

‘NOT ALL THAT IS WHITE IS LIME’—WHITE SUBSTANCES FROM ARCHAEOLOGICAL BURIAL CONTEXTS: ANALYSES AND INTERPRETATIONS*

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Archaeological burial contexts may include a variety of white substances, but few analyses have been published. This study reports on the physico-chemical characterization of such residues from seven archaeological sites. It is often assumed that white materials from burial contexts are lime. Our findings demonstrate that they can be gypsum, calcite (chalk), aragonite, brushite, degraded metal, natural (gum) resins or synthetic polymer-based products. These may be present as the result of diagenetic processes, funerary practices or modern contamination. This paper provides an analytical approach for the holistic investigation of white materials encountered in burial contexts.

KEYWORDS: TAPHONOMY, DIAGENESIS, FUNERARY DEPOSITS, GYPSUM, CALCITE, BRUSHITE, XRD

*Received 26 March 2018; accepted 12 November 2018

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INTRODUCTION

When white substances are found in burial contexts, it is often assumed that the white material is lime (CaO or Ca(OH)₂) or another derivative of limestone. Specific traditions of burial in lime are documented in archaeological contexts (Schotsmans *et al.* 2014, 2015, 2017), but archaeologists have to be careful of automatically interpreting white materials as intentionally applied limestone derivatives. The burial of the Blessed Idesbald provides one example (Van Strydonck *et al.* 2016). Initially, researchers thought that chalk, a soft form of limestone of late Cretaceous age, was present at the bottom of Idesbald's coffin. Analysis, however, showed that the white powder was lead carbonate, probably produced by a reaction between the lead coffin liner and the decomposing body.

The aim of the present study is to highlight the importance of physico-chemical identification of unknown white substances encountered in burials during excavations. This paper details the varied analytical outcomes of the analysis white materials, other than lime. Specific cases from different time periods were selected in order to demonstrate the range of materials that can be encountered. The results show that characterization of white substances can aid in understanding diagenetic processes and help with the interpretation of funerary practices.

METHODS

The following method for the analysis of unknown white substances was established. Raman spectroscopy was used as an initial, non-destructive screening technique for sample characterization. If the spectral signature appeared to be organic, further analysis by gas chromatography – mass spectrometry (GC–MS) was carried out. Characterization of inorganic components was performed on powdered samples by X-ray diffraction (XRD). Where the spectral signature obtained by Raman spectroscopy was unclear, elemental analysis was carried out by energy-dispersive X-ray fluorescence (ED-XRF). Sample micromorphology was explored with scanning electron microscopy (SEM). In addition, electron probe microanalysis (EPMA) was applied to bone samples of one site and X-radiography to powdered material from another site.

Raman spectra were recorded on powdered samples using a Renishaw InVia confocal Raman microscope operating at 785 nm excitation, to limit interference from fluorescence. The spectral data were scanned for the acquisition of up to 10 accumulations and 10 s laser exposure time with a spectral footprint of about 2 μm (×50 objective lens).

X-ray diffraction patterns were recorded on a Bruker D8 diffractometer (wavelength of X-rays 0.154 nm, Cu source, voltage 40 kV and filament emission 40 mA). Samples were scanned from 10 to 90° (2θ) using a 0.01° step width and a 1 s time count. The divergence slit was 0.3°. The powder patterns were evaluated using the DIFFRAC.EVA V3.1 software package.

The elemental composition of the samples was investigated using a portable SPECTRO xSORT X-ray fluorescence spectrometer (ED-XRF) from Ametek, equipped with a silicon drift detector (SDD) and a low-power W X-ray tube with an excitation source of 40 kV. Samples were positioned above a 7 mm diameter aperture and analysed over an acquisition time of 10 s.

Samples submitted for organic residue analysis were solvent extracted (dichloromethane: methanol, 2:1 v/v), derivatized (*N,O*-bis (trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane) and characterized using established protocols (Stern *et al.* 2008; Brettell *et al.* 2014). Analysis was carried out by combined GC–MS using an Agilent 7890A GC system, fitted with a 15 m × 0.25 mm, 0.25 μm DB-5MS UI 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple-axis mass selective detector. The

analytical column was directly inserted into the ion source, where electron ionization (EI) spectra were obtained at 70 eV with a full scan from m/z 50 to 800 amu.

For SEM analysis, white powdery deposits were mounted on a stub with conductive carbon cement (Leit C) and gold coated. They were analysed using a scanning electron microscope (JEOL JSM-6460LV) in high-vacuum mode with an energy-dispersive spectrometer (Oxford X-Max 20). Measurements were made with a beam accelerating voltage of 20 kV and a working distance of 8 mm.

In order to find out which minerals were present in the microcracks of the bone samples from Çatalhöyük, EPMA was undertaken. Histological slides of transversely sawn femora were prepared and embedded in an epoxy resin (Epo-Kwick, Buehler). After grinding and polishing, the slides were gold–palladium coated because gold–palladium provided a better electrically conductive surface than carbon coating. When examining the sections, the instrument was accelerated to a 15 kV electron beam at a 15 nA beam current.

In one specific case (Tours), X-radiographs were taken to visualize the burial inclusions within the white powder. Radiographs were acquired using an Odel Genius 5001 medical X-ray generator (125 kV and 500 mA).

SITES, SAMPLES AND RESULTS

White residues associated with funerary assemblages from six archaeological sites were analysed. The results from one additional site, Rathgall, are also included to demonstrate the need to consider modern contamination.

Çatalhöyük, Turkey (Pre-Pottery Neolithic B)

The Neolithic site of Çatalhöyük, located in central Turkey, was excavated in the 1960s and from the 1990s onwards. Dated to the seventh millennium BC, the site is well known for its mudbrick houses, elaborate symbolic assemblages and subfloor burial practices (e.g., Hodder 2006). Two excavation shelters (each ~1300 m²) were erected approximately 15 years ago for protection and display purposes. Since their construction, the mudbrick buildings appear to have been adversely affected. A wide range of temperature (−17°C to +54°C) and relative humidity (9–100%) variations have been recorded within both shelters (Lingle and Seifert 2017). In addition, the North Shelter has had problems with drainage related to water accumulating from rain and snow melt (Çamurcuoglu 2008). Soil samples from 15 burials in the North Shelter showed an alkaline pH between 6.8 and 8.0, with conductivity between 4 mS and 14 mS. Soil samples from 19 burials in the South Shelter showed a slightly more alkaline pH between 8.2 and 8.9 but a much lower conductivity, between 173 µS and 1361 µS. The measurement of conductivity is directly related to the concentration of ions from dissolved salts and inorganic material in the soil (Cronyn 1990).

In many of the burials in the North area, a white crystalline substance has frequently been observed on and in the bones (Figs 1 (a) and 1 (b)). These deposits, generally referred to as ‘salts’ by the excavation team, have also been interpreted as possible adipocere (Knüsel *et al.* 2012). Because lime and gypsum burials emerged in the Neolithic Near East (e.g., Erdal 2015), samples of this white material were analysed to ensure that they were not part of funerary practices. The material was identified by XRD as gypsum (CaSO₄·2H₂O) (Table 1 and Fig. 1 (c)) composed of lenticular-shaped euhedral monoclinic crystals, as observed by SEM (Figs 1 (d) and 1 (e)). EPMA analysis also showed the presence of calcium and sulphate in microcracks and natural pores in the bone.

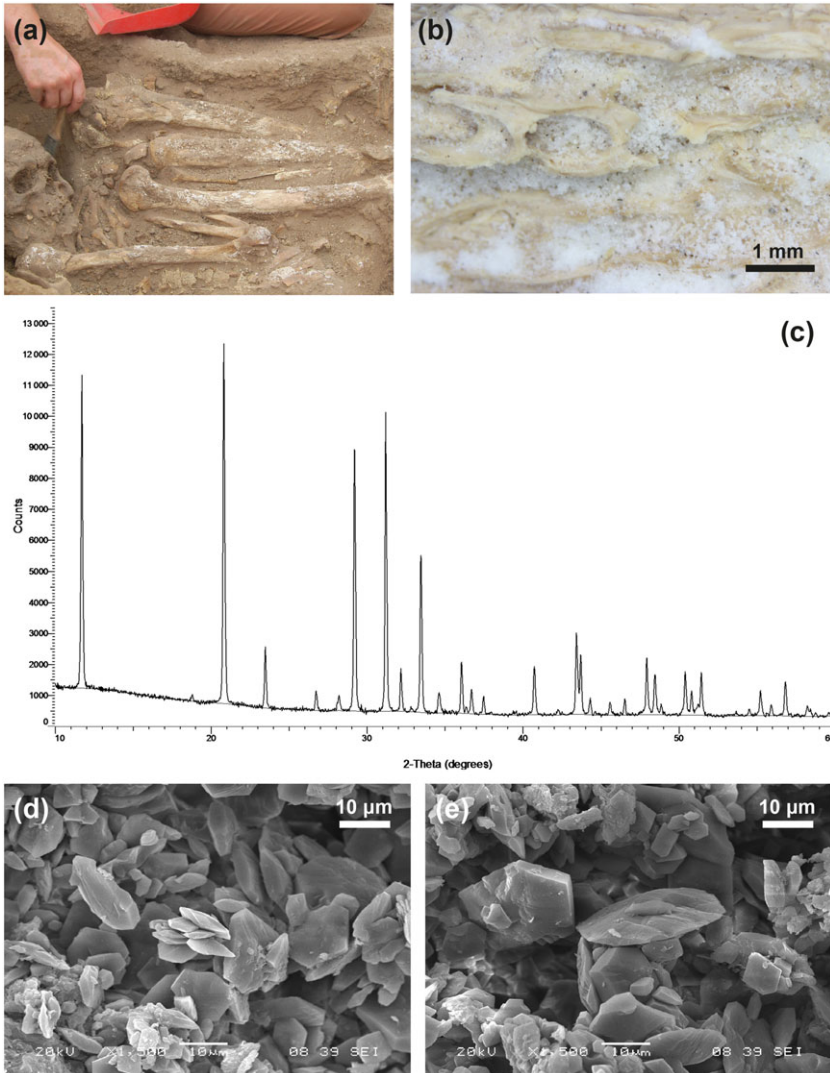


Figure 1 (a) Skeletal remains from the North Shelter in Çatalhöyük are covered with white secondary gypsum salt. (b) Detail of gypsum on trabecular bone. (c) XRD pattern demonstrating gypsum peaks ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). (d, e) SEM images showing monoclinic lenticular morphology of naturally formed gypsum. [Colour figure can be viewed at wileyonlinelibrary.com]

Rathgall, Ireland (Bronze Age)

Rathgall, in County Wicklow, Ireland, is a multi-period hillfort with Late Bronze Age funerary and industrial activity. Excavation in the late 1960s and 1970s revealed burials and cremation pyres as well as deposits related to glass and metalworking activities. During the re-examination of the finds, a white film was found on *Fraxinus* spp. charcoal from pit 118 (Raftery and Becker in press). Pit 118 was large, funnel shaped in cross-section (top, 220 cm × 190 cm; bottom, 90 cm × 95 cm) and consisted of large amounts of pottery, separated by a series of basket inserts. The white

Table 1 The characterization of white substances collected from seven archaeological sites

Site	Period	Figure	Grave details	Characterization	Taphonomy	Burial practice	Modern contaminant
Çatalhöyük (TR)	Neolithic	Fig. 1	White crystalline deposits on human bones in the North Shelter	Gypsum	✓		
Rathgall (IE)	Bronze Age	–	White layer on fraxinus charcoal from a funnel-shaped pit	Plasticizer		✓	✓
Mersea Island (UK)	Roman	–	White substance associated with cremated human remains in a glass urn	Gum resin		✓	
Rome (IT)	Roman	Fig. 2	Multiple burial of human remains wrapped in white plastered textile	Gypsum			
Bezannes (FR)	Roman	Fig. 3	White material above the skeleton (BZ1)	Calcite (chalk)	✓		
			White material below the base of the lead inner coffin (BZ3)	Gypsum		✓	
Sunderland (UK)	19th c.	Fig. 4	Two badly preserved skeletons with white substances on humerus and femur	Gypsum	✓		
			White material between right forearm and right os coxae (BZ4)	Brushite		✓	
Tours (FR)	20th c.	Fig. 5	White powder on coffin lid (1007.1)	Gypsum		✓	
			White powder on coffin lid (1007.2 and 1007.3)	Metal alloy	✓	✓	
			White powder on coffin lid (1007.3)	Aragonite	✓	✓	
			White lining at the bottom of the coffin (1010.1)	Gypsum	✓	✓	

substance was found on a sample of the basketry taken in the 1960s. Because the function of pit 118 was unclear, it was thought that analysis of the white residue could help with interpretation.

Analysis of this substance by Raman spectroscopy and SEM failed to characterize the material. Therefore, GC–MS analysis was carried out. The majority of the compounds present, including those of significant abundance, were found to be phthalate plasticizers (Table 1). These are components of many synthetic polymer-based products, including adhesives, glues and films, some of which are used as consolidants by conservators. A broad range of applied polymers are employed in conservation laboratories, with a more limited range of water-miscible consolidants suitable for field application. These include polyvinyl acetate emulsions and acrylic colloidal dispersions, to which phthalate plasticizers are added in order to improve flexibility and durability (Horie 2010). It seems likely, therefore, that the white layer had been applied in an attempt to consolidate the charcoal in the field. This result highlights the need to consider post-excavation processes and shows that information about specific treatments may no longer be available when materials are studied after a considerable period of time.

Mersea Island, Colchester, United Kingdom (Roman)

In 1912, excavation of a burial mound situated on Mersea Island, Essex, UK, uncovered a Roman cremation burial. Dated to the second century AD, re-evaluation of the find in 2012 demonstrated that the remains of a robust, mature male had been placed in a glass vessel inside a lead box within a tile-built chamber at the centre of the mound. A significant quantity of a whitish residue was found coating the bones (McKinley 2014). Visual inspection of a subsample of this substance revealed orange crystalline fragments embedded in a white matrix. Screening by Raman spectroscopy indicated that both fractions were resinous in nature. Analysis by GC–MS confirmed the presence of terpenic compounds (polycyclic hydrocarbons, terpenes and related, oxygen-containing, terpenoids) characteristic of natural resins/gum resins (Table 1). The diagnostic, higher-molecular-mass, compounds were identified as diterpenoids with abieta(e)ne and pimara(e)ne skeletons, cembrene- and verticillane-type diterpenes and triterpenic compounds with oleana(e)ne and ursane(e)ne skeletons. This combination does not occur in nature. Thus, two different resinous exudates, an abundance of the gum resin frankincense (*Boswellia* spp.) and a lesser amount of a conifer (Pinaceae) resin, had been deposited in the cremation urn. In addition, the survival of an array of mono- and sesquiterpenes demonstrated that this find represented an unburnt offering and that exceptional taphonomic conditions had prevailed within the burial chamber. Due to their volatility, these lower-molecular-mass compounds are normally rapidly lost upon heating and rarely endure over archaeological time (Brettell *et al.* 2018).

The central sector of the catacomb of Saints Peter and Marcellinus, Rome, Italy (Roman)

Excavations conducted from 2004 to 2010 in the central area of the catacomb of Saints Peter and Marcellinus, Rome, Italy, revealed multiple burials in six chambers, dated from the end of the first to the end of the third century AD. A mortality crisis following an epidemic is thought to be at the origin of these burials (Castex *et al.* 2011; Kacki *et al.* 2013). Burial chamber X80/T16 was rectangular (1.22 m × 2.05 m), with a height of 2.97 m (7.4 m³) and contained more than 70 individuals wrapped in plastered textile (Figs 2 (a) and 2 (b)), initially thought to be lime plaster. Due to the poor skeletal preservation, the exact number of skeletons was difficult to estimate. Likewise, the study of the biological identity of these individuals remained limited.

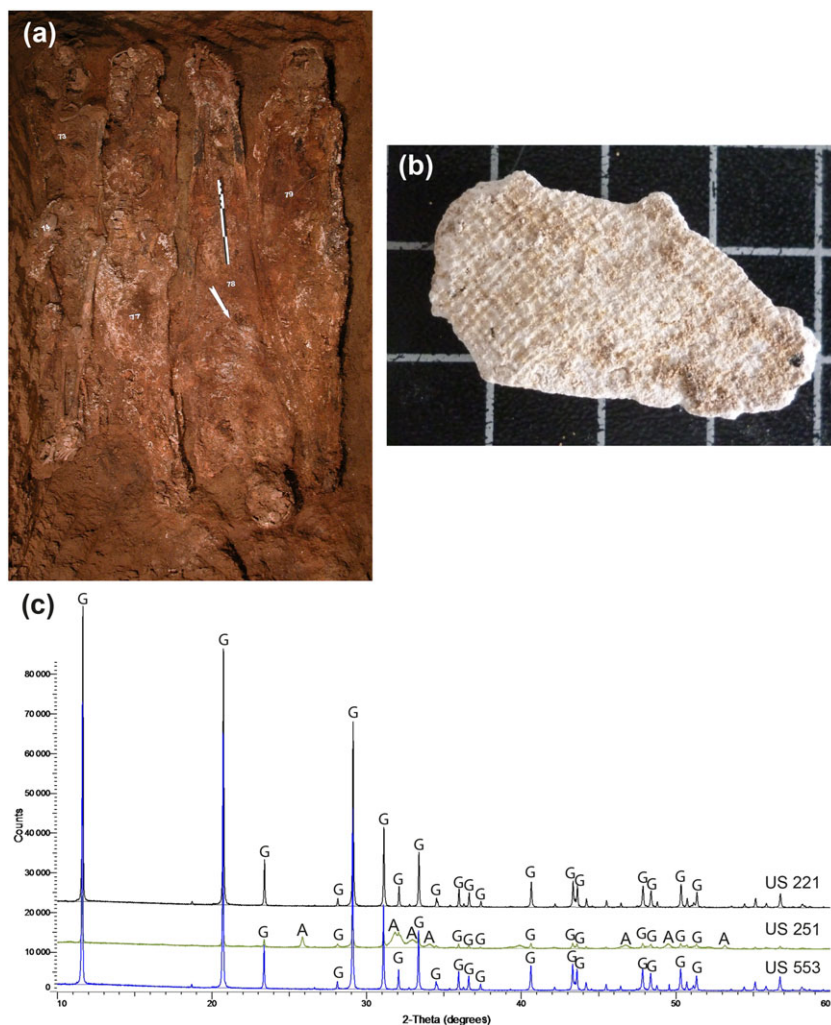


Figure 2 (a) Human remains wrapped in plastered textile in burial chamber X80/T16 of the catacomb of Saints Peter and Marcellinus in Rome. (b) A textile impression in the gypsum plaster. (c) XRD pattern of plaster samples from the upper layers (US 221), middle layers (US 251) and lower layers (US 553), with identified mineral phases of gypsum (G) ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and apatite (A) ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$). [Colour figure can be viewed at wileyonlinelibrary.com]

Environmental monitoring within burial chamber X80/T16 showed temperatures between 16.6°C and 17.6°C and an extremely high relative humidity of 100%, without fluctuations.

Three-dimensional (3D) reconstruction showed that not all of the individuals were deposited at the same time, based on a calculation of the volume of the estimated number of corpses with soft tissue that could fit within the space of the burial chamber at one time (Sachau-Carcel *et al.* 2013). This means that several phases of deposition were necessary to deposit the bodies, separated by a time interval of unknown duration, but sufficient enough to allow at least partial decomposition of the corpses.

Fifty samples of white material from different individuals were analysed on mineralogical composition, pH and colour in order to characterize this material and to ascertain if there were

differences in the plaster composition related to possible different phases of deposition. Analysis by XRD indicated that all samples consisted of gypsum plaster, sometimes mixed with calcium phosphate minerals derived from degraded bone (Table 1 and Fig. 2 (c)). Different compositions within the gypsum plaster could not be detected. The pH of the gypsum samples was mildly acid, ranging from 5.3 to 6.8. The colour ranged from white to very pale brown. No distinct differences in pH or colour could be discerned between the upper, middle and lower layers of human remains. Additional microscopic observations revealed that some of the white samples contained yellow–reddish-coloured particles. These were identified using GC–MS as Baltic amber and sandarac resin, with black matter interpreted as *Boswellia* sp. bark, used for embalming (Devièse *et al.* 2017).

Bezannes, France (Roman)

During excavations at Bezannes in Champagne-Ardenne, near Reims, France, 11 inhumations, dated to the second half of the third century AD, were uncovered. One of these, F77, consisted of a lead-lined wood coffin (1.85 m × 0.46 m × 0.31 m) within a rectangular grave cut. The burial contained supine, extended human skeletal remains encased in a white packing (Figs 3 (a) and 3 (b)). Although the cranium and lower extremities were well preserved, the bones from the upper body, pelvis and upper extremities were very fragile. Neither sex nor age at death could, therefore, be determined. The white packing material was observed both on top and below the skeletal remains with textile impressions on its inner surface (Fig. 3 (c)). In addition, the wooden outer coffin had totally decayed, leaving only iron coffin nails as evidence. The inner lead liner showed a mixed state of preservation. Thus, although the lid was in good condition, the base of the coffin was poorly preserved. As a result, white material from the plaster packing was also found under parts of the lead inner liner. However, since the geology of this region is chalk, questions were raised about the precise nature of material observed.

Three samples of white material associated with F77 were analysed. BZ1 was recovered above the skeleton, but not in contact with it; BZ3 was sampled from below the base of the lead liner, where it had decayed; and BZ4 was recovered from between the right forearm and right os coxae.

Using XRD, sample BZ1 was identified as calcite (CaCO₃) (Table 1 and Fig. 3 (g)) and samples BZ3 and BZ4 as gypsum (CaSO₄·2H₂O) (Table 1 and Fig. 3 (g)).

SEM analysis confirmed the difference between samples BZ1 and BZ3–BZ4. Sample BZ1 was identified as chalk, which is recognizable by the presence of coccoliths, planktonic single-celled algae with a calcite spherical skeleton (Fig. 3 (e)). The micromorphology of BZ3 and BZ4 showed monoclinic gypsum (Fig. 3 (f)).

Evaluation of the available samples by GC–MS revealed compounds indicative of heavily degraded animal and plant tissues. These may represent traces of the decomposing body and the remains of floral tributes. No biomarkers for resinous exudates were observed. Botanical analysis confirmed abundant and diverse pollen, showing high values of taxa that are not compatible with the environmental record. Two samples of dark organic residues recovered below the left hemithorax and the right hand were characterized by the dominance of Urticaceae pollen (*Urtica dioica* type) and two exotic taxa (*Acacia senegal* type and *Ecbolium*) (Fig. 3 (d)). Gypsum below the right femur contained high amounts of cereal pollen. Below the left femur, Urticaceae (nettle) and cereal pollen along with other non-European taxa (*Hyphaene thebaica* type and *Gardenia*) were discovered (Fig. 3 (d)). All four exogenous morphotypes probably originate from a wooded savanna area in, for example, Yemen, Sudan or Ethiopia. Finally, samples below the cranium and above the skeleton appeared to be very poor in pollen.

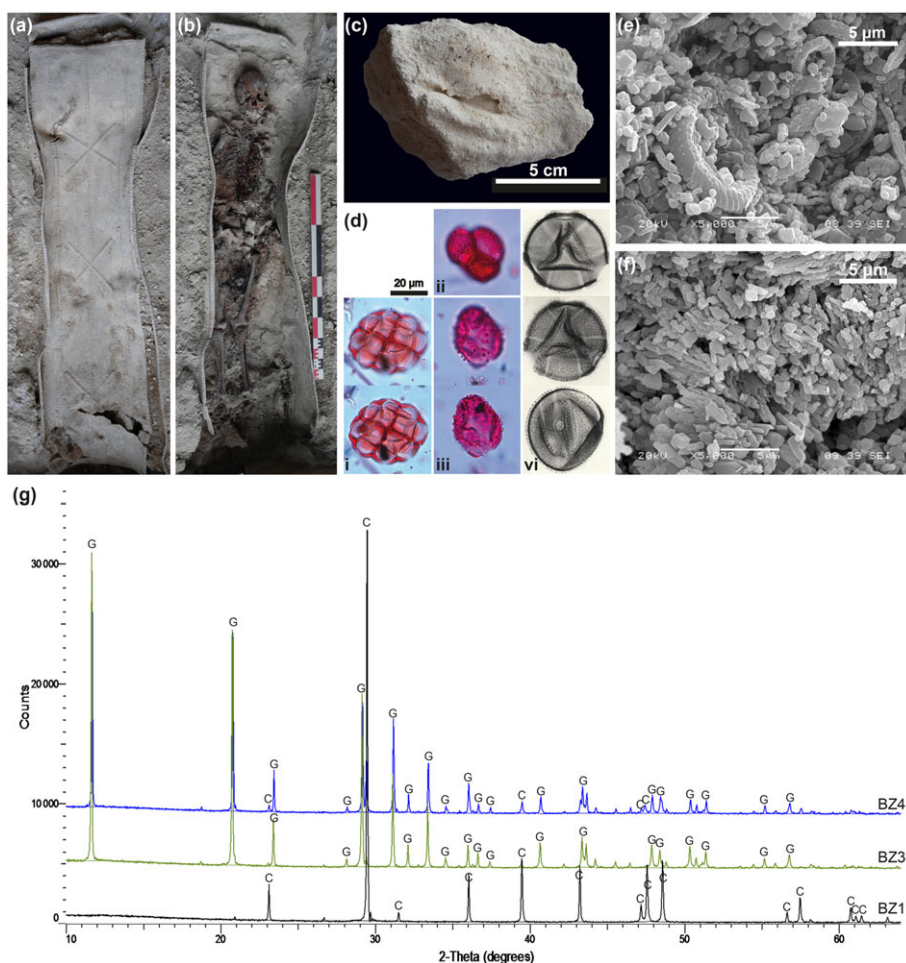


Figure 3 (a, b) Burial F77 from Bezannes consisted of a lead-lined coffin with a supine skeleton encased in a white packing. (c) Textile impressions in the gypsum plaster: (d) Botanical analysis revealed pollen grains of (i) *Acacia senegal* type, (ii) *Gardenia*, (iii) *Hyphaene thebaica* type (determination, G. Buchet; images, R. Corbineau) and (iv) pollen of *Ecobolium*: for the latter, a modern specimen from the reference collection of the CEREGE laboratory is shown. (e) The micromorphology of BZ1 shows the presence of coccoliths, indicating that this sample was part of the chalky burial environment. (f) The micromorphology of BZ3 and BZ4 showed monoclinic gypsum. (g) Analysis by XRD characterized sample BZ1 as calcite (C) (CaCO_3) and samples BZ3 and BZ4 as gypsum (G) ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). [Colour figure can be viewed at wileyonlinelibrary.com]

Bethel Chapel Crypt, Sunderland, United Kingdom (19th century)

In 2010, the Bethel Chapel Crypt, Sunderland, UK, was excavated, as part of an urban development programme. These underground burial vaults, built in 1826, held an estimated 409 individuals by the time they were decommissioned in 1852. The coffins were then covered with sanitizing sand (beach sand), as dictated by the 1852 Burial Act. During excavation, the project evaluated the preservation of the human remains in different parts of the crypt. White substances were noted on some of the bones. Vaults 4Ai and 6Aii from the southern side of the crypt were studied. Both vaults contained poorly preserved skeletons in surviving wooden coffins (Fig. 4 (a)). White

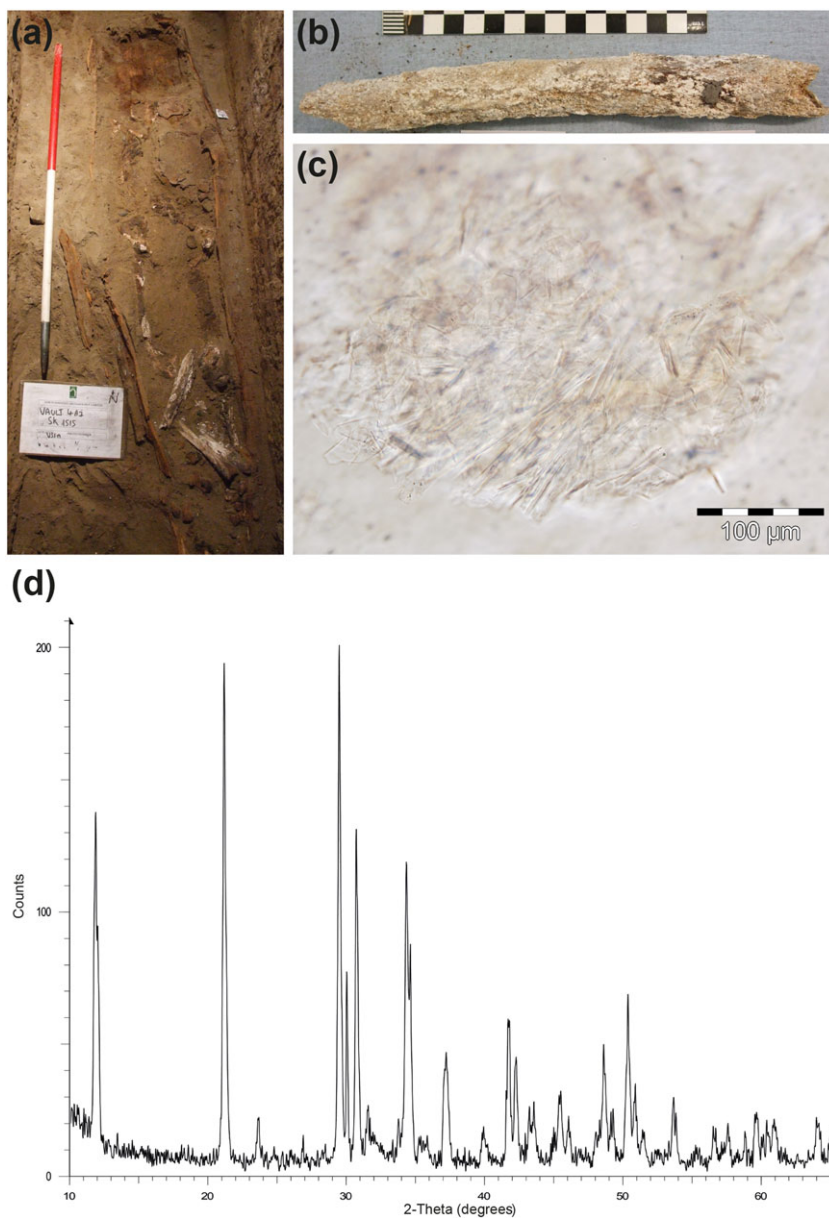


Figure 4 (a) Vault 4Ai in the Bethel Chapel Crypt in Sunderland contained a poorly preserved skeleton in surviving wooden coffin. (b) The badly preserved femur of the individual in vault 4Ai was covered with white residue. (c) Microscopic examination revealed needle-like crystals growth. (d) XRD analysis identified mineral phases of brushite ($\text{CaHPO}_4 \cdot (\text{H}_2\text{O})_2$). [Colour figure can be viewed at wileyonlinelibrary.com]

residue was found on the left femur of the individual in vault 4Ai (Fig. 4 (b)), and on the right humerus of the individual in vault 6Aii. The material displayed a needle-like morphology when observed under the microscope (Fig. 4 (c)).

Raman spectra obtained from both samples represented the mineral brushite ($\text{CaHPO}_4 \cdot (\text{H}_2\text{O})_2$) (Table 1). Raman spectra of calcium phosphates are dominated by strong HPO and PO stretching bands, which appear in the $900\text{--}1000\text{ cm}^{-1}$ spectral range and phosphate bending and PO bands in the $400\text{--}600$ and $1000\text{--}1100\text{ cm}^{-1}$ ranges, respectively. Peaks near 1060 , 988 , 878 , 525 and 380 cm^{-1} are diagnostic features of brushite (e.g., Frost and Palmer 2011). The presence of brushite was confirmed using XRD (Fig. 4 (d)). Soil analysis of the sand that was encasing the coffin showed a mildly acidic pH between 6.2 and 6.6 and the presence of calcite.

Cimetière des religieuses du Sacré Cœur, Tours, France (20th century)

In 2011, the area near the medieval abbey of Marmoutier at Tours, Indre-et-Loire, France, which had been used in the second half of the 19th and 20th centuries AD as the cemetery for the Sacré-Coeur convent, was partially excavated. Several inhumations of nuns were exhumed, with the aim of reburying the remains at the main cemetery of Tours. Burial 2 consisted of an adult female interred supine in a wooden trapezoidal coffin ($1.80\text{ m} \times 0.58\text{ m} \times 0.44\text{ m}$) (Fig. 5 (a)). The skeletal remains and the coffin were well preserved. Black remnants of textile indicated the presence of a traditional habit (Blanchard *et al.* in press) (Fig. 5 (b)).

A whitish powder with green and blue reflections was found on top of the coffin lid (US 1007) (Figs 5 (a) and 5 (c)). A part of the powder was recovered and put aside for archaeometric analysis. The bottom of the coffin appeared to be covered in a white material (US 1010) (Fig. 5 (b)).

The white powder on the coffin lid (US 1007) consisted of smaller and larger aggregates. First, X-radiographic analysis was carried out to determine the morphological features and degradation state. The larger piece of the aggregates ($78\text{ mm} \times 27\text{ mm}$, 20 g) had a recognizable anthropomorphic shape that corresponded to the lower parts of the human body, representing the abdomen and lower limbs of a figure of Christ (Fig. 5 (g)). The exact nature of the smaller fragments in the powder could not be identified radiographically, but were characterized as gypsum with XRD, possibly related to a gypsum plaster frame (Table 1 and Fig. 5 (h)).

Two other subsamples of white powder from the coffin were analysed (US 1007.2 and 1007.3). Microscopically, they showed white, blue, yellow and black colours (Fig. 5 (d)), indicating a possible mix of several materials. No clear defined morphology could be observed with SEM. XRD analysis showed that sample 1007.3 was a mix of aragonite (CaCO_3), quartz (SiO_2), aluminium and unclear (alloy) peaks (Table 1 and Fig. 5 (h)). Similarly, aluminium was observed in sample 1007.2 (Fig. 5 (h)).

The white powder was further characterized by ED-XRF as an alloy of aluminium, iron (Fe), copper (Cu), zinc (Zn), lead (Pb) and tin (Sn) (Fig. 5 (i)). ED-XRF confirmed the presence of calcium (Ca) (cf., aragonite) in sample 1007.3 and aluminium (Al) in both 1007.2 and 1007.3.

Upon visual examination of the white material that covered the bottom of the coffin (US 1010), it was observed that the underside of the plaster had taken the structure of the wood from the coffin (Fig. 5 (f)). Sample US 1010 was identified as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) by XRD (Table 1 and Fig. 5 (h)). Observations by SEM showed an elongate lath micromorphology (Fig. 5 (e)). The pH of this sample indicated a neutral to slightly acid pH of 6.5, which fits with the pH of gypsum plaster.

DISCUSSION

The analysis of white residues from seven archaeological contexts demonstrates that such materials can be variably characterized as gypsum, calcite, aragonite, brushite, degraded metal, natural (gum) resins or synthetic polymer-based products. Their presence in the burial record

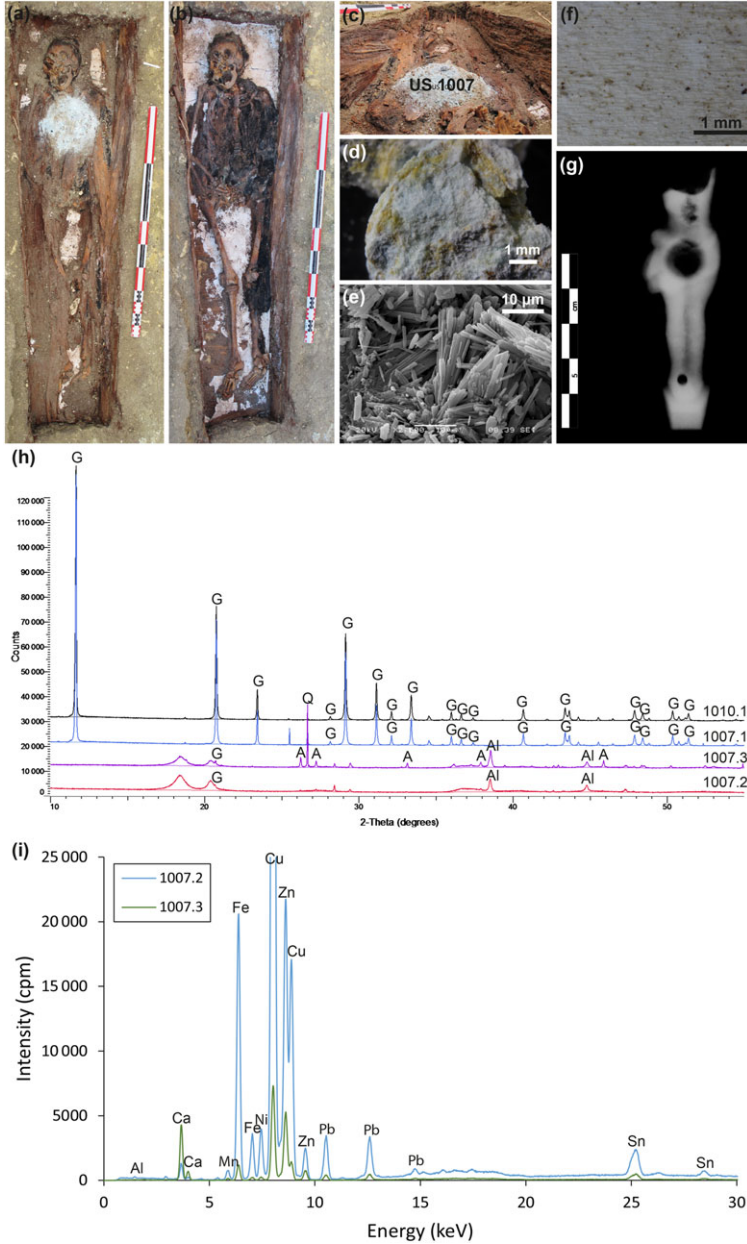


Figure 5 (a) Burial 2 from Tours consisted of an adult female buried supinely in a wooden trapezoidal coffin. (b) The bottom of the coffin was covered in a white material (US 1010). (c) White powder was present on the coffin lid (US 1007). (d) Microscopically, US 1007 showed white, blue, yellow and black colours. (e) SEM showed the elongated lath micromorphology of the gypsum (US 1010). (f) The underside of the gypsum layer at the bottom of the coffin (US 1010) had taken the structure of the wood from the coffin. (g) Radiographic analysis of US 1007 revealed the presence of the abdomen and lower limbs of a figure of Christ (X-ray parameters: 51 kV, 25 mAs and 50 mA). (h) US 1010 was characterized by XRD as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) (G) and sample US 1007 as a mix of aragonite (A) (CaCO_3), quartz (Q) (SiO_2), aluminium (Al) and unclear (alloy) peaks. (i) Further analysis with ED-XRF showed that US 1007 consisted of an alloy of aluminium (Al), iron (Fe), copper (Cu), zinc (Zn), lead (Pb) and tin (Sn). [Colour figure can be viewed at wileyonlinelibrary.com]

can be shown to be the result of (1) modern contamination, (2) interactions with the depositional environment and/or (3) funerary practices.

Modern contaminants

The traces of a synthetic polymer-based product on the charcoal sample from Rathgall shows that researchers should consider a broad range of possible sources for any white materials present on artefacts and ecofacts. It also highlights the issue of plasticizer contamination and cautions against the use of consolidants in the field, as such knowledge may be lost over time. Plasticizer contamination can arise from a wide range of modern practices, including storage in plastic containers or bags and the application of adhesive labels or glues, as well as the use of synthetic stabilizers. This result demonstrates the importance of documenting every step in the excavation and post-excavation process so that the chain of evidence can be reconstructed when analytical questions are raised.

Taphonomic processes and diagenesis

The results from the analysis of white residues recovered from Çatalhöyük, Sunderland, Tours and Bezannes (BZ1) provide insights into the depositional environment.

At Çatalhöyük, analysis of white crystals observed on the bones excavated in the North Shelter were found to be composed of secondary gypsum, which is created by the post-depositional crystallization of geological gypsum. Secondary gypsum was also observed in the midden formations (Shillito and Matthews 2013) and in thin sections from the plaster on the walls and floors (Anderson *et al.* 2014). Across Turkey, including on the Konya plain, where Çatalhöyük is located, gypsum is a common geological mineral, which was formed in the shallow, saline-water conditions associated with the hot and dry climate of the Early Holocene (Inoue *et al.* 1998). In arid and semi-arid environments where the soil is gypsum rich, such as those that prevail in the region, secondary gypsum is often formed due to extensive evaporation and subsequent supersaturation (Porta and Herrero 1990; Herrero and Porta 2000). However, its occurrence is not uniform, as a range of physical and chemical conditions involving pH, temperature and hydrogeology as well as atmospheric and soil humidity must be met (Buck *et al.* 2006). At Çatalhöyük, the crystallization of secondary gypsum was more commonly observed in burials from the North Area, with lenticular gypsum formed in conditions of an alkaline pH, a high Ca:SO₄ ratio and in the presence of organic matter (Cody 1979; Kushnir 1980; Poch *et al.* 2010). This can clearly be linked to the localized fluctuations in relative humidity and temperature on diurnal and annual cycles and the additional problems created by sorptivity and hysteresis (Lingle and Lercari 2017). Gypsum crystallization is known to be very damaging to archaeological materials (Goldberg and Macphail 2006). This is particularly true of bone because gypsum crystals eventually become embedded in the microstructure, as observed in the microcracks by EPMA, which can lead to its mechanical destruction. Although many archaeologists and osteologists are aware of this issue, only a small number of publications, which detail the recovery of archaeological artefacts from caves, mention the formation of gypsum (e.g., Wellman 1996; Bergada *et al.* 2015; Stephens *et al.* 2016). There has, therefore, been little research into occurrence and impact of gypsum on skeletal materials or at other types of archaeological sites.

In the Bethel Chapel Crypt, Sunderland, the white material on bone was identified as brushite, also known as calcium hydrogen phosphate dehydrate. This mineral has largely been studied in relation to modern osseous tissue regeneration (e.g., Penel *et al.* 1999; Xu *et al.* 1999; Alkhrasat

et al. 2013). Reactive crystallization of calcium phosphate, resulting in brushite formation, is clearly favoured by pH 6.5–7 (Arifuzzaman and Rohani 2004; Yanovska *et al.* 2012). As with secondary gypsum, brushite crystals have been observed in cave systems (Shahack-Gross *et al.* 2004; Frost and Palmer 2011; Frost *et al.* 2012), where formation has been shown to be complicated and dependent on localized concentrations of phosphate, calcium and hydrogen ions. The presence of calcite may also act as a catalyst, as a high concentration of calcium ions appears to facilitate the creation of brushite. The rapid growth of brushite crystals causes volumetric expansion, which can result in the destruction of archaeological bone (Piepenbrink 1986; Hanson and Buikstra 1987). In the Bethel Chapel Crypt, the sanitizing sand used to encase the coffins was found to contain calcite and had a mildly acidic pH of 6.2 and 6.6. These findings demonstrate that the micro-environment created by the deposition of sand in the vaults was conducive to brushite formation.

With regard to the religious burial from Tours, analysis of the powder on the coffin lid indicated that these fragments represented the remains of a decorative piece consisting of a figurine predominantly made from a metal alloy in a frame of gypsum. The origin of aragonite in this powder is unclear. It could indicate that molluscs, seashells or coral were used as decoration (Edwards and Farwell 2008; Weiner 2010) or aragonite could have been present in the burial environment. This cannot be confirmed, however, as reference samples of the surrounding soil were not available for analysis. The metal alloy used consisted of aluminium, iron, copper, zinc, lead and tin. As aluminium was not produced in Europe until AD 1827 (Heron 1996), this provides a *terminus post quem* that fits with the proposed time period for the burial (second half of 19th to early 20th centuries AD). The fact that the original shape of the item was no longer recognizable, and that the material was highly comminuted, may relate to the metal alloy used for the figurine. The rate of metal corrosion depends on a number of factors, including the nature of the alloy itself and the amount of moisture and oxygen present, as well as the pH, salinity and composition of the soil (Janaway 1996). With regard to this find, galvanic corrosion, also called bimetallic corrosion, needs to be considered. Galvanic corrosion occurs when two metals, with different potentials, are in electrical contact while immersed in an electrically conducting corrosive liquid (electrolyte). When this occurs, the natural processes of corrosion will be accelerated (Palanna 2009). It appears, therefore, that the interactions between the burial environment and the alloy used to create the figurine had a detrimental effect upon the survival of this coffin decoration.

As with the aragonite observed in the religious burial at Tours, sample BZ1 from the Roman inhumation at Bezannes is almost certainly derived from the burial environment. Characterized as chalk, it was collected from above the skeletal remains, which had been interred in a chalk substrate in a wood coffin (subsequently decayed) with an inner lead liner that had become partially corroded over time. The sample, therefore, probably entered the burial as part of the taphonomic process.

A clear understanding of the diagenetic processes that have created the archaeological record can inform us about the nature, rate and degree of degradation of the materials recovered and their relationship with the burial environment. Such knowledge about the differential impact of taphonomic factors on human remains and associated ecofacts and artefacts can assist greatly with the interpretation of funerary practices. It can also aid heritage management, as active monitoring and protective measures can be put in place to safeguard not only items in storage or on display in museums but also those that remain in the field (e.g., at Çatalhöyük).

As our findings show, materials utilized in funerary practices are often adversely affected by the depositional environment. They themselves, however, can also impact on the condition of any associated skeletal remains and these effects may be highly variable. Thus, in the gypsum burials

evaluated here, the skeletal remains from the Roman catacomb were particularly poorly preserved. In Bezannes, the upper extremities, upper body and pelvis were, likewise, in a poor condition, although the cranium and lower extremities were better preserved. The skeletal remains of the nun interred at Tours were, however, well preserved. It has often been assumed that gypsum has an adverse effect on bone preservation, although little research has been undertaken in this field. Farwell and Molleson (1993) observed that gypsum burials from Poundbury, UK, that remained dry were better preserved than gypsum burials that had been affected by groundwater seepage. The poor level of preservation of the skeletal remains in the Roman catacomb and the mildly acid pH would seem, therefore, to be due to the humid, moist environment demonstrated by a relative humidity of 100% during all months of the year. Similarly, at Bezannes, the variation in skeletal preservation can probably be related to the hydrogeological character of the site with moisture ingress and, perhaps, pooling of decomposition fluids localized in the region of the torso and pelvis. This also destroyed the base of the lead liner. The treatment of the bodies at these two sites may be another factor in their differential preservation, as those in the Roman catacomb were fully encased in plastered textiles whereas, at Bezannes, the gypsum appears to have been added as a packing and may have been more concentrated in the region of the trunk. The relatively good skeletal preservation of the remains in the religious burial at Tours is, therefore, probably the result of the individual having placed on a layer of gypsum rather than receiving a body coating. Lower moisture conditions must also have played a part.

Funerary practices

The analysis of white residues recovered from the plural burial in the central sector of the catacomb of Saints Peter and Marcellinus, the inhumation at Bezannes, the religious interment at Tours and the cremation urn in the Mersea Island burial mound add to our understanding of funerary practices.

These diverse inhumation burials provide examples of three different types of intentional burial with gypsum. For centuries (*c.* 11th millennium BP to present day), white plaster (variously gypsum, lime or chalk) has been added to burials for many different reasons, although such finds have rarely been analysed (Schotsmans *et al.* 2015). For example, the earliest intentional use of gypsum in a funerary context, as confirmed by analysis, comes from Körtik Tepe in Turkey and dates to the Pre-Pottery Neolithic A (Erdal 2015). In the Roman period, burials containing both lime and gypsum body coatings have been reported from many provinces of the Empire (de Larminat 2012) while, from the Middle Ages to the present day, most plaster burials appear to involve the use of lime. The gypsum lining at Tours is, therefore, of particular interest, as it is the only confirmed example from the post-medieval period of which the authors are aware.

Observations regarding the method of application of the gypsum also show that it was applied in three different ways: to textiles wrapped around the bodies in the Roman catacomb (Fig. 2 (a)), as plaster packing in Bezannes (Fig. 3 (b)) and as a coffin lining in Tours (Fig. 5 (b)). These findings raise questions about the intended function of the gypsum, as it is possible that different application methods may have held a different symbolic significance. However, more research is required before drawing such conclusions, as not all of the plaster originally deposited in these burials may have remained *in situ* and because adding plaster to burials could have had multiple purposes (Schotsmans *et al.* 2015). Nonetheless, on the basis of current evidence, it seems that it could have been applied for practical (e.g., encasing the body to reduce odour and delay invasion by decomposer organisms) or symbolic (e.g., maintenance of bodily integrity or marking a quality of the individual) reasons. The use of gypsum-plastered textiles as body wrappings in

the central sector of the catacomb of Saints Peter and Marcellinus could, therefore, have had both a symbolic and a practical purpose. These individuals were contained by gypsum plaster and perfumed with natural resinous substances. This may have facilitated handling and transportation, but could also have formed part of a cleansing rite or even denoted religious affiliation. It would certainly have minimized the odour of decay and delayed the egress of bodily fluids, which would have been important, as it appears that there were multiple deposition episodes within the burial chamber. The addition of straw and nettles to the gypsum plaster in the Bezannes inhumation might have had a similar purpose.

In contrast, the coffin lining in the religious burial at Tours probably had a symbolic (e.g., purity, chastity or bride of Christ) or visual function, as it would have provided a contrast with the black habit in which the nun had been buried. This thin layer at the bottom of the coffin could have been applied for practical reasons too, such as the absorption of putrefaction liquids, but this seems less likely, as gypsum, when used as wall plaster, is never applied with the intention of 'absorbing' liquids (Weiner 2010). The comminuted residues on the coffin lid of this burial definitely held a symbolic meaning, as they formed part of a decorative element, a crucifix.

Finally, the presence of pollen from exotic taxa in Bezannes, the provision of resinous substances as an unburnt offering in the Mersea Island burial mound and their presence, in conjunction with gypsum plaster, as part of a body treatment, in the catacomb of Saints Peter and Marcellinus broadens our knowledge of sociocultural aspects of the Roman world. Clearly, certain individuals were accorded highly elaborate funerary practices, which included the use of imported plants and their exudates. Such finds have now been attested in inhumation burials across the Roman Empire, with the example from Mersea Island providing the first molecular confirmation from a cremation burial (Brettell *et al.* 2015, 2018).

CONCLUSION

The findings of this study demonstrate that burials and their inclusions should be carefully analysed with reference to their depositional environment, knowledge of the local geology and an understanding of their sociocultural context. Funerary practices can only be interpreted correctly if taphonomic and diagenetic processes are clearly understood. Analysis of the materials employed provides us with fresh insights into past social, cultural and economic practices, and enhances our knowledge regarding the degradation of skeletal and associated materials. Both aspects should, ultimately, support the development and management of our archaeological heritage. The approaches used in this paper should be viewed as individual tools in an armoury of techniques that can provide complementary and confirmatory evidence for the holistic investigation of white materials. This research demonstrates the benefits of using a wide range of analytical techniques to study white residues from burial contexts and of creating interdisciplinary collaborations between archaeology, anthropology, archaeometry, geology, botany and conservation. By applying methods from different disciplines, new and important evidence related to the treatment of the dead, taphonomic processes and modern post-excavation practices can be revealed.

ACKNOWLEDGEMENTS

The authors would like to thank the following individuals: Guillaume Buchet (CEREGE), Jo Buckberry (University of Bradford), Nadia Cantin (IRAMAT-CRP2A), Stephan Dubernet (IRAMAT-CRP2A), Aroa Garcia-Suarez (University of Reading), Raffaella Giuliani (Vatican's Pontifical Commission for Sacred Archaeology), Sue Howlett (Mersea Island Museum Trust),

Yannick Lefrais (IRAMAT-CRP2A), Jérôme Livet (INRAP), Jackie McKinley (Wessex Archaeology), Africa Pitarch-Martí (PACEA), Alain Queffelec (PACEA) and Matt Town (Northern Archaeological Associates Ltd). Special thanks are offered to Pontificia Commissione di Archeologia Sacra (Vatican), Çatalhöyük Research Project (Turkey-USA), PACEA (UMR 5199 CNRS-Université de Bordeaux) and IRAMAT-CRP2A (UMR 5060 CNRS – Université Bordeaux Montaigne). Eline Schotsmans received financial support from the French State (IdEx Bordeaux ANR-10-IDEX-03-02) and from Collaborative Projects of the France–Stanford Center for Interdisciplinary Studies.

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