

A TECHNICAL EXAMINATION AND THE IDENTIFICATION OF THE WOOD, PIGMENTS, GROUNDS AND BINDER OF AN ANCIENT EGYPTIAN SARCOPHAGUS

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Abstract

This paper describes an ancient Egyptian polychrome wooden sarcophagus found in the Saqqara excavation, Egypt. Multiple analytical and examination techniques were carried out on that ancient Egyptian, polychrome, wooden sarcophagus, such as optical light polarizing microscopy, X-Ray diffraction (XRD), scanning electron microscopy (SEM) coupled with EDS and FTIR spectroscopy. The structure of the object was visually examined. The wood was identified as Ulmus wood, which helped and confirmed the history of that wooden sarcophagus. Ulmus wood type is not Egyptian wood, it was imported from Greece. The plaster layers were identified as calcite mixed with huntite, by using a combination of techniques. The pigments used to decorate the Sarcophagus were identified as Egyptian blue cuprorivaite ($\text{CaCuSi}_4\text{O}_{10}$) as the source of the blue pigment. The green pigment sample was identified by SEM-EDS, which indicated the presence of Cu, Si, Ca, as the main elements of the Egyptian green. The black pigment was carbon black. The organic binder used was probably egg yolk and white egg with linseed oil.

Keywords: Egyptian sarcophagus; Pigments; Egyptian blue; Ulmus; Egg Yolk.

Introduction

The compositions of the painted layer were analyzed by Optical Microscopy (OM) and Scanning Electron Microscopy – Energy Dispersive Spectroscopy (SEM–EDS). A multi-analytical study was carried out to evaluate the state of conservation and the constitutive elements of the decorated wooden sarcophagus. That artifact was part of an Egyptian collection belonging to an Egyptian mission in Saqqara. The sarcophagus consists of two side-panels with polychrome decorations, probably dating from the late period to the Greco-Roman period, ca. 3900–1800 years B.C. (Fig. 1).

The provenance of the wooden sarcophagus is unknown, but on the basis of our comparative, stylistic and formal study and according to the results we obtained, from the identification of the kind of wood, which was Ulmus wood, and its non-Egyptian source, as such wood was imported from Greece, or it was used by Greco-Roman craftsmen who brought their materials into Egypt, it appears similar in style to other items dated to the late Greco-Roman (Early Roman) period in Egypt.

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Fig. 1. Ancient Egyptian Greco-Roman sarcophagus.

During the late Pharaonic period they used cartonnage to make the inner coffins for mummies. It was shaped on the shape of the body, forming a one-piece shell. It was often richly decorated with various geometric designs, an assortment of deities and with inscriptions, which included verses from the Book of the Dead, colored with different pigments [1, 2].

The characterizations of materials in archaeological objects can contribute significantly to their preservation and to their understanding; however, sampling and characterization are ideally performed by using non-destructive methods.

Additional analytical data can inform us of the ancient technologies used in their preparation, which is especially useful to art historians, archaeologists and conservators [3, 4]. Investigations of archaeological objects provide much information about the development and propagation of technologies in the past, which helps us conserve and restore valuable and scarce objects; it is absolutely necessary to understand their chemical compositions and crystal structures.

The aim of the present paper is to identify the wood type that was used as support, to examine the pigments, ground layers and the binding media. Although there were previous conservation works performed to stabilize the sarcophagus, such as the pre-consolidation of the sarcophagus, after excavation, the sarcophagus was examined by using a variety of techniques: polarized light microscopy (PLM), X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy, coupled with Energy Dispersive Spectroscopy analysis (SEM-EDS).

Materials and Methods

Analytical Procedure

The Optical Microscopy Analysis (O.M).

Polarized Light Microscopy (PLM) was carried out on the samples by using a Wild M8 stereomicroscope, an Olympus BX51 optical microscope and we recorded with a photo-camera. In order to visualize the paint cross-section, some samples were prepared in a special manner:

the samples were thinned to 1 mm and subsequently immersed in fluid Meliodent resin. Meliodent can be obtained by mixing two components: first a powder of polymethyl methacrylate, second, a liquid mixture of dimethacrylate and methyl methacrylate. After being mixed in the correct proportions, it solidifies at room temperature. After its solidification, the resin of the samples was finely polished in steps, ending with a Buehler Metadi 1 micron diamond suspension, to obtain a flat surface exposing the cross-section of paint. Then it was polished with a Strikers DAPV machine, by using Si-C paper discs with a decreasing granulometry (600, 1200, 2400 and 4000 grit size), until the cross-section surface became smooth and specular [5, 6].

These preparation processes were applied at the LRMH Laboratory of Restoration of Historical Monuments, Paris, France. Cross-section observations provided information about the number of painted layers, their sequence and thickness and also about the size of the pigment grains and the stratigraphy of the samples taken from the sarcophagus. Then the mounted cross-sections were studied by Mac-Microscopy, then under a Scanning Electron Microscope (SEM), where Energy Dispersive Spectroscopy (SEM-EDS) was used to characterize the elements present in different layers.

Non-destructive X-ray Diffraction (XRD)

X-ray diffraction analysis was carried out with Phillips X-ray diffraction equipment, model PW/1840, with a Ni filter, Cu-K α radiation 1.54056 Å at 40kV, 25mA, 0.05°/sec. It had a high resolution graphite monochromator, rotating sample holder and a proportional detector. Measurements were carried out on the samples, without grinding or powdering, in the range $0^\circ < 2\theta < 70^\circ$ in steps of 0.02°. The Rigaku unit was run at 40kV, 35mA, for 22 minutes.

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was carried out by using a Perkin-Elmer Spectrum. Spectra were recorded in the 4000–400cm $^{-1}$ region, with a resolution of 4cm $^{-1}$, in order to identify the materials present.

The Scanning Electron Microscopy-Energy Dispersive Spectroscopy Analysis (SEM-EDS).

Spectrometry, SEM-EDS, employed a Philips XL30 instrument, with an INCA Oxford spectrometer package, with an LaB6 source and an EDAX/DX4 detector, at a working distance of 10mm, with an accelerating voltage of 20kV, a spot size of 4.7 to 5.3nm (INCA conventional units) and a process time of 5s, corresponding to a detector dead time of 25–40%; and an acquisition time of 75s. OM images were taken with a Leica DMR microscope equipped with a Leica DC300 digital camera. We recorded SEM images by backscattered electrons (BSE).

The Description and Condition of the Wooden Sarcophagus

The Sarcophagi fragment is made up of the following layers: support, ground layer and polychrome paint layer.

Support

The support of that sarcophagus was wood, identified as *Ulmus*, according to the characteristics specific to that kind of wood. The results of our cross-section, tangential section and radial section investigation under optical microscopy confirmed that the wood in the sarcophagus was *Ulmus* wood (Fig. 2). It was impossible to cut the sample properly, to observe the microscopic sections. That was possible because the wood was probably only superficially decayed (mildew, fungi or salt). Observations were done directly on the samples, with a reflection microscope. Three plans were observed: the transversal section (Fig. 2a and b), which showed that it was a dicotyledonous wood (wood with vessels). The sample was too small to

observe a complete ring, pores are solitary, or in radial rows of 3 cells. There is a line of parenchyma, tangential to slightly oblique bands on the tangential section (Fig. 2). The rays were generally in series of 4 to 5, occasionally narrower or wider. Ray height was 30 to 50 cells. The radial section confirmed the parenchyma disposition. Without being able to observe a bigger sample and by comparing our observations to wood that we know that description could correspond to *Ulmus* (elm). The wood could have been imported from Greece or Italy. The preparation processes for the wood samples were performed at LRMH (Laboratory of Restoration of Historical Monuments, Department of wood), Paris, France.

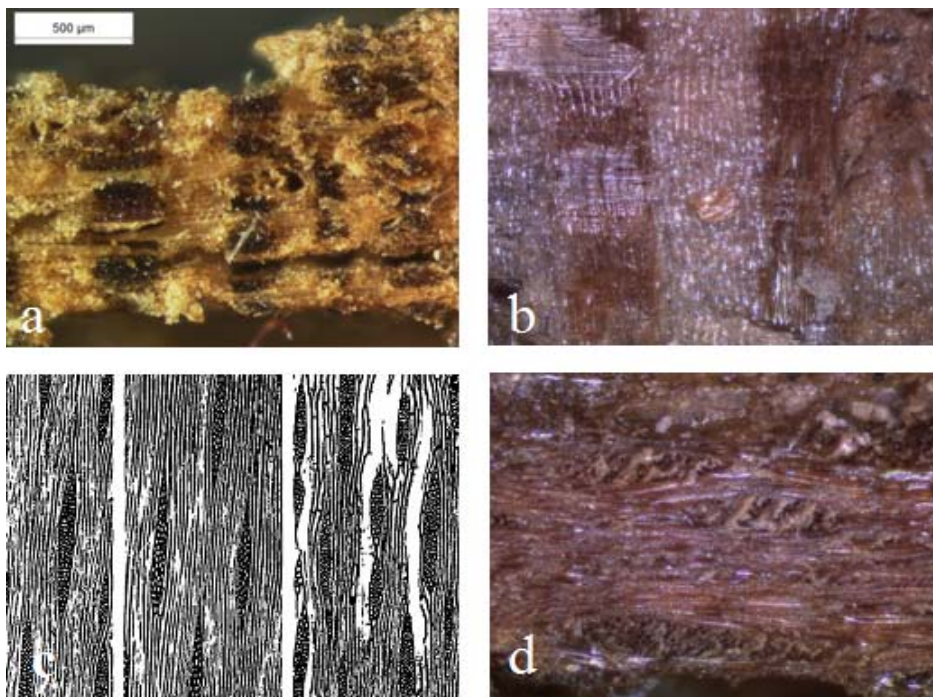


Fig. 2. Images of the wood section: a – cross-section from the sarcophagus (50X), b – radial section from the sarcophagus (50X), c – tangential reference sample – *Ulmus*, d – tangential section from the sarcophagus (100X)

Wood anatomy of *Ulmus* and *Celtis* species (Ulmaceae) is described. *Ulmus* differs from ring-porous species of *Celtis* in ray structure, crystal location, colour and fluorescence of water extracts. The soft elms/non-winged bark species differ from the hard elms/winged bark species in density, early wood pore diameter and appearance of crystal-containing axial parenchyma. Some species of hard elm can be distinguished from one another by a combination of characters: water extract color and fluorescence early wood pore diameter and spacing. The anatomy of ring-porous species of *Celtis* is uniform except that in Figure 2c, reticulate early wood pores have a smaller radial diameter than the other species. *Celtis pallida* is diffuse-porous and resembles other diffuse-porous species of the genus. Vessel element lengths are similar for all species within these two genera regardless of habitat [7].

Ground Layer

Ancient Egyptian craftsmen applied on the body of the wooden sarcophagus a layer of calcite, mixed with some amount of huntite and an organic binder. Our optical microscopy investigation indicated that the thickness of that layer was between 950-1200µm (Fig. 3). CaCO_3 and huntite ($\text{CaCO}_3 \cdot 3\text{MgCO}_3$) were used on the wooden coffin [8]. Some elements

found by our SEM-EDS analysis, such as Ca, Mg, Na and Cl, which means calcite with an amount of huntite (calcium and magnesium carbonates), were identified as the main compounds of that ground layer. In figure 4 our X-ray Diffraction analysis confirmed the presence of calcite (CaCO_3) and huntite ($\text{CaCO}_3 \cdot 3\text{MgCO}_3$), also the Na and Cl elements refer to sodium chloride (halite, NaCl). FTIR analysis showed that the organic binder used was almost entirely egg yolk and linseed oil (view paragraph on *The Binding Media*).

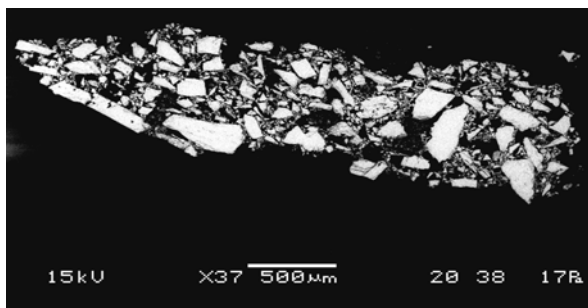


Fig. 3. SEM image of ground layer of Wooden Sarcophagus Coffin.

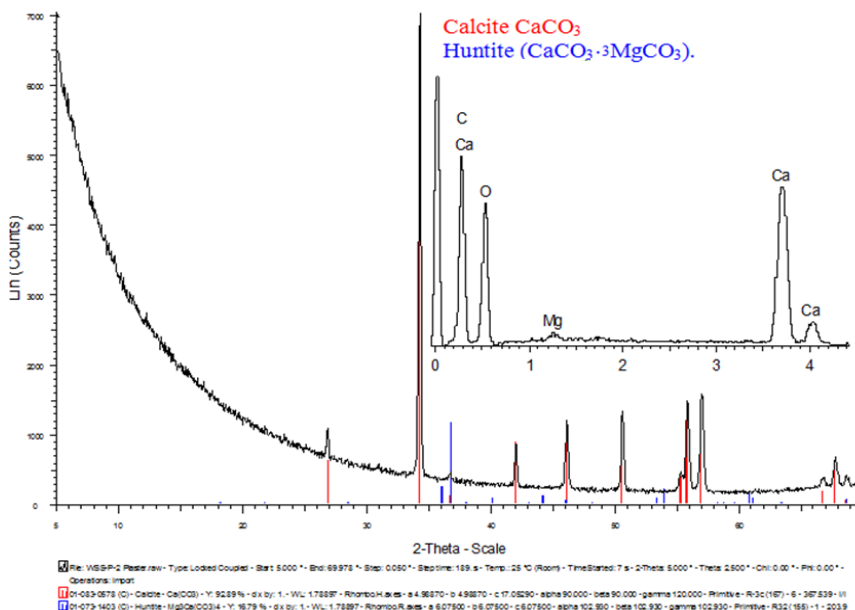


Fig. 4. X-ray Diffraction and EDS Spectra with the main elements of the ground layer

Pigments

Ancient Egyptian pigments have been analyzed extensively in laboratory studies and in the field [8-16]. It is self-evident that non-destructive and non-contact analysis in the field is indispensable for investigating ancient remains [8].

The Blue Pigment

Egyptian blue was identified in the dark green pigmented areas, as discussed below. Egyptian blue was the principal blue pigment used in Egypt from the early Fourth Dynasty to the Roman period [9]. The different shades of this color could be achieved in a number of different ways. The pigments may have been of varying quality. In 3000 BC, Egyptian craftsmen created the first synthetic pigment produced by man, Egyptian blue, which was

widely used during antiquity, spreading all around the Mediterranean basin until the 7th century AD. The Egyptian green pigment, also called green frit, appeared shortly after, presenting the same chemical elements and a turquoise color. These two pigments have been confused for a long time [10-12] in many cases; Egyptian blue was added in small amounts to enhance the brilliance of other colors, as was reported in various studies [13, 14]. The composition of Egyptian Blue and the recipe for its preparation have been established through experimental analytical techniques and experimental investigations. The pigment is a multicomponent mixture of cuprorivaite ($\text{CuCaSi}_4\text{O}_{10}$), cuproan wollastonite (CaCuSiO_3), silica polymorphs (tridymite, cristobalite or quartz) and an alkali-and chlorine-bearing cuproan glass phase. For the manufacture of Egyptian Blue three initial components are required: (1) a copper-bearing ingredient (e.g. malachite), (2) lime and (3) silica. All these initial materials were accessible to the ancient Egyptians: malachite in the oxidation zones of copper sulphide deposits in the Eastern desert and lime and quartz sand in sedimentary rocks along the Nile valley.

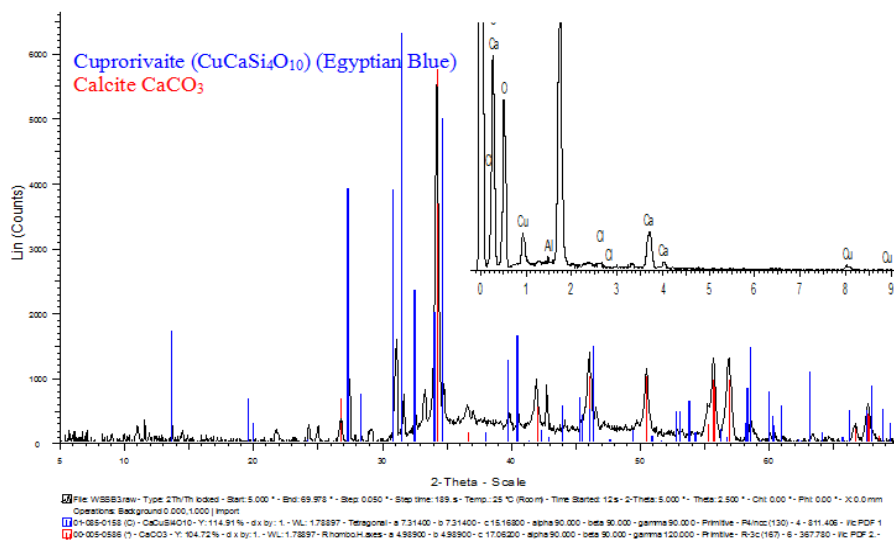


Fig. 5. XRD and EDS Spectrums of the blue sample with main elements

The occurrence of an alkali-rich copper-and chlorine-bearing glass between the cuprorivaite crystals provides evidence of the use of an alkali-bearing flux during the melting process, to lower the melting temperature [15]. Blue and green Egyptian pigments are typically Cu containing phases, with the coloration arising from d-shell electronic transitions. Egyptian blue is the first known artificially synthesized pigment. It was developed during the Old Kingdom (2600 BC) and used until the Roman period [16]. It is a synthetic copper silicate, with the chemistry of the naturally occurring mineral cuprorivaite prepared by firing a mixture of compounds containing silicon (silica as sand), calcium (chalk or lime), a source of copper (copper minerals or bronze filings) and a soda flux [17]. This produces a heterogeneous product comprised predominantly of manufactured blue cuprorivaite. The modification of mixing ratios directly influences the composition of the end product. The strong blue coloration of cuprorivaite is attributed to Cu^{2+} within a square planar arrangement [18].

Egyptian blue ($\text{CaO}\cdot\text{CuO}\cdot 4\text{SiO}_2$) was identified by X-ray diffraction. The pattern came together with small amounts of iron oxide hematite. The X-ray pattern was taken from a blue part on a sample. SEM-EDS spectra taken from the blue part, as shown in figure 5, corresponded to the chemical compositions mentioned above: Ca, Si and Cu as the main three elements for Egyptian blue.

The Green Pigment

From earlier periods, malachite ($\text{Cu}_2\text{CO}_3(\text{OH})_2$) and chrysocolla ($\text{Cu}_{2-x}\text{Al}_x(\text{H}_{2-x}\text{Si}_2\text{O}_5)(\text{OH})_4 \cdot n\text{H}_2\text{O}$) have been identified in green. Both were overshadowed by one of the great synthetic achievements of ancient Egypt, namely the Egyptian green, which was used from the first Intermediate Period onward. Greens were also produced in Egypt by using a combination of blue and yellow, either Egyptian blue mixed with orpiment or blue mixed with yellow ochre. Probably a secondary deterioration of these pigments is responsible for the identification of atacamite ($\text{Cu}_2\text{Cl}(\text{OH})_3$) or clinoatacamite ($\text{Cu}_2(\text{OH})_3\text{Cl}$) in a variety of Egyptian contexts. In the case of Egyptian artifacts, there is less published information available on the successful identification of green than any other color [19-22]. Evidence of the use of green frit is so far confined to Egypt. Alongside malachite, green frit was usually employed as a green pigment, its earliest occurrence being in tomb paintings of the 18th dynasty, but its use extends at least to the Roman period. The manufacture of green and blue frit relies on the same raw materials but in different proportions. To produce green frit, the lime concentration must outweigh the copper concentration. The firing temperature required for green frit may be slightly higher than that for blue frit, in the range of 950 to 1100°C. The ultimate product is composed of copper-wollastonite - (CaCu)₃Si₃O₉, crystals and a glassy phase rich in copper, sodium, and potassium chlorides. In certain circumstances (the use of a two-step heating process, the presence of hematite) scientists were able to make a cuprorivaite based blue frit that later became a copper-wollastonite based green frit at 1050°C. On some ancient Egyptian wall paintings, pigments that were originally blue became green: the blue frit can “devitrify” so that the “copper wollastonite predominates over the lesser component of cuprorivaite. As with blue frit, Hatton, Shortland, and Tite have analyzed evidence for green frit at Amarna in the form of cakes, powders and one vessel fragment and that inferred the sequential production of the three types of artifacts [23-26].

The green pigment sample from the wooden sarcophagus was identified with SEM-EDS, which showed the presence of elements as Cu, Ca, Si and Cl (Fig. 6).

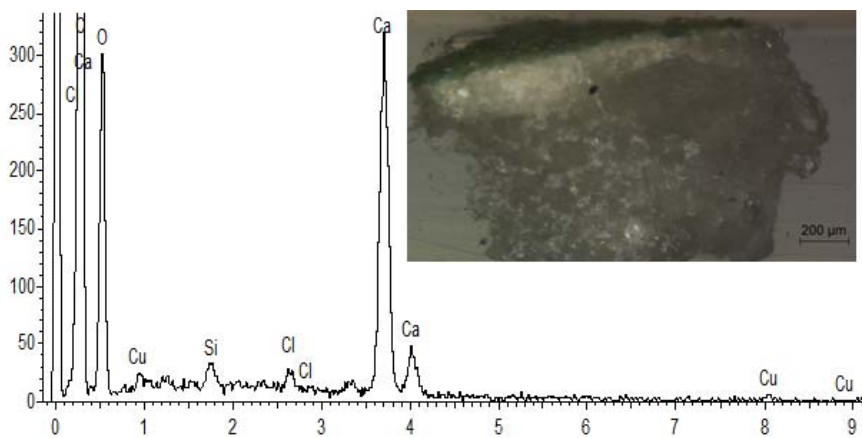


Fig. 6. SEM Image of green sample and the EDS Spectrum

Data obtained by X-ray Diffraction showed that calcite with huntite and quartz were in the final product, which was composed of copper-wollastonite and wollastonite (CaSiO_3), Spectral Card Number [027-1064]. A high silica ratio in the mixture leads to increased SiO_2 phases (e.g.; quartz, cristobalite, and tridymite). Additional chalk or lime increases the amount of wollastonite and an increase in the formation of copper oxides, such as cuprite and tenorite, was due to Cu having been added in excess [27]. However, in contrast to Egyptian blue frit, in which the copper oxide (CuO) content normally exceeds that of lime (CaO), the lime compound

normally exceeds the copper oxide content in the green [28]. Moreover, the alkali content tends to be higher in the green frit. In terms of microstructure, the green frit consists of a glass phase from which wollastonite and high temperature polymorphs of silica crystallized, together with partially reacted quartz particles. Green frit exhibits a range of colors from green to turquoise to pale blue and, as a result, it has been variously referred to as turquoise frit [29] and pale blue frit [30].

The Black pigment

The black pigment was identified as carbon black. It is the most frequently attested black and was used from ancient Egyptian periods. The data obtained by X-ray Diffraction showed that the compositions were calcite with huntite, elements that we found by SEM-EDS were confirmed as Ca, Mg, carbon black mixed together with iron oxide hematite to produce a kind of hue to the black pigment (Fig. 7).

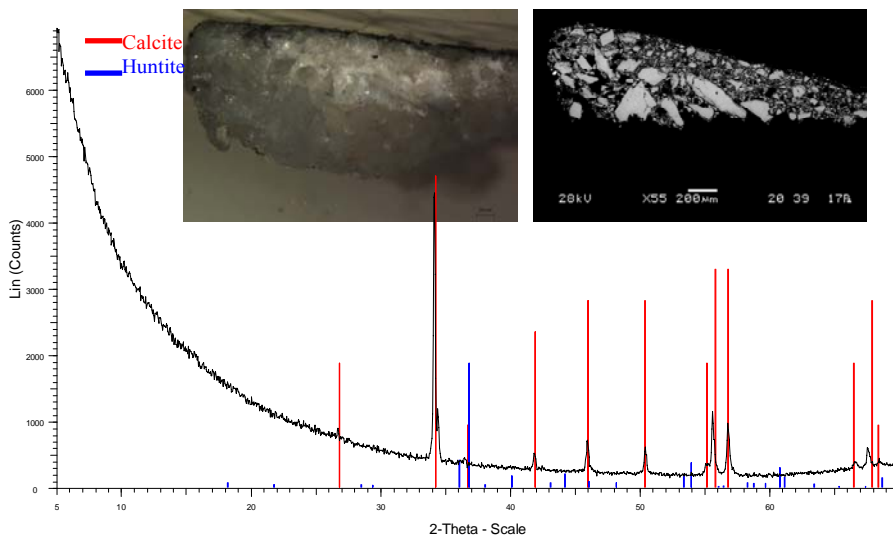


Fig. 7. XRD Spectrum and SEM and Optical microscopy Image of the black sample

We can see that its use is very frequent, both as a homogeneous paint layer and also mixed with other pigments to produce darker tones [31]. SEM observation revealed its chemical composition, characterized by a great amount of carbon (Fig. 8).

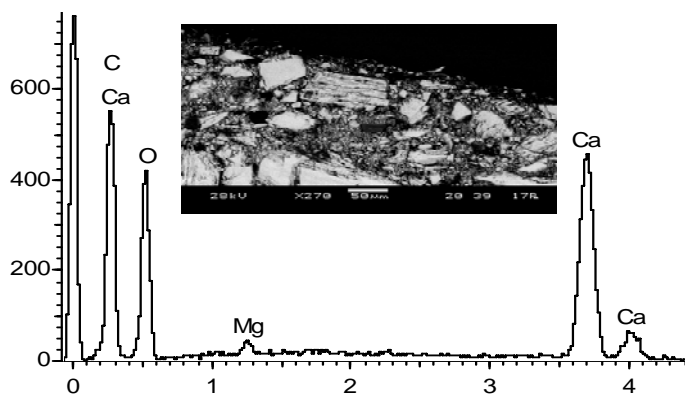


Fig. 8. EDS spectrum and SEM image of the black sample

The Binding Media

The spectroscopic study was essentially addressed to characterize the coloring medium used in the Sarcophagus samples. In all the analyzed samples the stretching vibrations of calcium carbonate CaCO_3 , peaked at 1409 , 705 and 611cm^{-1} , since the substrate was just a calcarenite [32]. FTIR analysis showed that the organic binder used was almost entirely egg yolk and egg yolk with linseed oil. In figures 9 and 10 we identified the use for the first time of a mixed media as a binder in a sarcophagus.

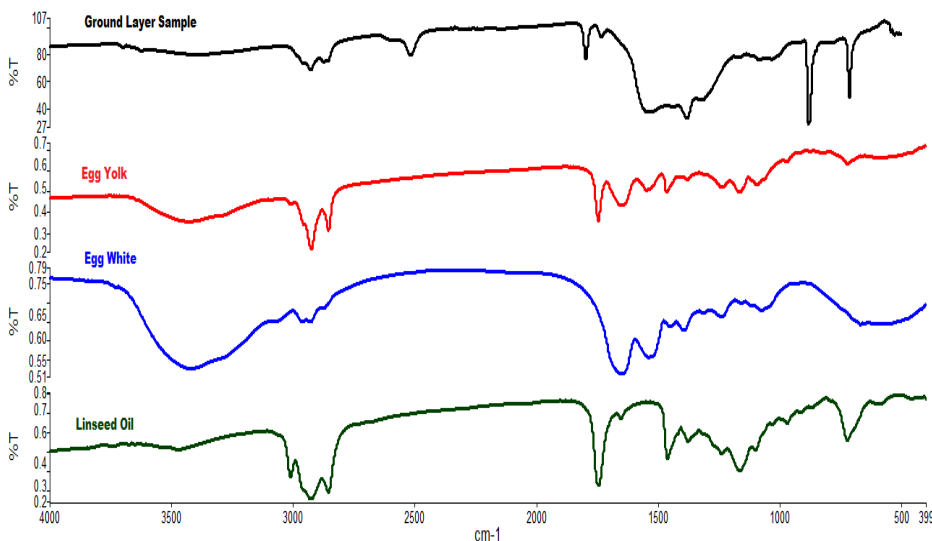


Fig. 9. FTIR Spectrum of ground layer comparison with egg yolk, egg white and Linseed Oil.

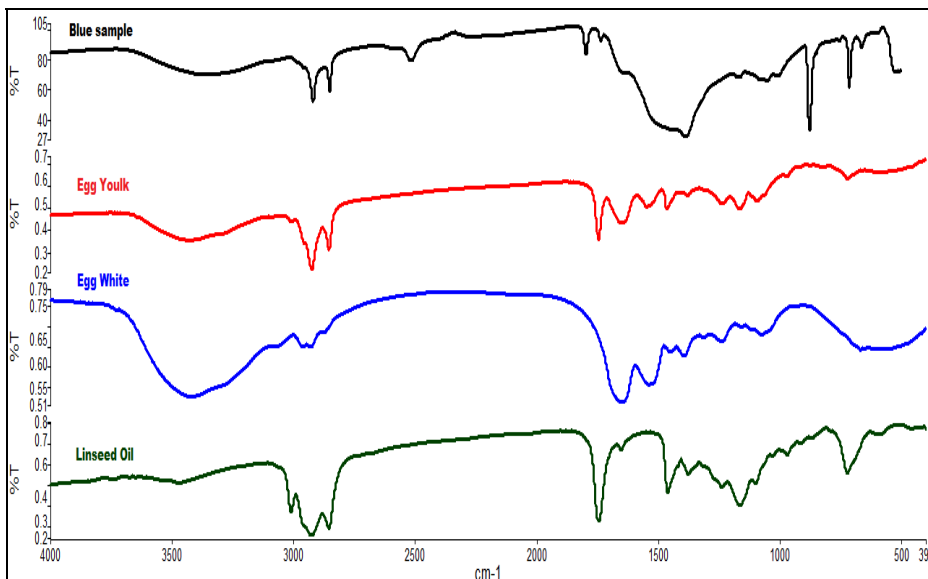


Fig. 10. FTIR Spectrum of the blue sample: comparison with egg yolk, egg white and Linseed Oil standard.

Discussions

According to our investigation and the analysis studies of the wooden sarcophagus, we confirmed that it consisted of two parts. The two polychrome parts were: a colored wooden lid and a colored wooden base. Ancient Egyptian craftsmen during the Greco-roman late period used *Ulmus* wood as a support for the sarcophagus coffin, then they applied a ground layer that consisted of calcite with some additives of huntite with egg yolk and egg white, with linseed oil as organic binder medium, then they decorated the structure of that sarcophagus with funerary decoration and texts.

Support

The results of cross-section, tangential section and radial section optical microscopy confirmed that the wood of the sarcophagus was *Ulmus* wood.

Ground layer

Within the results that obtained from our SEM-EDS analysis we found the presence of the elements Ca, Mg. X-ray Diffraction confirmed the composition of the ground layer calcite (CaCO_3), mixed with an amount of huntite ($\text{CaCO}_3 \cdot 3\text{MgCO}_3$).

Blue pigment

The blue pigment was confirmed by our SEM-EDS and X-Ray diffraction analysis. Our SEM-EDS analysis revealed that the highest concentrations were of the elements Cu, Ca and Si, which identify the use of cuprorivaite ($\text{CuCaSi}_4\text{O}_{10}$) in the Egyptian blue pigment. Egyptian blue ($\text{CaO} \cdot \text{CuO} \cdot 4\text{SiO}_2$) was identified by its X-ray diffraction pattern, together with a small amount of iron oxide hematite. The X-ray pattern was taken from a blue part on a sample. The SEM-EDS spectra taken from the blue part confirmed the chemical compositions mentioned above: Ca, Si and Cu as the main three elements for Egyptian blue.

Green pigment

The green pigment sample was identified with SEM-EDS, which revealed the presence of the following elements: Cu, Ca, Si and Cl, the main elements of Egyptian green (green frit). X-ray Diffraction confirmed the information we obtained and found the following elements: Cu, Ca, Mg, Si and Fe, which are also contained by Egyptian green. By X-ray Diffraction we found that this pigment contains calcite with huntite and quartz. The last pigment mentioned is composed of copper-wollastonite - $(\text{Ca,Cu})_3\text{Si}_3\text{O}_9$ and wollastonite - CaSiO_3 (Spectral Card Number [027-1064]).

Organic adhesive and binder

FTIR analysis showed that the organic binder used was almost entirely egg yolk and egg yolk with linseed oil.

Conclusions

The multi analytical methods that we have used provided information to identify the compositions of an ancient Egyptian Sarcophagus. For the support, the ancient Egyptian craftsmen used *Ulmus* wood. For the ground, the comparison between SEM-EDS and XRD results confirmed that the composition of the ground layer calcite (CaCO_3), mixed with an amount of huntite ($\text{CaCO}_3 \cdot 3\text{MgCO}_3$). The Egyptian painters invented binding media consists of egg yolk mixed with linseed oil, this egg-oil emulsion dries relatively quickly. The studied Sarcophagus is richy with its pigments; there is wide variety of colour combinations obtained

through the use of mixtures with different proportions and superimposing layers of different pigments, for examples using Egyptian blue with hematite to get different shades, Egyptian green was identified. Black carbon mixed with hematite used for black color, from all previous studies, the ancient Egyptian painter used to mix materials to achieve his target.

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