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A complementary integrated approach using non-destructive optical and X-ray methodologies for pigment characterisation on an ancient Egyptian coffin

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Abstract Museums and culture heritage institutions seek affordable, comparative, non-invasive imaging and analytical techniques to identify and study ancient materials and support their conservation. This study presents a multimodal research approach to assess the art-historical value of an isolated ancient Egyptian polychrome wooden coffin fragment from the KU Leuven archaeological collections, dating to the Third Intermediate Period (ca. 945–656 BCE). In a first step imaging techniques (standard even and raking photography, visible-induced luminescence, visible imaging spectrometry, white light and multispectral multilight reflectance imaging, narrow band multispectral imaging) were applied on the coffin's surface to document its state, to identify and differentiate original and restored areas, and to select spots for more in-depth spectroscopic molecular and elemental analysis (fibre optics reflectance spectroscopy, X-ray fluorescence spectroscopy). These allowed pigment identification, characterisation of mixtures and provided a deeper understanding of the object's condition and used painting techniques. All applied methodologies can be used in situ. The resulting datasets are curated into a multilayered IIIF Mirador 3 viewer, presenting all results in a complete and user-friendly environment.

1 Introduction

The application of non-destructive imaging and analytical techniques on archaeological artefacts has seen a steep rise in the past decade, including for the identification of pigments and minerals on polychrome objects [1–3]. These are especially welcomed in museums, where it is important to have detailed information on the composition and used materials of their objects, for conservation, restoration and storage purposes. Not all museums have access to (or the budget for) an extensive set of analytical techniques. This paper aims to present an approach using relative commonly available, non-invasive, non-destructive imaging techniques (i.e. visible reflection photography (VIS), narrow band multispectral imaging (NBMSI), visible-induced luminescence (VIL), white light multilight reflectance imaging (WL MLR), multispectral multilight reflectance imaging (MS MLR)) and analytical techniques (FORS, pXRF), applied to an ancient Egyptian polychrome coffin fragment, to assess its condition and identify the applied pigments, based on the acquired digital data. The study contributes to the discussion of how the combined use of spectral, reflectance spectroscopy and X-ray fluorescence data can be complementary for the identification of pigments on ancient artefacts [4] and how this information can assist in reconstructing the object life of isolated artefacts.

The Didactic Museum of Archaeology at the Faculty of Arts of the KU Leuven holds an ancient Egyptian coffin fragment (ARO000005, $62 \times 34 \times 4$cm) of an unknown provenance. The fragment was originally part of the 'Museum voor Oosterse Oudheden'/'Musée Oriental'/'Museum Egyptische en Voor-Aziatische Archeologie', which was installed during the Interbellum (i.e. from 1927 until it was heavily damaged during bombing raids in 1944) on the second floor of the University Hall in Leuven [5]. At that time the object had not yet received an inventory number. The object is also mentioned in the archival documents on the collection division, drafted on 2 May 1974, when half of the collection was moved to the University of Louvain-la-Neuve.

The fragment is the upper part of the central decorative panel of an anthropoid wooden coffin lid. It is divided in horizontal registers, including characteristic ancient Egyptian iconographic elements such as the winged solar disc, the four sons of Horus and the Wedjat-eye. Instead of the usual alteration of figurative and textual registers, each register only contains pictorial elements. The small yellow zones that were destined for hieroglyphic inscriptions remained empty; hence, it is impossible to identify the owner

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of the coffin. The inside of the lid contains no decoration, but is entirely coated with a 2–5 mm layer of an unknown black organic substance.

Based on the used colour palette and the iconography, the coffin fragment likely dates to the Third Intermediate Period (22nd to 25th Dynasty, ca. 945–656 BCE; [6]). This dating must be regarded as preliminary, given the lack of inscriptions. A discussion on the iconographic elements of the coffin is beyond the scope of this paper. It is nevertheless visually apparent that the pigments applied to the right side of the coffin fragment are not entirely consistent with the pigments elsewhere on the surface. This prompted further investigation into the object life of the fragment to find out to what extent these are traces of modern retouches. Therefore, given the absence of clear dating markers and provenance, the characterisation and identification of pigments could offer a more accurate date range for its production and provide insights on the overall authenticity of the object as it appears to the beholder today.[1]

In a first step, non-invasive imaging techniques such as VIS, NBMSI, VIL, WL MLR and MS MLR were applied on the entire surface in order to document its current state, to identify and differentiate original and restored areas, and to select spots for more in-depth spectroscopic and elemental analysis by means of fibre optics reflectance spectroscopy (FORS) and portable X-ray fluorescence spectroscopy (pXRF). This second step allowed us to identify the pigments or characterise the mixtures of pigments and to provide a deeper understanding on the condition of the object and the used painting techniques. All applied methodologies are portable and can be used in situ: All the equipment was transported to and applied in the storage location of the coffin fragment.

2 Materials and methods

2.1 High-definition standardised imaging (HDSI) for ground truth visual representations

Understanding and interpreting the materiality of the complex polychromy starts with the correct visual documentation of its current state. Due to damage and degradation, the appearance has most likely undergone changes that deviate from the initially intended result by its creator. This mainly concerns alterations in the colours, as well as in the micro and macro surface structure of the pigments. It is therefore important to establish a starting point that one can always revert to during the research and analyses, a digital visual ground truth. The fragment of the coffin is only slightly concave in shape and is in fact overall flat. In order to produce a digital representation of the original as truthfully as possible, the FADGI guidelines for still-images can be used [8]. To profile the light conditions and assess the quality (i.e. greyscale and colour response, white balance, lighting uniformity, resolution, colour encoding accuracy, sampling frequency, spatial frequency response (SFR), colour channel registration and noise) of the digital image of the coffin fragment, the GoldenThread FADGI ISO 19264 device level target was used, resulting in a FADGI 4-star rated representation [9]. The digital image was made with a Phase One IQ3 100MP camera system. To allow comparison with other visual representations of the coffin fragment, this image has been included in the multilayered Mirador 3 viewer (see infra). The lighting setup conceals variations in the relief in order to be FADGI compliant, so the HDSI was combined with two high-definition raking images (HDRI), raking 5° above the coffin's surface horizon, both from the top and bottom (Fig. 1). On the macro level, it clearly reveals the slight concave elevations on the outside of the coffin representing the deceased's knees. At the same time, small details on the surface reveal information on the physical materiality and structure of the applied pigment in its current condition. The combination of HDSI and HDRI ensures a standardised qualitative basic documentation of the coffin.

2.2 Multilight reflectance imaging (MLRI)

As the raking images in Fig. 1 reveal, this type of representations has great value in documenting and understanding the materiality and texture of a surface. Therefore, applying an interactive imaging system that expands this methodological approach has great potential. MLRI does that in particular. This technology, often referred to as Reflectance Transformation Imaging (RTI), computes a relightable composite image based on a source image set consisting of photographs of the same surface documented with multiple lighting angles. Depending on the applied processing methods, various characteristics of the surface (reflectance, albedo, surface orientations, …) can be enhanced and used to study macro surface features that would otherwise remain obscured [10–12]. Both white light (WL) MLRI and multispectral (MS) MLRI scans of selected zones on the coffin fragment were made by using the MicroDomes of the Portable Light Dome (PLD) system [13]. Their results proved to be particularly interesting to dynamically inspect and understand the succession and sequence of the preparation and pigment layers when visible at the surface. It also made it possible to examine the thickness, density and transparency (the latter especially when combined with the various multispectral responses) of the applied pigment layers. Depending on the activated visualisation style, both the thicker pigment layers and the structure below the preparation and pigment layers (condition of the wood panel) can be assessed (Fig. 2).

[1] No significant elements were picked up by the XRF analyses further supporting the organic nature of the substance. Ultimately the identification of this substance requires other techniques and is beyond the scope of this paper [7].

Fig. 1 White light colour representations with standard (centre) and raking (left and right) photography

2.3 Narrow band multispectral imaging

The photographical narrow band multispectral imaging (NBMSI) has been obtained in a single acquisition sequence using the 3PI-project's Spectral XV infrastructure by the KU Leuven Core facility VIEW. In short, it consists of a Phase one XF IQ4 150MP Achromatic Camera System (light sensor), a Schneider Kreuznach 120 mm LS f/4.0 Macro (lens), a filter wheel with bandpass and longpass filters and two EurekaLight panels with sets of 16 narrow band LEDs.[2] The coffin panel was positioned perpendicular to the camera system. The capture sequence includes both reflectance and fluorescence/luminescence shots. With the multispectral MLR system (see also supra), equally five sets of narrow band LEDs are producing MS images and create normal maps for five different micro depths with the estimations of surface orientations. For this study, in particular the NBMSI results, the multispectral images have been consulted individually or they have been placed in register in a stack or image cube (see also below, 'Dissemination Platform', par. 2.6) on which false colour and/or principal component analysis (PCA) have been performed (using the image processing interface ImageJ). We used an automated high-end NBMSI system, but equivalent results can be obtained with more manual mid-end capture infrastructure, as long as they produce a calibratable multispectral image cube [14, 15].

The standardised and calibrated workflow producing NBMSI datasets—when placed in a spectral cube—allow imaged materials to be characterised based on their changing spectral responses as registered across the electromagnetic spectrum, in our case ranging

[2] For a full description of the applied 3Pi Project's infrastructure, see https://doi.org/10.5281/zenodo.7410238.

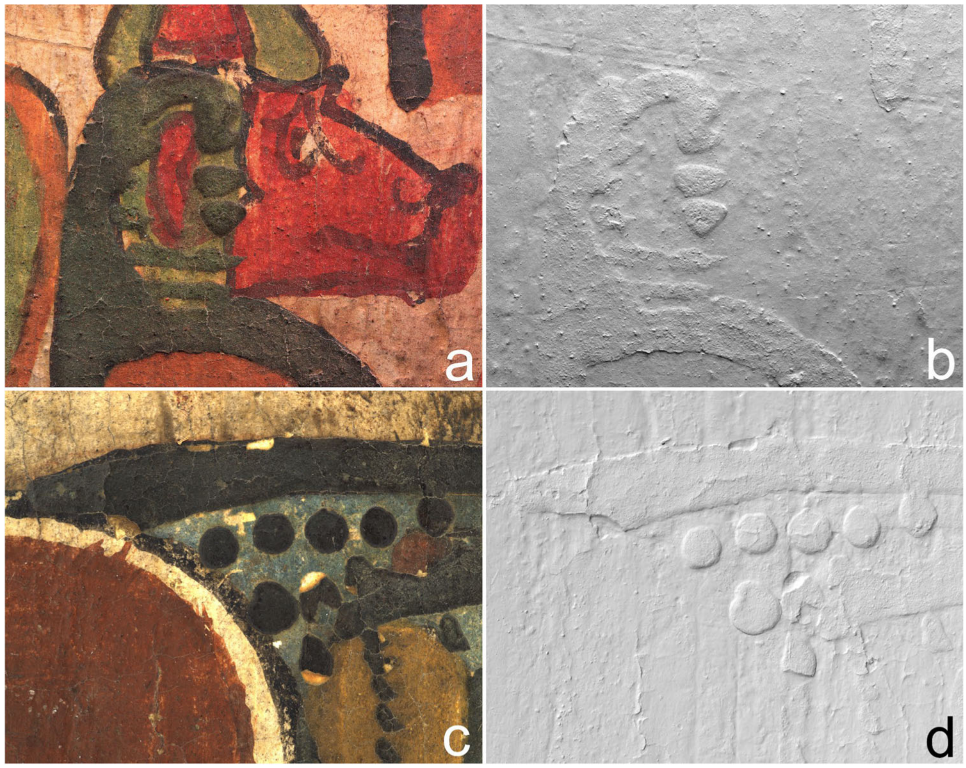

Fig. 2 **a** MS PLD image based on ultraviolet normals, RGB colour representation; **b** MS PLD image based on ultraviolet normals, albedo set to uniform value; **c** WL PLD image, RGB colour representation; **d** WL PLD image, albedo set to uniform value

Fig. 3 Left: Calibrated visual representation. Right: VIL image (radiation narrow band 660 nm, capture through IR longpass 850 nm filter)

365-940 nm. When compared with known references, especially when equally imaged with the same workflow, for a number of materials, this approach has the potential to allow identifications. To extract the spectral curves we followed the Wilson et al. [16] method, calibrations are based on a 99% Spectralon diffuse reflectance standard (imaged on all recordings), round spot size 20 pixels. This approach has the benefit that it produces spectral curves without the need to touch the original surface of the heritage object. And secondly, they can be generated on the fly when requested based on the original NBMSI spectral cube (SI Fig. 1 / file3). The Spectral XV capture sequence also included two visible-induced luminescence (VIL) recordings (Fig. 3) [17, 18]. One radiated with narrow band 600 nm and one with 660 nm, both captured through a longpass 850 nm filter. This detects the presence of the pigment Egyptian blue in which the copper ions (Cu^{2+}) have a strong luminescence effect in infrared (IR) with a maximum around ca. 910 nm.

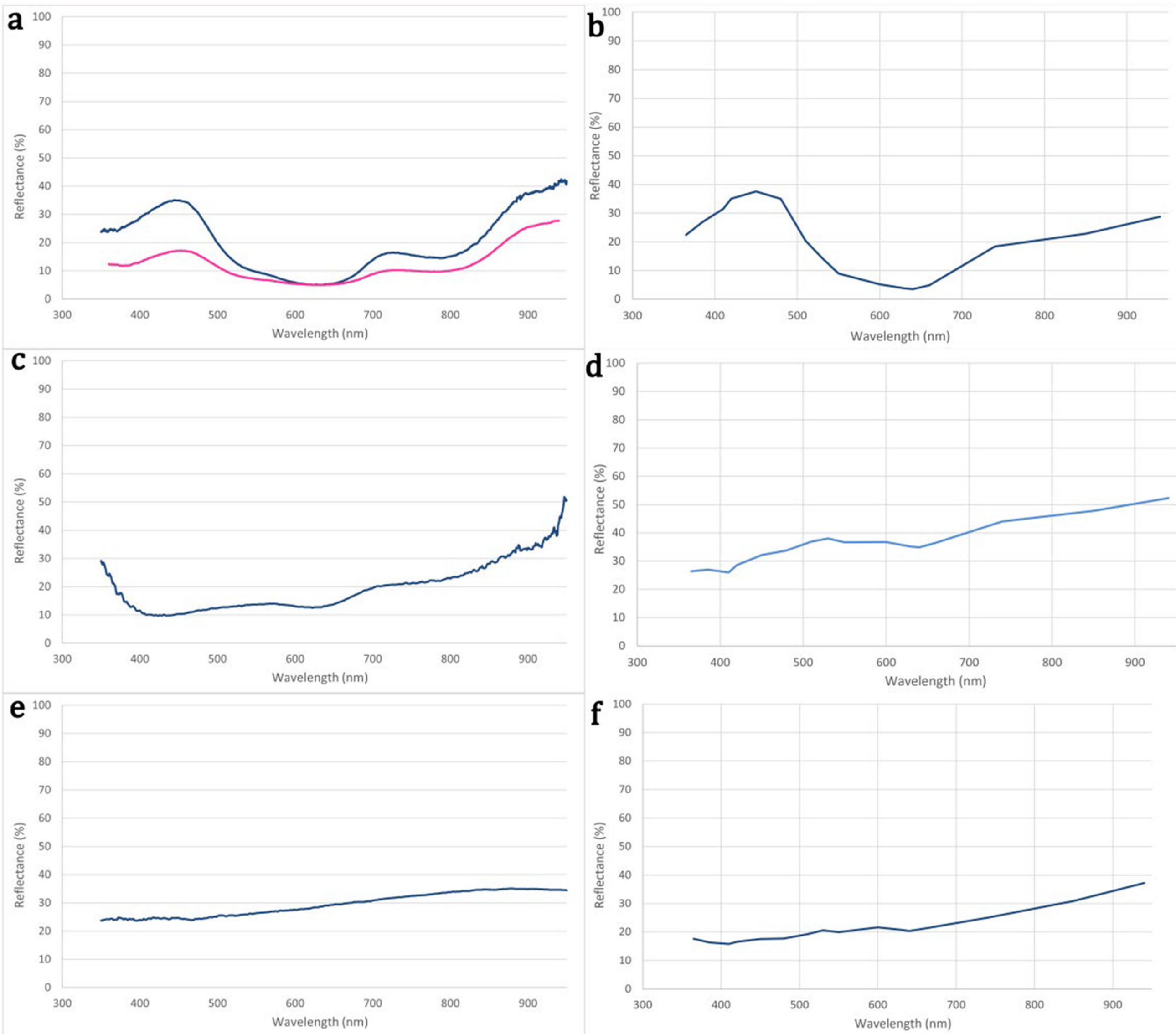

Fig. 4 Comparison of FORS and NBMSI spectral curve measurement on blue pigments. **a** FORS measurements on modern Egyptian blue samples, blue graph: on Pigment Checker v.5 with the KU Leuven device; pink graph: plot by CHSOS as included in the Gorgias database; **b** spectral curve measurement with NBMSI cube on modern Egyptian blue sample on Pigment Checker v5; **c** FORS measurement blue02 on pale blue pigment; **d** NBMSI measurement blue005 on pale blue pigment; **e** FORS measurement blue01 on darker blue pigment; **f** NBMSI measurement blue006 on darker blue pigment

2.4 Reflectance spectroscopy

For a fast, straightforward and relative low-cost spectral characterisation of materials the fibre optics reflectance spectroscopy (FORS) method can be applied [19, 20]. Although non-invasive, obtaining qualitative spectra does require soft touching of the original surface and therefore does not always present itself as an optimal method for fragile heritage objects. Based on a careful assessment, the robust stable condition of the pigment layers on the coffin allowed this method of measurement. The system produces, based on the ratio between the intensity of the reflected light and the incident light, for each wavelength the reflectance and plots it in a graph (in real-time). The reflectance measurements targeting specific spots with pigments were applied with the Gorgias FORS spectrometer which uses a Toshiba TCD1304DG linear array detector with a spectral range of 300-1000 nm (100 microns slit, 2 nm resolution). Light source is a 10W halogen lamp; the connected reflectance fibre probe consists of six excitation fibres (600 μm) and one collection fibre (600 μm). In our study, the spectral information extracted based on the NBMSI spectral cubes and the FORS measurements were closely aligned (Fig. 4). Their employability is therefore perceived as interchangeable.

2.5 Portable X-ray fluorescence (pXRF)

The coffin fragment required further chemical analyses in order to identify the used pigments, characterise the mixtures of pigments and to provide a deeper understanding on the condition of the object.

Non-destructive chemical compositional analysis was conducted using portable X-ray fluorescence equipment operated using a tripod system for measurement stability. The system used was a Bruker Tracer III-SD. The instrument used is equipped with an Rh anode X-ray tube and a Peltier-cooled Silicon Drift Detector (~145 eV at Mn Kα) with a spot size in fixed collimation of approximately 4.5 mm^2. The goal of the measurements is primarily to qualitatively assess the presence, ratio and distribution of different elements. In order to achieve this, analysis was conducted using 40kv and 10 μA during a live time of 100 s in air. All measurements are taken in close proximity to the target areas. Precision and machine drift of the instrument was monitored by replicated analyses of a certified reference sample CRM 2710a (NIST certificate: https://tsapps.nist.gov/srmext/certificates/2710a.pdf). Spectral analysis was conducted using proprietary ARTAX software to calculate the net intensities of the different identified elements (Supplementary Information (SI) Table 1 / file2) through Bayesian Deconvolution. All data were normalised to the Rhodium anode of the instrument to correct for instrument variation (if any), enhance comparative purposes of the semi-quantitative data and are subsequently used to generate bivariate plots. The analytical strategy entails a documentation of the elemental enrichments and depletions. The variable thickness and layered structured can induce small amounts of additional elements next to accessory elements present.

2.6 Dissemination platform

A multimodal approach driven by research questions automatically leads to a variation and complexity of visual and analytical data. Each applied method contributes specific information, facilitates in-depth research and provides insight in the material genesis and state of the object. They become part of the narrative that has substantiated the study step by step, and scientifically document the followed thought process. Managing, aligning and publishing these multimodal and multilayered datasets (online) is challenging. A solution is found in integrating and curating all these various datasets into one easy-to-use interoperable consultation platform, i.e. a Mirador 3 implementation (SI Fig. 2 / file4). The coffin fragment is presented in this virtual environment as it appears to the human eye, and it provides access to the full dataset used throughout the study. It also aligns and maps all of the visual data of the complete panel (in register) with the processed data (visual and analytical) for which details and measure points are annotated on the surface. To explore the multimodal results, the viewer is allowed to interact with all this layered information, which is accompanied by labels and curated metadata. By implementing this workflow in the existing end-to-end digitisation process of the home institution where the coffin fragment is kept, this curated dataset acts as an accessible scientific documentation of the data collecting strategy.

3 Results and discussion

The various materials and pigments visible on the surface were mainly analysed through the obtained data via the NBMSI, FORS (n = 16) and pXRF (n = 63) point measurements (cf. Figure 5 for locations). The section below discusses all the materials applied by the ancient Egyptian artisan(s) on the surface of the coffin fragment, based on the current knowledge of ancient Egyptian polychromy and painting materials [21–24]: the white preparation layer, the beige coloured background and the yellow, red, blue, green and black shades of pigments of the paints layer. Compiled XRF spectra for each of the colours can be obtained and evaluated in the supplementary information (SI Figs. 3–9 / files 5–11).

3.1 White

The coffin contains a white preparation layer, applied, most probably, all over the surface. This preparation layer becomes visible in the areas where the coloured paint layers have been damaged, e.g. on the right side of the panel between the green wing tips and especially at the panel's border on the left hand side, where modern damages clearly reveal this white layer (measuring ca. 3 to 1.5 mm) (SI Fig. 10 / file12) along the entire stretch. The pigments were applied directly on top of this preparation layer.

In one area, where this particular layer was visible due to surface damages, pXRF analysis was conducted (White 01a in Fig. 5). Only a single analysis was possible due to the small available surface area. Based on the very high presence of calcium and traces of sulphur (SI Fig. 3 / file5), this would be consistent with a calcium sulphate layer (gypsum) rather than calcium carbonate [1]. Magnesium (as a marker for a huntite pigment identification) is typically difficult to measure with a portable XRF system, especially without a vacuum or helium flush in place. In this particular setting, the presence of magnesium can thus not conclusively be identified.

Traces of titanium were attested, which is not unusual in the case of ancient Egyptian painted coffins [1], possibly explained by the presence of sand or natural Ti-bearing minerals in the ground layer applied directly to the wood.

Fig. 5 Documentation of measurement points of the various analysed areas. Top: pXRF measurements. Bottom: FORS measurements (yellow), NBMSI measurements (white, only those included in this paper)

3.2 Yellow

Yellow (visually yellow ochre) pigments were mainly used on the cobras, the torso of the four sons of Horus, and in the multicoloured metope bands. It was also used to fill the smaller zones foreseen for hieroglyphic inscriptions next to the heads of the four sons.

All yellow zones appear to be the same hue/intensity, suggesting that no alterations were made in later periods. Qualitative analysis of five obtained XRF spectra points to the identification of a Fe-based pigment specifically enriched in As (Fig. 6a) (SI Fig. 4 / file6).

The most frequently used yellow pigment in ancient Egypt was yellow ochre, an iron earth pigment consisting of iron oxide hydroxide goethite (α-FeO·OH). It was used from the Predynastic Period to the Roman Period. In some cases, traces of the so-called limonite ($FeO \cdot nH_2O$) were found in yellow ochre pigments [25]. Alternatively, orpiment (As_2S_3), an arsenic sulphide mineral, was attested as early as the 2nd Dynasty. It was, however, often identified on coffins from the New Kingdom to the end of the Third Intermediate Period [26]. It is known that yellow pigments were used to imitate gold, especially on coffins. Of all Egyptian yellow pigments, the hue of orpiment was regarded as the closest to actual gold [21]. The presence of As in the yellow pigment suggests that orpiment was also used on this coffin.

3.3 Beige

A large area of the coffin has been covered with a distinctive variable beige to cream colour. As can be observed also visually over the entire surface, the green, yellow, black and red pigments have been applied on top of this beige pigment. For the thick packages of blue pigments, this is more difficult to determine. The section detail on the small damaged zone on the left side of the coffin (SI Fig. 10 / file12) reveals no clear layer of the beige pigment in between the white preparation layer and the thick layer of Egyptian blue.

XRF analyses of 20 surface locations point at a Ca-rich material and will therefore be strongly influenced by the preparation layer. There is, however, a clear presence of As or Zn, and traces of S as well (Fig. 6b) (SI Fig. 5 / file7). Based on the unusual combination of both Ca and As in the same material, this likely can signify the admixture of an arsenic sulphide like (para)realgar (As_4S_4) or orpiment (As_2S_3) to a white pigment (e.g. calcium carbonate, gypsum or huntite [27]) in order to obtain a pale brownish yellow colour [24, 27]. A number of measuring points on areas with beige are rich in Zinc (locations: Beige 4a, 4b, 18a, 19a and especially 20a on Fig. 5). These are clearly distinguishable from the others, and thus, suggest the use of a more recent [28] or modern beige coloured pigment, applied for retouches on these specific locations on the surface.

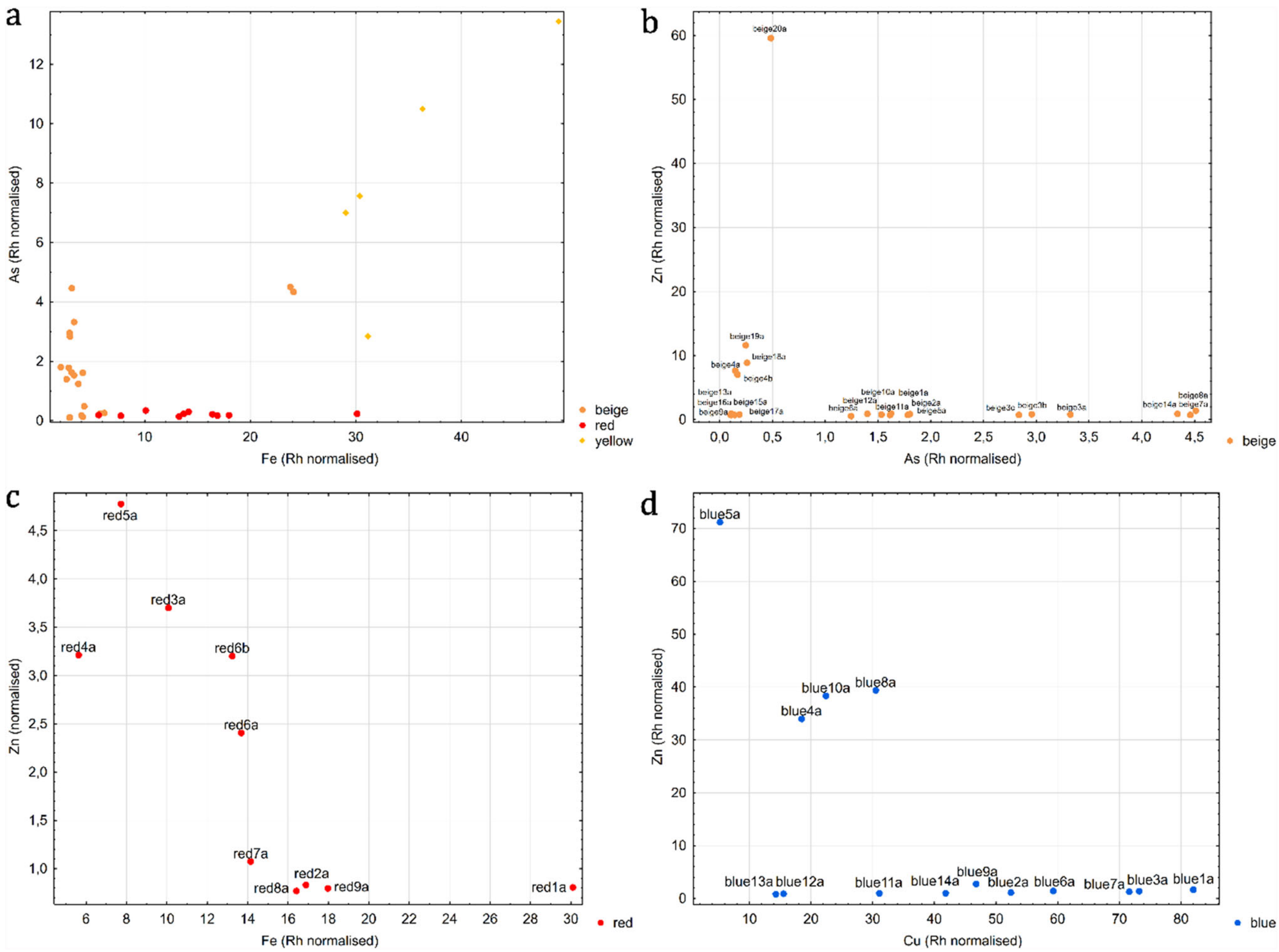

Fig. 6 Rh normalised scatter plot of net peak intensities of **a** beige, red and yellow pigments showing enrichments in both Fe and As for the yellow pigments, as well as enrichment in As for the beige layers and Fe for the red layers; **b** the beige areas showing either Zn or As enrichments; **c** Fe-rich and Zn-rich red pigments **d**; Zn and Cu for the blue pigments, clearly delineating Zn enriched measurements from purely copper based pigments

Coffins with a beige background layer are known from the 26th Dynasty (672–525 BCE, [29]). Coffins from other periods might have a similar background, due to the discoloration or degradation of the original (white) pigment, or due to the use of varnish.

3.4 Red

Two types of red appear to have been used on the fragment. On the central part of the fragment, a rather transparent red pigment was used, with a slight orange undertone. On the right side of the fragment, a darker red was applied. The cohesion of the latter with the preparation layer appeared to be less strong, causing the red pigment layer to flake, e.g. at the wing tips.

In the XRF data of the transparent red (SI Fig. 6 / file8), only Fe and Ca are significantly present, of which Ca probably contributes from the white preparation layer. Red pigments 3a, 4a, 5a, 6a and 6b (all located on the right side of the panel, Fig. 5) contain a significant amount of Zn next to Ba (Fig. 6c).

The predominant red pigment in ancient Egypt was red ochre, used from the 5th Dynasty (ca. 2435–2305 BCE) to the Roman Period (30 BCE-395 CE). Red earth pigments generally contain iron(III) oxide hematite (α-Fe_2O_3), quartz and clays [20]. Hematite is said to be only in use from the 6th Dynasty (ca. 2305–2120 BCE) to the First Intermediate Period (ca. 2120–2030 BCE), with some examples specifically dating to the 18th Dynasty (ca. 1550–1295 BCE) [29]. Realgar (As_4S_4), an arsenic sulphide, is an orange-red pigment. It is an extremely light-sensitive pigment that degrades into pararealgar (orange-yellow). Red lead pigments were not attested before the late Ptolemaic/Roman Period [22], while cinnabar was mainly used in the late Ptolemaic and Roman Period [26].

The red pigment on the central part of the coffin can be identified as a red ochre. The presence of Zn (and Ba) in the red pigments at the right side of the coffin, however, suggests that these areas have been altered (e.g. through restoration/conservation) at a later date, commonly mixed with lead white, zinc white or organic lakes to enhance opacity. From the late nineteenth century and until the 1960s, zinc oxide (zinc white) was used widely as a white pigment, but it was also added as a lighting agent to other pigments [28] (see also below, 4.1). Barium sulphate can be present as an extender or filler basis [30].

3.5 Blue

Four shades of blue pigments can visually be distinguished on the coffin fragment (Figs. 4 and 5). They also vary in density and thickness. Blue pigments on Egyptian artefacts are generally identified as Egyptian blue, an artificially manufactured cuprorivaite pigment ($CaCuSi_4O_{10}$) first synthesised around the late 4th millennium BCE in ancient Egypt and Western Asia [31, 32].

The presence of Egyptian blue on the coffin fragment was confirmed by the visible-induced luminescence (VIL) test produced with the NBMSI system (supra Fig. 3). Though visibly showing four shades of blue, the test identifies Egyptian blue in only three of them, of which the zones with the palest blue reveal the luminescence effect most prominently. This identification is confirmed by the clear presence of Cu in the XRF measurements (SI Fig. 7 / file9), and therefore clearly consistent with authentic (ancient) Egyptian blue ($CaCuSi_4O_{10}$).

The varying shades might be explained due to smaller (pale) or larger (darker) particle sizes of the Egyptian blue powder [33]. What also plays a role in terms of the physical materiality is that the darkest blue is applied in much thicker layers compared to thinner layers that show a visibly paler blue (see the MLR images, Fig. 2). This is related to the fact that Egyptian blue pigment is transparent, and requires multiple, very coarse layers in order to create a deep blue shade. The differences in shades cannot be explained due to the addition of gypsum or calcite to the blue mixture, as discussed by Hussein et al. [34], as the XRF data show no significant changes in the Ca.

The palest shade, containing the finest pigment grains, was easier to apply than the very dark shade, with the larger grains. Therefore, the dark shades of Egyptian blue were probably not painted, but dabbed onto the surface [35].

Discolouration of Egyptian blue is not related to the degradation of the pigment itself (as it is considered to be a very stable pigment, being also resistant to strong light), but is probably due to the accumulation of dirt on the surface or the darkening of the (organic) varnish or binders [35]. The coarseness of Egyptian blue pigments creates a rough surface that traps more dirt than the other (finer) pigments [35]. The darkening of Egyptian blue appears to be only happening at the surface, with the fresh blue pigment still preserved underneath [35]; this phenomenon has been documented at several macro spots on the coffin panel where the thick (darkest) blue paint layers were locally damaged and more bright underlying blue paint can be observed (see SI Fig. 11 / file13).

The chemical composition of the fourth and darkest shade of blue, however, appears to be an anomaly. XRF measurements on these spots show these are the only pigments containing relatively high amounts of chromium and zinc (Fig. 6d), suggesting these are not original/ancient pigments. It appears only on the right-hand side of the panel fragment and includes retouches of several wing tips. Interestingly, in those same zones the VIL image (see infra and Fig. 3) shows that this section underwent an undatable post-production intervention during which Egyptian blue was smeared in a downwards vertical direction. This might have been considered an aesthetical unpleasing damage that, in modern times, led to the overpainting of various figurative elements.

3.6 Green

All measured XRF spectra (SI Fig. 8 / file10) show a consistent Ca-Cu composition, which signifies that only one shade and type of green was used on the fragment. In both the wing tips and in the rectangular green coloured borders in the lowest register, the colour appears to be somewhat transparent. The VIL images of the coffin fragment do not detect any luminescence in the green parts (Fig. 3). Based on the chemical composition, the green pigment can be identified as a copper-rich pigment.

Egyptian green, a synthetic green pigment, was made with the same ingredients as Egyptian blue, but with variations in the ratios. For the green variant, more silica and lime were required, and less copper [36]. In some cases, green was identified as a mixture of Egyptian blue and either yellow ochre or orpiment [1]. Despite being made of the same ingredients as its blue counterpart, Egyptian green does not have the effect of luminescing in the IR when radiated with visible rays and is thus not visible on VIL images.

Another green pigment, malachite ($Cu_2(CO_3)(OH)_2$), has been attested from the Old Kingdom to the 26th Dynasty [25, 37]. From the 5th Dynasty onwards, several other green copper chlorides have been attested [22].

Based on the chemical composition at hand, it is clear this pigment is Cu-Ca based. This particular elemental composition can relate both to Egyptian Green as well as Malachite. However, the extensive presence of varying shades of Egyptian blue in other areas of this coffin shows that the artistic workshop applying the decorations was closely acquainted with its production technology and it might, as such, also hint at their knowledge of producing and applying Egyptian green. This is an indirect argument, as the XRF data are insufficiently conclusive in this matter. Contradictingly, the FORS and NBMSI spectra on the green measure points show responses for which the reflectance peak is situated closer to that of malachite (540-550 nm), than that of Egyptian Green (510 nm) [38] (Fig. 7). Therefore, the final identification of the green pigment remains inconclusive.

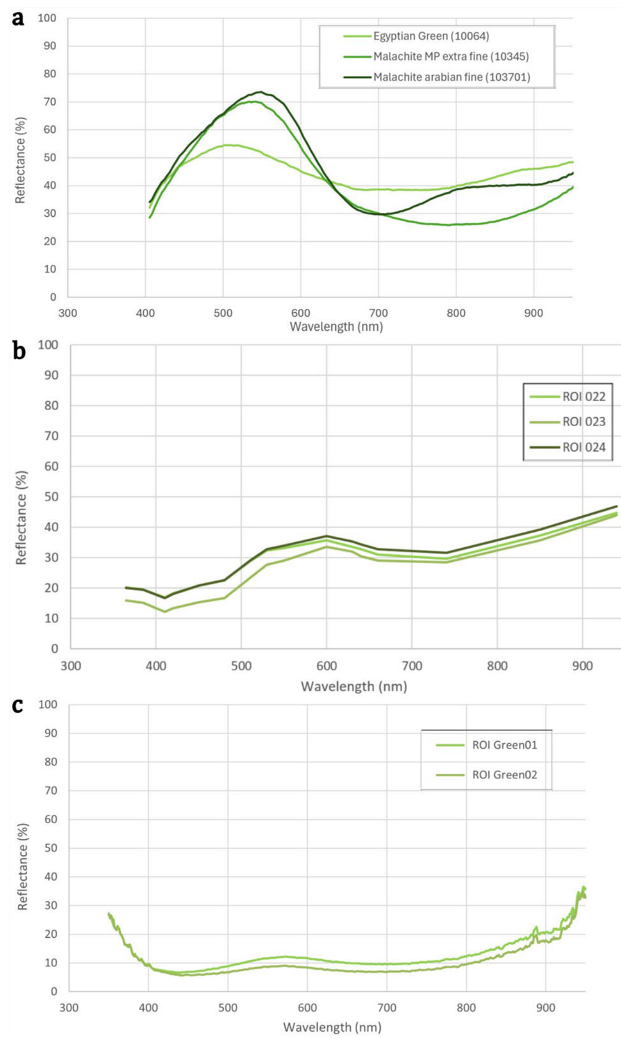

Fig. 7 Comparison of NBMSI and FORS measurements on green pigments: **a** Spectral curves from Deborah database [38], as reference for Egyptian green and Malachite. https://hyppigments.streamlit.app/; **b** NBMSI measurements green022, green023 and green024 and **c** FORS measurements green01 and green02

3.7 Black

Black was only sporadically used on the fragment. It can be found in the pupils of the Wedjat-eyes, on the beak of the Sokar falcon and on the heads of the four sons of Horus. It was also used in the multicoloured metope bands. Furthermore, the underdrawing of the coffin seems to have been drawn with a black pigment.

Five XRF measurements (SI Fig. 9 / file11) were conducted that seem to detect solely the underlying preparation layer—i.e. Ca based—and not the pigment itself, confirming the identification of the black pigment as carbon.

Ancient Egyptian black pigments have been analysed by several scholars, and the general agreement is that these are (almost) always identifiable as carbon. Lucas identified the pigment as soot, probably scraped from cooking pots, making it a widely available source [39]. It is a very fine-grained pigment, especially when compared to charcoal black [40]. The latter pigment is made by burning organic materials, such as wood or oil, pulverising the material and mixing it with water. In most cases a binder is used, often a plant gum from the acacia tree family [41]. The use of pyrolusite (MnO_2) as a black pigment was questioned for a long time, but has been confirmed on two 12th Dynasty reliefs (one at Beni Hassan [39]; one at Dayr al-Barsha [42]) and artefacts in the Theban tomb of Kha and Merit (18th Dynasty) [43]. One must take into account that lead pigments, vermilion and Egyptian blue can blacken when decaying [44]. Although there are some examples where this happened, none of these pigments could be identified on the coffin fragment.

The pupil of the Horus eye on the right side of the coffin, however, contains a significant amount of Zn, consistent with the lighter pigment next to it. As carbon is not detected by the pXRF, this might suggest that the entire eye was repainted with a—possibly modern—zinc admixed pigment before the black pupil was painted.

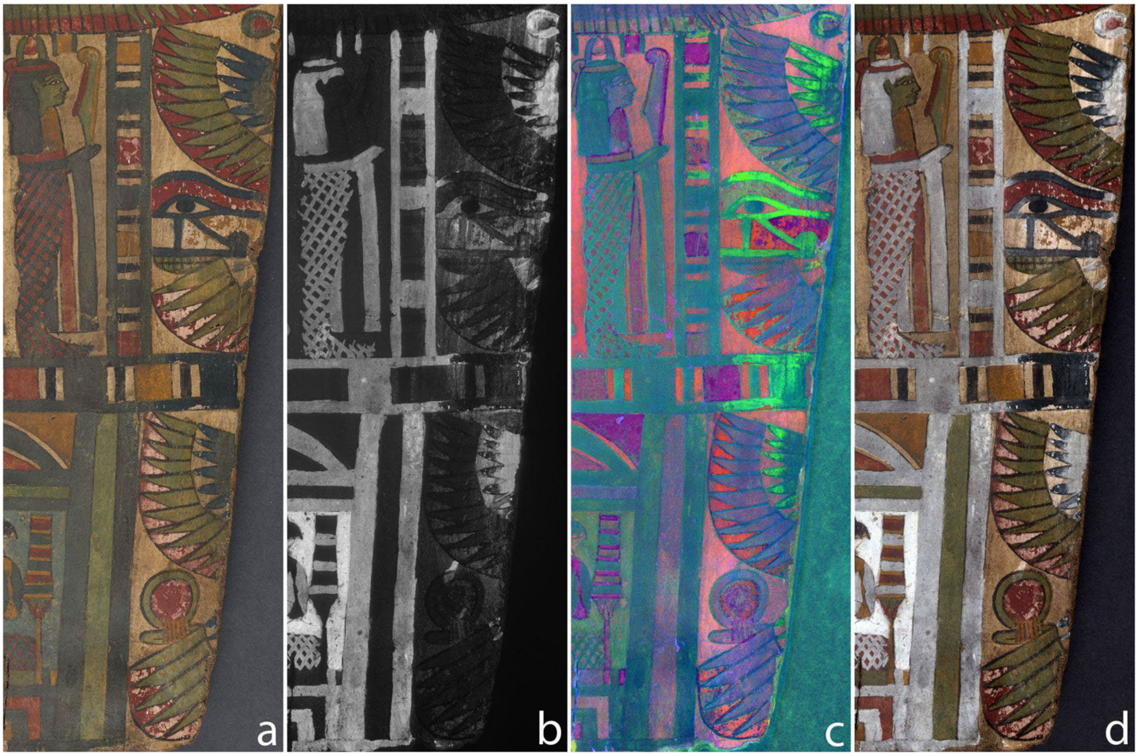

Fig. 8 Detail of right edge of the coffin panel, visualising smear marks and re/overpainted blue areas. **a** Calibrated visual representation; **b** VIL image (radiation narrow band 660 nm, capture through IR longpass 850 nm filter); **c** PCA image (based on all NBMSI spectral and luminescence shots); **d** overlay by ‘a’ (50% transparency) on ‘b’

4 Conclusions

The analysis of all the collected data makes it evident that both optical and chemical data are imperative for the comprehensive investigation of complex archaeological objects of this nature. Each of the types of data produced contributed in part to compiling the full story.

4.1 Restoration

NBMSI images show the right side of the panel has been affected by a, most probably post-excavation, intervention. Clear vertical (starting from the top right corner) smear marks are visible, in particular in the VIL image (Figs. 3 and 8). The reason for this action remains unclear. A failed attempt to clean the surface, possibly with an undefinable organic compound, is a likely explanation. In the VIL image, it is clear the action has partly dissolved the Egyptian blue pigment and smeared it downwards on the panel, leaving behind thinned-out traces of it. This event also affected the presence of other original paints, causing a (partial) repaint of the right side of the panel. By producing PCA images based on the NBMSI spectral and luminescence captures, the spread of these secondarily applied paints can be visualised and differentiated from patches painted in closely similar colours. In Fig. 8, this is shown by the spread of the secondary blue, highlighted in the image as bright green.

High amounts of zinc (Zn) in several zones that have been repainted on the same right side of the panel could point to the use of an industrial-era pigment (Zinc white) [28]. Zinc white additionally has the possibility to impact on the colour temperature of other pigments due to its semi-transparency properties and was used in that way from the middle of the nineteenth century [45]. The presence of chromium in the blue pigment and Zn in the red, blue and beige pigments in that zone confirms that the decoration of the coffin has been extensively altered in the industrial era. In the nineteenth century, coffins and other polychrome artefacts were often repainted by art dealers in order to make the objects more appealing for potential buyers. In our case, this may have happened in particular after a failed cleaning with a solvent product that partially damaged the original paint layer.

Traces of a shiny substance on the side of the fragment (on the wood) might suggest that the fragment was covered with a sort of varnish at some point. The fact that remains of this substance are also visible on the wood (i.e. on a post-production fracture) suggests that this must have happened after the coffin was already broken, and might therefore be a modern addition. This varnish has not been detected by the pXRF. Apart from some smudges on the edge, the varnish layer appears to be very thin.

4.2 Polychromy

A white pigment was probably mixed with a yellow arsenic (As) pigment (presumably orpiment) to create the main beige layer of the panel. Notably, arsenic is frequently attested at many measure points on the (original) pigments, suggesting that the beige layer was originally applied to the entire surface. However, several zones that were repainted in the industrial era do not contain any arsenic at all. This does not mean that the original pigments were entirely removed, but that covering the original pigments with a modern paint causes As to be no longer detectable by the pXRF. An approximate calculation of the penetration depth of As in pigments with densities of ~ 3.08 g/cm3—3.5 g/cm3 would be roughly around 100 μm.[3]

Based on the collected data, the stratigraphy of the layers was reconstructed. No traces of the use of a linen layer to smoothen the original wooden surface are visible, nor are there traces of a ground layer consisting of mud (*mouna*). The white gypsum preparation layer is considerably thicker than the top layer containing the pigments.

The stratigraphy of the layers presents itself as follows:

1. Wood
2. Preparation layer (gypsum—white)
3. Background (beige layer)
4. Paint layer: decorations (all colours)
5. Varnish (modern)

While the sequence in which the pigments were applied to the surface cannot always be determined, some observations can be made. It is clear the beige colour was the first to be applied, followed by the green, yellow, black and red pigments. While the application of the (thick layers) of blue pigments is more difficult to determine, some areas clearly show it was placed on top of the yellow. However, as this cannot be confirmed in all areas, we must refrain from drawing all too strict conclusions regarding the applied technical-artistic traditions.

Dating the coffin purely based on the composition of the used pigments is difficult. The majority of the pigments was in use during a long period, covering more than two thousand years. Apart from the modern alterations on the right side of the panel, all pigments were identified as ancient and confirm the authenticity of the fragment. In order to establish the date and provenance of the coffin, the style and iconography require further in-depth research, but this has already been marked as being beyond the scope of the current paper. All acquired insights into the materiality of the original pigments with the applied techniques confirm the initial dating based on the iconographic analysis, that is, Third Intermediate Period (22nd to 25th Dynasties, see also [6]).

4.3 Research protocol

This study advocates that the integration of widely accessible mobile equipment should not only be optional, i.e. only being applied in the case of specific research questions, but rather should be considered as being part of a standardised protocol. While the search for low-cost and easy-to-use techniques is ongoing, several techniques have already proved to earn their place in this explorative protocol. This is definitely the case for the combination of (multispectral) imaging and chemical analyses for the study of pigments. As the results show, this combination not only allows to identify individual pigments, but also to distinguish and clearly visualise the original pigments from the modern restorations. The anomalies that became visible during the imaging of the object were not only confirmed, but also to a significant extent explained by the chemical composition of the pigments obtained through XRF. Vice versa, the apparent differences in the chemical compositions were often supported by the imaging results, showing for example differences in the density of the pigments layers.

The authors acknowledge that the high-end systems used in this multimodal study are not one-on-one accessible for all institutions, e.g. due to equipment and operation costs and the requirement of trained operators. It should be noted that several steps in the protocol can be replaced by more accessible lower-end systems, such as using conventional photography equipment (Canon, Nikon, …) instead of the Phase One Camera, and handheld Reflectance Transformation Imaging (RTI) instead of the PLD system for MLR imaging. Furthermore, the study shows spectroscopy systems such as FORS produce similar results as extracting reflectance information from photographical narrow band multispectral (NBMSI) datasets. Applying both within the execution of standard documentation and research protocol for this type of objects is therefore undue. Finally, it must also be emphasised that the protocol with non-invasive methods followed here does not allow addressing any research questions concerning the analyses and presence of organics in the coffin's decoration materials.

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[3] Based on commercially available pigment, e.g. https://www.kremerpigments.com.

Data Availability Statement The authors declare that the data supporting the findings of this study are available within the paper and its supplementary Information files. Should any raw data files be needed in another format, they are available from the corresponding author upon reasonable request. The manuscript has associated data in a data repository

Declarations

Competing interests The authors have no competing interests to declare that are relevant to the content of this article. No funding was received to assist with the preparation of this manuscript.

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