



Technological and compositional variation in pink and white pastes from a coffin set of the late Third Intermediate Period (Thebes, ca. 680–664 BCE)

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ABSTRACT

Ancient Egyptian artefacts often feature an intriguing assortment of plaster-like materials. These range from authentic plasters, derived from heated lime and gypsum, to mud, calcium carbonate and calcium sulphate-based pastes bound with organic media and sometimes integrated with clay minerals and plant fibres. Although architectural plaster use in ancient Egypt has received some attention, a gap remains in the analysis and precise characterisation of the materials applied to objects. Understanding their composition can shed light on technological changes, provide contextual insights and support provenance studies. A minimally invasive approach was applied to the two coffins of Pakepu (intermediate and inner), a funerary ensemble from Thebes dating back to about 680–664 BCE, and to a group of coffin fragments from the same area and period, employed as a comparison group. The inner coffin of Pakepu and the fragments all exhibit a complex surface layering reminiscent of cartonnage. This is in contrast to the much simpler surface construction seen on the intermediate coffin. Optical microscopy, scanning electron microscopy with energy dispersive spectroscopy and Fourier transform infrared spectroscopy were employed to characterise the inorganic constituents. Ground, unheated calcite was detected in all the pastes, but differences in micromorphology and the distribution of minor elements (particularly Mg and Fe) in the Pakepu inner and intermediate coffin, and in the fragments, point to the involvement of different workshops, geological sources and manufacturing protocols in the production of these materials. (For a summary in Arabic, see **Online Resource**)

1. Introduction

From stylistic and iconographic perspectives, coffins are amongst the Egyptian archaeological artefacts that have received the most study and research attention. However, only relatively recently has detailed research been directed to their manufacturing technology and materials. This has focused on structural components of wooden and cartonnage¹ coffin construction and on the pigments, varnishes and resins used in the surface decoration of these objects (see for example Dawson, Rozeik et al. 2010; Amenta and Guichard 2017; Strudwick and Dawson 2019). An under-researched aspect is the pastes which, on coffins, are found as gap-fillers in the wooden carcasses, as substrates for painted decoration, in the creation of relief decoration, as key components of cartonnage, and in the manufacture of mould-made funerary masks (Zaggia, Dawson et al. 2024).

Architectural pastes in ancient Egypt have been the focus of more detailed analytical studies. However, in Egyptological literature, the use of inconsistent and imprecise terminology—both historically and in modern studies—has compounded the challenge of identifying these materials. Terms like gypsum, chalk, lime plaster, and whitening are often applied without consideration of their actual chemical composition, making it difficult to determine the precise nature of the materials being discussed. (Lucas 1924; Hatchfield and Newman 1991; El Hadidi and Hamed 2017).

The term “paste” is adopted in the current study, as a generic that does not a priori denote particular chemical or technological characteristics (Strudwick and Dawson 2016), and is preferred to the term “plaster”, which in reality denotes more specific technologies (namely, high-temperature treatment of limestone and gypsum) (Lucas 1924; Gourdin and Kingery 1975; Rodríguez-Navarro 2004; Philokyprou

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¹ A free-standing composite material crafted from layers of linen, glue and paste.



Fig. 1. The coffin set of Pakepu, E.2.1869 (image © Fitzwilliam Museum).

2012). Pastes on Egyptian artefacts range from mud, calcium carbonate and calcium-sulphate based preparations, bound with organic media and sometimes integrated with clay minerals and plant fibres, to these authentic plasters derived from heated lime and gypsum.

Analytical work in technical studies has led to better identification of the composition and use of pastes on individual objects, but it varies considerably in approach, techniques used and level of detail presented. Within coffins' research specifically, studies have identified the occurrence of the major components listed above and presence of minor elements. They have, in some cases, included discussion of mixtures or layering and the presence of minerals such as the magnesium calcium carbonates dolomite and huntite as natural or, potentially, intentional additions; binding media, principally plant gums or animal glues have also often been identified. Some studies go further into discussion of morphology or possible evidence of pyrotechnology. For examples, see Scott, Warmlander et al. 2009, Stein and Lacovara 2010, Abdelaal, Mahmoud et al. 2014, Mahmood and El Fatah 2011, Abdrabou et al. 2017, Badr et al. 2018, Cavaleri et al. 2023. However, comprehensive investigations of the physical and chemical characteristics of coffin pastes, which demonstrate the possibility of greater insight on provenance of raw materials, production process and working properties of these materials have, as yet, only rarely appeared in the published literature (examples: Rowe, Siddall et al. 2010, Scott 2018, Kasso, Stenger et al. 2024, Brunel-Duverger 2020).

The main aim of this paper is the full characterisation of the inorganic components of two kinds of paste identified in a single coffin set (the coffins of Pakepu, Fitzwilliam Museum Accession Number E.2.1869), which comprises an inner and intermediate coffin. The pastes are employed for different purposes:

- a pink paste used as a gap-filler in the wooden construction;
- a white paste used as a simple preparatory layer for the painted decoration of the intermediate coffin and to develop a complex multi-layered surface on the inner coffin.

We describe their morphology, microstructure and chemical composition as a starting point to infer production steps and uses, to

examine differences and similarities within and between the pastes on the two coffins and, hence, to contribute to our understanding of the biography of the coffin set. In addition, the results are compared with the data obtained from the examination of "comparison fragments", which come from coffins of the same period and same geographic area and which present a surface stratigraphy similar to that of the inner coffin of Pakepu (as described in the following section).

For pastes employed on Egyptian coffins from the period under analysis, our starting hypothesis and understanding was that they were obtained from ground geological materials, mixed with organic binders of different origin (so no real plasters are expected on the structure). However, we cannot dismiss the possibility that some calcined materials, even if in small quantities, might be present in mixtures, an inclusion which could bring more information regarding manufacturing techniques and pyrotechnology available at a particular time and in a specific area (Rowe, Siddall et al. 2010, Scott 2018).

Our research is based on the premise that the microstructure and composition of pastes can be informative about manufacturing processes, sourcing of raw materials, object context and provenance, and broader issues of technological tradition, development and craft organization and would be particularly valuable when studying groups of (temporally or geographically) related material. Recent work by Brunel-Duverger² (Brunel-Duverger 2020), on technological aspects of "yellow" coffins of the Twenty-first Dynasty (c. 1070–945 BCE) in the collection of the Louvre, considers the significance of these materials across a group. However, in the broader context of coffin studies, the potential of this type of highly detailed characterization remains largely unexplored.

Initial analysis of the construction and decoration of the coffin set of Pakepu and research into its possible meaning and significance in social and religious terms were carried out and published as part of the Fitzwilliam Museum Egyptian Coffins Project (Dawson, Turmezei et al. 2022; Strudwick, Dawson et al. In press; Fitzwilliam Museum Ancient Egyptian Coffins Project 2019). Those results are summarized below as

² Within the context and investigation protocol of the Vatican Coffin Project (Amenta 2014).

Table 1

Current understanding of internal and external structure of surface layers on Pakepu’s intermediate coffin (E.2.1869).

Box	Paint	Lid	Paint
Exterior surface	White paste Pink filler paste	Exterior surface	White paste Pink filler paste
Wood			
Box	Pink filler paste	Lid	Partial pink paste
Interior surface	White paste Paint	Interior surface	

Table 2

Current understanding of internal and external structure of surface layers on Pakepu’s inner coffin (E.2.1869).

Box and Lid	Paint
Exterior surface	White paste* White paste Linen textile (and non-fibrous glue)* White paste Linen textile (and non-fibrous glue) White paste Fibrous glue layer Pink filler paste
Wood	
Box and Lid	Pink filler paste
Interior Surface	Linen (and non-fibrous glue) White paste

* These layers are not present everywhere.

context for the further case study on the pastes which is presented here.

2. Materials investigated

2.1. The coffin set of Pakepu (Fitzwilliam Museum Accession Number E.2.1869)

The coffin set of Pakepu, acquired by the Fitzwilliam Museum in 1869 as a gift from the Prince of Wales (later King Edward VII), was said to have come from the West Bank of the Nile at Luxor (ancient Thebes) (Sheikholeslami 2019).

Stylistically, the coffins may be dated to the end of the 25th Dynasty, around 680–664 BCE. They form a “nested” set. The inner coffin is a classic “bivalve” type, housed within an intermediate coffin (Fig. 1), which may originally have been placed in a *qersu* coffin (Strudwick and Dawson 2016).

The inscriptions on the coffins are appeals to the gods, but also include Pakepu’s name and his job titles. He was a “water pourer on the west of Thebes”, a person responsible for maintaining the funerary cult of the dead on behalf of their families and a member of the “lower elite” in the area (Taylor 2016).

Both coffins are crafted from the native Egyptian tree sycomore fig (*Ficus sycomoros*) (Cartwright 2019). They share similarities in their use of a simple color palette—primarily copper-based green, Egyptian blue, red ochre, yellow ochre, carbon black, and orpiment. Additionally, their decoration is of mediocre quality, characterized by predominantly freehand work, and the hieroglyphic inscriptions exhibit noticeable imprecision in several areas (Strudwick, Dawson et al. In press). In construction terms, however, they are strikingly different to each other. As reported in the previously mentioned studies, the intermediate coffin is made of numerous small, badly-fitting pieces of wood, in which a coarse pink paste was used as filler to smooth irregularities in the wood and gaps between joints. The exterior surface of the whole coffin and the interior of the box were then coated with a white paste preparation layer prior to decoration. The interior of the lid does not have decoration or a

white layer, only a partial pink paste layer (Table 1).

The inner coffin is much better carpented than the intermediate coffin. It is made of a relatively small number of large pieces of wood with tight joins between them. The lid and the box were each assembled from roughly cut pieces of wood, then pegged together through the rims. This allowed the external surface to be carved down as if the coffin was a single piece of sculpture, rather than a separate box and lid. Filler paste followed by a layer of linen and a white preparation layer were applied on the interior. The complex, multi-layered structure detailed in Table 2, was then created over the exterior of the entire coffin, including across the box to lid closure, and the painted decoration was added (Dawson, Turmezei et al. 2022; Strudwick, Dawson et al. In press).³

Prior to the 25th Dynasty, Egyptian wooden coffins were made for centuries with a relatively simple surface layer structure similar to that seen on the intermediate coffin of Pakepu. The multi-layer treatment of the inner coffin, by contrast, bears a distinct resemblance to free-standing cartonnage mummy cases of the 22nd Dynasty (945–735 BCE), such as the mummy case of Nakhtefmut at the Fitzwilliam Museum (Fitzwilliam Museum Accession Number E.64.1896) (Strudwick and Dawson 2016). The complex surface structure over a tight shape and careful carpentry of Pakepu’s inner coffin, may have been a “pseudo-cartonnage”, intended to mimic a cartonnage mummy case.

Complete inner coffins from the same period in other museum collections demonstrate variations on this type of construction (Taylor 2006; Strudwick, Dawson et al. In press). The evidence suggests that during the 25th Dynasty there was significant experimentation with ways of creating inner coffins.

2.2. Comparison fragments

Four coffin fragments from the Fitzwilliam’s collection (Fitzwilliam Museum E.Misc. 109.i, E.Misc. 117, E.Misc. 118, E.Misc.119) (Fig. 2), which have a similar layered structure (Fig. 3) to that found on the inner coffin of Pakepu and which bear decoration of the period, were investigated and the results were employed as a comparison group in relation to the coffin set of Pakepu. This was used to evaluate the presence (or absence) of shared technological skills and traditions in the area under study.

Their broken edges and areas of significant wood loss through decay and termite damage, allowed examination under the optical microscope (Fig. 3). A stratigraphy resembling the one described for the inner coffin of Pakepu is detected, with a painted layer, at least one layer of white paste, linen with glue, a second layer of white paste, the unusual layer of fibrous glue and a wooden carcass. No pink paste is visible. However, given the small size of the fragments and the highly deteriorated state of the wood, it is possible that filler paste between wooden elements was present but has not survived. Its absence does not affect the comparison of the complete surface structures above the wooden construction from the fibrous glue layer upwards.

Together, the different treatments of the inner and outer coffins of Pakepu and the additional resource of the comparison fragments, provide a compelling range of examples with which to begin a detailed exploration of the pastes on these late Third Intermediate Period Theban coffins.

³ The Fitzwilliam Museum project team has proposed that the body was put into the coffin before this phase of the decoration (Strudwick, Dawson et al. In press). Research at the Louvre Museum has produced evidence that in this type of coffin, the parts may have been separated again after the external decoration was applied, so that the body could be inserted (Thomas, 2022). For the current paper on the nature of the layers, this difference is not significant. The important point is that the external layers were applied with the coffin closed to create a particular effect.



Fig. 2. From the top left, comparison samples E.Misc.109.i, E.Misc.119, E.Misc.117 and E.Misc.118.

3. Sampling and methods

3.1. Sampling

54 specimens in the form of cross-sections and powders were examined. Cross-sections were employed for optical microscope and scanning electron microscopy with energy dispersive spectroscopy, while the powders for Fourier transform infrared spectroscopy. A more detailed description of the preparation of these samples is provided in the methods section, as the preparation process varies depending on the analytical technique employed.

The samples range from 3.5 mm to 200 μm and a significant number was required to ensure a reliable representation of the materials under consideration. 10 of the samples pre-existed from the Fitzwilliam's earlier project. and were re-analysed using new techniques, 44 additional samples were acquired for the current research.

In Table 3, an outline of these acquisitions is reported, focusing on the area and nature of the sampling. The sample preparation varied depending on the analytical technique employed. In Online Resource 1, a detailed representation of the sampling location on the coffin set of Papepu and comparison fragments is available.

3.2. Methods

3.2.1. Optical microscopy

Two optical microscopes were used for initial observation of

micromorphology and to obtain reference images for further analyses. A Zeiss AxioImager optical microscope was employed, using reflected light, to obtain images at three different magnifications ($\times 50$, $\times 100$, and $\times 200$). A Keyence VHX 6000 digital microscope was also utilized, providing higher depth-of-field, and producing 2D and 3D images with a free-angle observation system. Images at magnification of $\times 20$, $\times 30$ and $\times 50$ were acquired.

Before microscopic observation, the samples (typically hundreds to thousands of μm in size) were embedded as cross-sections in epoxy resin, ground on silicon carbide papers, with a grit size from 1200 to 4000, and then polished with a 6 μm to 0,25 μm diamond paste. Between steps, the samples were washed in an ultrasonic bath, using ethanol (Middendorf, Hughes et al. 2005; Turco, Davit et al. 2015). Because of their smaller size, comparison fragments could also be examined microscopically before invasive sampling, to characterise the inner stratigraphy and identify the best sampling location.

Following this first examination, all the samples were analysed under scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), while the powders were analysed by Fourier transform infrared spectroscopy (FTIR).

3.2.2. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS)

The cross-sections used for optical microscopy were carbon-coated for further micro-morphological examinations and elemental composition analyses, using a Hitachi TM3000 scanning electron microscope

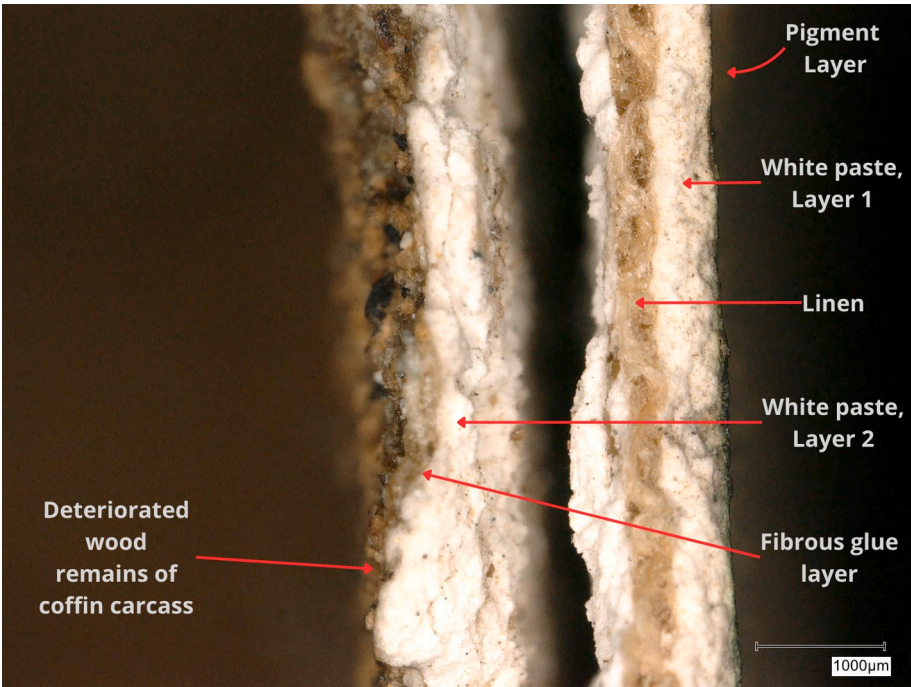


Fig. 3. Layers of the pseudo-cartonnage present in the comparison fragment E.Misc.117, observed under a digital microscope. The central gap is a crack resulting from deterioration and it is not characteristic of the original stratigraphy. No pink paste was identified. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 3

Outline of sampling strategy for the coffin set of Pakepu and the comparison fragments, highlighting the area chosen for the acquisition and the typology of sample acquired. The different quantities of samples are related to the conservation status of the artefacts and to the position of these pastes on the objects, which allowed easier sampling in the case of the intermediate coffin and comparison fragments.

Object	Area	Cross Section	Powder	Total
Coffin Set of Pakepu	Inner Coffin	10	4	37
	Intermediate Coffin	12	11	
Comparison Fragments	E.Misc.109.i	2	2	17
	E.Misc.117	1	2	
	E.Misc.118	2	3	
	E.Misc.119	2	3	

coupled with an Oxford Instruments energy dispersive spectrometer, in the following conditions: working distance 8–12 mm, probe current 200 pA, accelerating potential 15 kV. All the EDS data are presented as stoichiometric oxides, measured as weight%, and normalized to 100 %.

For every sample, backscattered electron (BSE) images were acquired under the same magnification ($\times 200$, $\times 1000$, $\times 1200$, $\times 2500$), to facilitate comparisons. In addition, X-ray maps were obtained to evaluate and illustrate similarities and differences in the homogeneity and composition of pastes.

The SEM-EDS was particularly useful to differentiate the overall composition of both paste types based on area analyses, as well as the abundance, size, shape and composition of the different minerals detected within, which can be reflective of different raw materials selection or processing.

Moreover, previous studies (Gourdin and Kingery 1975; Philokyprou 2012) have shown that it is possible to use the size of calcite grains to differentiate real plaster (grains size typically $<1\text{ }\mu\text{m}$) from pastes made of crushed limestone mixed with binder ($>1\text{ }\mu\text{m}$). We therefore measured

7 to 11 grains per section, employing the software ImageJ on SEM images.

3.2.3. Fourier transform infrared spectroscopy (FTIR)

Further characterisation of the samples was obtained employing a Nicolet iS5 FTIR, from Thermo-Fisher Scientific. For this, a few milligrams of each sample were crushed and homogenized, using an agate pestle and mortar, and mixed with KBr powder. The mixture was then placed in a mould and inserted into a press, under 2,7ton for 15–20 s (Chu, Regev et al. 2008; Regev, Zukerman et al. 2010). These resulting KBr pellets were analysed 64 times in the range 4000–400 cm^{-1} , with a speed of 8 cm^{-1}/s and a resolution of 4 cm^{-1} . The results were processed using OMNIC software, presented as transmission spectra and compared to the reference spectra in the Kimmel online library.

FTIR was primarily employed to determine the mineralogy and technological origin (anthropogenic or geological) of the pastes.

In case of presence of mud or gypsum, the identification is relatively straightforward, based on the comparison with standards for mud and on the observation of impurities and anhydrite in the gypsum (Bracci, Caruso et al. 2015; Cao, Yi et al. 2022).

For lime pastes, the v_2/v_4 ratio was calculated, which represents the extent of the atomic disorder in the calcite structure, and therefore serves as an additional proxy to differentiate between anthropogenic (plaster) or natural calcite (Fig. 4). As established by Chu et al. (Chu, Regev et al. 2008), a sample of calcite is classed as anthropogenic if the ratio falls between 3.3 and 7.7, where 3.3 represent the lower temperature employed for the treatment of the limestone ($<700\text{ }^\circ\text{C}/3\text{h}$) while 7.7 results from a complete calcination of the material (obtained at 900 $^\circ\text{C}$). Lower values are reflective of untreated calcite (Regev, Zukerman et al. 2010; Turco, Davit et al. 2015; Calandra, Cantisani et al. 2022).

4. Results and discussion

The SEM-EDS and FTIR results obtained for the funerary set of Pakepu (both intermediate and inner coffin) and the comparison fragments are summarised in the Online Resource 2. Microscopic analyses

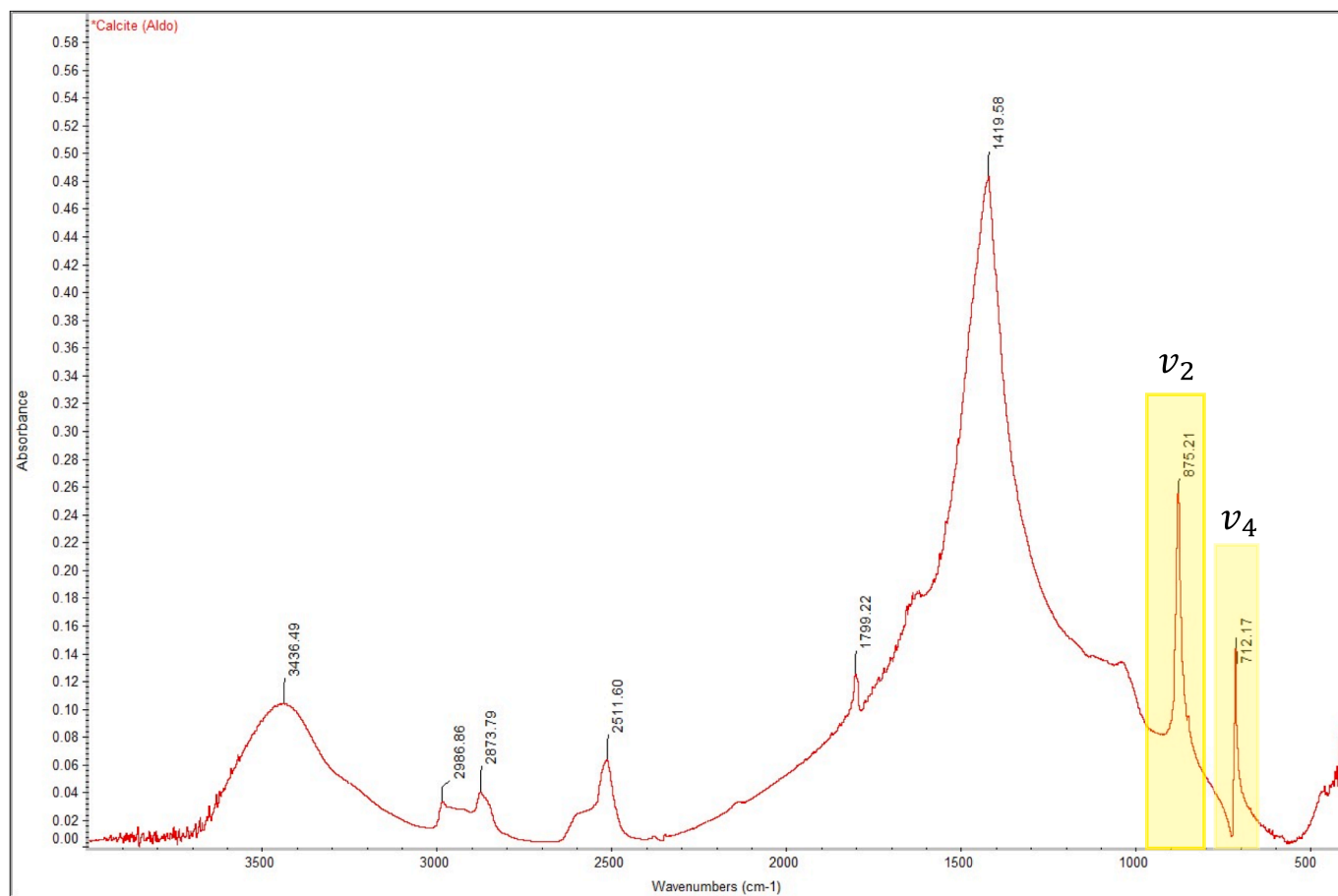


Fig. 4. FTIR reference spectrum of calcite, available in the Kimmel library database, highlighting the peaks of ν_2 and ν_4 , respectively at ca. 875 cm^{-1} and 712 cm^{-1} , used to determine whether calcite has undergone thermal treatment or not.

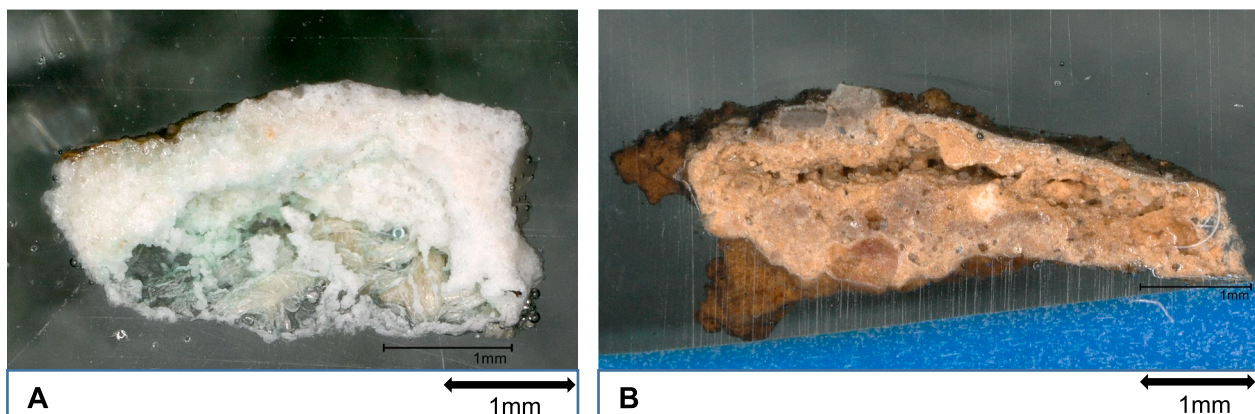


Fig. 5. Microscope images of white and pink pastes at $\times 50$, showing differences in homogeneity and colour. A. Sample CA220351 (CR06_ICI), from the inner coffin, showing white paste and linen, B. CA220322 (CR01_OL), from the intermediate coffin, in which just pink paste is present. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

of cross-sections show that, besides the colour, the main difference between white and pink pastes is grain size and homogeneity in the distribution of the mineral grains (Fig. 5): the white paste appears homogeneous while the pink one is characterised by larger matrix grains, mineral inclusions – sometimes in clusters – and an overall coarser texture.

Microscopic examination further allowed the identification of distinct layers of paste in each sample, ranging from two to four, potentially corresponding to several applications of very similar

material. Analytical results for each of these layers are presented separately (see below). As seen in Fig. 6, it is also worth noting that white and pink often occur together in the Pakepu samples from the intermediate coffin; therefore, the possibility of some cross-contamination between pink and white paste samples should be considered in the evaluation of the results.

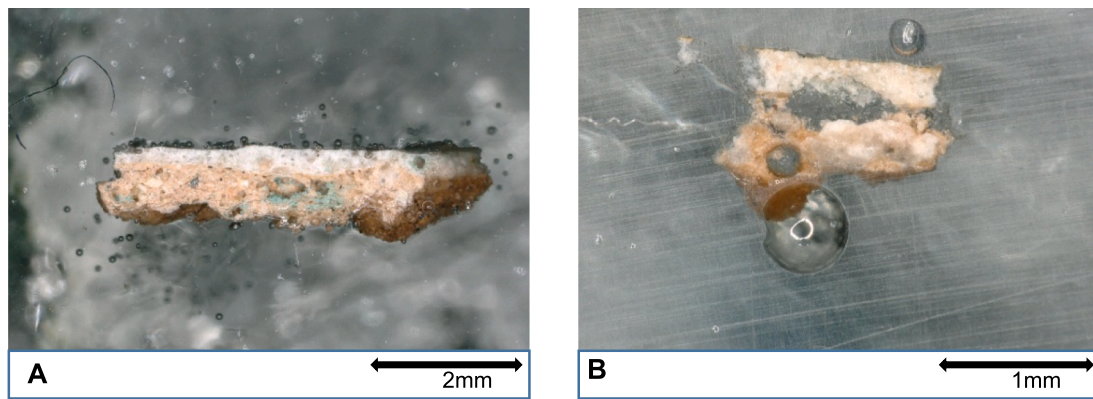
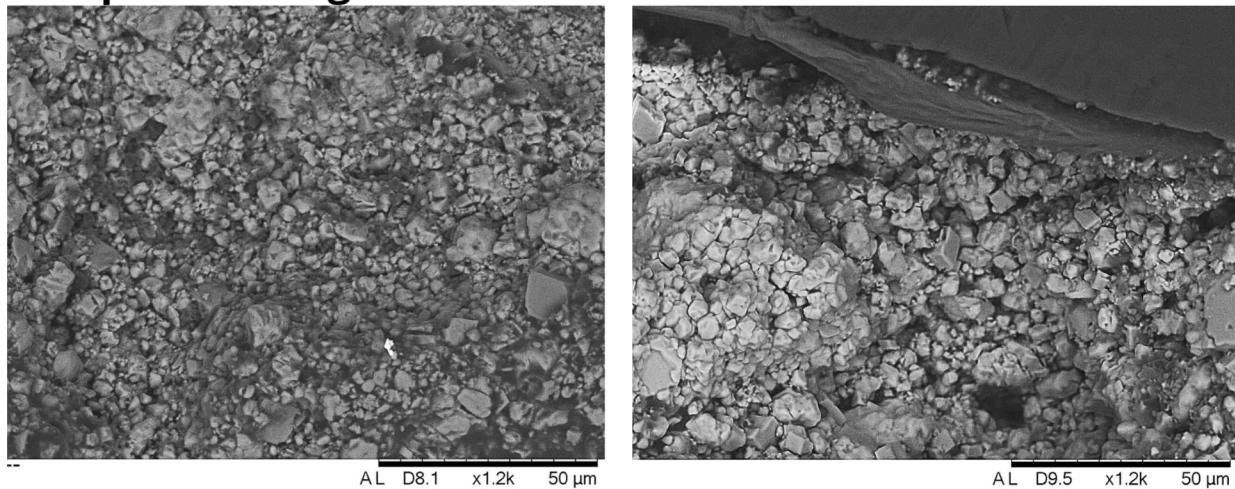


Fig. 6. Example of layering of pastes in samples, as observed in digital microscope images of the Pakepu intermediate coffin. A. Sample CA220346 (CR01_OBi) at $\times 50$, showing three layers: Egyptian Blue pigment on the surface, white and pink. B. Sample CA220348 (CR03_Obo) at $\times 100$, showing a yellow colour on top of white, which is on top of pink. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Comparison fragments:



Coffin set of Pakepu:

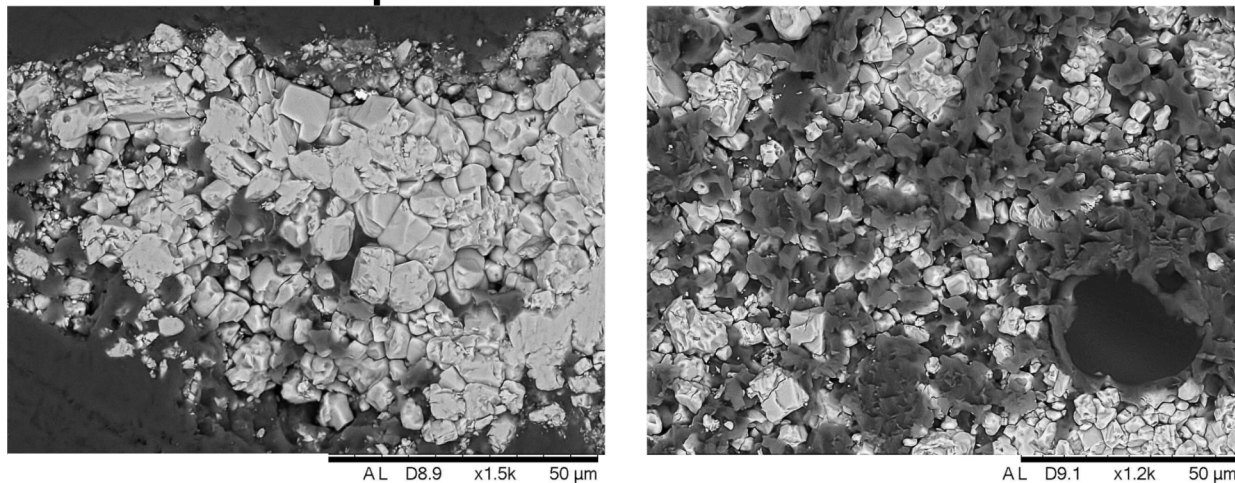


Fig. 7. SEM-BSE images showing the micromorphology of white pastes at $\times 1200$ magnification. Note the pseudocubic shape of calcite grains. A: sample CA220220, comparison fragment E.Misc.118, B: sample CA220218, comparison fragment E.Misc.109.i, C: sample CA220349 (CR04_ICB), inner coffin, D: sample CA220353 (CR04_OBO), intermediate coffin, in which the darker organic binder is more abundant.

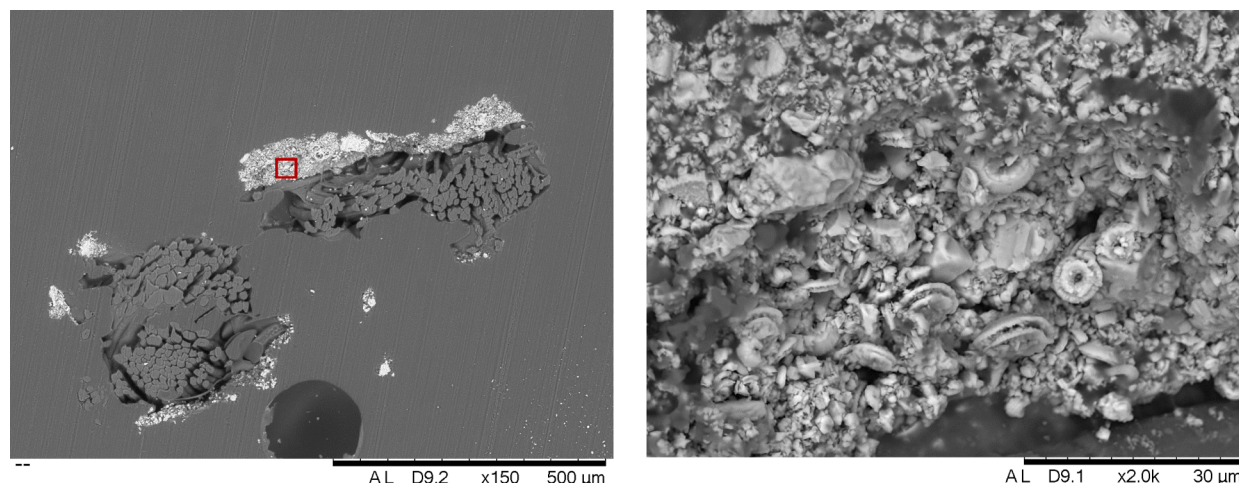


Fig. 8. SEM-BSE image of white paste sample CA220352 (CR07_ICB) from Pakepu inner coffin showing the presence of microfossils. The darker grey part on the sample on the left image is a cross section of the linen attached to the white paste.

4.1. White paste

4.1.1. Composition

All the white paste samples (both from the comparison fragments and the Pakepu coffin set) reveal a homogeneous internal structure, dominated by isometric (pseudocubic) calcite grains in a microcrystalline matrix. Even though sulfur was detected in some samples (see below), no grains with the characteristic shape of gypsum were observed (neither untreated geological ones nor the needle-shaped gypsum of Paris; cf. Gourdin and Kingery 1975; Philokyprou 2012); the only other mineral visible was quartz (SiO_2) in small amount (Fig. 7).

These calcite grains are usually intermixed with an organic binder, which appears dark in SEM-BSE, and which was identified as proteinaceous by FTIR analysis (see below). Further analysis of this material, by means of paleoproteomic techniques, is underway.

In a few samples, calcareous microfossils could be identified too (abundant in samples CA220349 and CA220352 from the inner coffin and sample CA220322 from the intermediate one, while rarer in the comparison fragments) (Fig. 8). Their classification, carried under the supervision of Liam T. Gallagher,⁴ is consistent with Late Paleocene–Early Eocene age fossils, judging by the presence of *Coccolithus pelagicus*, various *Toweius* species, and *Neochiastozygus* species. These analyses may help narrow down the possible origin of the parent geological material (Goren and Goldberg 1991; Klemm and Klemm 2008; Rowe, Siddall et al. 2010; Quinn 2017).

Based on the type of microfossils and the description by Klemm and Klemm (Klemm and Klemm 2008) of limestone quarries in the area nearby Thebes, we were able to narrow down with the help of Dr. Trevor F. Emmett⁵ the possible origin of the geologic material employed for the white pastes to three sources: Abydos, Sidi Musa and Qurna. All these quarries present white and pure samples of limestone, characterised by a high concentration of microfossils.

FTIR data also confirmed the predominant presence of calcite in the white paste of all samples, with the three characteristic peaks around 1420 cm^{-1} (assigned to CO_3^{2-} asymmetric stretching band), 875 cm^{-1} (O–C–O out-of-plane bending) and 712 cm^{-1} (in-plane bending) (Gruchow, Machill et al. 2008; Regev, Poduska et al. 2010; Yao, Xie et al. 2013) (Figs. 4 and 9).

In addition to the unequivocal identification of calcite, as the dominant phase in all samples, bands of variable intensity at ca. $1090\text{--}80\text{ cm}^{-1}$ and 790 cm^{-1} are consistent with the presence of quartz, a

common impurity in geological calcite. A weak band at 1660 cm^{-1} , characteristic of collagen (used here as a proxy for the presence of proteinaceous materials), is also noticeable. Having said that, white paste samples from the Pakepu inner coffin stand out in their higher purity, with sharper peaks for calcite and weaker signals for any other phase. This is particularly noticeable when the FTIR spectra are compared to the Kimmel's library slate-red (with characteristic band at ca. 1160 cm^{-1} – 1084 cm^{-1} – 1032 cm^{-1} , 779 cm^{-1}). The latter was employed as a reference of an iron-bearing siliceous material, containing higher percentage of quartz, chlorite and ferric oxides. In the region from 1160 to 1030 cm^{-1} , broad bands are detected in the Pakepu intermediate coffin and the comparison samples, which are virtually absent in the Pakepu inner coffin. Finally, dolomite is detected in the intermediate coffin and, especially, in the comparison samples. This result is consistent with those of previous studies of “yellow coffins” from the same area, but from an earlier dynasty (21st–22nd) (Brunel-Duverger 2020). It might indicate a different source of geological raw materials compared to the coffin set of Pakepu. The above observations were confirmed by hierarchical cluster analysis of the FTIR data (Fig. 10), where the Pakepu inner coffin white paste samples form a distinct group. Notwithstanding one possible outlier, comparison fragments form two separate subgroups, which are in turn different from a more mixed group containing all the remaining samples.

Elemental data from the SEM-EDS also demonstrate the broad correspondence between sample types and compositions, while adding further detail. In a scatterplot of CaO vs MgO, white inner coffin samples show the highest calcite purity, while other groups show different amounts of CaO and MgO –the latter, likely related to variable dolomite ($\text{CaMg}(\text{CO}_3)_2$) content. The possibility that huntite ($\text{CaMg}_3(\text{CO}_3)_4$) was present in the mixture instead of dolomite was considered too, since this mineral was typically employed in Ancient Egypt as white pigment (Heywood 2001; Scott, Warmlander et al. 2009). However, the typical structure of an ordered rhombohedral double-carbonate (with the characteristic rhombic platy habit) was not detected in the paste by SEM imaging (Faust 1953; Graf and Bradley 1962; Barbieri, Calderoni et al. 1974). Moreover, considering the results obtained with FTIR (Fig. 9), the characteristic peaks of huntite at 1538 cm^{-1} , 1507 cm^{-1} and 1443 cm^{-1} were not detected (Seki, Sever et al. 2013). These considerations, combined with the relatively low amount of MgO detected in the white paste samples (Online Resource 1), lead us to the conclusion that huntite had not been mixed in the paste, while more probably dolomite, a very common impurity of calcite, was present in the mixture, due to natural processes during rock formation (Hsu 1967; Allen 2017).

Considering more generally the SEM-EDS data, white pastes from the Pakepu inner coffin cluster relatively tightly, whereas those from the

⁴ Director / Nanopalaeontologist, Network Stratigraphic Consulting Ltd.

⁵ Geologist, former Principal lecturer at Anglia Ruskin University.

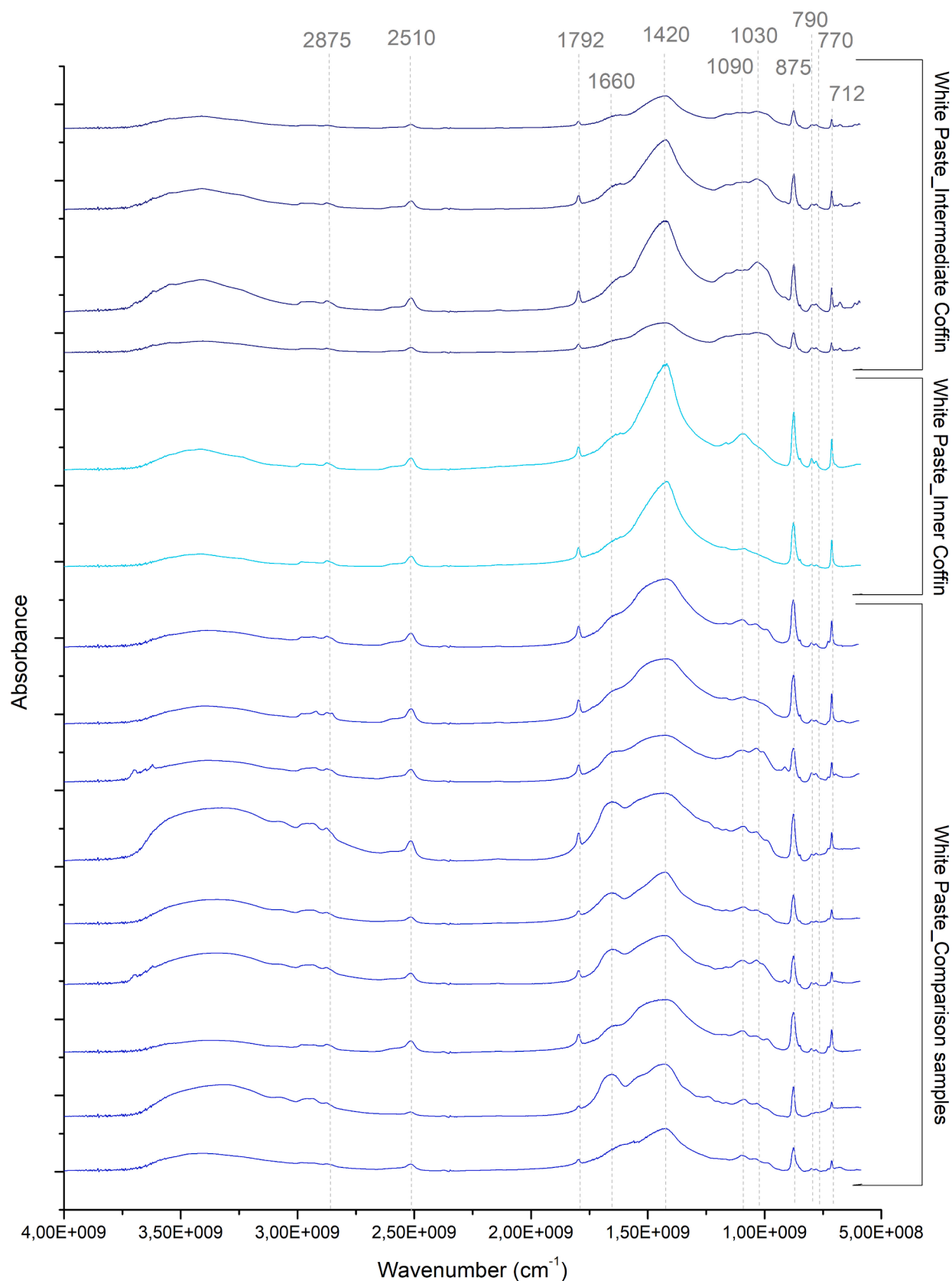


Fig. 9. FTIR spectra of white pastes, in the range between 4000 cm^{-1} to 500 cm^{-1} . The characteristic peaks of calcite (1420 cm^{-1} , 874 cm^{-1} and 712 cm^{-1}) are recognisable in all the samples. Bands comparable to the reference samples of collagen, slate red, quartz and dolomite have been recorded mostly in the intermediate coffin and comparison samples, while the inner coffin appears purer, with just a small signal from the binder (collagen). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

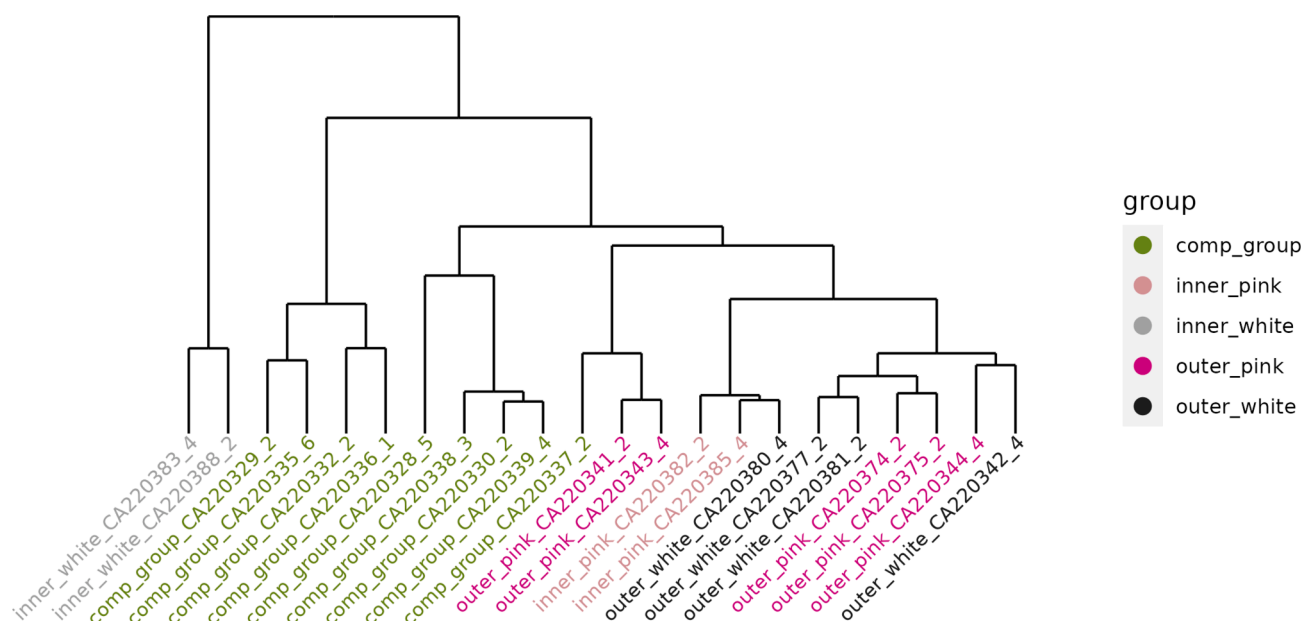


Fig. 10. Hierarchical cluster analysis of the FTIR data for all pastes (courtesy of Ismael Rodriguez-Palomo). The white pastes from Pakepu inner coffin stand out as different from all the others, probably because of their higher purity. Comparison fragments also form distinct subgroups. The codes of samples CA220333, CA220376 and CA220379 appeared to be corrupted, so it was not possible to include them in the cluster analysis.

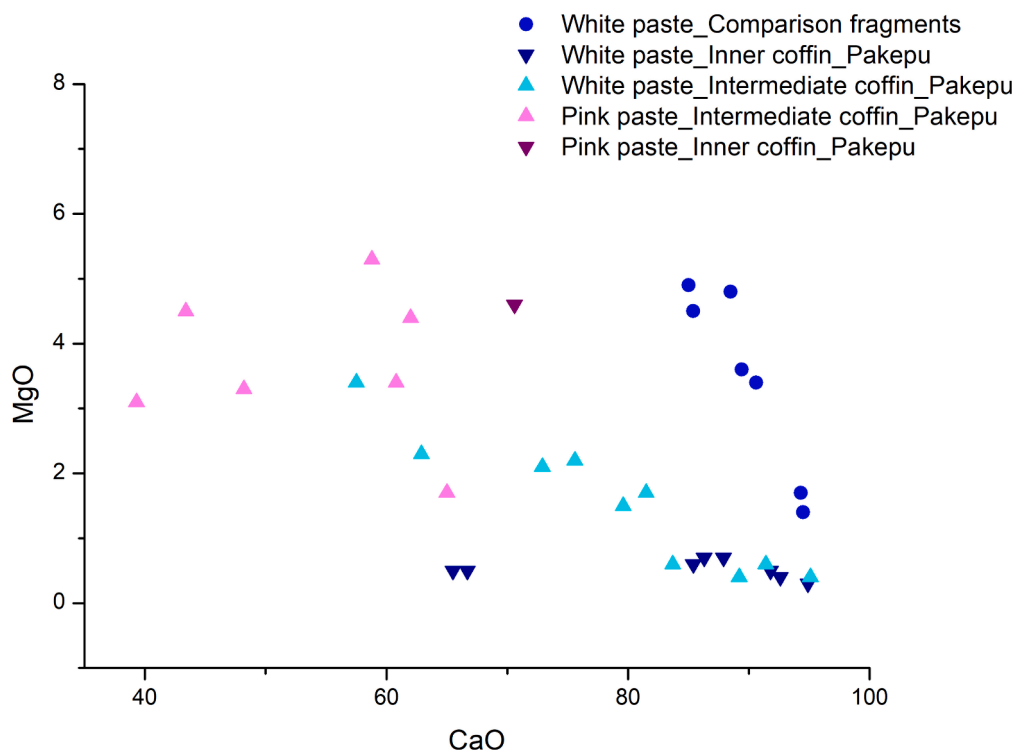


Fig. 11. Scatter plot of the concentration of MgO and CaO in the samples. The white paste from the Pakepu inner coffin seems consistently the purest, with very low MgO and correspondingly high CaO (see text for two exceptions likely effected by contamination). All the other sample types tend to show slightly higher MgO, consistent with the presence of dolomite in the mixture (as supported by the FTIR data). White pastes from Pakepu intermediate coffin show more variable composition. See text for explanation of two outliers in the white pastes from the Pakepu inner coffin.

intermediate coffin show a more scattered compositional distribution (Fig. 11 and [Online Resource 2](#)).

More specifically, the composition of the white paste in the Pakepu inner coffin is similar in all the layers present in the pseudo-cartonnage and consistent with that of relatively pure calcite, with median values of 87 % CaO, 6 % SiO₂, and all other values oxides in concentrations

typically around 1 % or less. Occasional traces of heavy oxides such as CuO and As₂O₃ are likely contamination from pigments used on the surface and detected during preliminary analyses, such as Egyptian Blue (Cu-rich) and Orpiment (As-rich).

There are two exceptions to this pattern, but they can be explained by external factors: sample CA210159 showed very high Na₂O, but the

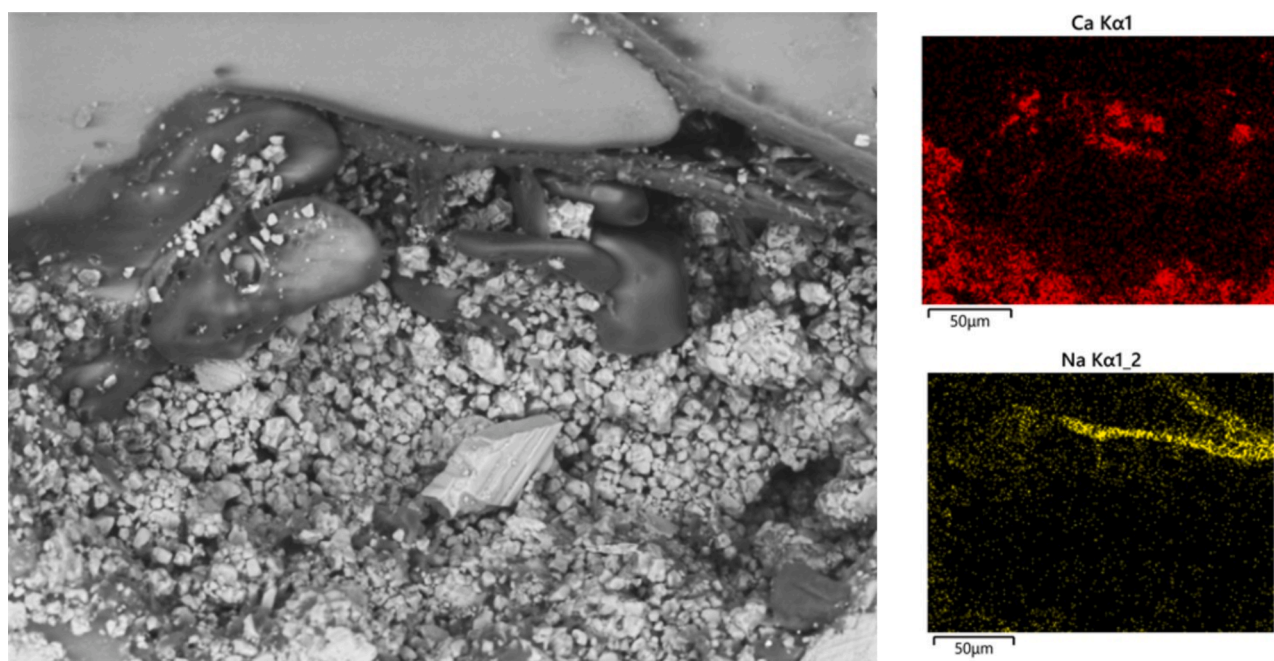


Fig. 12. SEM-BSE image and X-ray map of elemental distribution in sample CA210159, (white paste from the Pakepu inner coffin) showing a high concentration of Na in the area surrounding the linen layer, probably due to treatment of linen with Na-rich substances during fibre processing. The area towards the middle of the image appears blank in the X-ray maps because it is slightly deeper in the sample and hence further from the EDS detector.

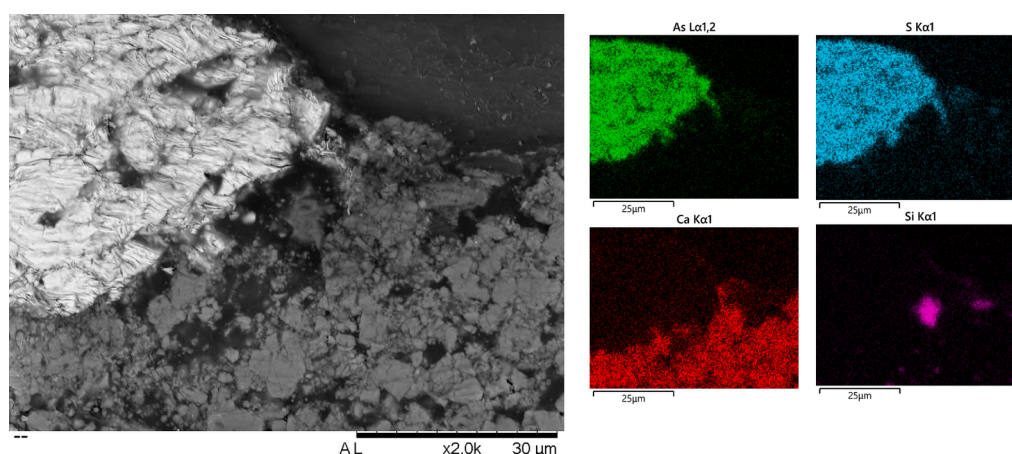


Fig. 13. SEM-BSE image and X-ray map of elemental distribution in sample CA220087 (white paste from Pakepu's inner coffin, from yellow surface), showing a high concentration of S and As in the upper layer, closer to the upper paint layer. The exceptionally high values are, probably, a contamination from the latter yellow pigment orpiment (As_2S_3) present on the surface. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

X-ray distribution map clearly shows that this relates to the presence of adjacent linen rather than being part of the paste itself (Fig. 12). Linen is a textile obtained by the processing of the fibres of the flax plant and it does not have an inherited high amount of sodium. The latter could be related to external factors such as environmental conditions or processing and manufacturing methods, such as bleaching, which could involve the use of natron (Sloan 1974; Forbes 1987; Vogelsang-Eastwood 2009).

For sample CA220087, sulfur showed exceptionally high values (quantified by stoichiometry as 12 % SO_3), but this was correlated with similarly high levels of arsenic (14 % As_2O_3). As the sampling location was adjacent to a pigmented yellow layer, it is likely that these values reflect contamination from the yellow pigment orpiment (As_2S_3) (Fig. 13), which was identified in the earlier study as a component of the painted decoration.

Turning to the white pastes in the intermediate coffin, while some are comparable to those of the inner coffin, most display a lower purity, with higher levels of SiO_2 , Al_2O_3 , MgO and FeO (Online Resource 3, Figs. 11 and 21), in addition to more frequent detection of sulfur. The X-ray maps show that these oxides are distributed relatively uniformly within the matrix rather than concentrating in individual minerals (Fig. 14), which would support the hypothesis of impurities in the geological material employed for the pastes (rather than deliberate addition of external material). This is consistent with the proposition that different geological sources were employed for the different components of the coffin set of Pakepu and perhaps made at different workshops, as suggested by the broader analysis of the coffin construction (Strudwick, Dawson et al. In press).

Finally, considering the elemental composition of the comparison fragments, the concentration of CaO is similar to that in the inner coffin

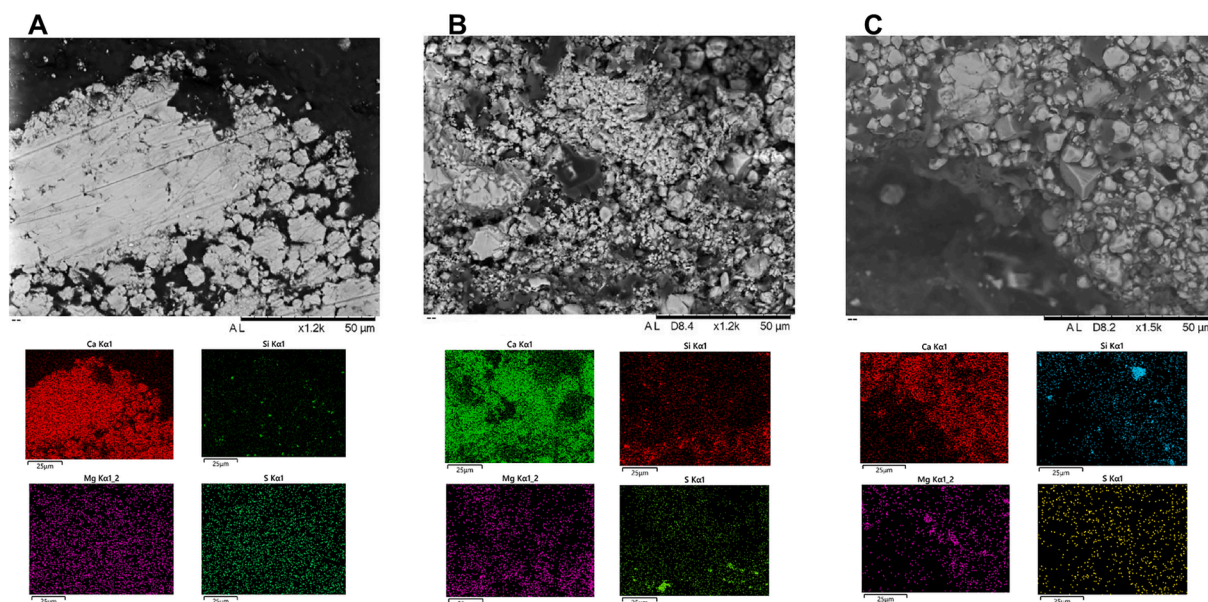


Fig. 14. SEM-BSE images and X-ray maps of elemental distribution in white pastes. A: sample CA220089 (E.2.1869 IL13), Pakepu inner coffin. B: sample CA220347 (CR02_OBi), Pakepu intermediate coffin. C: sample CA220221 (E.Misc.118 15), comparison fragments. Ca is the dominant element in all the white pastes, followed by Si. In sample B, an increase of Si, S and Mg concentrations are recorded towards the bottom, due to the presence of the boundary with the pink paste layer. Discrete mineral impurities are more frequent in B and C, especially Mg-rich particles in C, denoting the higher abundance of dolomite crystals in the comparison fragments. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

white paste (median 89 %), while the presence of higher MgO (3.5 %) and FeO (0.3 %) places them closer to the composition of the white paste from the intermediate coffin of Pakepu. However, the distribution of these oxides, particularly the more frequent presence of Mg-rich grains in the comparison fragments, would support a different geological source (Fig. 14).

4.1.2. Pyrotechnology

The size of the calcite grains, as illustrated in Fig. 15 and reported in Online Resource 2 is mostly comprised in an interval between 1 μm and 7 μm , and therefore larger than calcite obtained through true calcination for the production of the plaster (<1 μm). On average, comparison fragments showed larger grain sizes. Some smaller values were recorded for the individual grains in samples from Pakepu's intermediate coffin, suggesting the possibility that some calcined material was crushed and added to the mixture – but these are the exception rather than the norm. In fact, consistently high temperature treatment as required for plaster preparation would have led to the decomposition of any calcareous nanofossils, contrary to our observation above. Grain size information is largely consistent with the results of the v2/v4 ratios calculated from the FTIR data, which again show most of the samples plotting in the 'low temperature' region.

Thus, overall, it is likely that the majority of the white paste was obtained by mixing finely ground calcite with an organic binder (subject of ongoing analyses to be reported in a future publication). It is possible that heat was applied in some cases, perhaps to facilitate the crushing of the rocks, and that, particularly with the comparison fragments, some calcined materials may have been added to the mixture, suggesting a slightly different *chaîne opératoire*. Although imperfect, the clustering of sample types based on grain size and especially v2/v4 ratios might denote the presence of different batches being prepared for individual coffins or coffin parts.

4.2. Pink paste

4.2.1. Composition

As noted above, no pink filler pastes were identified within the

deteriorated wood below the 'pseudo-cartonnage' construction of the comparison fragments. Regarding the Pakepu coffin set, while multiple samples of pink paste were available for the intermediate coffin, the better preservation of the inner coffin meant that we could only obtain one cross-section (CA231048, from the exterior of the inner coffin) and two powder samples from the latter, and hence the scope of our results and comparison is more limited.

Microscopic examination shows that the pink paste from the inner coffin of Pakepu appears very similar to the associated white paste, dominated by isometric grains of calcite with only very occasional presence of clusters of quartz (SiO_2). Distinctive is the presence of fibers of organic origin interspersed within this calcite matrix, which X-ray maps show to be enriched in Si, Al and Mg (Fig. 19). These are part of the unusual fibrous glue layer (Strudwick, Dawson et al. In press) noted in Table 1, which is currently under further investigation.

Conversely, the pink pastes from the intermediate coffin also contain abundant calcite grains, but they are much more heterogeneous, including much larger grains and clusters of minerals (Fig. 16) such as quartz, feldspars and dolomite (Fig. 12), as reflected in generally higher concentrations of oxides such as SiO_2 , Al_2O_3 , MgO, and FeO.

The observation of the different appearance and, probably, preparation of these pink pastes is also supported by the comparison of FTIR results of the pink and white pastes present on same surfaces. Visually, FTIR spectra for white and pink pastes in the inner coffin appear quite similar, with the most notable difference being a peak at 1030 cm^{-1} for the latter, corresponding to ferric oxides (Fig. 17, but note their separation in the cluster analysis in Fig. 10).

In contrast, the comparison of the FTIR spectra from the intermediate coffin (Fig. 18) shows a marked increase in the concentration of quartz and iron oxides in the pink paste, recorded in the stronger and sharper peaks at 1160 cm^{-1} –1084 cm^{-1} –1032 cm^{-1} and at 779 cm^{-1} .

For both inner and intermediate coffin, area analysis of the matrix by SEM-EDS show CaO as the main oxide (median 62 %), consistent with the presence of calcite as the main matrix material, but SiO_2 , Al_2O_3 , MgO and FeO are much more abundant than in white paste samples. Respectively, these oxides show average concentrations of 16.9 %, 3.4 %, 4.6 % and 2.6 % for the inner coffin and 25.8 %, 5.9 %, 3.7 % and 3.2

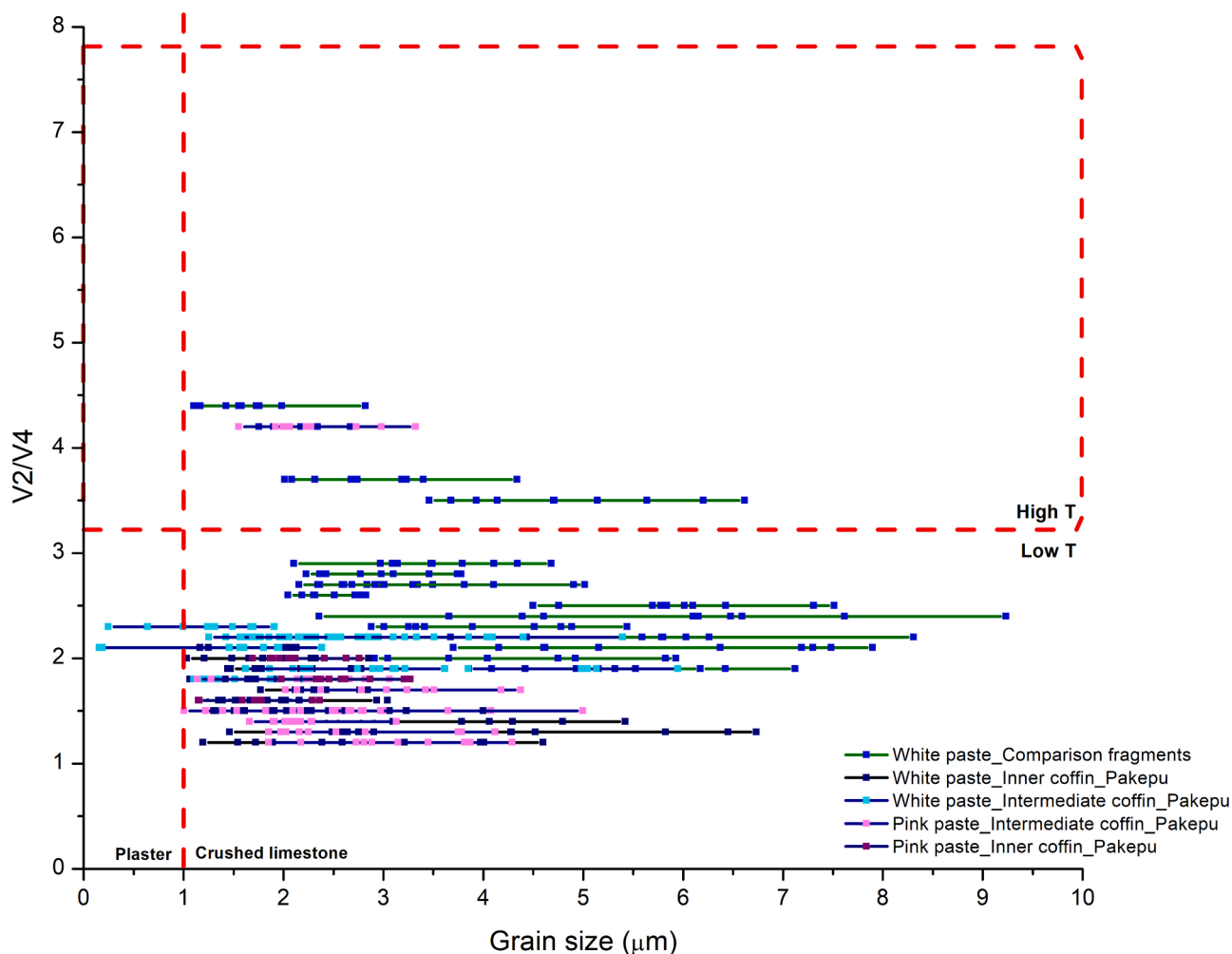


Fig. 15. Summary graph representing the relationship between the v_2/v_4 ratios (from FTIR data) and grain size (from SEM images), to assess if there is a coherent distribution of the pyrotechnology evaluation. Almost all the samples are reported with a grain size larger than $1\ \mu\text{m}$ and with a v_2/v_4 ratio lower than 3.3, consistent with a geological origin and limited or no pyrotechnological treatment. There are, however, a few outliers, namely smaller grains in white pastes from Pakepu intermediate coffin, and higher v_2/v_4 ratios in white comparison fragments and a sample of pink paste from Pakepu intermediate coffin. Note that, with some exceptions, sample types appear to cluster based on the v_2/v_4 ratio, although comparison fragments show an overall higher variability, potentially reflecting that the samples come from a larger range of coffins. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

% for the intermediate coffin ([Online Resource 3](#)), consistent with the presence of dolomite, iron oxides and aluminosilicates in the mixture, and with the use of less pure material for the production of these fillers.

Still, a lower proportion of inclusions and impurities is registered in the pink paste of the inner coffin. This is supported by the SEM images and X-ray maps, and verified by SEM-EDS analyses of individual phases. Concerning the intermediate coffin, there are relatively large quartz grains (Si) as well as feldspars (Si, Al, Na) ([Fig. 19](#)). There is some compositional overlap between a few pink and white paste samples of the intermediate coffin, but this may at least in part derive from cross-contamination between layers ([Online Resource 2](#) and [Online Resource 3](#)).

Of particular note is the concentration of iron oxide (measured as FeO), on average three times higher in the pink paste than in the white paste from the same coffin for both inner and intermediate structure. While relatively low compared to other geological materials, this additional amount of iron is the most likely explanation for the characteristic shade of the pink paste. The presence of ferric oxides in the bulk material was confirmed by FTIR, showing characteristic peaks in the range of 1084 cm^{-1} – 1030 cm^{-1} and in the band at 779 cm^{-1} in addition to calcite ([Fig. 20](#)).

We considered the possibility that hematite was deliberately crushed

and mixed with the white and pure limestone. However, given the absence of discrete particles of hematite detected with SEM and the lack of the characteristic FTIR peaks for hematite at 1604 cm^{-1} , 1482 cm^{-1} , 1293 cm^{-1} and in the region of 700 cm^{-1} ([Farahmandjou and Soflace 2015](#)) this hypothesis was discarded. The hypothesis that the pink colour was due to the addition of organic red dyes, such as red madder or kermes, largely employed in Ancient Egypt ([Phipps 2010](#); [Karapanagiotis, Verheeken-Lammens et al. 2019](#); [Newman and Gates 2020](#)), was also considered. In this case, this addition would not have been visible with SEM-EDS, due to its organic nature, but it could have been detected through FTIR. However, as in the previous case, neither the characteristic peaks of red madder at 1529 – 1428 cm^{-1} and at 1359 – 1270 cm^{-1} ([Wertz, Quye et al. 2017](#)) nor the ones of kermes at 1632 – 1559 cm^{-1} , 1444 cm^{-1} , 1371 cm^{-1} , 1240 cm^{-1} and 1048 cm^{-1} ([Mujodi, Achachluei et al. 2020](#)) were detected. Further analyses, for example by HPLC, may help clarify this issue. In any case, while not impossible, the deliberate addition of a pigment or dye to a material that would be hidden from view would seem unlikely.

Thus, based on the data acquired with SEM and FTIR, we believe that iron oxides were natural impurities of the geological material, rather than added separately to the mix. Reddish limestone has been reported from El-Salamuni, El-Dababiya and Sidi Musa near Thebes ([Klemm and](#)

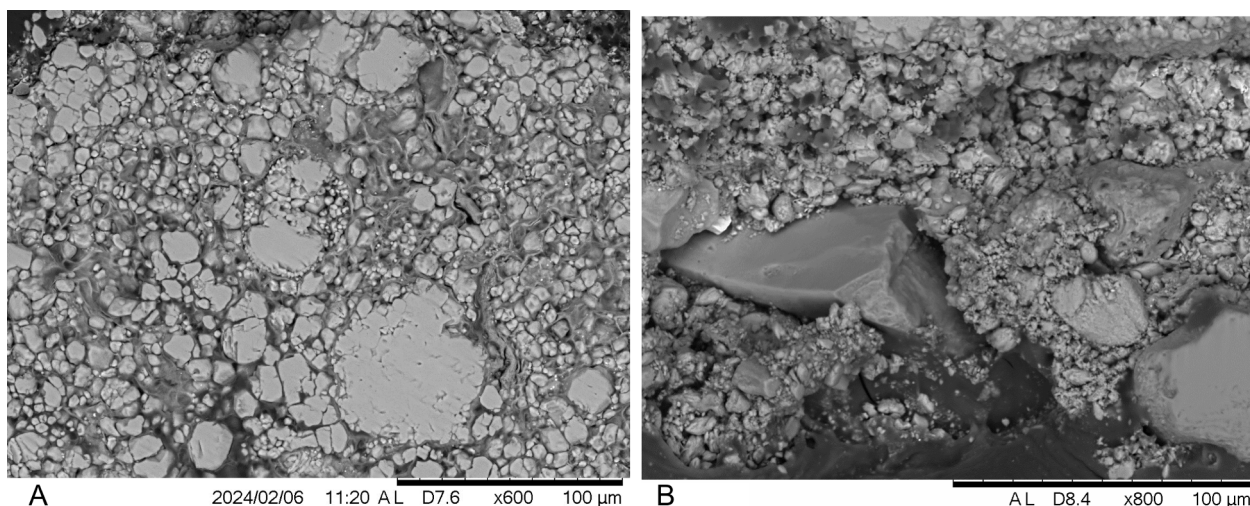


Fig. 16. SEM-BSE images showing the micromorphology of pink pastes. A: sample CA231048, Pakepu inner coffin, $\times 600$ magnification, B: sample CA220347 (CR02_OBi), Pakepu intermediate coffin, $\times 800$ magnification. Pseudocubic grains of calcite are abundant in both, but the Pakepu intermediate coffin sample B appears more heterogeneous, including also silicate minerals (larger, darker grey phases). In the Pakepu inner coffin A, filaments of fibrous glue entangled in the calcite grains can be observed.

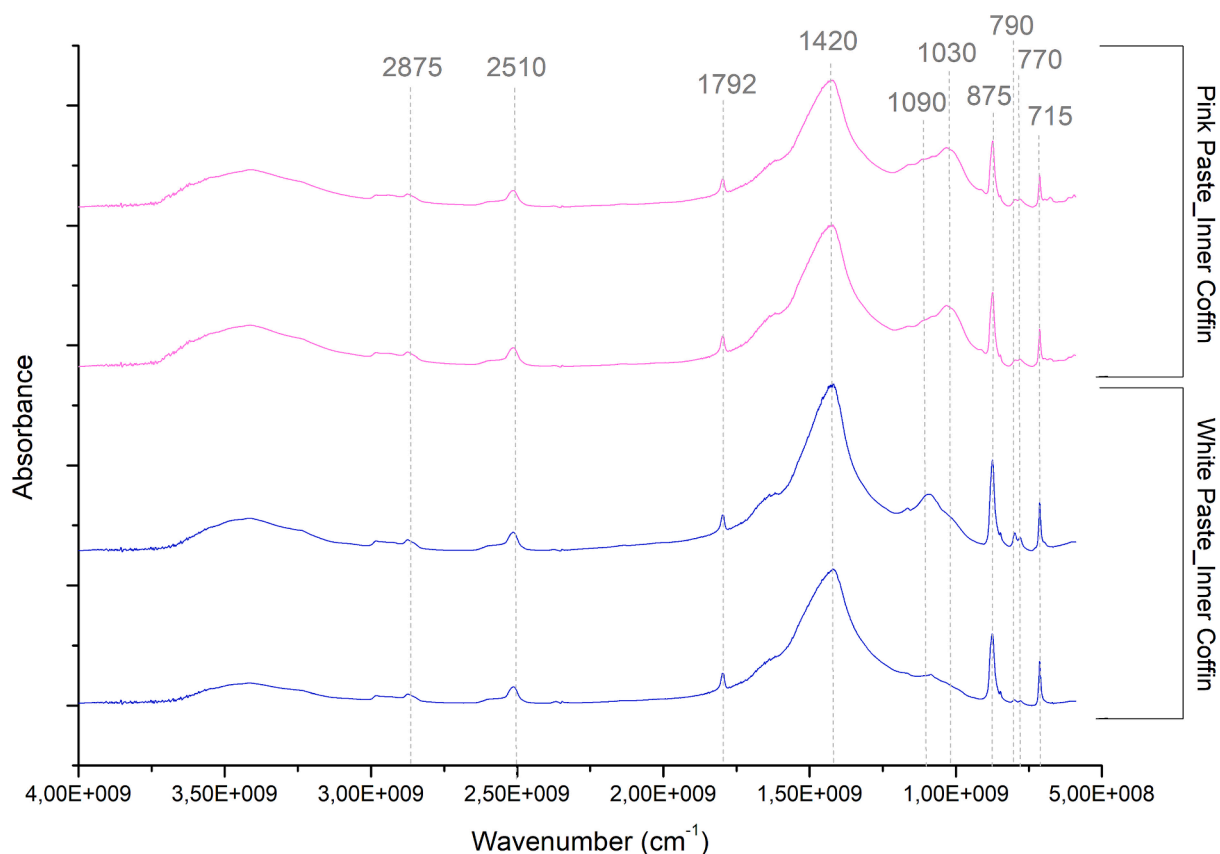


Fig. 17. FTIR spectra of white and pink pastes from the inner coffin of Pakepu, in the range between 4000 cm^{-1} to 500 cm^{-1} . The characteristic peaks of calcite (1420 cm^{-1} , 875 cm^{-1} and 712 cm^{-1}) are recognisable in all the samples, but pink pastes show a sharper peak at 1030 cm^{-1} , related to the characteristic pink shade of the filler. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Klemm 2008) and these should therefore be considered as possible sources for this geological material.

The scatterplot of FeO vs MgO for all the samples analysed corroborates the above findings. The latter shows consistently high values of both oxides for the pink pastes, low values for the white pastes from the intermediate coffin, and an intermediate position for the white pastes

from the inner coffin; white paste comparison fragments generally show high MgO and low FeO (Fig. 21).

4.2.2. Pyrotechnology

As with the white paste, v_2/v_4 ratios were calculated from the FTIR to determine whether pyrotechnology was involved in the preparation

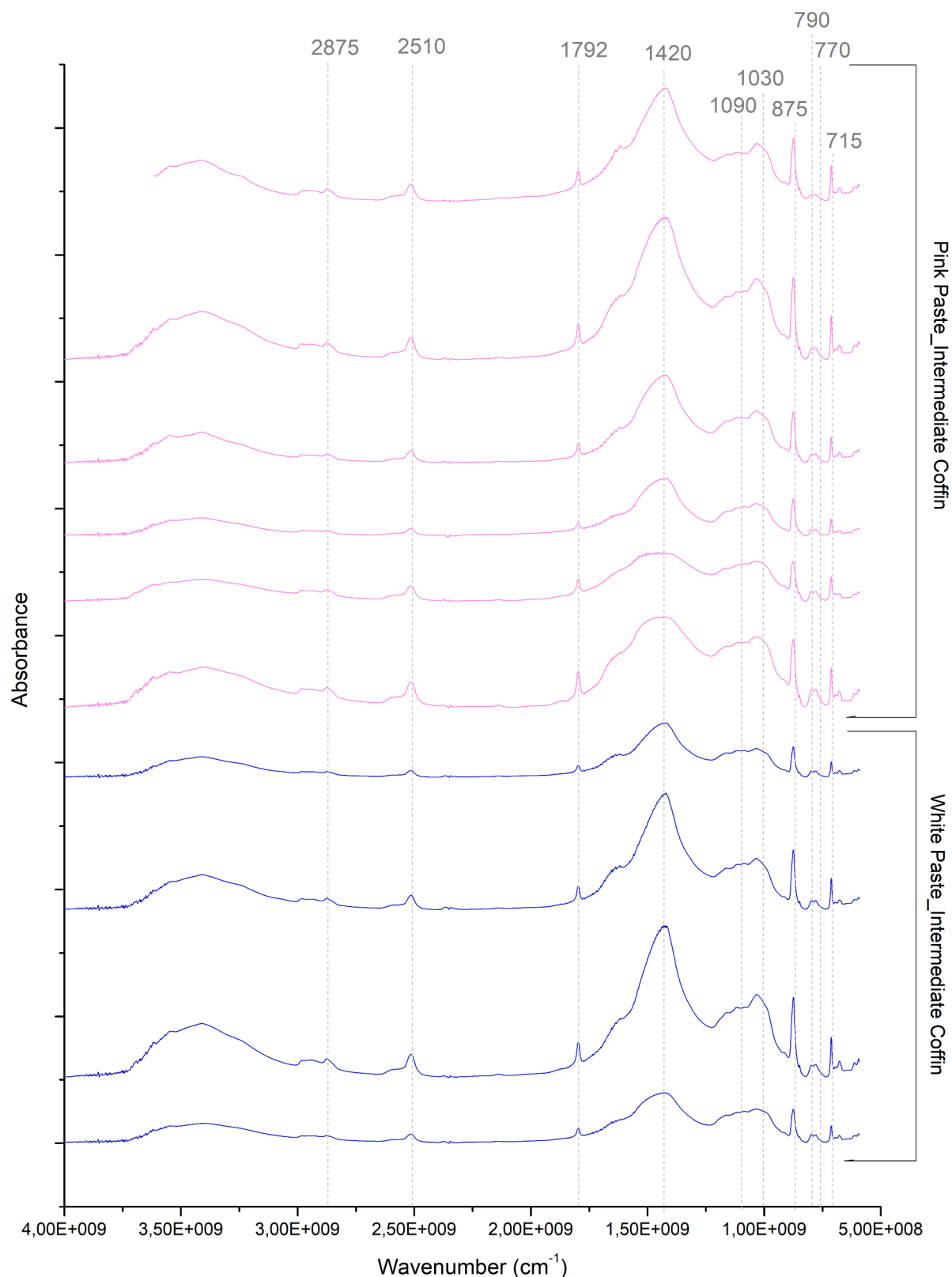


Fig. 18. FTIR spectra of white and pink pastes from the intermediate coffin of Pakepu, in the range between 4000 cm^{-1} to 500 cm^{-1} . The characteristic peaks of calcite (1420 cm^{-1} , 875 cm^{-1} and 712 cm^{-1}) are recognisable in all the samples. Bands corresponding to collagen, slate red, quartz and dolomite are also recorded for all the samples, but stronger signals for quartz and iron oxides are recorded for the pink pastes in the range between 1160 cm^{-1} – 1084 cm^{-1} – 1032 cm^{-1} and in the band at 779 cm^{-1} . (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

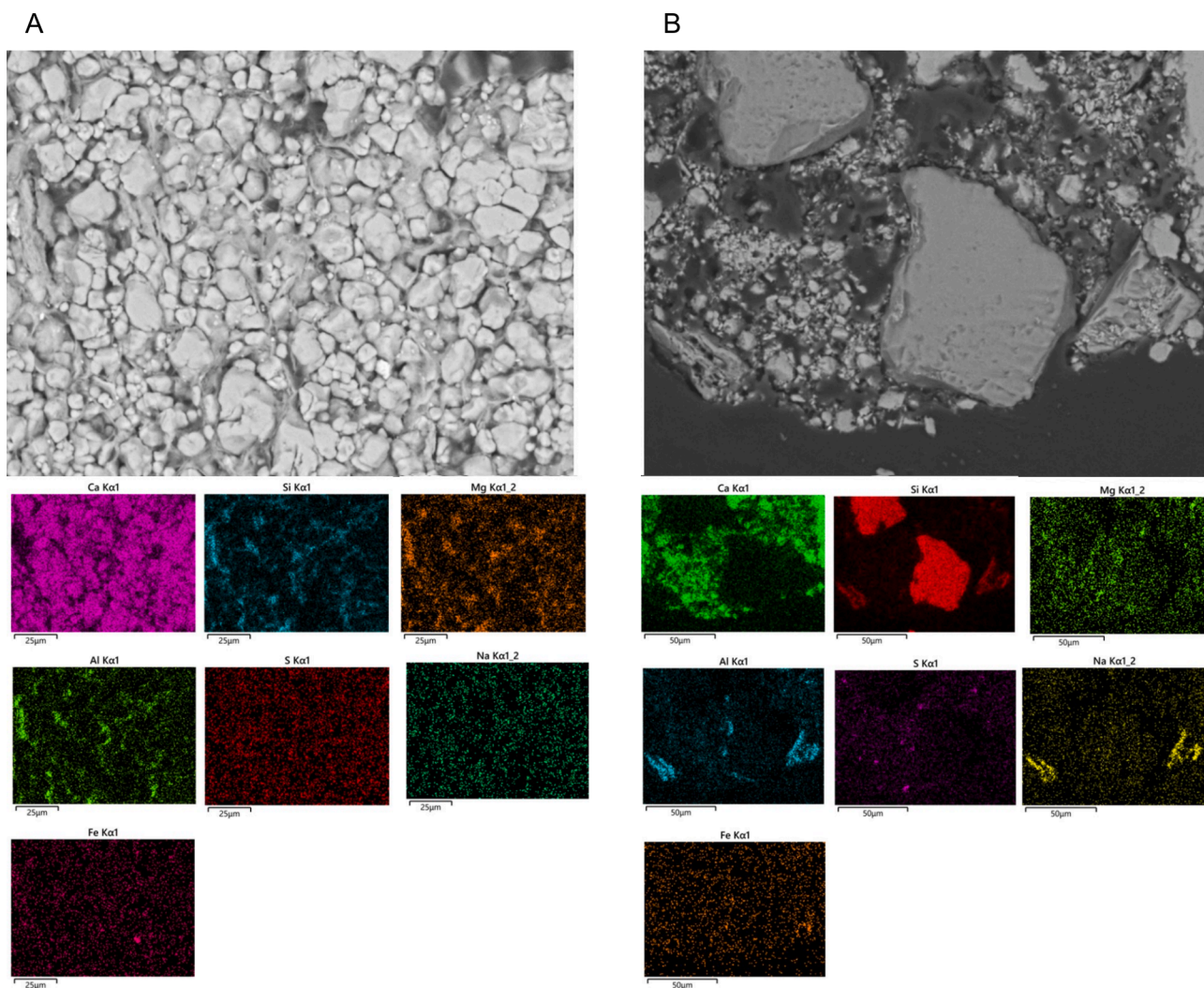


Fig. 19. SEM-BSE images and X-ray maps of elemental distribution in pink pastes. A: sample CA231048, Pakepu inner coffin. B: sample CA220347 (CR02_OBi), Pakepu intermediate coffin. Ca is the dominant element for all the pink pastes. In sample A, Si, Mg and Al appear to correlate with the presence of the fibres of fibrous glue. Sample B shows higher heterogeneity with a variety of coarser minerals. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

of this paste (Fig. 15). The v_2/v_4 ratios for the samples from the inner and intermediate coffin are below 3.3, indicating a lack of heat treatment. Only one outlier is found from the intermediate coffin, with a value of 4.15, potentially denoting heat treatment but well below calcination temperatures. These inferences are supported by the calcite grain sizes (Fig. 15 and Online Resource 2) invariably larger than $1\ \mu\text{m}$ and hence ruling out the presence of heat treatment for the production of these pastes.

In sum, it appears that, while natural calcite is the main component of the pink paste, this appears to have derived from red limestone and was mixed with siliceous mud, either deliberately or through natural weathering.

5. Conclusions

The makers of the coffin set of Pakepu used a variety of pastes: pink pastes as filler for gaps in the wooden construction and white pastes as preparatory grounds for painted decoration (either in simple layers or in more complex “pseudo-cartonnage” layered structures). Their study by optical microscopy, SEM-EDS and FTIR, and comparison with additional white paste samples from other pseudo-cartonnage samples from the same region and period have allowed us to reveal aspects of the material

selection and processing that may in turn inform about different workshop practices and raw material sources.

Calcite was recognized as the bulk component of all these pastes. All the white pastes were obtained by mixing homogeneous, finely powdered limestone with an organic binder. This material sealed the surface of the coffins while providing a white, smooth ground for the application of polychromy. However, notable differences were identified in their homogeneity and chemical make-up. The white paste from the Pakepu inner coffin appears to have been made with the purest form of calcite, and is also the richest in calcareous nanofossils. The white pastes from the intermediate coffin are more variable, but they are generally richer in iron and magnesia – the latter, likely due to minor presence of dolomite. Although variable too, the comparison fragments show generally low iron and high magnesia, in addition to frequently larger grain sizes.

The pink pastes were invariably richer in iron. While that from the inner coffin was very similar to the analogous white paste but richer in iron, the pink paste from the intermediate coffin was much more heterogeneous and included a variety of siliceous minerals of different sizes. In this case, either a calcareous mud or a mixture of mud with crushed calcite was employed. As this paste was predominantly used as a filler for gaps and not visible from the surface, appearance and texture

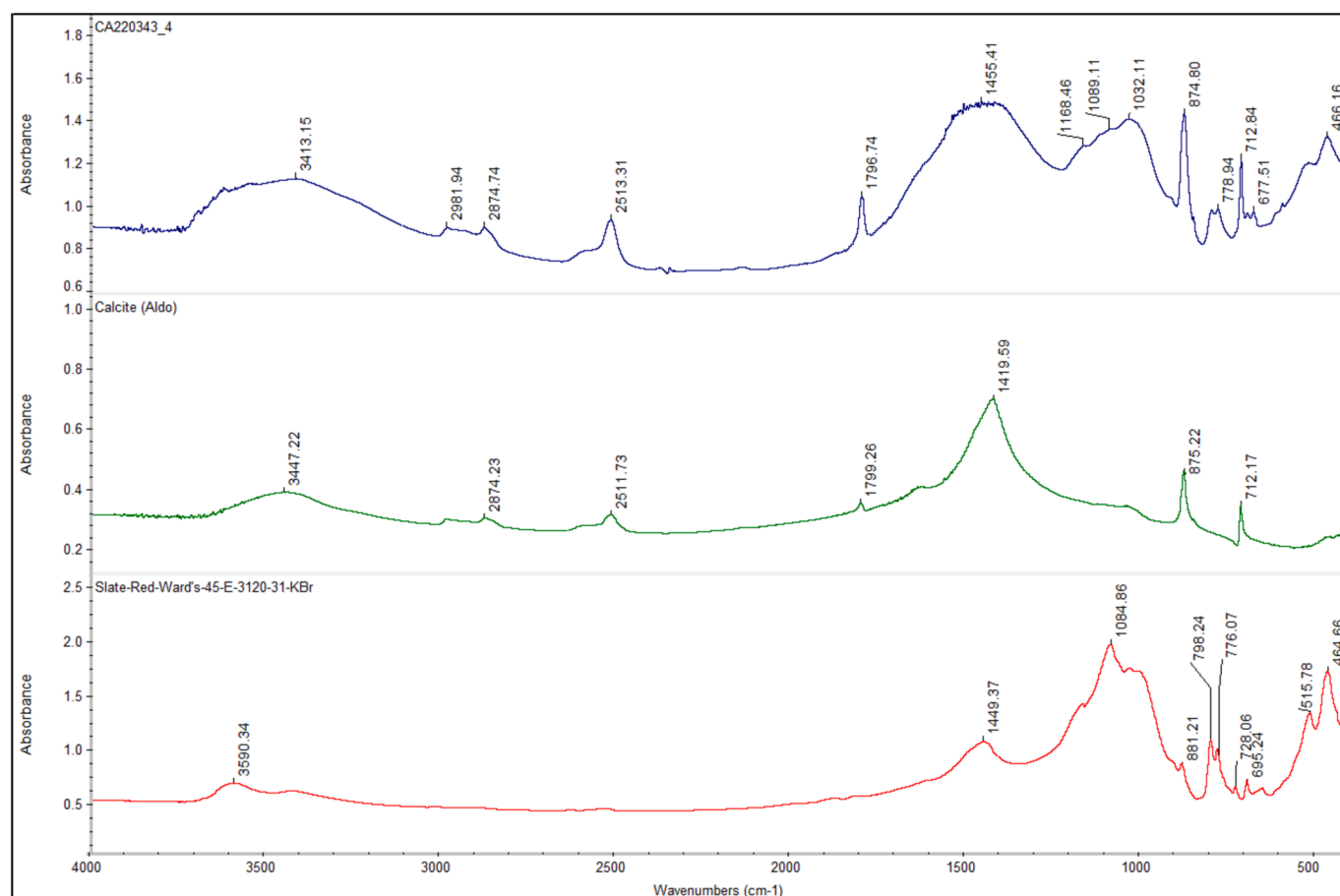


Fig. 20. FTIR spectra of pink paste sample CA220343 (E.2.1869_30L) from the intermediate coffin of Pakepu and closest matches in the Kimmel online library employed as reference, in the range between 4000 cm^{-1} to 500 cm^{-1} . The characteristic peaks of calcite (1420 cm^{-1} , 875 cm^{-1} and 712 cm^{-1}) are recognisable in the archaeological sample, but also matches with the slate red reference s at 1084 cm^{-1} – 1032 cm^{-1} and at 779 cm^{-1} , consistent the presence of iron oxides. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

may have been of less importance, while the presence of larger mineral grains may have helped prevent shrinkage during drying and thus facilitate structural stability.

Focusing on the coffin set of Pakepu, it was evident that the outer surface decoration for both components originated from a single workshop. However, macroscopic analyses of the coffin structures indicated that their manufacture likely took place in different workshops. The differences in grain size and composition of the pastes, only detectable through microanalysis, potentially reflect slightly different choices in terms of bulk materials and this supports the hypothesis that the pastes for the two coffins were not made together and at the same time. It appears that greater care was given to selecting and processing the geological material employed on the pseudo-cartonnage of the inner coffin, the structure closest to the deceased body, than those of the intermediate coffin. The differences in quality of the pastes revealed through this study add to the understanding of the purposes of each coffin, suggesting that the inner coffin served, like an earlier cartonnage mummy case, as the outermost layer of the mummy itself (Taylor 2016). Conversely, the intermediate coffin, with its pastes containing more impurities and contamination, served as a protective vessel for the pseudo-cartonnage structure.

The analysis of the comparison fragments showed some internal consistency that sets them apart from the Pakepu set, whilst differences within the group support our understanding from other technical and decoration variations that they come from more than one coffin. Despite the close relationship in layer structure, the inorganic components of these particular fragments did not have clear similarities on the micro

scale with the inner coffin pastes.

Overall, it appears that the makers of coffins in Third Intermediate Period Thebes shared a broad tradition in the use of crushed calcite based pastes, but different workshop practices and technological skills were present in the area at the same time (Taylor 2006), leading to the creation of different pastes at a microscopical and elemental level. These differences also highlighted the use of different raw material sources, present in the nearby area.

The analyses presented here demonstrate the potential of our methodological approach to identify different workshop practices and technological traditions in the production of coffins, by focusing on relatively inconspicuous and largely under-researched components. Expanding the sample to other regions and chronologies, as well as to lime-based pastes in other artefact types, will likely show further material and technological variability. It might then be possible to cross this information with research of other aspects of coffin construction from timber supply to iconography and polychromy, progressively revealing the complex craft structure behind the production of funerary materials.

Declaration of Interest-Caterina Zaggia

- **Research Support:** This research received no external financial or non-financial support
- **Relationships:** There are no additional relationships to disclose
- **Patents and Intellectual Property:** There are no patents to disclose
- **Other Activities:** There are no additional activities to disclose

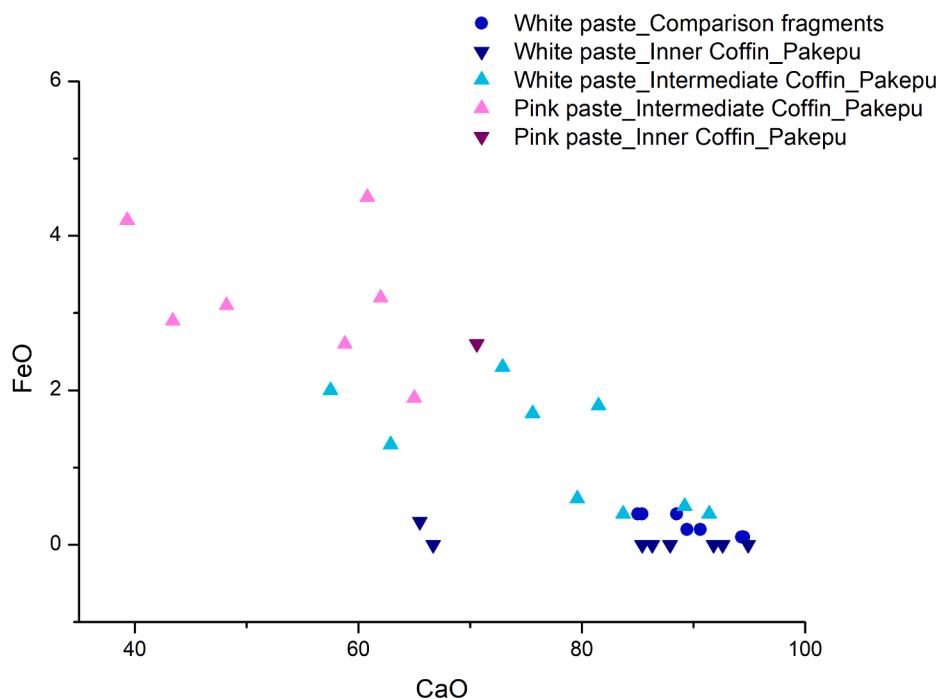


Fig. 21. Scatter plot of the concentration of FeO and CaO in the samples. The pink paste from Pakepu inner and intermediate coffin present a similar behavior, with lower CaO and higher FeO. All other sample types tend to show a lower FeO content, but they form relatively coherent groups based on the CaO concentration. The white pastes from the Pakepu inner coffin and the comparison fragments show similarly low concentrations of FeO (but see Fig. 8 for their different MgO levels). White pastes from the Pakepu intermediate coffin show more variable composition. See text for explanation of two outliers in the white pastes from the Pakepu inner coffin. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

CRediT authorship contribution statement

Caterina Zaggia: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Marcos Martín-Torres:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Matthew Collins:** Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Conceptualization. **Julie Dawson:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization. **Helen Strudwick:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization.

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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.jasrep.2025.105019>.

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