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ARCHAEOMETRIC STUDY OF A POLYCHROME WOODEN COFFIN FROM 26TH DYNASTY-EGYPT

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ABSTRACT

This study focuses on a multi-analytical method to identify the pigments, ground layer, and previous restoration materials used on a polychrome wooden coffin (Late Period) from Saqqara, Egypt. It utilized technical imaging, optical microscopy, scanning electron microscope attached to X-ray dispersion unit, X-ray diffraction, and Fourier transform infrared spectroscopy. The results showed the use of hematite and cinnabar for the red painted layers, Egyptian blue for the blue painted layer, yellow ochre for the yellow painted layer, Atacamite and Paratacamite for the green painted layer and carbon for black painted layer. The ground layer was calcium carbonate. The original binding medium was animal glue, while the previous consolidation materials were poly vinyl acetate (PVAC) and beeswax.

KEYWORDS: Polychrome wooden coffin; Technical imaging; Pigments; SEM-EDX; XRD; FTIR

1. INTRODUCTION

The polychrome wooden coffins were one of the most important archeological materials as they contained colors, scenes, texts, and ancient writings, revealing important historical information, facts and secrets(Salem et al., 2016). The identification of the components of ancient Egyptian polychrome coffins is very important to understand production methods (Cosentino, 2018), which reflect the state of art and the life in the age of the Pharaohs (Crupi et al., 2016), as well as it is required during proposal for future conservation works (Zidan et al., 2018). These coffins have received much investigation in the last years by many authors using several imaging and analytical techniques to reveal their construction techniques and painting materials, as well as the previous conservation materials (Afifi et al., 2019; Afifi et al., 2018; Moustafa et al., 2017; Amin., 2018).

The studied coffin dates back to late period (26^{th} dynasty) in ancient Egypt, and was found at archaeological area of Saqqara under registration number 18610. Its dimensions is (218) cm long, (67.7) cm wide and (51.7) cm high. It consists of body and lid, which formed by a hollowed-out tree trunk as one piece. There are numerous longitudinal cracks, some of which penetrated through the depth of the wood and caused warping. In addition, there is a missing part in the foot area of the lid. The outer surface of the coffin covered with white preparation layer and decorated with painted layers (green, blue, yellow, red and black), while the inner surface covered with black resin layer. The painted layers of this coffin suffered from many deterioration aspects including powdering, flaking, cracking and missing parts, as well as it was covered with a thick layer of dust resulting from bad storage inside the store rooms of Saqqara archaeological area so, this study aims to identify the pigments, ground layer, and previous restoration materials used on the studied coffin using different analytical techniques in order to provide necessary information for suitable future conservation works.(Bader et al., 2013; Afifi et al., 2011; Roundhill, 2004; Bratitsi et al., 2019).

2. MATERIALS AND METHODS

Samples

The loose painted layers of the object detach and flake, and six fallen samples (1, 2, 3, 4, 5, 6) (Fig.1) representing the different layers were carefully chosen for the analyses. From the fallen samples, a sample from the previous consolidating material was carefully chosen from the preparation layer for analysis too.

Optical Microscopy (OM)

A stereo microscopy (Zeiss Stereo Discovery V 20) equipped with Axio Cam MRC5, was used to study the stratigraphic structure of the painted layer, as well as to view the details that cannot be seen with unaided eyes (Fatma, et al 2011; Ibrahim and Mohamed., 2019).

Technical photography (TP)

Technical images were acquired by using a modified digital camera Nikon D90 equipped with Nikon Nikkor 18-55mm f/3.5:5.6G AF lens (Table 1).

Table 1: Specifications of lights and filters which were used for technical photography.

Method	Light	Filter
Visible(Vis) photography(Cosentino,2015)	LED lamps	X-Nite CC1
UV-induced luminescence (UVL)	UV 365nm LED lamps	Kodak Wratten 2E + Schneider B + W 486 filter+ X-Nite CC1
Visible-induced infrared luminescence (VIL)	LED lamps	Schott RG840 cut-on filter
IR infrared(Cosentino,2014)	IR LED lamps	Schott RG840 cut-on filter
Infrared false- color images	Are made by digitally editing the VIS and IR images by Adobe Photoshop.	

Scanning Electron Microscope attached to Energy Dispersive X-Ray (SEM-EDX)

A Quanta 250 FEG with scanning electron microscope attached with EDX Unit (Energy Dispersive X-ray Analyses) was used to examine and identify the elemental composition of the painted layers. The accelerating voltage was 30 K.V and the magnification was 500X. (see Table 2).

X-ray diffraction analysis (XRD)

X-ray diffraction was performed on the fallen samples using X-ray diffraction model PW3040 /60– Analytical Equipment – PANalytical pro model with a Cu anode, working at 40mA/45kV. Nondestructive mode without any sample preparation. X'Pert High score data software was used to identify the components of the painted layers (Abdrabou et al., 2014).

Fourier transform infrared spectroscopy (FTIR)

A Bruker VEREX 70 FTIR equipped with the Universal ATR, 4 cm^{-1} resolution spectra in the 500–4000 cm^{-1} region, averaging 32 scans (Gorassiniet al., 2016), was used to identify the organic binding medium and the inorganic functional groups in the painted layer (Badr et al., 2018), as well as the previous consolidation materials (Zidan et al., 2016).

3. RESULTS AND DISCUSSION

SEM.EDX results of the painted layers (green, yellow, red and black) and ground layer were summarized in Table 2. All the EDX spectra acquired on the surface of the coffin, showed the peaks of oxygen and calcium related to the calcium carbonate in the ground layer. Magnesium, aluminum, silicon and potassium were also found due to the dust on the surface of the coffin (Bader et al., 2013). In several areas, we found chloride and sodium, which are indicative for the existence of halite that may be related to the burial soil.

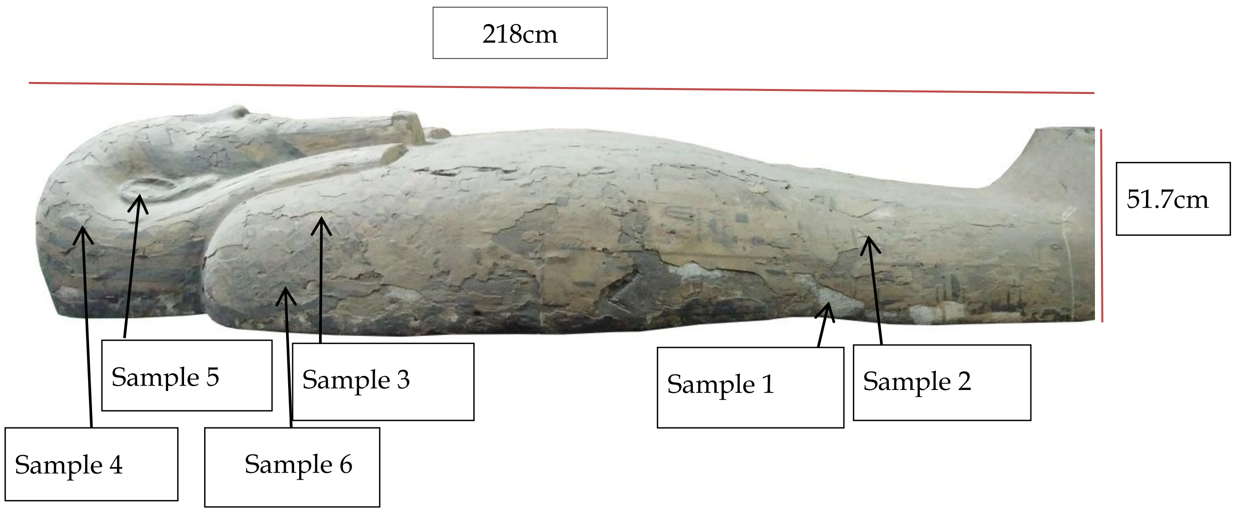

Figure1. A map of the sampling areas

Analysis of organic materials

Previous consolidation materials

OM of the ground layer and painted layers (Fig. 2a) showed that these layers are covered with shiny layer resulting from the previous consolidation materials.

UV-induced luminescence revealed the presence of a greenish emission from luminescent material on the surface. This luminescence presumably relates to natural resins or waxes used in the previous restoration interventions (Fig. 4b). FTIR spectrum of this material (Figure. 8a) showed the characteristic peaks of C-H stretching bands at 2918 cm^{-1}, C=O stretching band 1728 cm^{-1}, C-H binding bands at 1461cm^{-1} and C-H torsion bands at 719 cm^{-1}, which ascribed to beeswax. It was used as surface coating for the painted layers in the last decades (Nakhla, 1986; Johnson et al., 1995).

In the same spectrum C-O stretching band at 946 - 1230cm^{-1}, C-H bending bands 1372 and 1431 cm^{-1}, and C=O stretching band 1728 cm^{-1}, which ascribed to organic poly vinyl based material suggesting the use of poly vinyl acetate as it was extensively used as surface treatment for the painted layers in the last decades (Frances et al.; Rivers et al.,2003; Wei et al.,2012).

The bending medium

FTIR-KBR spectrum of the green painted layer (Figure. 8b) showed the characteristic peaks of C=O stretching of amide I band at 1629 cm^{-1}, N–H bending of amide II band at 1517 cm^{-1}, N-H stretching band at 3328 cm^{-1} and C-H binding band at, 1451cm^{-1}, which ascribed to proteinaceous based material such as animal glue (Derrick et al.,1999), which was commonly used as binding medium in ground layer from very early period in ancient Egypt(Abdrabou et al., 2018; Abdrabou et al., 2017) .

Analysis of in organic materials

Ground layer

SEM. EDX analysis of the ground layer (sample 1) (Fig. 6a) showed that the presence of carbon (C), (O) and Calcium (Ca). These elements suggest the use of calcium carbonate as a ground layer.

FTIR spectrum of the ground layer (Fig. 8c) showed the characteristic peaks of O-C-O binding band at 871 cm^{-1}, and CO_3 stretching band at 1391 cm-1, which ascribed to calcium carbonate confirming the attribution by SEM. EDX. Calcium carbonate was used for preparation layers.

Yellow painted layer

(OM) image (sample 2) (Fig. 2b) showed that the yellow painted layer was coated with a previous consolidation material. Thus, the grain of the yellow painted layer could not be seen. The thickness of the yellow painted layer (Fig. 3a) was 100µm. Yellow painted layer appeared dark under UVF (Fig.4b and 5b) which suggests the use of yellow ochre (Abdrabou et al.,2018; Cosentino et al., 2015)

SEM-EDX analysis (Fig. 6b) showed that the elements were calcium (Ca), iron (Fe), and silicon (Si) that proved the presence of yellow ochre pigment. The elements of sodium (Na) and chloride (Cl) indicated the presence of sodium chloride (NaCl). XRD data confirmed that the yellow painted layer (Fig. 7a) was made of yellow ochre in the form of Goethite (FeOOH) (Westlake et al., 2012).

Table 2: SEM. EDX results of the painted and ground layers

Sample number	Color	Elements	XRD components
1	**Ground Layer**	C, O, Ca.	-----
2	**Yellow**	O, Na, Mg, Al, Si, S, Cl, K, Ca, Ti, Fe.	FeOOH
3	**Red**	C, O, Mg, Al, Si, S, Cl, K, Ca, Fe,Hg.	HgS- Fe_2O_3
4	**Blue**	-----	$CaCuSi_4O_{10}$ $CaSO_4.2H_2O$
5	**Green**	C, O, ,Mg, Al, Si, S, Cl, K ,Ca, Fe, Cu.	$Cu_2Cl(OH)_3$ - $(Cu,Zn)_2(OH)_3Cl$
6	**Black**	C, O, Na, Mg, Al, Si, S, Cl, K, Ca, Fe.	-----

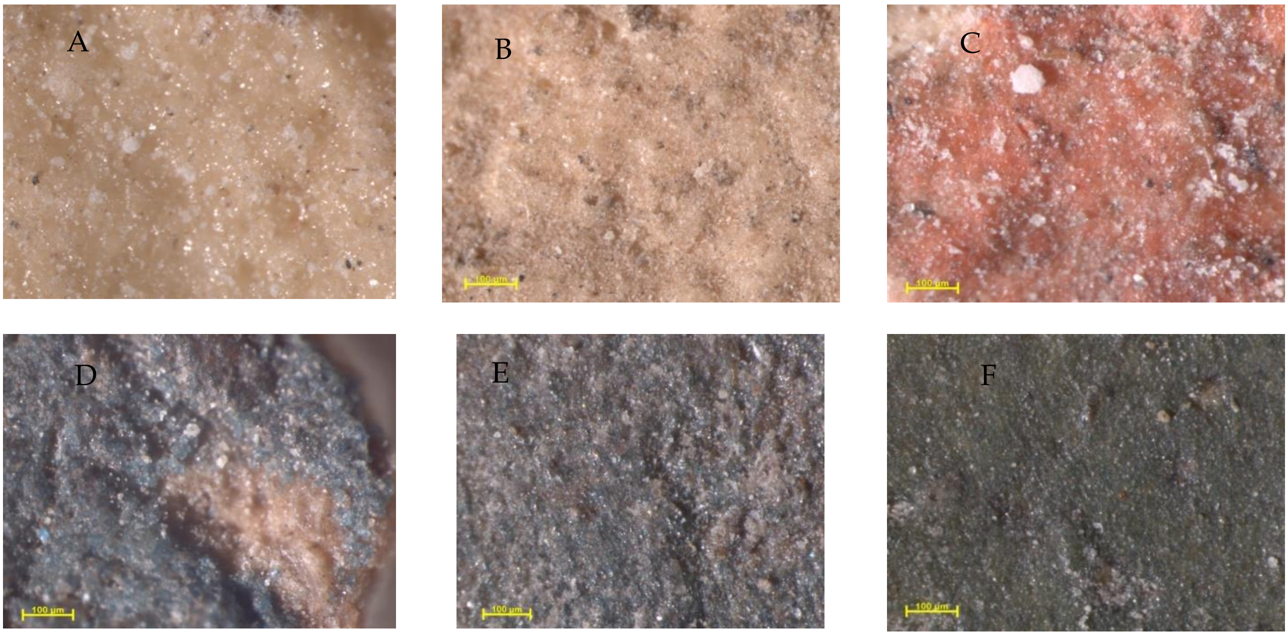

Figure 2. Optical photomicrographs showing the course morphology of the painted layers surface used in the coffin: A - Ground layer; B - Red pigment; C - Green pigment; D - Blue pigment; E - black pigment; F – Yellow pigment

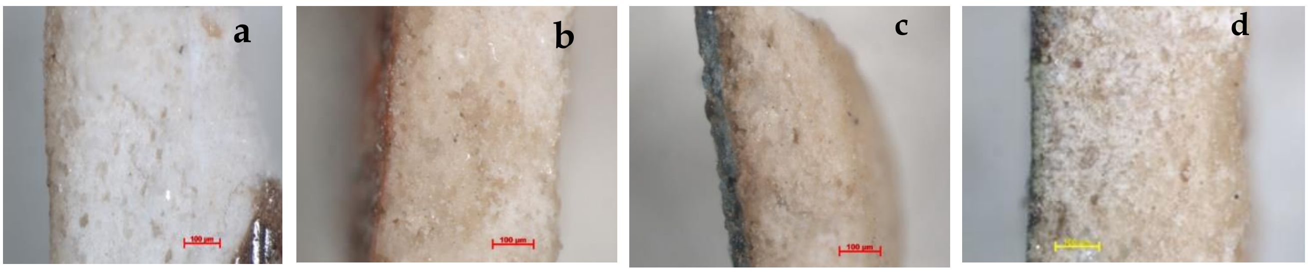

Figure 3. Stratigraphic structure of the painted ground layers by OM: a) yellow, b) red, c) green, d) blue pigment showing two layer (ground layer,thin paint layer)

Red painted layer

The majority of red pigments used in ancient Egypt were earthen based colors containing iron oxide (Hühnerfu et al., 2006; Green, 2001) until recently scholars believed that cinnabar was not used in Egypt before late Ptolemaic or Roman period. Nevertheless, many updated studies allowed moving back the early appearance of cinnabar in Egyptian art (Bracci et al., 2015; Hühnerfu et al., 2006). OM image (sample 3, Fig. 2c) showed that the red painted layer was coated with previous consolidation material and salt crystallization on the surface of the color. Stratigraphic structure of the red painted layer (Fig. 3b) showed that its thickness was 100μm. Areas of red painted layer appeared darker in the (UVF) image (Fig. 5b), which may suggest that the red painted layer is iron-based pigments. SEM. EDX analysis (Figure. 6c) showed that the elements were cinnabar (Hg) iron (Fe), sulphur (S), and silicon (Si), calcium (Ca), Oxygen (O), which refers to the use of red ocher and cinnabar. XRD data confirmed that the red painted layer made of red ochre in the form hematite (Fe_2O_3), as well as cinnabar (HgS) was also observed (Fig. 7b). This result shows that the red painted layer is composed of hematite and cinnabar to obtain a deep chromaticity (Prieto et al.,2016; Badr et al., 2018).

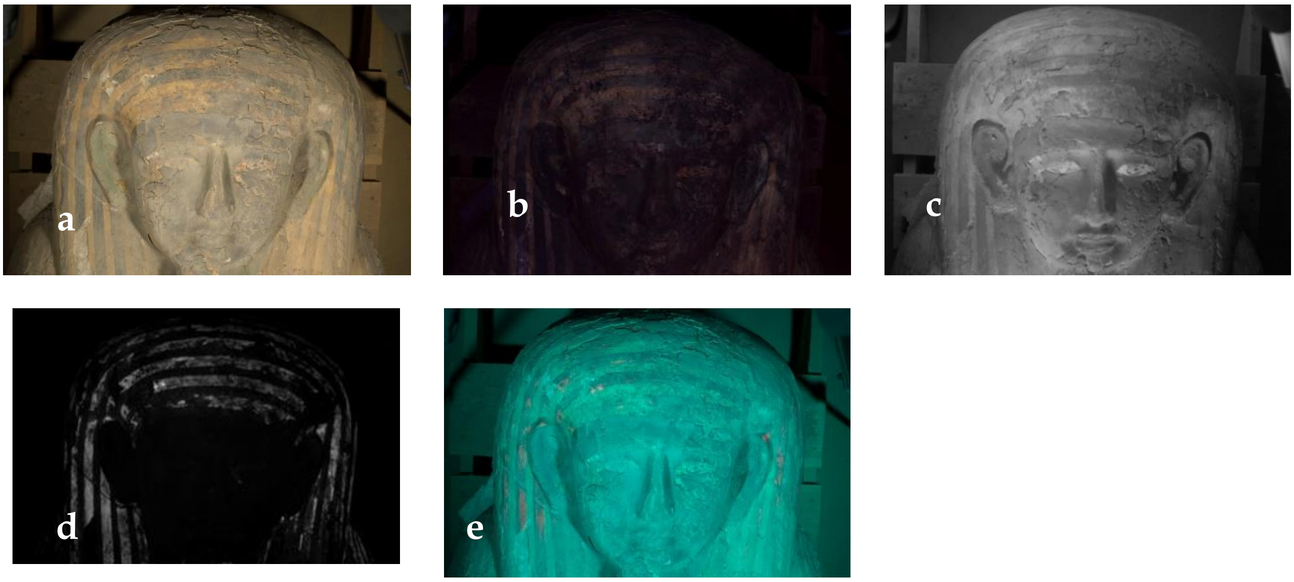

Figure 4. Technical images for the face area of the coffin made by different methods a) visible (VIS), b) Ultraviolet Fluorescence (UVF), c) infrared (IR), d) Infrared fluorescence (IRF), e) infrared false color (IRFC).

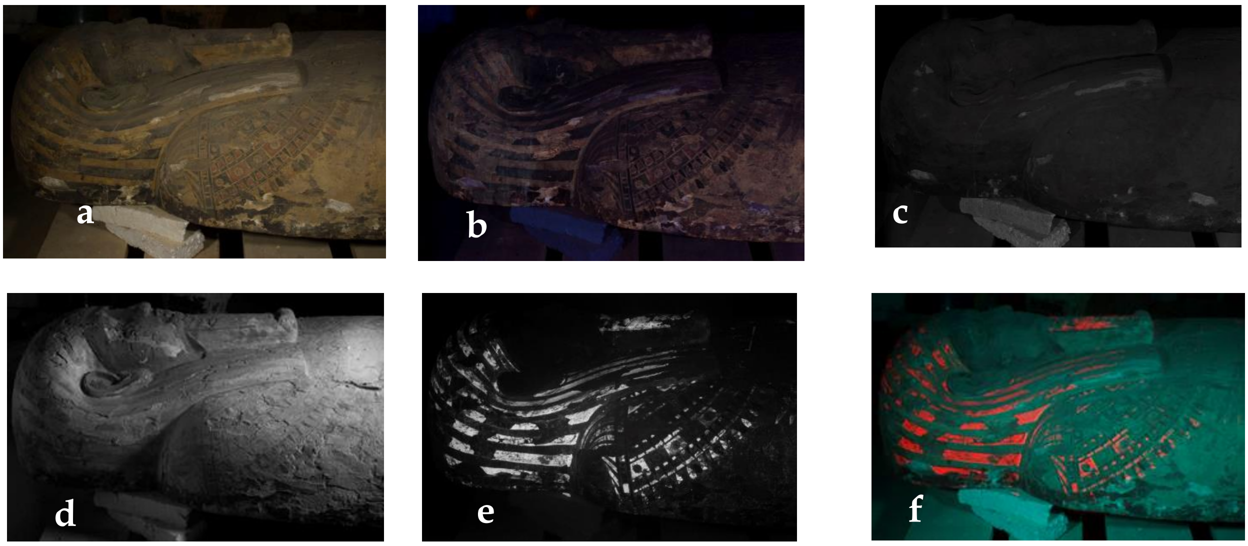

Figure 5. Technical images for the middle area of the coffin made by different methods: a) visible VIS, b) Ultraviolet Fluorescence (UVF), c) Ultraviolet Reflected (UVR), d) infrared (IR), e) Infrared fluorescence (IRF), f) infrared false color (IRFC).

Blue painted layer

OM images (sample 4) **, (**Fig. 2d) revealed that the samples were coated with a previous consolidation material. Therefore, the grain of the blue painted layer could not be seen. The thickness of the blue painted layer (Fig. 3c) was 100µm. The blue painted layer appeared white bright in the VIL image (Fig. 4d and 5e), while all other materials appeared dark, referring to the use of Egyptian blue (Verri et al., 2010), as well as the IRFC image (Fig. 5f) showed that the areas painted with the Egyptian blue appeared red (Abdallah et al.,2018).

XRD data the blue painted layer showed the presence of Gypsum ($CaSO_4.2H_2O$) and cuprorivaite ($CaCuSi_4O_{10}$)(Rosalie et al., 2011). (Figure. 7c), which is the main component of Egyptian blue, in accordance with the attribution made by VIL. The Egyptian blue – one of the earliest synthetic pigments with its bright blue inorganic compound was extensively used throughout the Mediterranean from the Fourth Dynasty in Egypt (c. 2500 B.C.) until the end of the Roman period in Europe (Verri et al.,2009; Accorsi et al., 2009).

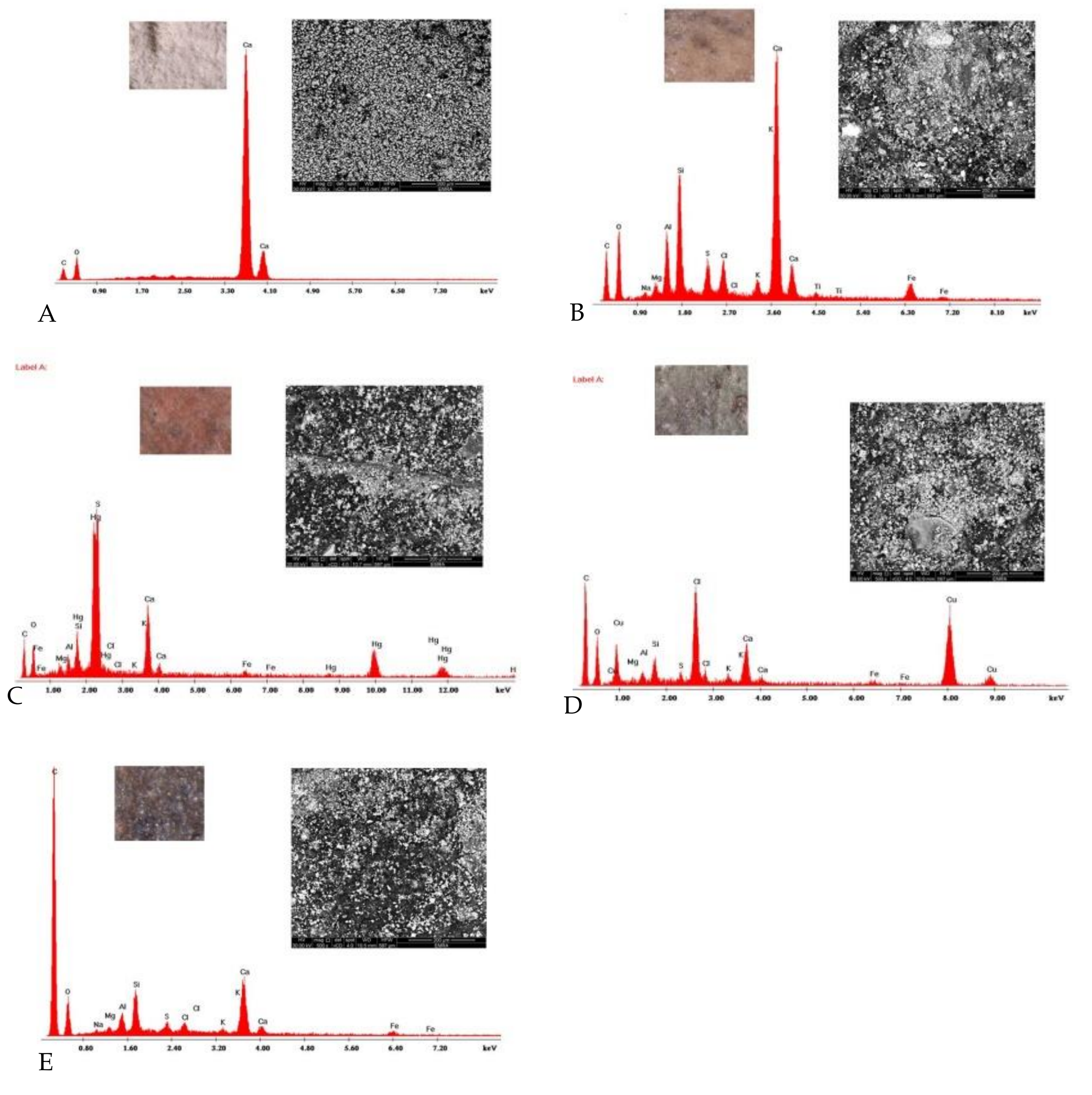

Figure 6. SEM-EDX spectrum of a) ground layer, b) yellow pigment, c) red pigment, d) green pigment, d) black pigment

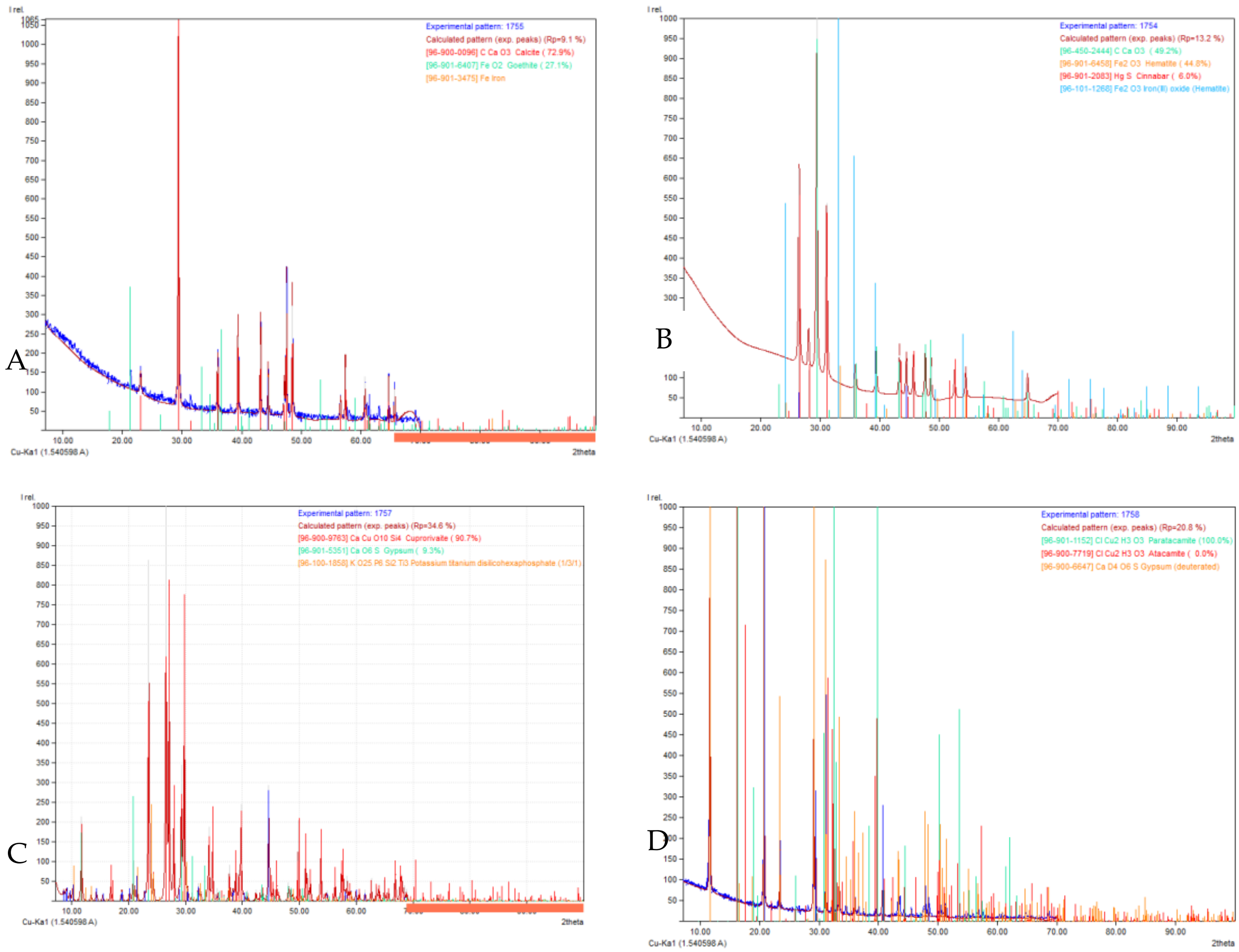

Figure 7. XRD spectrum of pigment; a) yellow ; b) red ; c) blue; d) green

Green painted layer

OM images (sample 5) , (Fig. 2e) seemed darker because of the previous consolidation material. Thus, the grain of green painted layer cannot be seen. The thickness of the green painted layer (Fig. 3d) was 100μm. VIL images (Fig. 5e) did not show any fluorescence, therefore the hypothesis of a mixture of Egyptian blue and yellow pigments can be discarded. EDX analysis (Fig. 6d) showed that the elements were copper (Cu), calcium (Ca), iron (Fe), silicon (Si), chloride (Cl), which suggest the use of copper based pigments. XRD data showed that the green painted layer contained atacamite ($Cu_2Cl(OH)_3$) and paratacamite ($(Cu,Zn)_2(OH)3Cl$) (Figure. 7d). Thus, we suggest that the main copper based pigment changed to atacamite and paratacamite due to moisture and salts (Schiegl et al., 1989; Lee et al.,2001; Brecoulaki et al., 2006; Scott et al., 2016).

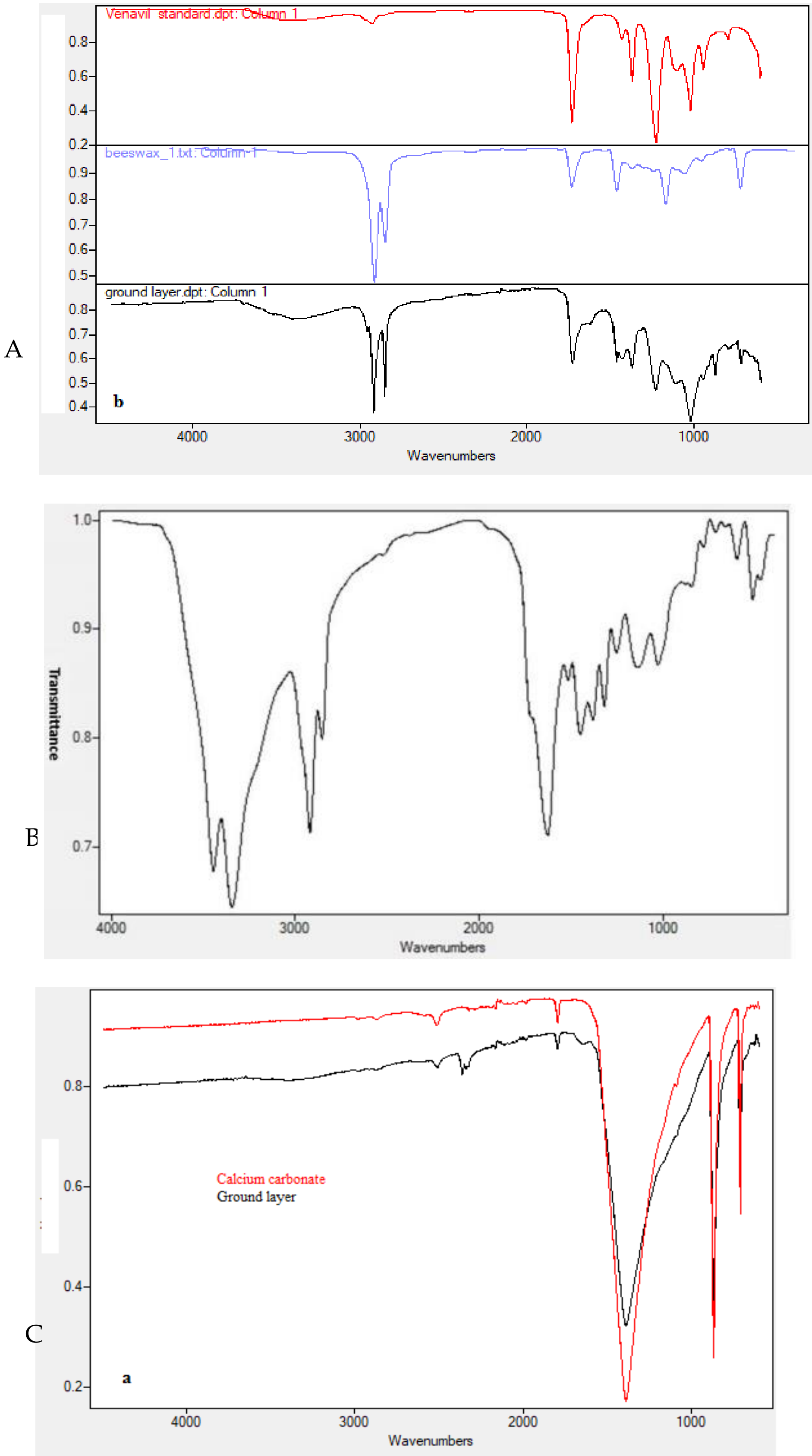

Figure. 8. Comparing FTIR spectra between references and samples, a) previous restoration ground layer, b) binding media, c) ground layer.

Black painted layer

OM images (sample 6) (Fig. 2f) revealed that the samples were coated with a previous consolidation material. Therefore, the grain of the black painted layer could not be seen. The black painted layer appears darker in the infrared image (Fig. 5d). This is particularly evident on the coffin, which may suggest that the black pigment is a carbon-based black (Abdrabou et al., 2017; Abdrabou et al., 2018). Moreover, a carbon-based black is supported by the infrared false color image (Fig. 5f). EDX analysis (Fig. 6e) showed that the main element is carbon (C) which confirms the use of carbon for the black painted layer (Afifi, 2011).

4. CONCLUSION

The study investigated polychrome wooden coffin at Saqqara using technical photography and SEM-EDX to identify the ground and painted layers. Additionally, complementary techniques such as XRD were used to confirm the painted layer. Our analysis using FTIR allowed us to characterize the materials added during the previous restorations, which were Poly vinyl acetate (PVAC) and beeswax. The binding media was animal glue. The optical microscope provided more details about the surface condition of the painted layers and a cross-section showed the stratigraphic structure of the painted layer. The pigments were hematite mixed with cinnabar for the red painted layer to obtain a deep chromaticity, the Egyptian blue for the blue painted layer, and the yellow ochre was goethite for the yellow painted layer. Furthermore, Atacamite and Paratacamite were used for the green painted layer. This was evidence for the copper pigment that turned into Atacamite and Paratacamite due to moisture and salts. Carbon was used for the black painted layer and calcium carbonate was used for the ground layer. The analyses provided more information concerning the original and previous treatment materials, which will help in choosing the most suitable materials in the future conservation works.

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