

## INVESTIGATION OF AN ANCIENT EGYPTIAN POLYCHROME WOODEN STATUETTE BY IMAGING AND SPECTROSCOPY

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### **Abstract**

*This paper describes the combined use of imaging and spectroscopic techniques performed on a polychrome wooden statuette of the god Ptah –Sokr- Osiris from 26th dynasty in ancient Egypt, with the aim of identifying the painted materials layered on wood surface and emphasizing the surface texture of the painted preparation layers. The identification of wood was also included in the study. The wood sample was identified by observing the thin sections with optical transmission light microscope; the painted materials layered on the wood surface were analyzed by both imaging and spectroscopic techniques including optical microscopy (OM), reflectance transformation imaging (RTI), technical photography [visible, visible-induced ultraviolet luminescence (UVL), ultraviolet reflectance (UVR), infrared (IR), visible-induced infrared luminescence (VIL), infrared false color (IRFC)], dino-lite USB microscope (UV/IR), handheld X-ray fluorescence spectroscopy (XRF), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The microscopic observation of wood thin sections allows identifying it as a native Egyptian Sycamore fig (*Ficus sycomorus*). The preparation layer was proved to be a mixture of calcite and quartz. The chromatic palette used in the statuette was identified as hematite, Egyptian blue, arsenic sulphides, possibly malachite and carbon from charred animal origin. The application of RTI provided new and valuable information about surface irregularities of the painted layers and marks relating to surface preparation that are imperceptible to the naked eye or through other inspection techniques. Dino-Lite USB digital microscope (UV/ IR) has proven to be a simple and a cost-effective tool for recognizing the spatial distribution of the single crystals of the pigments.*

**Keywords:** *Egyptian polychrome statuette; Reflectance transformation imaging; Technical photography; XRF; Wood identification.*

### **Introduction**

Ancient Egyptian polychrome wooden artifacts belonging to museum collections have gained much interest in the last decades. For their study invasive and non-invasive analytical techniques have been applied by many authors. For example, multidisciplinary approaches combining imaging and spectroscopic techniques were applied for the study of some ancient Egyptian polychrome artifacts [1-3]. The elemental analysis provided by portable X-ray fluorescence (XRF), allowed for the non-invasive identification of a number of pigments on some ancient Egyptian wooden artifacts [4]. A non destructive approach involving IR fluorescence, optical microscopy, environmental scanning electron microscopy, X-ray diffraction and Fourier transform infrared spectroscopy equipped with attenuated total reflectance unit (ATR), was applied with the aim of the identity of the original and added

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materials, to explain the deterioration processes and establish the state of conservation of Pharaoh Tutankhamun's Painted Boat Model [5]. Further, scientific analytical techniques involving optical microscopy, scanning electron microscopy, X-ray fluorescence spectroscopy (XRF), X-ray diffraction and Fourier transform infrared spectroscopy were applied to different polychrome wooden artifacts in order to identify and characterize wood species, ground layer, paint layers, binding medium and previous restoration materials, to yield information useful for the conservation processes [6-10]. In addition, reflectance transformation imaging (RTI) was applied by some authors as an innovative instrument for the documentation and analysis of texture of cultural objects, which yielded new and valuable information in the identification of manufacturing techniques and assessments of surface condition that were not previously recognized through direct examination or any other photographic techniques [11-13].

In this study, we demonstrate the ability of combining imaging techniques and hand held XRF as a very efficient and non-destructive method for analyzing the painted layers. Complementary techniques such as X-ray diffraction and Fourier transform infrared spectroscopy were used in some cases to confirm the identity of certain pigments. The authors were much interested in the application of reflectance transformation imaging (RTI) to emphasize the surface texture of the painted preparation layers to provide a deeper understanding of manufacturing techniques and assess the surface condition. The identification of wood was also included in the study.

## Materials and methods

### *The Object*

Our study focuses on a polychrome wooden statuette of the god Path – Sokar - Osiris, which dates back to 26<sup>th</sup> dynasty (B.C) in an ancient Egypt. The god Path – Sokar - Osiris appeared at the end of the New Kingdom, and became a very common feature of burials through late period to the roman period [14]. It represents in a wooden mummiform figure standing on a rectangular base and wearing beard and tripartite wig surrounded by a crown of ram's horns with plumes and sun-disk. On the wooden base a cavity prepared to contain a small wooden falcon usually sits looking at the figure (in the studied object, the falcon was lost). In a number of instances, the statuette body or the base may contain a funerary papyrus (Often parts of the book of the dead) or sometimes contain part of a mummified corpse [15]. It is constructed from pieces of wood covered with painted preparation layers. The front surface of the statuette is decorated with broad collars, made of layers of a floral multicolored necklace. Under the necklace there are three columns of hieroglyphic inscriptions and four sons of god Horus on both right and left sides, two for each side, which end with goddesses Isis and Nephthys. The representation of god khpr is depicted on the foot area. The back surface of the statuette is decorated with four columns of hieroglyphic inscriptions and four gods. The base is decorated with the common hieroglyphic motif of the sign of life (ankh), flanked by the sign for dominion, was, all on the basket, neb, is lightly painted and repeated around the four sides of the base. In 2014, the statuette was transported from Egyptian museum to the Wood Conservation Laboratory of the Grand Egyptian Museum-Conservation Center (GEM-CC) under GEM number 5214. Its dimensions are about 75cm in height, 23cm in Width.

### *Methodology*

In this work, based on the data from the visual inspection under stereo microscope and technical photography, X-ray fluorescence spectrometry (XRF) analyses were performed directly on differently colored areas, after that four samples fallen off some painted preparation layers (preparation, red, blue and greenish blue) were analyzed using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). It should be noted that the XRF measurements were also performed on the fallen samples. For wood identification very small sample was taken from hidden area of the base. It was cut into the three principal anatomical directions: transverse, tangential longitudinal and radial longitudinal. These three thin sections

were mounted on glasses to be observed under transmitted light using Optika microscopy (Italy) equipped with an Optika B9 digital camera. The observed anatomical features were then compared to reference wood anatomy atlases and database [16-17].

#### *Optical Microscopy (OM)*

For preliminary morphological observations of the painted preparation layers, we used a Zeiss Stereo DV 20 (portable stereomicroscope) equipped with an Axio Cam MRC5 with an optical zoom of 28 up to 560 $\times$ . The images were taken at 28 $\times$  covering an area of 18x16 mm.

#### *Reflectance transformation imaging (RTI)*

The object is photographed along with two shiny black spheres and illuminated by a light source (a flash) manually held in position using a digital camera Nikon D 810 DSLR (CMOS sensor) fitted with a Nikon Nikkor 60mm f/1:2.8D AF lens. The camera was operated in fully manual mode and was tethered to a computer using live view mode. For each photograph, the light is moved to a different position and angle, always maintaining the same distance from the subject, thus creating an imaginary dome around it. Through a set of mathematical calculations made by RTI Builder, the surface normals are identified and consequently, the object's topography and reflective properties.

#### *Technical photography (TP)*

For acquiring the technical images (visible (VIS), visible-induced ultraviolet luminescence (UVL), ultraviolet reflected (UVR), visible-induced infrared luminescence (VIL) and infrared (IR)) presented in this study, a modified digital camera Nikon D90 was used in different spectral ranges (from 360 to about 1100nm). The camera was equipped with a Nikon Nikkor 18-55mm f/3.5:5.6G AF lens. The X-rite Color Checker Passport was used as a reference alongside the object. Visible (Vis) images were acquired by using two sources of two fluorescent lamps and a Schneider filter B + W digital UV/IR blocking 486 filtering system in front of the camera lens. Two UV 365nm LED lamps were used for ultraviolet (UV). During the acquisition of the ultraviolet-induced visible luminescence images, a combination of Kodak Wratten 2E and Schneider B + W digital UV/IR blocking 486 filters was used on the lens to cut both ultraviolet reflected and the possible infrared stray radiation generated by the lamps. This technique is particularly useful in locating the presence of organic materials and colorants (lake pigments, ancient binders or varnishes) as well as many modern retouching or coating materials [18, 19]. Moreover, some inorganic materials also show luminescence properties that can give an indication of the identity of the pigments [20, 21]. While a Schneider filter B + W digital UV band pass 403 filtering system (c.365–400nm) was placed in front of the camera lens for the acquisition of ultraviolet reflected images (UVR) in order to block the visible component and investigate ultraviolet range between 365 and 400nm.

In the case of visible-induced infrared luminescence imaging (VIL), the statuette was illuminated by two LED lamps and a Schott RG840 cut-on filter was placed in front of the lens, to block all stray radiations from visible spectrum and investigate only the emission of IR radiation in the 850–1000nm region. This technique was used to reveal the presence of Egyptian blue pigment that shows photo-induced luminescence properties in the infrared range [22-25]. In the VIL images, Egyptian blue pigment shows up as bright white areas against a dark background.

Infrared imaging was performed with two LED IR lamps and a Schott RG840 cut-on filter was placed in front of the lens to block the visible component and investigate the range between 850 and 1000nm. This technique is useful in revealing the presence of any preparatory drawings or carbon-based pigment [26]. Infrared false color images are made by digitally editing the VIS and IR images by adobe Photoshop.

#### *Dino-Lite USB digital microscope (UV/IR)*

Dino-Lite USB digital microscope (with UV-IR light) equipped with 1.3 MP digital camera with an optical zoom of 20 $\times$  up to 250 $\times$  was used to detailed technical images. In addition, a Dino-Lite digital microscope (IR/UV) was developed to obtain imaging techniques by VIL and UVL. For VIL imaging, it was fitted with a Fuji IR 840 cut-on filter and two

external LED light lamps [13]. For UVL imaging, it was fitted with a UV/IR Baader filter and two external small LED UV lamps.

#### *X-ray fluorescence (XRF)*

The measurements were performed with a Niton™ XL3t GOLDD hand held XRF spectrophotometer instrument using the NITON XL3t X-ray tube based analyzer with Ag anode, 50kV and 0-200µA max. The instrument head was placed in contact with the selected area and the irradiated area was about 3mm radius. All points were exposed for a minimum of 50 seconds using mining mode. XRF spectra were produced using™Niton Data Transfer (NDT) software.

#### *X-ray diffraction (XRD)*

The fallen samples were analyzed in a nondestructive mode without any sample preparation by X-ray diffraction using X-ray Diffractometer System PW3040 – Analytical Equipment – PANalytical pro model with a Cu anode, working at 30mA/40kV (An approximately flat surfaced sample was attached into the sample holder inside the XRD apparatus). X'Pert High score data acquisition and interpretation software for determining the components of the painted layers was used.

#### *Fourier transformed infrared spectroscopy (FTIR)*

Fourier transform infrared spectroscopy was done using FTIR spectrometer (IRPrestige-21, Shimadzu) in the 400 – 4000cm<sup>-1</sup>, range with resolution of 8cm<sup>-1</sup>. Inorganic materials were identified by comparing the obtained spectra with literature data [27] and standards created in FTIR laboratory.

## **Results and Discussion**

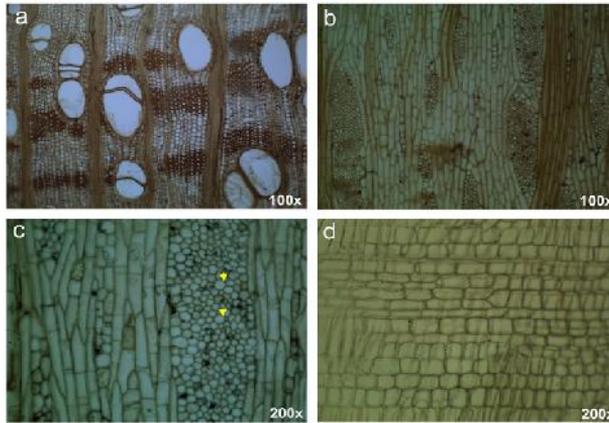
Corroborating the analytical techniques of imaging and spectroscopy we discuss separately the wood support, the preparatory layer and respectively the painted layers with all identified pigments.

### ***Wood support***

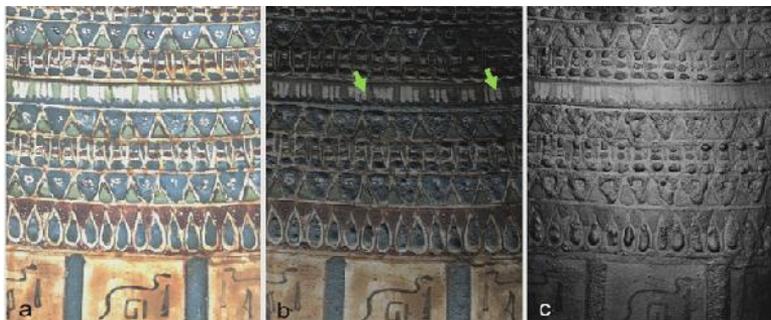
The microphotographs of wood thin sections showed that the wood sample obtained from the wooden base is sycamore fig (*Ficus sycomorus*). Its transversal section is characterized by the presence of diffuse porosity with vessels solitary and in radial multiples of 2 to 4 and axial parenchyma vasicentric and in bands more than three cells wide as observed in Figure 1a. The tangential longitudinal section (TLS) is characterized by the presence of simple perforation plates and two distinct sizes of rays (1-4 seriate up to 10 high and larger rays commonly 5 to 12 seriate up to 20) (Fig. 1b). Some laticifers are observed in rays (arrow head) as shown in Figure 1c. Heterocellular rays with square and upright cells only on marginal rows and strongly procumbent central cells were observed in the radial longitudinal section (RLS) (Fig. 1d). This kind of wood has been used extensively for manufacturing different kinds of wooden artifacts in ancient Egypt since at least the fifth dynasty till the Graeco - Roman period [28 - 29]. Sycamore fig wood is light, not of high quality and is prone to insect attack; however preparation layers and pigments that cover the wood objects reduce these drawbacks [30].

### ***The preparatory layer***

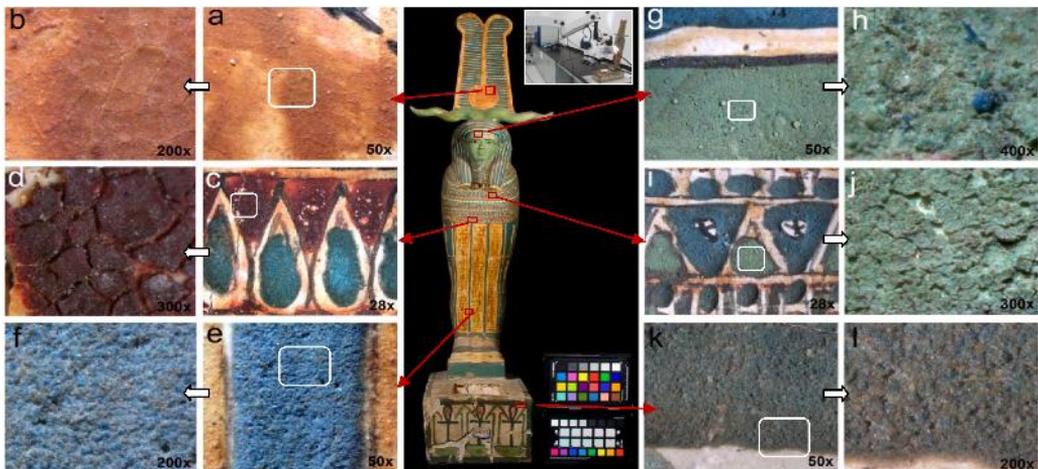
In the RTI images (Fig. 2a and b), comparing the default and the diffuse gain images allowed uncovering clarified marks relating to surface preparation and tool slips. The XRF spectrum of the preparation layer showed the presence of Calcium (Ca) as the main element with small amount of silicon (Si), and traces of sulphur (S), potassium (K), iron (Fe), arsenic (As), titanium (Ti) as impurities. This result suggests that the preparation layer is a mixture of calcium carbonate (CaCO<sub>3</sub>) and quartz (SiO<sub>2</sub>). The XRD analysis (Fig. 9a) confirmed that the preparation layer is composed of calcite and quartz. The FTIR spectrum (Fig. 10a) was also consistent with the presence of calcium carbonate (bands at 1440cm<sup>-1</sup>, 875cm<sup>-1</sup> and 711cm<sup>-1</sup>) and quartz (bands at 1087 cm<sup>-1</sup>, 1053 cm<sup>-1</sup> and 779 cm<sup>-1</sup>) [1, 2, 31]. This composition agrees with the composition of preparation layers previously determined by some authors [1, 6, 32].



**Fig. 1.** Microphotographs of wood sections under the microscope in transmitted light showing the anatomical characteristics of *Ficus sycomorus*: a- Transverse section (TS); b- Tangential section (TLS); c- Details of tangential section (TLS); d- Radial section (RLS).



**Fig. 2.** Detail images visualized in RTI with flat light (left), diffuse gain (middle) and specular enhancement (right).



**Fig. 3.** Detailed images for different areas of the painted layers under stereo microscope: a and b - Yellow painted layer; c and d - Red painted layers; e and f - Blue painted layer; g and h - Green painted layer (face); j and i - Green painted layer (necklace); k and l - Dark green painted layer (base).

### *The painted layers*

The chromatic palette includes yellow, red, green, blue and black. Table 1 summarizes the XRF analysis results for the different painted preparation layers that shown in Figure 7 a.

All the XRF spectra, acquired on the surface of the statuette, showed the peaks of calcium and silicon, related to the calcite and quartz in the preparation layer.

**Table 1.** The results of X-ray fluorescence (XRF) for the painted preparation layers

Spot	Color	Elements
1	Preparation layer	<b>Ca, Si</b> , S, K, As, Fe, Sr, Cl, Cu
2	Preparation layer	<b>Ca, Si</b> , S, K, <b>As</b> , Fe, Cl, Sr, Cu
3	Yellow	<b>Ca, S, As, Si</b> , Fe, K, Cl, Ti, Sr
4	Red	<b>Ca, Si, S, Fe, Cu, K</b> , As, Cl, Ti, Sr
5	Red	<b>Ca, Fe, Si</b> , S, K, As, Ti, Sr, Cl, Cu
6	Green	<b>Ca, Cu, Si</b> , S, K, Cl, Fe, As
7	Green	<b>Ca, Cu</b> , Si, S, K, Fe, As, Ti, Cl, Sr
8	Green	<b>Ca, Cu</b> , Si, K, S, Fe, Ti, Cl, Sr, As
9	Green	<b>Ca, Si, Cu</b> , S, K, As, Fe, Cl, Sr,
10	Blue	<b>Ca, Si, S, Cu</b> , Fe, As, Cl, K, Ti, Sr, Sn, Pb
11	Blue	<b>Si, Ca, Cu, S</b> , Fe, K, Cl, As, Ti, Sn, Sr, Pb
12	Dark green	<b>Si, Ca, Cu, Cl, Fe</b> , S, , As, K, Sn, Sr, Pb
13	Black	<b>Ca, Si, S, Cu, K, p</b> , Fe, Cl, As, Ti, Sr

Bold element indicates correlation with the main pigment mineral

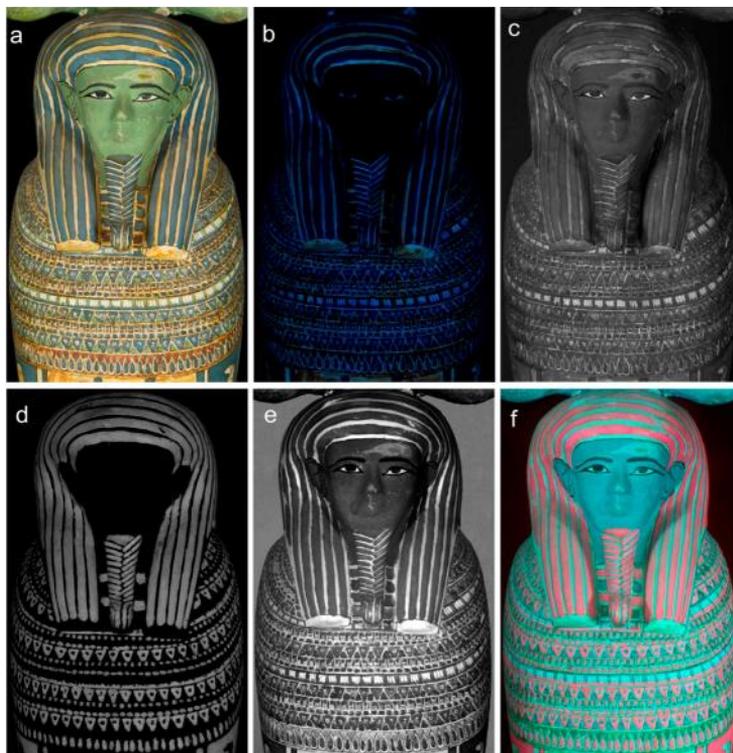
### *Yellow painted layer*

In the RTI, Diffuse gain image (Fig. 2b) shows the grainy surface of the yellow painted layer and shows also inhomogeneous in thickness, most probably due to inadequate preparation of the paint layer. The optical examination of the yellow pictorial layer (Fig. 3a and b) shows fading of the color in some places with micro cracks distributed on the entire pigment surface. The ultraviolet-induced luminescence (UVL) images (Fig. 4b and 5b) and ultraviolet reflected (UVR) images (Fig. 4c and 5c) showed that no evidence for the presence of varnishes or organic colorants, while fluorescence of yellow areas under UV-induced luminescence (UVL) suggests that a yellow painted layer probably made of arsenic based pigments since it gave fluorescent properties. The result of XRF analysis (Fig. 8b) showed presence of calcium (Ca), arsenic (As), sulphur (S) and silicon (Si) with traces of iron (Fe) and potassium (K). This result provides evidence for the presence of arsenic sulphides based pigment that is possibly orpiment  $As_2S_3$ . Orpiment is one of the most known arsenic sulphides pigments and early known uses as a pigment occur in middle kingdom in ancient Egypt as painted decoration on wooden artefacts [1, 7, 31].

### *Red painted layer*

RTI images of the red paint layer (Fig. 2b and c) show rough morphology of surface and homogeneous distribution of the pigment with many deformation and losses of pictorial layers. The microscopic examination (Fig. 3 c and d) offered a clearer and more detailed view of the red painted layers showing the paint layered separated into many pieces distributed on the entire pigment surface with exfoliated colored layer in flakes shapes and dropping off in small particles. Areas of red pigment appeared darker in the UV-induced luminescence (UVL) image (Fig. 4b and 5b). This is particularly evident on the collar of the statuette, which may suggest that the red pigment is red ochre (colored by hematite,  $Fe_2O_3$ ), consistent with the strong quenching properties of iron-based pigments. The results of XRF analysis for some points on the red painted layer (Fig. 8c) showed presence of calcium (Ca), iron (Fe) and silicon (Si) with a high intensity, in addition to a small amount of sulphur (S), copper (Cu) and arsenic (As). This result provides strong evidence for the presence of an iron-based pigment that is most likely hematite  $Fe_2O_3$ . It is interesting to note that copper, arsenic and sulfur are detected in the red areas, clearly related to the adjacent yellow and blue layers as the irradiated area is about 3 mm radius; that is, larger than the red decoration patterns that we are investigating. On the basis of the characteristic diffraction peaks recorded in XRD results for a sample fallen off the red painted layer (Fig. 9b), the presence of hematite was confirmed. In addition, calcite and quartz were also detected. The majority of red pigments used in ancient Egypt were earthen based

colors containing iron oxide, Especially, the mineral hematite ( $\alpha\text{Fe}_2\text{O}_3$ ) was very common [33], and has been reported by many authors [1, 9, 34].

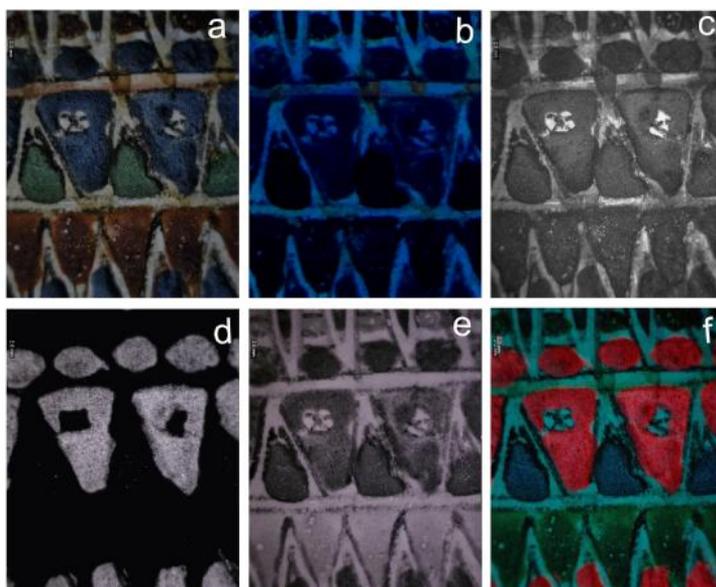


**Fig. 4.** Technical images of the painted layers made by different spectral ranges using modified Nikon digital camera: a- visible (VIS); b- UV-induced luminescence (UVL); c- UV Reflected (UVR); d- Visible- induced luminescence (VIL); e- Infrared (IR); f- Infrared false color (IRFC).

### ***Blue painted layer***

RTI images (Fig. 2b and c) reveal the grainy surface of the blue painted layer. Its grains were not evenly covered or flow off the brush showing the difference in the thickness from part to another. In addition, the application of the blue pigment may have been dabbed into place for small areas rather than brushed on in the normal way. The microscopic examination of the blue painted layer (Fig. 3e and f) shows coarse morphology of the surface and homogeneous distribution of the grains. In the visible-induced infrared luminescence image (Fig. 4d and 5d), the blue color appeared as bright white. The luminescence of such areas could indicate the presence of Egyptian blue. In addition, infrared false color image (Fig. 4f and 5f) indicated that the areas painted with blue pigment appeared red, which confirm the presence of Egyptian blue. According to XRF spectrum of the blue painted layer (Fig. 8d), the elements that presented the highest concentrations were silicon (Si), copper (Cu), and Calcium (Ca). This result provides evidence for the presence of copper -based pigment that is most likely Egyptian blue according to the data acquired by VIL. The presence of impurities of tin (Sn) and lead (Pb) in the XRF analysis (table 1) allowed us to assume that a leaded bronze scrap was used to produce the Egyptian blue pigment [35]. XRD analysis of a sample fallen off the blue painted layer (Fig. 9c) showed the presence of calcite ( $\text{CaCO}_3$ ), quartz ( $\text{SiO}_2$ ) and cuprorivaite ( $\text{CaCuSi}_4\text{O}_{10}$ ), which is the main component of Egyptian blue. In the FTIR spectrum of the same sample (Fig. 10b), we can detect, besides calcium carbonate (bands at  $1450$ ,  $870$  and  $712\text{cm}^{-1}$ ), Egyptian blue pigment (bands at  $1246$ ,  $1161$ ,  $1083$ ,  $1012$ ,  $665$  and  $482\text{cm}^{-1}$ ) [1, 2, 31, 36]. This pigment is so far the

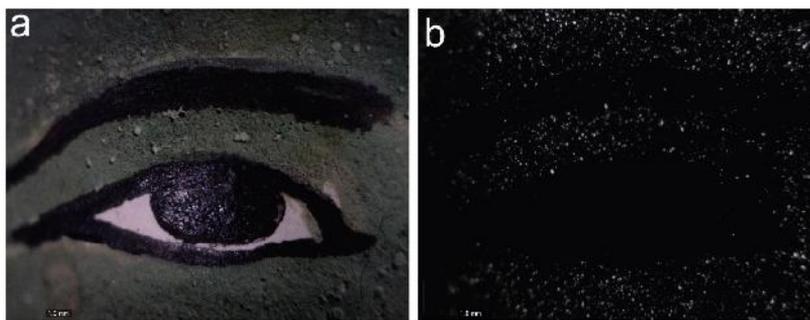
first synthetic pigment ever produced by man and was extensively used from the fourth Dynasty in Egypt until the end of the Roman period [5, 37, 38]. Its identification, characterization, production and degradation are discussed in previous works by many authors [38- 40].



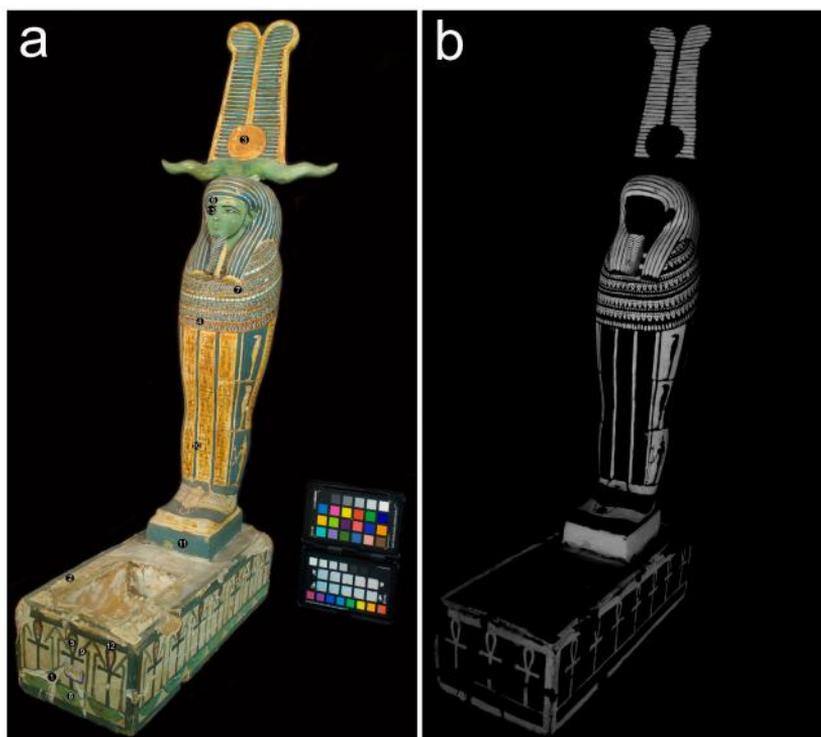
**Fig. 5.** Detailed technical images of the painted layers made by different spectral ranges using **USB microscope**: a- visible (VIS); b- UV-induced luminescence (UVL); c- UV Reflected (UVR); d- Visible- induced luminescence (VIL); e- Infrared (IR); f- Infrared false color (IRFC).

### ***Green painted layer***

RTI images of the green painted layer (Fig. 2b and c) show rough and grainy morphology of surface and in homogeneous distribution of the pigment with many deformation of pictorial layers. The microscopic examination shows the presence of some single particles of the blue pigment in the green paint layer in the face area (Fig. 3e and f) which could be accidental impurities due to the use of a dirty brush. In addition, some places of the green painted layer separated into many pieces with exfoliated colored layer in flakes shapes and dropping off in small particles as observed in Figure 3 g and h. The green painted areas didn't show any UV fluorescence (Fig. 4b and 5b). This could be an indication of the presence of copper- based pigments, since they are known to quench fluorescence of surrounding media. Copper- based pigments are supported by the infrared image (Fig. 4e and 5e); where the green painted layers appear dark due to the high absorption properties of these pigments in the near-infrared. VIL images (Fig. 4d and 5d) didn't show any luminescence of the green painted areas except for single particles of the Egyptian blue pigment, which clearly visible as a bright white by USB microscope (Fig. 6b), therefore the hypothesis of a mixture of Egyptian blue and yellow pigments can be discarded. Infrared false color images (Fig. 4f and 5f) show that the painted green areas appear with a lighter blue hue, which suggests the use of malachite [41]. From XRF analysis (Fig. 8e), the elements that presented the highest concentrations for some spots on the green painted layer were calcium (Ca) and copper (Cu). This result provides the possibility for the presence of malachite  $\text{CuCO}_3\text{Cu}(\text{OH})_2$ . This hypothesis could not be supported by laboratory analyses -for example, XRD - as no sample was available. However, the use of malachite has been reported in some works [42, 43].



**Fig. 6.** Detailed images by USB microscope: a- visible (VIS); b- Visible- induced luminescence (VIL)



**Fig. 7.** The difference between VIS and VIL images for the statuette: a- Visible (VIS) image; b- Visible-induced luminescence image in the infrared range (800–1000 nm) showing the presence of Egyptian blue in the areas that appear bright white. Locations from which the spots listed in Table 1 were performed for analysis by XRF (indicated by English numbers)

In addition, dark green painted layer was observed on the base of the statuette. The microscopic examination of this green painted layer (Fig. 3k and l) shows coarse morphology of the surface and inhomogeneous composition as well as a mixture of yellow and blue grains was observed. Its luminescence in VIL image (Fig. 7b) evidences the use of Egyptian blue, probably mixed with a yellow pigment. The result of XRF analysis (Fig. 8f) shows the presence of silicon (Si), copper (Cu), and Calcium (Ca) as the main elements, with significant amount of iron (Fe) and chlorine (Cl). This result provides evidence for the presence of Egyptian blue and yellow ochre pigment. The presence of chlorine (Cl) could probably point to the presence of halite

which may be related to the burial soil that surrounded the object. XRD analysis for a sample fallen off the dark green painted layer (Fig. 9d) showed the presence of calcite, cuprorivaite, quartz and halite. The absence of yellow ochre in the diffractogram could be due to its lower percentage; consequently the XRD technique is not able to detect it. Mixtures of Egyptian blue and yellow ochre to create a green color have been reported in some works [1, 2, 44].

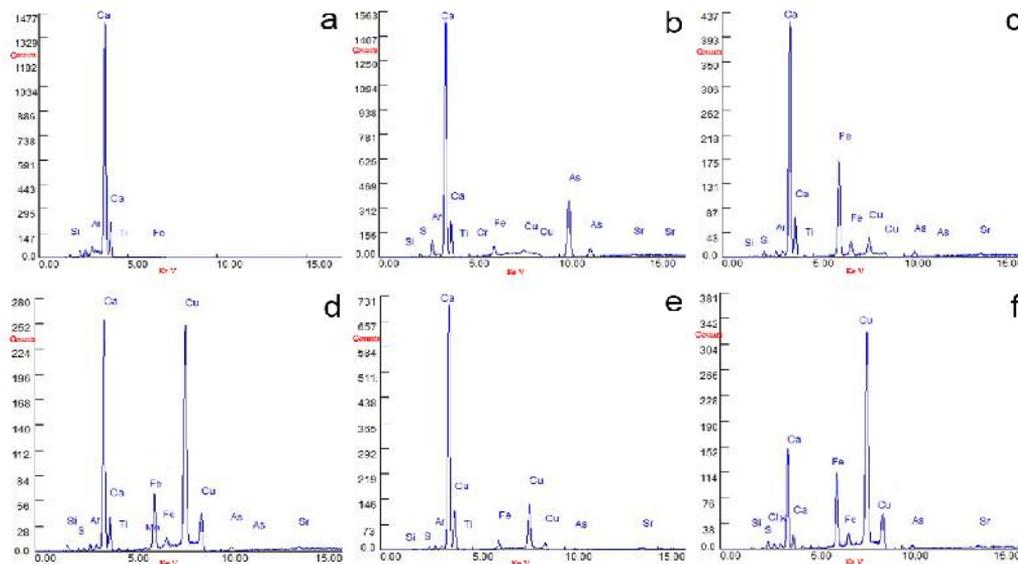


Fig. 8. XRF spectra of the preparation and painted layers used on the statuette: a- Preparation layer; b- Yellow painted layer; c- Red painted layer; d- Blue painted layer; e- Green painted layer; f- Dark green painted layer.

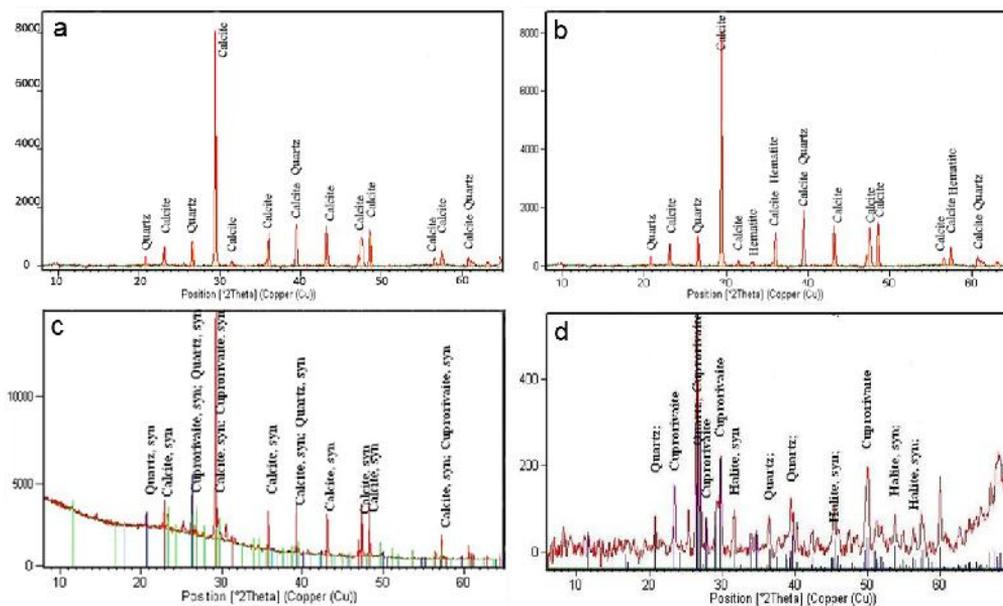


Fig. 9. XRD patterns for the fallen samples of the preparation and paint layers used on the statuette: a- Preparation layer; b- Red painted layer; c- Blue painted layer; d- Dark green painted layer.

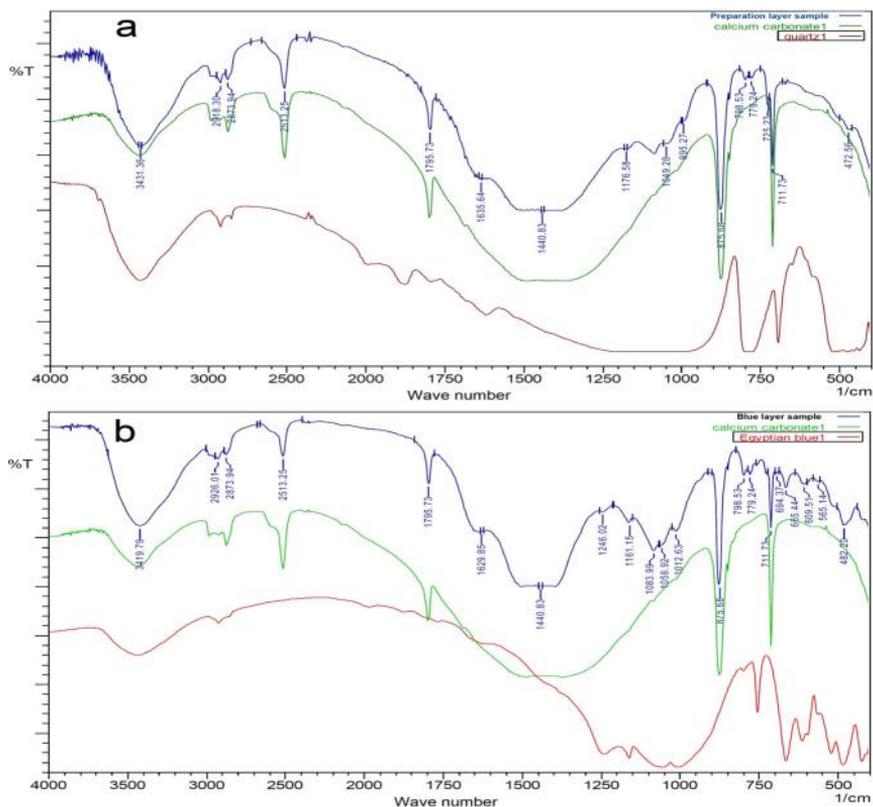


Fig. 10. FTIR spectra for the fallen samples of: a- Preparation layer; b- Blue painted layer.

### ***Black painted layer***

Areas of black pigments appear darker in the UVL image (Fig. 4b). This is particularly evident on the collar of the statuette, which may suggest that the black pigment is a carbon-based black, consistent with the strong absorption properties of carbon [1]. In the IR image (Fig. 5e), hidden black outlines below the over- red painted layers appeared, while it was difficult to see the black outlines under the blue and green painted layer. This is due to carbon black or copper-bearing pigments such as Egyptian blue are opaque under infrared emission and appear dark. Moreover, a carbon-based black is supported by the infrared false color image (Fig. 5f), where the black lines appeared black. The microscopic investigation of the black pigment indicated the fineness and evenness of particles and didn't show any fibrous structure, so it is possible to exclude burnt vegetable origin of the black pigment. In XRF analysis, phosphorus was detected. This result provides evidence for the presence of carbon obtained from the animal origin that is most likely bone black, which is one of the oldest pigments known to humans, and was originally made by charring animal bones [45].

## Conclusion

The paper presents the investigation of a polychrome wooden statuette of the god Ptah – Sokr- Osiris (26th dynasty) by the combined use of technical photography and handheld XRF spectroscopy as a non-invasive technique to map and identify the painted preparation layers, as well as complementary techniques such as XRD and FTIR spectroscopy were used in some cases to confirm the identity of certain pigments, while reflectance transformation imaging (RTI) and optical microscopy were applied to emphasize the surface texture of the painted preparation layers. In addition, the identification of wood was included in the study. The microscopic observation of wood thin sections allows identifying it as a native Egyptian sycamore fig (*Ficus sycomorus*), which was extensively used in ancient Egypt. The application of RTI provided new and valuable information about the surface condition of the painted layers showing the grainy surface of the pigments and the presence of many deformations and provided also more details about the manufacturing techniques (for example, marks relating to surface preparation that was imperceptible to the naked eye or by other inspection techniques). The stereo microscope provided more details about the surface condition of the painted layers showing some painted layers had lost their cohesion and separated into many pieces with exfoliated colored layer in flakes shapes and dropping off in small particles especially the red and green pigments, which require consolidation. TP showed that no evidence for the use of varnishes or any materials related to previous conservation interventions and provided useful information about the spatial distribution of the pigments, in particularly VIL, recognized the spatial distribution of areas containing Egyptian blue, even in mixed with other pigments, such as yellow. TP obtained by Dino-Lite USB digital microscope (UV/ IR) provided more details about the surface of the painted layers and determined the spatial distribution of the pigments, in particularly VIL, which played an important role in recognizing the spatial distribution of single crystals of Egyptian blue, to be clearly visible as a bright white in a very easy and convenient way. The chromatic palette used in the statuette was identified as hematite for the red painted layer, Egyptian blue for the blue painted layer, possibly orpiment for the yellow painted layer, Malachite for the green painted layer and carbon from an animal origin for the black painted layer, while a mixture of Egyptian blue and possibly yellow ochre was used for the dark green painted layer on the base of the statuette. The application of a protocol based on imaging techniques combined with data obtained from single-spot techniques such as X-ray fluorescence spectroscopy (XRF) provided useful information about the painted preparation layers without sampling. However, complete characterization of some painted layers required the use of other techniques such as XRD and FTIR spectroscopy.

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## References

- [1] A. Abdrabou, M. Abdallah, H. M. Kamal, *Scientific investigation by technical photography, OM, ESEM, XRF, XRD and FTIR of an ancient Egyptian polychrome wooden coffin*, **Conservar Património**, **26**, 2017, pp. 51-63, <https://doi.org/10.14568/cp2017008>.
- [2] S. Bracci, O. Caruso, M. Galeotti, R. Iannaccone, D. Magrini, D. Picchi, D. Pinna, S. Porcinai, *Multidisciplinary approach for the study of an Egyptian coffin (late 22nd/ early 25th dynasty): Combining imaging and spectroscopic techniques*, **Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy** **145**, 2015, pp. 511-522, <https://doi.org/10.1016/j.saa.2015.02.052>
- [3] M. Ganio, J. Salvant, J. Williams, L. Lee, O. Cossairt, M. Walton, *Investigating the use of Egyptian blue in Roman Egyptian portraits and panels from Tebtunis, Egypt*, **Applied Physics A**, **121**(3), 2015, pp. 813-821. DOI 10.1007/s00339-015-9424-5
- [4] C. Calza, R.P. Freitas, A. Brancaglioni Jr, R.T. Lopes, *Analysis of artifacts from ancient Egypt using an EDXRF portable system*, **International Nuclear Atlantic Conference - INAC - Belo Horizonte**, MG, Brazil, 2011, pp. 978-85.
- [5] Y. Ismail, A. Abdrabou, M. Abdallah, *A Non-Destructive Analytical Study and the Conservation Processes of Pharaoh Tutankhamun'S Painted Boat Model*, **International Journal of Conservation Science**, **7**(1), 2016, pp.15-28.
- [6] A. Abdrabou, M. Abdallah, M. Abd El Kader, *Analytical study and conservation processes of a painted wooden Graeco - Roman Coffin*, **International Journal of Conservation Science**, **6**(4), 2015, pp. 573-586.
- [7] N.A.A. Bader, Walid K. Al-Gharib, *Assessment of deterioration and conservation of a polychrome wooden coffin, From al-Arish museum, Egypt*, **International Journal of Conservation Science**, **4**(4), 2013, pp. 397-412.
- [8] M. Abdallah, H.M. Kamal, A. Abdrabou, *Investigation, preservation and restoration processes of an ancient Egyptian wooden offering table*, **International Journal of Conservation Science**, **7**(4), 2016, pp.1047-1064.
- [9] M. Moustafa, M. Abd Allah, R. Magdy, A. Abdrabou, I. Shaheen, H. M. Kamal, *Analytical study and conservation processes of scribe box from old kingdom*, **Scientific Culture**, **3**(3), 2017, pp. 13-24, DOI: 10.5281/zenodo.840011
- [10] H. Afifi, M.A. Abd El Fatah, *Analytical Study of Ground Painting Layers and Conservation Processes of an Egyptian Painted Coffin*, **Journal of Life Sciences**, **5**, 2011, pp. 661-669.
- [11] M. Mudge, C. Schroer, G. Earl, K. Martinez, H. Pagi, C. Toler-Franklin, S. Rusinkiewicz, G. Palma, M. Wachowiak, M. Ashley, N. Matthews, T. Noble, M. Dellepiane, *Principles and Practices of Robust, Photography-based Digital Imaging Techniques for Museums*, **The 11<sup>th</sup> International Symposium on Virtual reality, Archaeology and Cultural Heritage VAST** (Editors: A. Artusi, M. Joly-Parvex, G. Lucet, A. Ribes and D. Pitzalis), The Eurographics Association, 2010, pp. 179-186.
- [12] S.N. Manrique Tamayo, J.V. Andres, J.O. Pons, *Applications of reflectance transformation imaging for documentation and surface analysis in conservation*, **International Journal of Conservation Science**, **4**(Special Issue), 2013, pp. 535-548.

- [13] A. Abdrabou, M. Abdallah, I.A. Shaheen, H.M. Kamal, *The application of multispectral imaging and reflectance transformation imaging to an ancient Egyptian polychrome wooden stele, wood, laque and furniture group*, **The 18<sup>th</sup> Triennial Conference of ICOM-CC**, Copenhagen, Denmark, on 4–8 September 2017.
- [14] G. First, *The Ptah – Sokar - Osiris statuettes in the Cracow collections*, **Studies in Ancient Art and Civilization** (Jagiellonian University Krakow), **13**, 2009, pp. 109-139.
- [15] M.J. Raven, *Papyrus - Sheaths and Ptah-Sokar-Osiris Statues*, **Oudheidkundige Mededelingen uit het Rijksmuseum van Oudheden**, **59-60**, 1979, pp. 251- 296.
- [16] E.A. Wheeler, P. Bass, P.E. Gasson, *List of microscopic features for hardwood identification*, **IAWA Bulletin**, **10(3)**, 1989, pp. 219–332.
- [17] A. Crivellaro, F.H. Schweingruber, **Atlas of Wood, Bark and Pith Anatomy of Eastern Mediterranean trees and Shrubs with Special Focus on Cyprus**, Springer-Verlag Berlin Heidelberg, 2013.
- [18] M. Simpson Grant, *The use of ultraviolet induced visible-fluorescence in the examination of museum objects, Part II*, **Conserve O Gram - National Park Service**, **1(9)**, 2000, pp. 1-3.
- [19] G. Verri, C. Clementi, D. Comelli, S. Cather, F. Pique, *Correction of Ultraviolet-Induced Fluorescence Spectra for the Examination of Polychromy*, **Applied Spectroscopy**, **62(12)**, 2008, pp. 1295-1302.
- [20] R. Garcia –Moreno, F. Hocquet, F. Mathis, E. Van Elslande, D. Strivay, P. Vandenaabeele, *Archaeometry research on wall painting in the tomb chapel of menna*, **The Tomb Chapel of Menna (TT69), The Art, Culture, and Science of Painting in an Egyptian Tomb** (Editor: M. Hartwig), An American Research Center in Egypt, 2013.
- [21] B.H. Stuart, **Analytical Techniques in Materials Conservation**, England, 2007, p.73.
- [22] G. Accorsi, G. Verri, M. Bolognesi, N. Armaroli, C. Clementi, C. Miliani, A. Romani, *The exceptional near-infrared luminescence properties of cuprorivaite (Egyptian blue)*, **Chemical Communications**, **23**, 2009, pp. 3392–3394.
- [23] G. Verri, T. Opper, T. Deviese, *The ‘Treu Head’: A case study in Roman sculptural polychromy*, **Technical Research Bulletin**, **4**, 2010, pp. 39-54.
- [24] G. Verri, D. Saunders, J. Ambers, T. Sweek, *Digital mapping of Egyptian blue: Conservation implications*, **Studies in Conservation**, **55(Sup. 2)**, 2010, pp. 220-224.
- [25] G. Verri, M. Gleba, J. Swaddling, T. Long, J. Ambers, T. Munden: *Etruscan women’s clothing and its decoration: the polychrome gypsum statue from the ‘Isis Tomb’ at Vulci*, **Technical Research Bulletin**, **8**, 2014, pp. 59-71.
- [26] E. Passmore, J. Ambers, C. Higgitt, C. Ward, B. Wills, S.J. Simpson, C. Cartwright, *Hidden, looted, saved: The scientific research and conservation of a group of Begram Ivories from the National Museum of Afghanistan*, **The British Museum Technical Research Bulletin** **6**, 2012, pp. 33-46.
- [27] R.M. Derriik, D. Stulik, M.J. Landy, **Infra Red Spectroscopy in Conservation Science**, The Getty Conservation Institute, Los Angeles, 1999.
- [28] R. Gale, P. Gasson, N. Hepper, *Wood, Ancient Egyptian Materials and Technology* (Editors: T. Nicholson and I. Shaw), Cambridge University Press, 2000, pp. 334-371.

- [29] C.R. Cartwright, L. Spaabæk, M. Svoboda, *Portrait mummies from Roman Egypt: ongoing collaborative research on wood identification*, **The British Museum-Technical Research Bulletin**, **5**, 2011, pp. 49-58.
- [30] J. Dawson, J. Marchant, E. Von Aderkas, C.R. Cartwright, R. Stacy, *Egyptian coffins: materials, construction and decoration*, **Death on the Nile (Uncovering the Afterlife of Ancient Egypt)**, forwarded by T. Knox, Fitzwilliam Museum, 2016.
- [31] L. Bonizzoni, S. Bruni, V. Guglielmi, M. Milazzo, O. Neri, *Field and laboratory multi-technique analysis of pigments and organic painting media from an Egyptian coffin (26th dynasty)*, **Archaeometry**, **53**(6), 2011, pp. 1212-1230,
- [32] M. Rifai, N.M.N. El Hadidi, *Investigation and analysis of three gilded wood samples from the tomb of Tutankhamen*, **Decorated Surfaces on Ancient Egyptian Objects Technology, Deterioration and Conservation** (Editors: Dawson, J., Rozeik, C. and Wright, M.M.), 2010, pp.16-24.
- [33] K. Hühnerfuss, A. von Bohlen, D. Kurth, *Characterization of pigments and colors used in ancient Egyptian boat models*, **Spectrochimica Acta Part B – Atomic Spectroscopy**, **61**(11-12), 2006, pp. 1224-1228.
- [34] C. Johnson, K. Head, L. Green, *The Conservation of a Polychrome Egyptian Coffin*, **Studies in Conservation**, **40**(2), 1995, pp. 73-81.
- [35] H. Jaksch, W. Seipel, K.L. Weiner, A. El Goresy, *Egyptian blue—cuprorivaite a window to ancient Egyptian technology*, **Naturwissenschaften** **70** (11), 1983, pp. 525–535
- [36] M.C. Edreira, M.J. Feliu, C.F. Lorenzo, J. Martin, *Spectroscopic study of Egyptian blue mixed with other pigments*, **Helvetica Chimica Acta** **86** ,2003, pp. 29-49.
- [37] P. Mirti, L. Appolonia, A. Casoli, R.P. Ferarri, E. Laurenti, A. Amizano, G. Chiari, *Spectrochemical and structural studies on a roman sample of Egyptian Blue*, **Spectrochimica Acta, Part A: Molecular and Biomolecular Spectroscopy**, **51**(3), 1995, pp. 437-446.
- [38] T. Pradell, N.T. Salvado, G.D. Hatton, M.S. Tite, *Physical Processes Involved in Production of the Ancient Pigment, Egyptian Blue*, **Journal of the American Ceramic Society**, **89**(4), 2006, pp. 1426-1431. DOI: 10.1111/j.1551-2916.2005.00904.x
- [39] M. Uda, *Characterization of Pigments Used in Ancient Egypt (Chapter I-1)*, **X-rays for Archaeology** (Editors: M. Uda, G. Demortier and I. Nakai), Springer, 2005, pp. 3-26.
- [40] L.R. Lee, *Investigation into degraded pigments on the Coffin of Amenemope*, **EA 22941**. British Museum, Conservation Research Group CA1997/20, 1997.
- [41] A. Cosentino, *Effects of different binders on technical photography and infrared reflectography of 54 historical pigments*, **International Journal of Conservation Science** **6**(3), 2015, pp. 287-298.
- [42] S. Pages-Camagna, H. Guichard, *Egyptian colours and pigments in French collections: 30 years of physicochemical analyses on 30 objects*, **Decorated Surfaces on Ancient Egyptian Objects Technology, Deterioration and Conservation** (Editors: J. Dawson, C. Rozeik and M.M. Wright), Archetype Publications, London, 2010, pp. 25-31.
- [43] L. Lee, S. Quirke, *Painting materials (Chapter 4)*, **Ancient Egyptian Materials and Technologies** (Editors: P.T. Nicholson and I. Shaw), Cambridge University Press, 2000, pp. 104-121.

- [44] D.A. Scott, S. Warmlander, J. Mazurek, S. Quirke, *Examination of some pigments, grounds and media from Egyptian cartonnage fragments in the Petrie Museum, University College London*, **Journal of Archaeological Science**, **36**(3), 2009, pp. 923-932.
- [45] H.H.M. Mahmoud, *Investigations by Raman microscopy, ESEM and FTIR-ATR of wall paintings from Qasr el-Ghuieta temple, Kharga Oasis, Egypt*, **Heritage Science**, **2**, 2014, paper no. 18.
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