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Original article

CHARACTERIZATION AND EXAMINATION OF PIGMENTS, GROUNDS AND MEDIA FROM ANCIENT EGYPTIAN CARTONNAGE

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Abstract
An ancient Egyptian gilded Cartonnage with polychrome decoration period to late - Greek-Roman period found in Saqqara, Egypt was examined to characterize structure components (pigments, grounds layers and binder. It was studied by Optical Microscopy, X-ray diffraction (XRD), Laser-Induced Breakdown Spectroscopy (LIBS), Scanning electron microscopy coupled with energy dispersive X-ray (SEM-EDS) and Fourier transform infrared spectroscopy (FTIR) analysis. These techniques were used to identify the composition and morphology of grounds, nature of pigments and media used in the Cartonnage. The gilded Cartonnage is made on a double layer of plain weave linen soaked in gum. The first Coarse ground layer being a mixture of calcite and huntite. The second layer (finer one) being pure white calcite, the pigment colors employed were red, yellow and gold. Red was identified as hematite blended with gypsum, yellow as Calcite mixture with orpiment and gypsum. Gilded layer was identified as gold and silver with very thin layer of hematite and orpiment mixed with organic binder applied under the gilded layer. The binding medium on a double layer of plain weave linen , the coarse and the fine layer and binder in the yellow and red pigment areas was Arabic gum.

Keywords: *Cartonnage, Saqqara, Greek-Roman, XRD, Pigments, EDS, SEM.*

1. Introduction

The Egyptians believed that only a body that was preserved could fulfil the prerequisite of living forever. This belief came from religious observations that the dry sand of the desert acted to preserve buried bodies. Such beliefs were extant as early as the Neolithic and Predynastic periods of 5000-4000 B.C. [1][2]. An example of the importance of the preservation of the body is seen in the invocation from The Ancient Egyptian Book of the Dead: 'my body is everlasting, it will not perish and it will not decay for ages' [3][4]. According to Johnson et al. [5], the first use of Cartonnage coffins can be back to the Twelfth Dynasty (1991-1786 BC), then it becoming fashionable by the 22nd Dynasty (945-715 BC). Cartonnage may be made from a variety of materials. Adams [6] states that a core of mud and straw could be covered with linen soaked in gum, followed by a final layer of plaster. During the Greco- Roman period, papyrus was sometimes used instead of the layer of linen, fig. (1). The extraction of papyrus fragments from Cartonnage has been the subject of research by Wright [7][8]. The technology of the materials used in the fabrication of ancient Egyptian Cartonnage is still not fully investigated [9]. The use of Cartonnage material was prevalent during the last half of the first millennium BC, especially in the Ptolemaic and Roman periods [10]. The provenance of the Cartonnage is unknown but, on the basis of comparative stylistic and formal study, it appears similar in style to other examples dated to the late Greco/early Roman period in Egypt. The characterization of materials in historical artifacts can contribute significantly to their preservation and understanding; however, sampling and characterization are ideally performed using non-destructive approaches. Analysis of organic materials such as resins, gums and waxes and oil/fats from archaeological sites and excavated artifacts provides important information about trade routes and cultural preferences for decoration and usage [11][12][13]. Since resins (in particular) were used in both functional and decorative contexts [14], additional analytical data that can inform the methodology of their application or ancient technologies used in their preparation are especially useful to art historians, archaeologists and conservators [15][16]. The conservation of culturally significant artifacts is greatly assisted by the scientific analysis and identification of materials used in their construction. The knowledge of the material composition of an artwork may be used to inform decisions relating to treatment, storage and display as well as aid in provenance, attribution and authentication. The identification of specific pigments used for coloration is a key piece of information that is often employed for authentication. Manufactured pigments only become available after their invention and acceptance as a colorant, and, due to trends in artistic preferences, have a tendency to be used within specific windows of time. An approach such as this is only possible when we have pre-existing knowledge of which pigments were used during particular historical periods, which in turn is reliant on the collective documentation of a number of occurrences. This work analyzed the gilded textile Cartonnage in order to establish its elemental composition and to verify if the pigments used in the decorative paintings and the structure of Cartonnage are in accordance with those used by Egyptian craftsmen in the Greco-Roman period. The aim of the present paper is to report on the examination of pigments, grounds Layers, binding media and structure components employed in a Cartonnage. Technical and analytical studies were carried out to identify the pigments, textiles, binders and plaster ground of the Cartonnage. It was examined using a variety of techniques: polarized light microscopy (PLM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy with energy-dispersive analysis of elements from carbon upwards(SEM-EDS) has been undertaken with a view to establish the identification of the pigment composition and nature of the interaction with the substrate. The specimens date from the late period 712-332 BC to the Greco-Roman period 332 BC to 642 AD.

Figure (1) Flax harvesting, "Sennedjem" tomb, ancient Egypt.

1.1. *Condition assessment of the Cartonnage*

The Cartonnage, fig. (2-a) of unknown provenance appears as a double layer of plain weave linen backing, about 21 mm thick in total. Polarized light microscopy analysis showed it to consist of two plain weave layers, covered with a calcite as a double ground layer, figs. (2-b, c). A kind of similar type of structure was found by Seiler [17] who conserved a painted mummy mask, damaged during the Second World War, made of multiple layers of linen covered by a chalk base. Through studying the schematic drawing of the Cartonnage fragment, fig. (3-a, b), it could be noticed that it is made up of the following layers: **1)** Gilded layer-painted layer, **2)** Polychrome paint layer, **3)** Preparation red layer of hematite + orpiment + organic binder, **4)** Preparation plaster layer of white pure calcite, **5)** Ground layer of calcite + huntite + organic binder and **6)** Support layer, which is made from thick linen textile layers.

Figure. (2) **a** fragments of Egyptian gilded sarcophagus, **b** the left side of Egyptian gilded sarcophagus shows the deterioration state of a double layer of textile support, grounds layers and the gilded surface, **c** the right side of Egyptian gilded sarcophagus shows the weakness state of all textile layer and gilded surface.

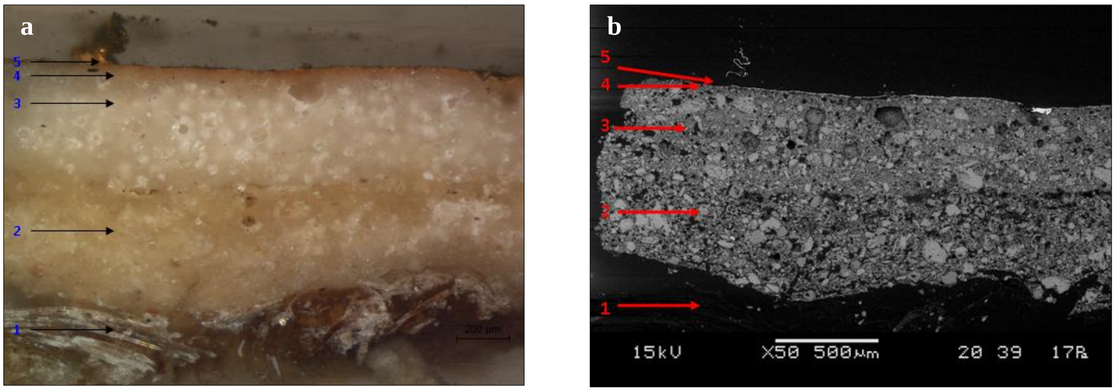

Figure. (3) sample surface from Egyptian gilded sarcophagus **a** OLM photo shows deferent layer of sarcophagi Cartonnage. 1. Textile support, 2. Ground layer, 3.plaster layer, 4. preparation red layer, 5. Gilded surface layer, **b** **SEM** photomicrograph shows the contractures of the gilded sarcophagus with the layer textile support, double ground layer, red preparation layer and gilded surface.

2. Analytical Procedures

2.1. *Optical Microscopy analysis*

Polarized light microscopy (PLM) was carried out on the samples which observed by a Wild M8 stereo-microscope, an Olympus BX51 optical microscope and recorded with a photo camera. In order to visualize the paint cross-section some samples were prepared in a special manner. Samples were thinned to about 1 mm and subsequently immersed in the fluid Meliodent resin. Meliodent can be obtained by mixing two components: The first one is powder polymethyl methacrylate and other one is liquid mixture of dimethacrylate and methyl methacrylate. After being mixed in the correct proportions it solidifies at RT. After solidification of the resin, the samples were fine grain polished in steps ending with Buehler Metadi 1 micron diamond suspension to obtain a flat surface exposing the cross-section of paint, then polished with a Struers DAPV machine by using Si–C paper discs with a decreasing granulometric (600, 1200, 2400 and 4000 grit size), until the cross-section surface became smooth and specular. The preparation processes were applied LRMH (Laboratory of Restoration of Historical Monuments), Paris, France. Cross-section observations provided information both about the number of painted layers, their sequence and thickness, and also on the size of pigment grains and to study the stratigraphy of samples taken from the Cartonnage. Then the mounted cross-sections were studied with Mac-microscopy then with Scanning electron microscope (SEM), where energy-dispersive spectroscopy (SEM–EDS) was used to characterize the elements present in the different layers. OM images were taken with a Leica DMR microscope equipped with a Leica DC300 digital camera.

2.2. *X-Ray diffraction (XRD)*

X-ray diffraction analysis was carried out with Phillips X-ray diffraction equipment model pw/1840 with Ni filter, Cu radiation 1.54056 A° at 40 KV, 25mA, 0.05 /sec. High resolution graphite monochromator, rotating sample holder and a proportional detector. Measurements were carried out on the samples with out grinding or powdering, in the range $0° < 2\theta < 70°$ with a step of 0.02°. The Rigaku unit was run at 40 kV, 35 mA, for 22 minutes.

2.3. *Fourier transform infrared spectroscopy (FTIR).*

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

2.4. *Laser-induced breakdown spectroscopy (LIBS).*

LIBS principle relies on the analysis of plasma induced by focusing a pulsed laser beam on the surface of a sample. The characteristic light emitted by the excited elements contained in the sample when they turn back to equilibrium is collected via optical fibers and sent to spectrometers. Spectra treatment allows an elemental identification of materials and a possible quantification through the calibration of the signal. The portable instrumentation is composed of a 1064 nmNd-YAG laser (Minilite II, Continuum, USA), with a 5 ns pulsing time and a maximum energy of 50 mJ/pulse; with a detection system located behind a lens (100 mm focal length) and based on a 7-fiber optical bundle connected to 3 integrated spectrometers (HR2000 Ocean Optics, USA). Those combined spectrometers cover the range from 200 to 940 nm. The two instruments working from 200 to 340 nm and 335 to 465 nm have a 1800 mm-1 grating and a 0.1 nm resolution while the third one, scaled from 510 to 940 nm, has a 600 mm-1 grating and a resolution of 0.31 nm. The emission spectrum is then recorded with an internal 2048 CCD array detector (delay: 1 s after laser Q-Switch, minimum integration time: 2.1 ms). The optical fibers are coupled with alignment lights to control the focal spot, placed at a distance of 130 mm from the convergent lens. The laser is piloted by the spectrometers software and pulses are triggered manually, in single shots or in series. The enclosure of the lenses in a rigid case, mounted on a camera tripod with wheels, makes on-site use possible. [18]

2.5. *Scanning electron microscopy investigations (SEM-EDS).*

Scanning Electron Microscopy coupled with energy dispersive x-ray spectrometry SEM–EDS was employed for samples investigations. A Philips XL30 instrument with an INCA Oxford spectrometer package, with an LaB6 source and an EDAX/DX4 detector at a working distance of 10 mm, with an accelerating voltage of 20 kV; a spot size of 4.7 to 5.3 (INCA conventional units) and process time 5, corresponding to a detector dead time of 25-40%; and an acquisition time of 75 s. SEM images, by backscattered electrons (BSE),

3. Results

3.1. *Identification of textile fibers*

Few textile fibers of Cartonnage examined by low-power binocular micro-scope, at 40-X magnification revealed that the textile was 'S'-spun and plain weave. Also, it has slight thickening at the nodes. The ends were not twisted, and tapered gently. The diameter of the fibers was uniform at around 1.5 m., as shown in fig. (4). they were pale yellow to Brown, oval in cross-section, and showed strong birefringence colors under crossed polars. Furthermore, the observations of longitudinal and cross sectional morphology of the cells perfor-med using an optical stereomicroscope Leica DMRM with magnifications of 100-X, 200-X attested that the fibers have a vegetable origin, and the twist direction of the yarn structure is S. In longitudinal view; fiber occurs as large groups. Individual fibers are cylindrical and partly have striated cross-marks. This characterized the bast fibers. The fibers belonging to polygonal cells with thin walls and large lumina characterized of linen, which was very commonly employed in ancient Egypt, both for everyday wear, use and in funerary contexts. [19][20].

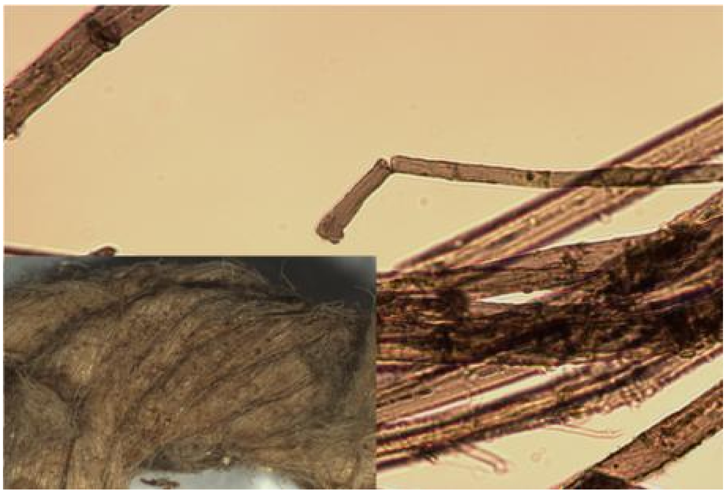

Fig. (4) fiber identification made by examination of both longitudinal and cross sectional morphology of the cells, Fibers are from vegetable origin. The twist direction of the yarn structure is S. The morphology is characteristic of bast fibers. In cross-section, the fibers correspond to polygonal cells with thin walls and large lumina characteristic of linen.

3.2. *Ground Layers*

The grounds layers consisted of three layers, fig. (3-a, b); they are coarse dark layer, plaster or fine layer and preparation bole layer. The **1st type** lies after Lenin textile support, with thickness between 500µm-700µm. the colure of that layer is darker (its may be because using of Arabic gum binder and huntite with the main compound calcite). Through SEM-EDX, and LIPS analysis; some elements can be found such as Ca, Mg, Na, Cl, that means calcite with amount of dolomite and huntite (calcite magnesium [$Mg_3Ca\ (CO_3)_4$]) were the main compounds of this ground layer. Na and Cl elements refer to sodium chloride as halite salt, also, X-Ray diffraction analysis refer to the same minerals (calcite and huntite) FTIR analysis showed that the organic binder is Arabic gum. The **2nd type** is plaster or fine layer; it is the second part of ground layer, through the microscopic observations, it could be noticed that this layer can named as plaster layer, which prepared to be ready for painted. It is whiter (lighter than the first course one), its thickness almost between 300 µm -400 µm. its component is calcite (Pure white calcite). Regarding, the presence of some huntite in the calcite ground layer is interesting in view of its possible use as a demographic indicator [21]. Relatively few objects of the Late Period through the Roman period (c. 712 BC-AD 476) were found to contain any huntite, so it is also of interest from a temporal aspect that huntite was found in this study. The use of calcite and huntite as ground may have been employed to create a color contrast in the whites themselves, or at least in the painted areas of the design. The 3^{rd} **type** is very fine layer applied above plaster layer, the purpose of that red layer was using as prepared layer for reviving the gilded layer about it. It composed of red grain of iron oxide hematite and orpiment mixed together with organic binder. Orpiment As_2S_3 It has a dense gilded yellow color with high brightness and an outstanding gold gleaming shiny glaze. The ancient Egyptians recognized its special quality in comparison to other yellow pigments. It was first mentioned by Lucas and Harris (1962) who reported that it first appeared in the second half of the 18^{th} dynasty. Terrace and Braziller (1968) reported orpiment from decorations on the coffin of Djhuti-nakht (11^{th} dynasty) in the necropolis of Deir el Bershah. The yellow colors appeared to have faded to a very weak yellow in many areas, such fading characterizes this pigment, particularly with light exposure as attested previously by Green [22]. The pigment may degrade photo chemically to arsenic oxide. The only yellow arsenic-containing pigments are orpiment and Realgar. Further identification by PLM also revealed the presence of orpiment. The very light yellow ground pigment was weakly pleochroic and strongly birefringent. Orpiment would have been available to the Egyptians as naturally occurring ground sulphide. The earliest example being on a coffin about 1900 BC, from the 12^{th} dynasty. Although its use as a pigment is not unknown during this period, orpiment is relatively rare, with most other yellows employed being ochres [23]. An attempt at X-ray powder diffraction characteriz-ation of this pigment was failed due to sample size and crystallinity were presu-mably insufficient to obtain a satisfactory result and no pattern whatever could be discerned on the resulting diffracto-gram.

3.3. *Polychrome paint layer*

Painting materials used in ancient Egypt can be strictly classified into two distinct categories *Natural pigments*; which make up a palette of natural polychromatic minerals (or their mixtures) exploited from domestic or foreign ore deposits, **Synthetic pigments* prepared from natural raw materials and metal scrap by firing the mixtures at moderate or high temper-atures[24][25].

3.3.1. Gilded layer sample

All face of the Cartonnage from the top to the bottom, fig. (2) was gilded with gold leaf in water gilding techniques. That craftsman prepared that red layer which consisted of hematite with orpiment mixture together with organic binder as a very thin layer then applied a single leaf made of alloy Au-Ag. Investigation by SEM-EDX presented the highest concentrations were Ag, Au, Ca, Fe, As & S, fig. (5). X-Ray Diffraction (XRD) analysis confirmed that gold and silver were used as gilded materials, the thickness of that layer almost between 10-20 µm., fig. (6). And presented of elements Fe, As, S and Ca are coming from calcite as plaster layer and Hematite mixture with orpiment as preparation layer under gilded layer [26], as shown in fig. (7).

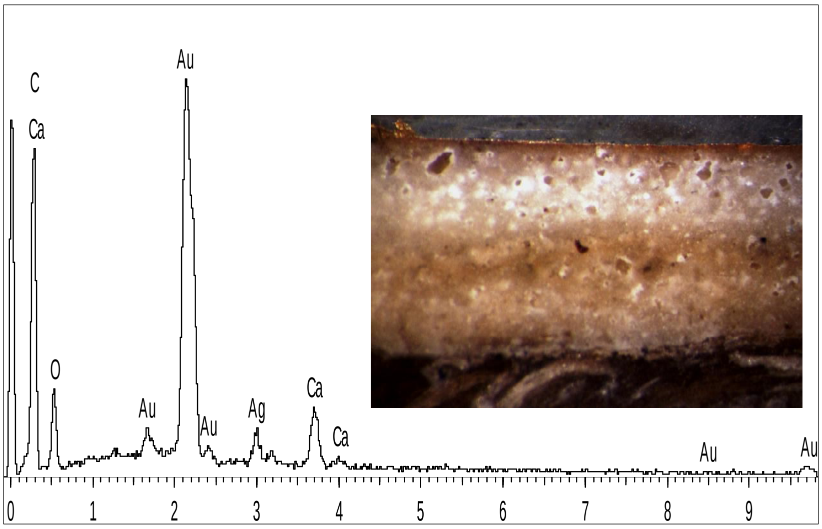

Figure (5) optical light microscopy photo and EDX spectrum of gilded sample surface from Egyptian gilded sarcophagus shows the elements found (Au, Ag, Ca).

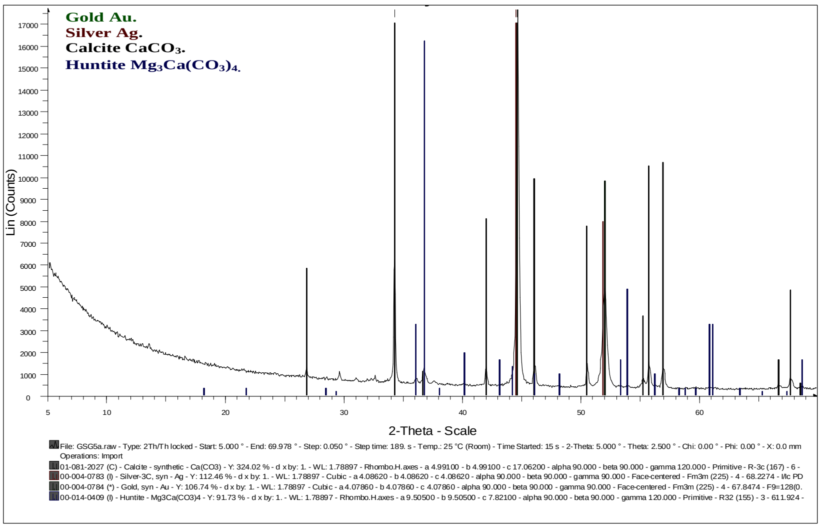

Figure (6) XRD pattern of gilded surface layer confirmed the present of Gold Au, Silver Ag, Calcite $CaCO_3$ and huntite $Mg_3Ca\ (CO_3)_4$

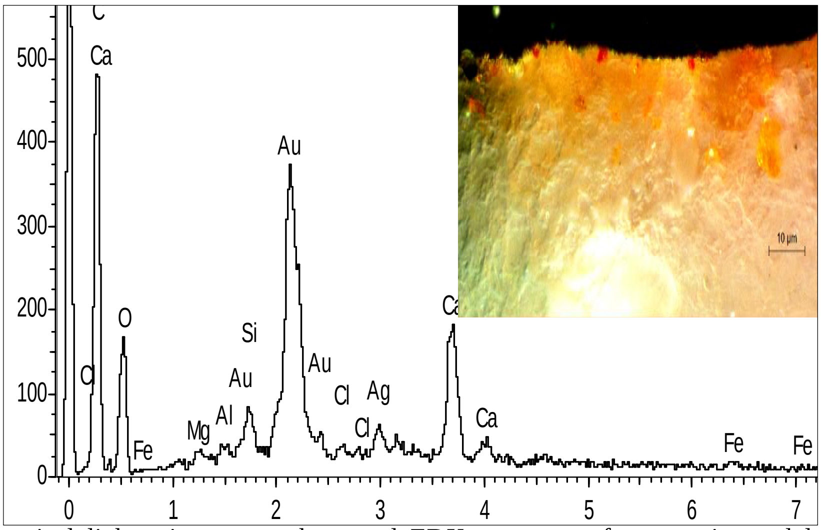

Figure (7) optical light microscopy photo and EDX spectrum of preparation red layer shows the elements found (Au, Ag, Ca, and Fe, As, Al, Cl) as very thin fin preparation layer under gilded layer.

3.3.2. Red pigment sample.

The principal red pigments in Egypt are of two main types, red iron oxide (hematite) and red ochre (hydrated) [25]. Natural iron oxides occur plentifully in Egypt and then anhydrous and hydrated oxides could be used as red pigments without any heat treatment. The use of red ochre in ancient Egypt was reported by many authors [27][28][29]. Red ochre used from the beginning of 5^{th} dynasty till the Roman times [30][21]. The result of SEM-EDX analysis of indicates that the main elements are (Fe, Ca, & S.) fig. (8). Also, EDX analysis showed the strong contribution of Al and Si which indicated the existence of an alumino-silicate material, probably from the clay minerals associated with ochre. Crafts mixed hematite with orpiment and gypsum to give some kind of hue of colure degree of pigment. These results are in full agreement with the other made by XRD, where, Fe is the main component of red ochre ($Fe_2O_3 .H_2O$) and the high Ca & S concentration is due to the Cartonnage matrix, which contains gypsum ($CaSO_4$. $2H_2O$) and calcite ($CaCO_3$), fig. (9). Moreover, the investingation of macro microscopy, it could be noticed that red pigment is very thin layer, with thickness aprox. 13-20 µm.

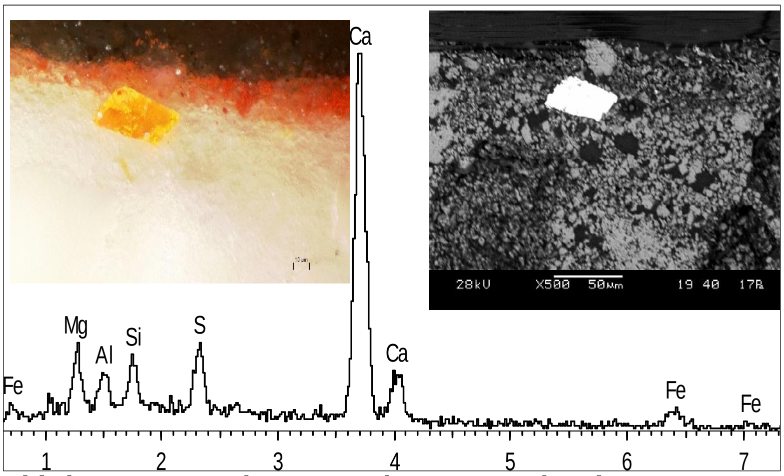

Figure (8) optical light microscopy photo, SEM photomicrograph and EDX spectrum of red pigment sample shows the elements found (Ca, Mg, Fe, As, S)

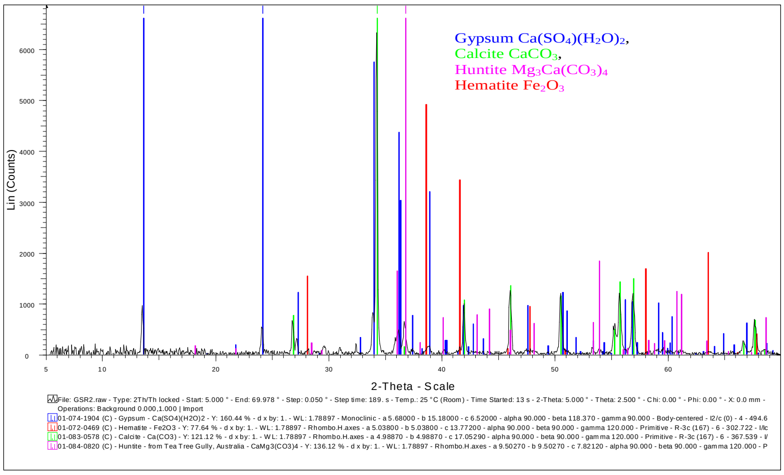

Figure (9) XRD pattern of red pigment shows orpiment crystal grains mixed together with gypsum as red pigment layer and confirms the presence of Gypsum $CaSO_4$ $2H_2O$, Calcite $CaCO_3$, Huntite Mg_3Ca $(CO_3)_4$ and Hematite Fe_2O_3

3.3.3. Creamy pigment sample (White pigments)

White pigments is belonging to the earliest painting materials in use since predynastic times as attested previously by many authors such as Lucas and

Harris., 2001 [31]. In addition, recent reports written by some authors El Goresy, et al., 1986 and Schiegl, et al., 1989 [32][33] have provided chronological evidence for almost uninterrupted use from the 5th dynasty to the Roman period. According to SEM-EDX investigation and LIPS identification spectrum, the white creamy area of the Cartonnage ground is principally of pure white calcite with a little of gypsum, huntite mixed together with orpiment [24][34]. The identified elemental components which are As, S, Ca, , Due to the material was so dilute it impossible to obtain an X-ray diffraction confirmation, fig. (10). Under the Macro-Microscopy, it could be noticed on one hand that the creamy pigment layer is very thin layer with thickness aprox. 10-15 µm. On the other hand, the analytical results of SEM-EDX and XRD showed that the elements found in the samples were: Si, S, Cl, K, Ca, As. the present of Ca and S are coming from Calcite and gypsum, As and S are coming from orpiment and Cl as halite salt found on Egyptian soil, fig (11). From specialized point of view and based on the XRD analysis, it could be claimed that the Gypsum was blinded with orpiment and they mixed with calcite by Egyptian crafts to give some kind of hue of colure degree of pigment

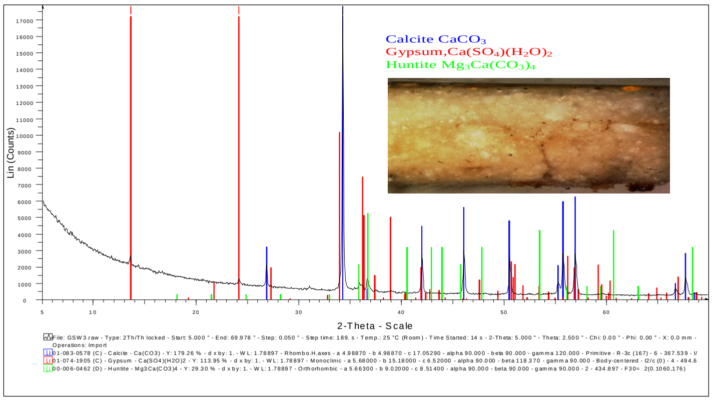

Figure (11) XRD pattern of white creamy pigment surface confirmed the present of Gypsum $Ca(SO_4)(H_2O)_2$, Calcite $CaCO_3$ and Huntite $Mg_3Ca(CO_3)_4$.

3.4. *Organic binder*

The investigation made by FTIR spectroscopy proved that Arabic gum was the unique binding media in all layers of Cartonnage. This media was identified in 5 layers of Cartonnage, e.g. Linen textile, support layer, double ground Layers (coarse and finer, thin red layer and gilded layer.. From specialized point of view, it could be said that the characteristic bands of Arabic gum as main organic binder in the ground and paint layers are 3600-3200cm- O-H stretching band, 300-2800cm- C-H stretching bands, 1650cm- O-H bending band, 1480-1300cm- C-H bending band, 1300-900 cm- C-O stretching bands, fig. (12).

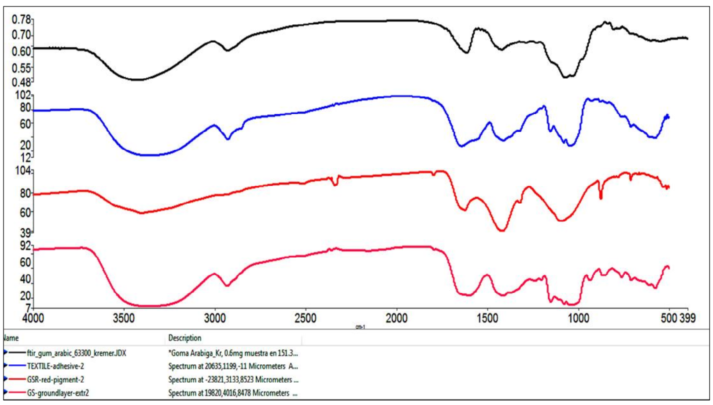

Figure (12) FTIR spectroscopy spectrum of ground layer, red, textile samples compared with reference sample, confirmed that Arabic gum was used as binder for all layers of Cartonnage

4. Discussion

According to the stratigraphic layers and the investigations of the gilded textile Cartonnage, it could be confirmed that it consisted of tow parts. The first one is the body (the front of body face and legs). The second part was separated; it is the base which the Mummy was putting inside it. These two parts were fixed together by using strong fiber of textile on the side of two edges of the Cartonnage. Through our explanations it could be argued that ancient Egyptian crafts in Greco-roman late period used linen textile as a support for his gilded Cartonnage with two thick layer of linen soaked on Arabic gum as binder material of adhesive (usually stone, wood is used as support for their Cartonnage). Then the crafts applied two different kinds of ground layers. the 1st one was much darkness and bigger in its thickness than the 2nd one. The 1st layer (ground layer) that applied directly on textile consisted of calcite ($CaCO_3$) with some additives from Huntite ($Mg_3Ca(CO_3)_4$) with Arabic gum as binder organic medium for ground layer with textile support. The 2nd layer was whiter and much brightness and less in thickness than the previous one, it named plaster layer; it consists of pure white calcite with little of Arabic gum as a binder. Then completing the structure of that Cartonnage with preparation layer, it's very thin layer and consists of hematite and orpiment with Arabic gum as binder and adhesive, it was prepared to receive the gilded layer above it.

5. Conclusions:

Through the examination with the previous multi analytical techniques (Optical microscopy and SEM-EDS, LIPS, and XRD), it could be affirmed that these techniques lead to some important information about the structural of ancient Egyptian Cartonnage from Saqqara area. The gilded layer of this Cartonnage consists of gold (Au) mixed with silver (Ag), hematite which was used as a red pigment which was blending with gypsum and orpiment to give some kind of red hue. Furthermore, the creamy pigment was mixture of calcite and gypsum with hematite blending with orpiment giving the creamy hue of the color, organic binder and adhesives in all the structural were Arabic gum.

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