazin:** Writing – original draft, Supervision, Project administration, Funding acquisition. **Armelle**

Charrié-Duhaut: Writing – original draft, Methodology, Investigation, Formal analysis. **Sébastien Cormier:** Investigation. **Christophe Loiseau:** 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.

Data availability

No data was used for the research described in the article.

Acknowledgments

We would like to thank Dr E. Leize-Wagner, Director of the LSMIS (UMR7140, Strasbourg), for providing us with the opportunity to carry out the chemical analyses. Our warmest thanks also go to A. Fluck (UMR 7140, Strasbourg), R. Mayer, W. Kazone, and Prof. Joseph Moran (Laboratory of Chemical Catalysis at ISIS, Strasbourg) for granting us access to the equipment.

The archaeological excavations were directed by Bruno Bazin, and funded by the Chartres Archaeology Department and by the French Ministry of Culture through the "Service Régional d'Archéologie" of the Centre Region.

References

Acovitsioti-Hameau, A., Hameau, P., Rosso, T., 1993. Fours à cade, fours à poix : de l'étude expérimentale à la distillation expérimentale. Techniques & Culture 22, 105–143.

Adam, J.-P., 1989. La construction romaine : matériaux et techniques, second ed. A. et J. Picard, Paris, 368 p.

A. Coutelas et al.

- Aufan, R., Thierry, F., 1990. Histoire des produits résineux landais : goudrons, poix et brais gras depuis l'Antiquité en Buch, Born et Marensin. Société Historique et Archéologique d'Arcachon, Arcachon, p. 190.
- Bailly, L., Adam, P., Charrié, A., Connan, J., 2016. Identification of alkyl guaiacyl dehydroabietates as novel markers of wood tar from Pinaceae in archaeological samples. Org Geochem. 100, 80–88.
- Bailly, L., 2015. Caractérisation moléculaire et isotopique de goudrons et résines archéologiques dérivés de conifères en contexte maritime, Thèse de doctorat de l'Université de Strasbourg, 494 p.
- Ball, L.F., 2002. How did the romans install revetment? Am. J. Archaeol. 106 (4), 551–573.
- Bazin, B., 2013. Le complexe monumental suburbain de Saint-Martin-au-Val (Chartres, Eure-et-Loir). État de la recherche (2006–2011). Gallia 70 (2), 91–195.
- Bazin, B., Bouilly, E., 2021. Le complexe cultuel gallo-romain de Saint-Martin-au-Val, Rapport annuel de fouilles archéologiques programmées triennales 2019-2021, Ville de Chartres, Direction de l'archéologie, 502 p.
- Bazin, B., Hérouin, S., Joly, D., 2014. Démantèlement et abandon d'un grand sanctuaire à Chartres (*Autricum*) dès la première moitié du III^e siècle apr. J.-c. Gallia 71 (1), 13–24.
- Bitterer, T., 2013. Marmorverkleidung stadtrömischer Architektur Öffentliche Bauten aus dem 1. Jahrhundert v. Chr. bis 7. Jahrhundert n. Chr., Thèse de doctorat à l'université Ludwig-Maximilian de Munich, 585 p.
- Blanc, A., Blanc, P., 2010. Quelques carrières de marbre oubliées du centre de la France, in: Ducastelle, J.-P. (Ed), Aspects de travail de la pierre en France et en Belgique de l'Antiquité à nos jours, Colloque international du 6 et 7 novembre 2009, Musée de la pierre de Mffle (Belgique), pp. 87–99.
- Carre, M.-B., 2021. Transports de matières premières par voie maritime : les données des épaves antiques dans la Méditerranée occidentale, in: Boisseuil, D., Rico, Ch., Gelichi, S. (Eds), Le marché des matières premières dans l'Antiquité et au Moyen Âge. Collection de l'École française de Rome 563, Rome, pp. 9–32.
- Charrié-Duhaut, A., Burger, P., Maurer, J., Connan, J., Albrecht, P., 2009a. Molecular and isotopic archaeology: top grade tools to investigate organic archaeological materials. C. r. Chimie 12, 1140–1153.
- Charrié-Duhaut, A., Connan, J., Darnell, M., Spangenberg, J., Szymczyk, E., Bissada, A., Albrecht, P., 2009b. Molecular and isotopic characterization of organic samples from the wreck of the Saint-Etienne merchant ship (XVIIIth century): identification of pitch, fat, hair and sulphur. Org Geochem. 40 (5), 647–665.
- Charrié-Duhaut, A., 2019. Molecular study of waterproofing agents, in: Koncani Uhač, I., Boetto, G., Uhač, M. (Eds), Zambratija. Prehistoric Sew Boat. Results of the Archaeological Research, Analysis and Study. Prapovijesni šivani brod, Rezultati arheološkog istraživanja, analiza i studija. Arheoloski muzej Istre, Pula (Monograjije i katalozi, 33), pp. 134–139.
- Colinart, S., 1998. Faces cachées de quelques portraits du Fayoum. Dossier D'archéologie 238, 18–21.
- Colinart, S., Grappin-Wsevolojsky, S., Matray, C., 1999. La cire punique: Etude critique des recettes antiques et de leur interprétation application aux portraits du Fayoum, in: ICOM committee for conservation, 12th Triennial meeting, Lyon, pp. 213–220.
- Colombini, M.P., Giachi, G., Modugno, F., Pallechi, P., Ribechini, E., 2003. The characterization of paints and waterproofing materials from the shipwrecks found at the archaeological site of the Etruscan and Roman harbour of Pisa (Italy). Archaeometry 45, 659–674.
- Colombini, M.P., Modugno, F., Ribechini, E., et al., 2005. Direct exposure electron ionization mass spectrometry and gas chromatography/mass spectrometry techniques to study organic coatings on archaeological amphorae. J. Mass Spectrom. 40 (5), 675–687.
- Connan, J., Maurin, B., Long, L., Sebire, H., 2002. Identification de poix et de résine de conifère dans des échantillons archéologiques du lac de Sanguinet : exportation de poix en Atlantique à l'époque gallo-romaine. Revue D'archéométrie 26, 177–196.
- Coutelas, A., Büttner, S., Oberlin, Ch., Palazzo-Bertholon, B., Prigent, D., Suméra, F., 2009. Le mortier de chaux, éd. Errance, Paris, 160 p.
- Coutelas, A., Hourcade, D., Bujard, S., Morin, Th., 2010. De la peinture murale au placage calcaire : données techniques sur les deux phases de décoration du *frigidarium* sud des thermes de Longeas (Chassenon, France), in: Bragantini, I. (Ed.), Atti del X congresso internazionale dell'AIPMA (Association Internationale pour la Peinture Murale Antique), Annali di archeologia e storia antica, quaderno N. 18/2, Naples, pp. 717–722.
- Davara, J., Jambrina-Enríquez, M., Rodríguez de Vera, C., Herrera-Herrera, A.V., Mallol, C., 2023. Pyrotechnology and lipid biomarkers variability in pine tar production. Archeol. Anthropol. Sci. 15 (133), 1–11.

- Duce, C., Orsini, S., Spepi, A., Colombini, M.P., Tiné, M.R., Ribechini, E., 2015. Thermal degradation chemistry of archaeological pine pitch containing beeswax as an additive. J. Anal. Appl. Pyrol. 111, 254–264.
- Elsen, J., 2006. Microscopy of historic mortars, a review. Cem. Concr. Res. 36, 1416–1424.
- Farcas, F., Touzé, P., 2001. La spectrométrie infrarouge à transformée de Fourier (IRTF), une méthode intéressante pour la caractérisation des ciments. Bulletin Des Laboratoires Des Ponts Et Chaussées 230, 77–88.
- Font, J., Salvadó, N., Butí, S., Enrich, J., 2007. Fourier transform infrared spectroscopy as a suitable technique in the study of the materials used in waterproofing of archaeological amphorae. Anal. Chim. Acta 598 (1), 119–127.
- Fröhlich, F., Schubnel, H.-J., 2000. Le lutétien : la pierre de Paris. Muséum National d'Histoire Naturelle, Paris, p. 46.
- Garnier, N., Cren-Olivé, C., Rolando, C., Regert, M., 2002. Characterization of Archaeological Beeswax by Electron Ionization and Electrospray Ionization Mass Spectrometry. Anal. Chem. 74 (19), 4868–4877.
- Giuliani, C.F., 1990. L'edilizia nell'antichita. La Nuova Italia scientifica, Rome, p. 226. Guidobaldi, F., Angelelli, C., 2005. I rivestimenti parietali in marmo (incrustationes). La tecnica di fabbricazione e posa in opera come base del progetto di conservazione, in: International Committee for the Conservation of Mosaics (ICCM), Wall and Floor Mosaics. Conservation, Maintenance. VIIIth Conference of the International Committee for the Conservation of Mosaics, Thessaloniki, pp. 33–43.
- Izzo, F.C., Zendri, E., Bernardi, A., Balliana, E., Sgobbi, M., 2013. The study of pitch via gas chromatography-mass spectrometry and Fourier-transformed infrared spectroscopy: the case of the Roman amphoras from Monte Poro, Calabria (Italy). J. Archaeol. Sci. 40 (1), 595–600.
- Loewen, B., 2005. Resinous Paying Materials in the French Atlantic, AD 1500–1800. History, Technology, Substances. Int. J. Naut. Archaeol. 34 (2), 238–252.
- Loiseau, L., 2012. Les métaux dans les constructions publiques romaines: applications architecturales et structures de production (1^{er} – III^e siècle ap. J.-C.), in: Camporeale, S., Dessales, H., Pizzo, A. (Eds), Arqueología de la Construcción III. Instituto de Arqueología de Mérida, Anejos del Archivo español de arqueología vol. 64, Merida, Spain, pp. 117–129.
- Middleton, J.H., 1892. The Remains of Ancient Rome, volume 1, ed A. and C. Black, London – Edinburgh, 393 p.
- Neri, E., Bouvier, C., de Viguerie, L., Brunelle, A., Nasr, N., 2021. Wax finishing in Roman polychrome statuary: Ganosis on the colossal head from Dougga (Tunisia). J. Cult. Herit. 51, 29–36.
- Pavía, S., Caro, S., 2008. An investigation of Roman mortar technology through the petrographic analysis of archaeological material. Constructionand Build. Mater. 22 (8), 1807–1811.
- Pecchioni, E., Fratini, F., Cantisani, E., 2020. Atlas of the ancient mortars in thin section under optical microscope, Nardini Editore, Quaderni di Kermes, Florence, 78 p.
- Ponti, R., Sinibaldi, M., 2004. Analisi delle malte di due frammenti di opus sectile parietale dalla Villa dei Quintili sull'Appia, in: Angelelli, C. (Ed), Atti del IX Colloquio dell'Associazione Italiana per Io Studio e la Conservazione del Mosaico (AISCOM), Ravenna, pp. 677–684.
- Prochaska, W., Attanasio, D., Bruno, M., 2018. Unraveling the Carrara Göktepe entanglement, in: Matetić Poljak, D., Marasović, K. (Ed), Proceedings of the XI ASMOSIA Conference (Split May 2015), pp. 175–185. DOI: https://doi.org/10.31 534/XL.asmosia.2015/02.01.
- Ribechini, E., Orsini, S., Silvano, F., Colombini, M.P., 2009. Py-GC/MS, GC/MS and FTIR investigations on LATE Roman-Egyptian adhesives from *opus sectile*: New insights into ancient recipes and technologies. Analytica Chimica Acta 638, 79–87.
- Robinson, N., Evershed, R.P., Higgs, W.J., Jerman, K., Eglinton, G., 1987. Proof of a pine wood origin for pitch from Tudor (Mary Rose) and Etruscan shipwrecks: application of analytical organic chemistry in archaeology. Analyst 112 (5), 637–644.
- Romanus, K., Baeten, J., Poblome, J., Accardo, S., Degryse, P., Jacobs, P., De Vos, D., Waelkens, M., 2009. Wine and olive oil permeation in pitched and non-pitched ceramics: relation with results from archaeological amphorae from Sagalassos, Turkey. J. Archaeol. Sci. 36 (3), 900–909.
- Seigne, J., 1999. Technique de construction en Gaule romaine, in: Bessac, J.-C., Journot, F., Prigent, D., Sapin, C., Seigne, J., La construction, la pierre. Ed. Errance, Paris, pp. 53–99.
- Trintignac, A., 2003. La production de poix dans la cité des Gabales (Lozère) à l'époque gallo-romaine. Revue Archéologique De Picardie 1–2, 239–248.
- Wurch-Kozelj, M., Kozelj, T., 1999. Fours à chaux et fours à poix à Thasos de l'Antiquité à nos jours, in: Schvoerer, M., Archéomatériaux, Marbres et autres roches, Actes de la 4e conférence internationale Asmosia IV, Presses universitaires de Bordeaux, Bordeaux, pp. 359–368.