

NEW APPROACH OF CHARACTERIZATION AND STATE OF PAINTED RELIEFS IN PETOSIRIS TOMB, TUNA EL-GEBEL, EGYPT

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Abstract

The aim of this study is to identify the components and assess the current state of the painted reliefs in the funerary tomb of Petosiris, which is located in Tuna el-Gebel in the province of Minia, Southern Egypt. The tomb has been dated to the late 4th and early 3rd centuries. It is considered one of the most important archaeological tombs in Egypt because its inscriptions include some of the Pharaonic rituals. These inscriptions were considered an important archaeological record, since the containment of historical information, Pharaonic scenes, rituals and those painted reliefs have been carried out with a very impressive technology in the implementation and use of pigments. The tomb and its painted reliefs suffered from severe damage factors that threaten and even foreshadows the disappearance and loss of these inscriptions. Accordingly, the aim of the work in this article was to study the techniques that were used in the painting process, assess the conditions of the painted inscriptions, the characterization of the pigments and the organic medium used. A range of modern and sensitive examination and analysis methods have been used to provide us with information about the components of the tomb and painted compositions. The analytical study was performed using X-ray diffraction, Scanning Electron Microscopy / Energy Dispersive X-Ray Spectroscopy (SEM/EDS), Fourier-transform infrared spectroscopy (FTIR). The examination was carried out using optical microscope, polarized microscope and scanning electron microscopy. The investigation of samples showed the use of Egyptian blue pigment as a pigment for blue color. Hematite used to get the red color, Goethite for yellow, a mixture of carbon and hematite for black and Celadonite ferrous as pigment material for green. Moreover, egg yolk used as organic binder in the painted reliefs of the tomb.

Keywords: Tomb; Painted Reliefs; Pigments; Organic Medium; XRD; Egypt.

Introduction

The Funerary tomb of Petosiris was discovered in 1919 and excavated by Gustave Lefebvre, who published all of his results in a series of preparatory reports and finally in his inclusive (Tombeau de Petosiris), at which he set forth from the beginning he dated the tomb to the late 4th and early 3rd centuries. Petosiris tomb (Fig. 1) continued to be used by successive family members through the brief reign of Alexander and into the Ptolemaic period [1].

Petosiris tomb consists of a hall consecrate to the memory of Petosiris a subsequent part of the structure and an almost square chapel with four pillars.

These cavities were decorated and painted by Egyptian artists with painted reliefs of great benefit for the history of Egyptian art, some of them are in well preserved painted. This tomb reveals one of the most beautiful and wonderful scenes in ancient Egyptian daily life and the most important works in ancient Egypt. The scene represents a view of carpenters (the first

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clarification of carpenters) working on a reamer. The wooden bed of this reamer appears to be mounted vertically and is promptly set into the ground. [2].

The tomb seems to have been recently cleaned and has new lighting built-up, which showed the adorable painted reliefs, some of the original paint is still in place and the paintings are greasy and graceful with a great deal of dim blue. [3].



Fig.1. Painted reliefs from Petosiris tomb including different Pharaonic rituals.

In this study, a modern set of analysis and examination techniques has been used to provide us with abundant, useful and sufficient information about the Petosiris tomb, such as building materials, the pigments materials were used in the painted reliefs and the organic medium. This information will explain the current state of the tomb and indicate a true vision of its condition and the damage it suffers from. [4].

The analysis was carried out using X-ray Diffraction, which is one of the most important analytical techniques. It provides us with the components of building materials and inorganic pigments. [5- 6].

Scanning Electron Microscopy / Energy Dispersive X-Ray Spectroscopy (SEM/EDS) with elements analysis unit was used to identify the various elements found in building material samples or painted inscriptions, and to identify the salts and aspects of deterioration on the painted surfaces. [7- 8].

Fourier infrared spectroscopy (FTIR) was used to identify the organic medium in the painted reliefs, and other information about the presence of varnish layer on painted reliefs of the Petosiris tomb. [9-10].

Examination and investigation of painted samples were done on the cross-section samples using the optical microscope, polarized microscope and scanning electron microscopy. [11-12].

All the used analysis and examination techniques provided us with information about the nature of painted reliefs as well as the current situation and the damage it suffers from. Furthermore, provide information about the painted layers, their numbers, thickness, and the painting techniques used in the tomb. [13-14].

It is worth mentioning that, there are many studies have already been done on the tomb, but most of them were archaeological and historical studies and there was no scientific study on the tomb, its compositions, pigments materials and organic media [15-16-17].

Materials and Methods

Microscopic Examination

Optical microscope: is an important and useful method of examining archaeological materials, it provides valuable and important information about the sample's characterizations, weakness and deterioration. [18-19].

Samples were prepared in a suitable manner, with to minimum thickness of the sample \approx 1 mm and then immersed completely in the resin. The resin used in the preparation is called Melodent, the samples were left for a period of time (5- 10 minutes) for total drought then polished and softened by very fine sanding to obtain polished and suitable sample surface level suitable for examination.

The study and examination of cross-sections samples provides us with abundant, important and useful information about the nature of the samples, the number of layers, the thickness of each layer. Besides, it provides us with the size of crystals and grains of pigment materials as well as information about the deterioration of samples compositions [20 - 21].

The study and examination were carried out using a polarized photovoltaic microscope the wild M8 stereomicroscope provided by the Olympus BX51 optical microscope, which is directed and recorded by the image camera.

Cross-sections samples were examined with *Scanning electron microscope* (SEM) with a kind of microscope named a Leica DMR microscope equipped with a Leica DC300 digital camera. [22].

X-Ray Diffraction Analysis

Samples were prepared for the analysis using X-ray diffraction model of P/1840 with Ni-filter, with copper radiation 1.54056° at 40°C , 25mA, 0.05/sec. The system attached with high-precision graphite monochromatic, revolving sample possessor and prorated detector.

It is worth mentioning that, the prepared samples were very simple, the samples were in the case of solid state and no grinding operations were done. The aim was to keep these samples intact, without any damage; in order to preserve them. [23-25].

The measurements of the samples prepared previously for X-ray diffraction analysis were performed with the operating system in the range of $0^\circ < 2\theta < 70^\circ$ with a step of 0.02° . It is worth mentioning that the Regaco unit was operated at 40kV, 35mA, for 22 minutes as a fixed time.

Fourier Transform Infrared Spectroscopy

Analysis of the archaeological samples by FTIR (Fourier transform infrared spectroscopy) is of a great importance and was applied in a wide range by researchers in order to identify an organic medium was used in paintings process a set of samples have been prepared for this type of spectroscopy.

The analysis was done using the Perkin Elmer Spectrum, measurements and readings were performed in the area $4000\text{--}400\text{ cm}^{-1}$ to identify existing organic materials and to determine the organic functional groups found in archaeological samples. [26–29].

Scanning Electron Microscopy Investigations

Scanning Electron Microscopy coupled with energy dispersive x-ray spectrometry SEM/EDS, is one of the most important and accurate methods of analysis of archaeological materials due to the abundance of information and results that can be obtained from the analysis of this instrument, as well as its high accuracy and sensitivity.

The operating system for the analysis and study of those samples was A Philips XL30 device with an INCA Oxford spectrometer bundle and with an LaB6 head witch an EDAX/DX4 retriever at a running range of 10 mm, active with a hasten voltage of 20 kV. A spot size was 4.7 to 5.3 and the operation time of action was 5, identical to a detector dead time of 25–40%; and a procuracy time of 75 s. [30–33].

Results

Red Pigment. Sample.

The red pigment sample was examined and studied by the optical microscopy which showed that, the red painted layer is not thick painted layer and characterized by irregular surface. The surface showed the general weakness, and the pigment was applied on a single preparation layer which is characterized as a soft white layer (Fig. 2).

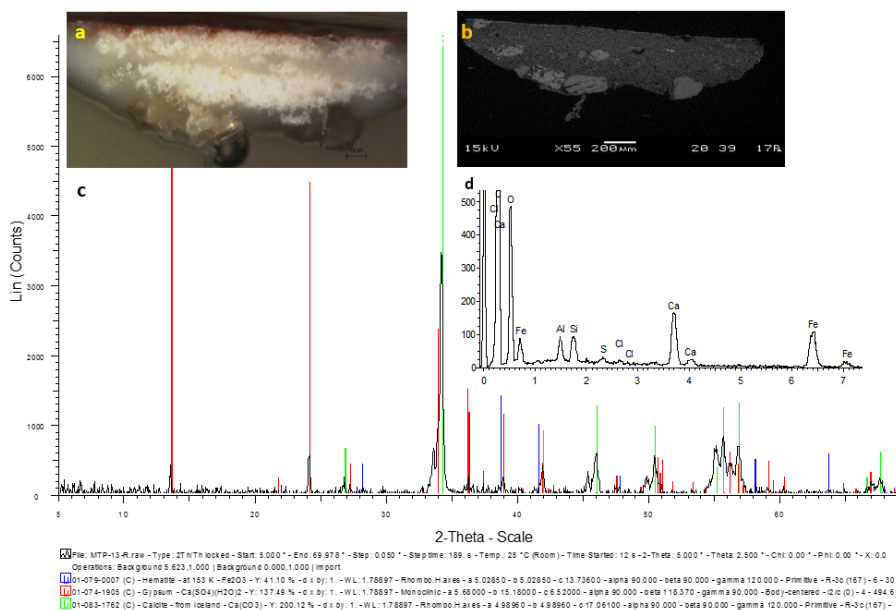


Fig. 2. a) Cross section investigation with optical microscopy of red sample. b) Scanning electron microscopy of sample shows that the red pigment is a very thin line, the deterioration in the ground preparation layer and the number of the layers. c) XRD pattern of red painted sample indicated the presence of hematite as a main component of red one plus the presence of gypsum and calcite which are coming from the fine preparation layer. d) SEM/EDS pattern confirmed the presences of Fe, O as main elements of hematite, Ca, O, S as main elements of gypsum and calcite.

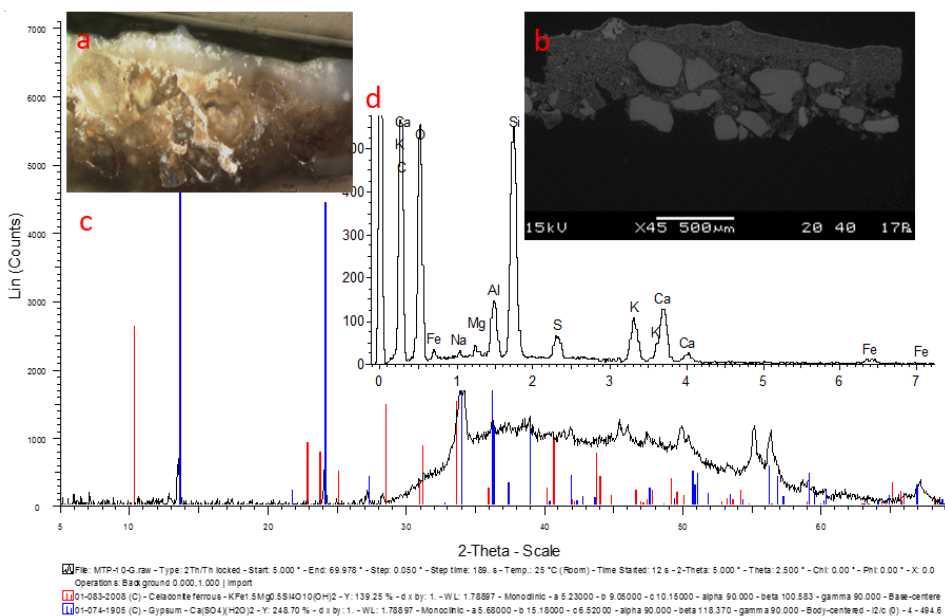
Scanning electron microscopy of the same sample emerges from the figures that, there is a thin painted layer with losses in some parts of the sample, cracks and weakness. The picture showed that, the painted layer was applied on a smooth ground preparation layer. (Fig. 2).

XRD analysis: The Study and analysis by the X-ray diffraction of the red painted sample confirmed the presence of hematite (Fe_2O_3) which is the primary component of the red pigment, with gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and calcite (CaCO_3), the two main components of soft preparation ground layer. (Fig. 2).

SEM-EDS analysis: The results of the analysis by SEM/EDS indicated the presence of the following elements: Fe and O, the basic constituents of hematite and red pigment respectively. The results also showed the presence of Ca, S and Cl elements, indicating that, gypsum and calcite were the main components for the preparation of ground layer. Also, the results indicated that, sodium chloride salt NaCl (halite) is present in the preparation layer as well. (Fig. 2).

Green Pigment Sample.

Through studying and examination of the green sample by optical microscope, it is clear from the image that, there were four layers. The first one is the green painted layer that is a small thickness and characterized as an irregular surface, the second layer was a white layer, the third is a rougher ground layer and the fourth last layer is much rougher than the previous



SEM-EDS analysis: scanning electron microscopy images showed that, the sample consists of 3 layers, arranged in the order that, the top is painted layer, the middle is white layer and the third is rougher, larger and thicker than another layers. It is clear from the images that, there is a weakness in the sample structure, cracks, joints especially the priming of the preparation and the green pigment surface is irregular.

XRD analysis: the results obtained by X-ray diffraction showed the presence of Celadonite ferrous (K (Mg, Fe₂ +) (Fe₃ +, Al) [Si₄O₁₀] (OH)₂). It is pigment material for green, which was not Egyptian synthetic green as it was thought by researchers. The ancient Egyptian artist preferred to use some of the available earth materials in his local environment for painting in green. This is a fundamental change in the painting technique in this tomb. the results show the presence of Gypsum CaSO₄.2H₂O, resulting from the white preparation layer below the green layer.

The data obtained from scanning electron microscopy (SEM-EDS) revealed the presence of the following elements (Ca, S, Fe, Mg, K, Na, C, and O) indicating the availability of the basic elements of the substance (Celadonite ferrous). Also, the results indicated the presence of gypsum resulted from the soft preparation of the ground layer below the green layer. Moreover, the results indicated the presence of halite salt NaCl.

Dark Blue Pigment Sample.

The examination of dark blue sample was carried out using both the optical microscope, and polarized microscope. Cross section investigation showed the presence of large, thick and coarse layer applied over a white plaster, the layer characterized by the presence of bright blue crystals tend to dark and luster. (Fig. 4).

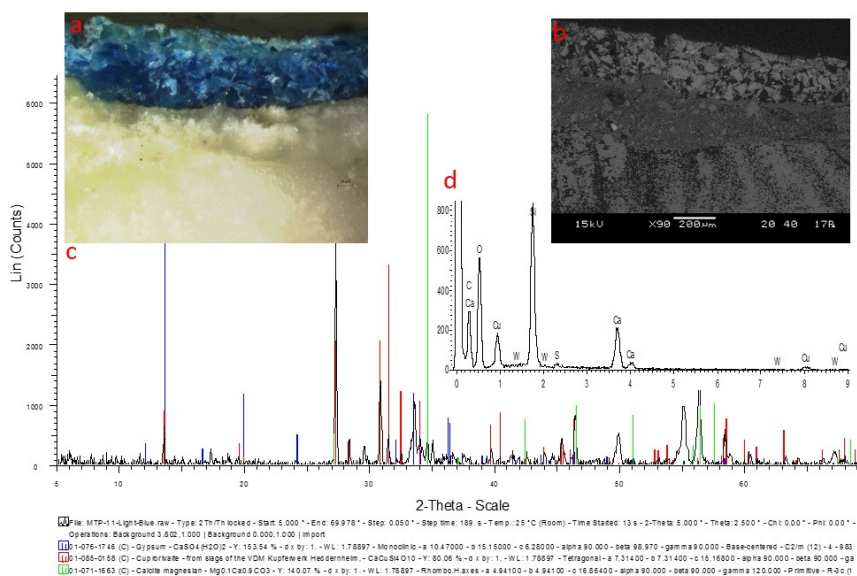


Fig. 4. a) Cross section investigation by optical microscopy of Dark Blue sample. b) Scanning electron microscopy of dark blue one showed that blue color is a very thick layer, the number of the layers are four layers. c) XRD pattern of dark blue painted sample indicated the presence of (cuprorivaite), also known as calcium copper silicate (CaCuSi₄O₁₀ or CaO CuO (SiO₂)₄) which is the main component of the Egyptian blue color plus the presence of gypsum which coming from the fine preparation layer. d) SEM/EDS pattern confirmed the presences of Cu, Ca, Si as main elements of (cuprorivaite), the elements Ca, O, S as main elements of Gypsum and Calcite.

Scanning electron microscopy of the blue sample clarified that, it consists of four consecutive layers.

The first layer is called rough preparation ground, it is the first layer placed on the wall of tomb to hide the defect and fill the gaps and cracks. This layer is characterized by its large thickness and rough granules, because the components and materials in the structure are characterized by the large size of granules.

The second layer is called soft ground, is less coarse and thicker than the coarse layer, placed to make a flat surface, the size of the granules of its constituents is smaller and less

rough. Then, the third layer which is the plaster layer, is thinner than all layers and is characterized by a smooth, flat surface, of white color applied to receive the painted layer on it. The fourth layer is blue painted which characterized by its thickness as well as large grains and also appear to have some deterioration and lost parts and the separation of the other parts.

XRD analysis: the results of the study of this samples by of X-ray diffraction indicated the presence of (cuprorivaite), known as calcium copper silicate ($\text{CaCuSi}_4\text{O}_{10}$) or $\text{CaOCuO}(\text{SiO}_2)_4$ which is the main component of the Egyptian blue, it is known to researchers as the first synthetic pigment. The ancient Egyptian artist was able to manufacture and use them in painting to have different shades of blue, other results indicated the presence of both gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and Calcite (CaCO_3), as a result of the preparation layer under the blue painted layer.

SEM-EDS analysis: the analysis was carried out using scanning electron microscopy with the elements analysis unit, indicated the following elements (Ca, Cu, and Si) which are the three main elements in the composition of the Egyptian blue. The results indicated the presence of elements (Ca, and S), which refers to the presence of gypsum, originates from the plaster as well as the presence of elements (Cl, and Na) that indicated the presence of halite salt.

Light Blue Pigment Sample.

The light blue sample was examined using optical microscopy, the image showed that, the pale blue crystals (large crystals) were $75.61 \mu\text{m}$ in length and approximately $24.70 \mu\text{m}$ wide and the height was approximately $15.99 \mu\text{m}$. these crystals tend to pale blue light

The pictures showed that, blue crystals were immersed in the preparation ground layer, other examination indicated that most of the crystalline body appears to be completely immersed in the plaster preparation layer (Fig. 5).

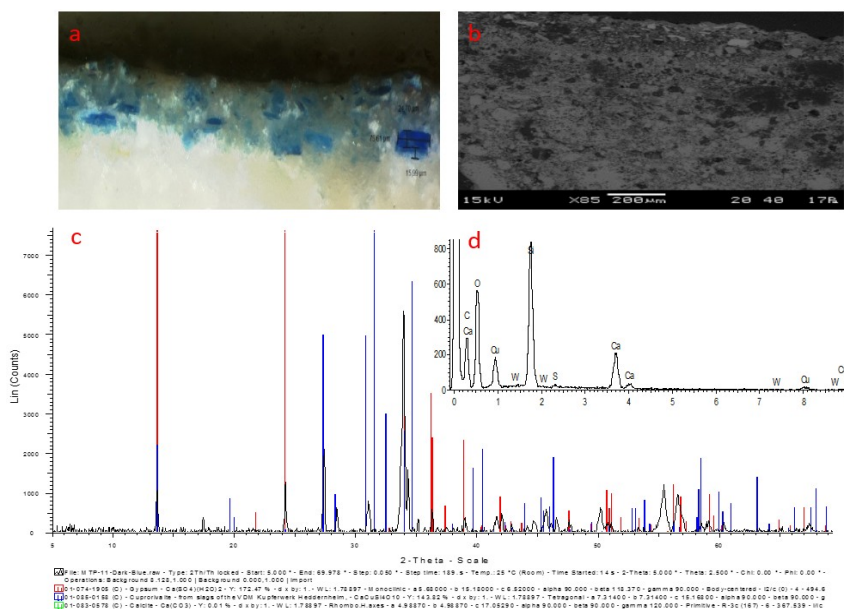


Fig. 5. a) Cross section investigation with optical microscopy of Light Blue sample showed that the pale blue crystals (large crystals) were $75.61 \mu\text{m}$ in length and approximately $24.70 \mu\text{m}$ wide and the height was approximately $15.99 \mu\text{m}$. b) Scanning electron microscopy of light blue one showed that light blue color is a very thick layer and the numbers of the layers are four layers. c) XRD pattern of light blue painted sample indicated the presence of (cuprorivaite), also known as calcium copper silicate ($\text{CaCuSi}_4\text{O}_{10}$ or $\text{CaOCuO}(\text{SiO}_2)_4$ which is the main component of the Egyptian blue color plus the presence of Gypsum which coming from the fine preparation layer. d) SEM/EDS pattern that confirmed the presences of Cu, Ca, Si the main elements of Egyptian blue, also the elements Ca, O, S as main elements of gypsum and calcite.

The images by scanning electron microscopy indicated that, there are large rough crystals appear to be immersed in the plaster preparation layer. Another layer of preparation is rougher, seems to be suffering from drought, weakness and damage.

XRD analysis: analysis by X-ray diffraction of this sample indicated the presence of (cuprorivaite), ($\text{CaCuSi}_4\text{O}_{10}$) which is the main component of the Egyptian blue. The results also indicated the presence of gypsum and calcite, which are the components of the plaster preparation layer.

SEM-EDS analysis: the results of the Scanning electron microscopy with the elements analysis unit indicated the presence of the following elements (Si, Cu, and Ca). These three main elements are the main components of the Egyptian synthetic blue pigment. Other results indicated the presence of elements (Ca, S, and O) referring to the presence of gypsum and calcite, which are the components of plaster layer.

Yellow Pigment Sample.

The yellow pigment sample was investigated using both by the optical microscope, and polarized microscopy. The images showed that, there was a soft, light yellow mixed with red granules, there is a loss of yellow pigment in some places, the picture indicated a kind of drought and obvious damage to the surface of the sample (Fig. 6).

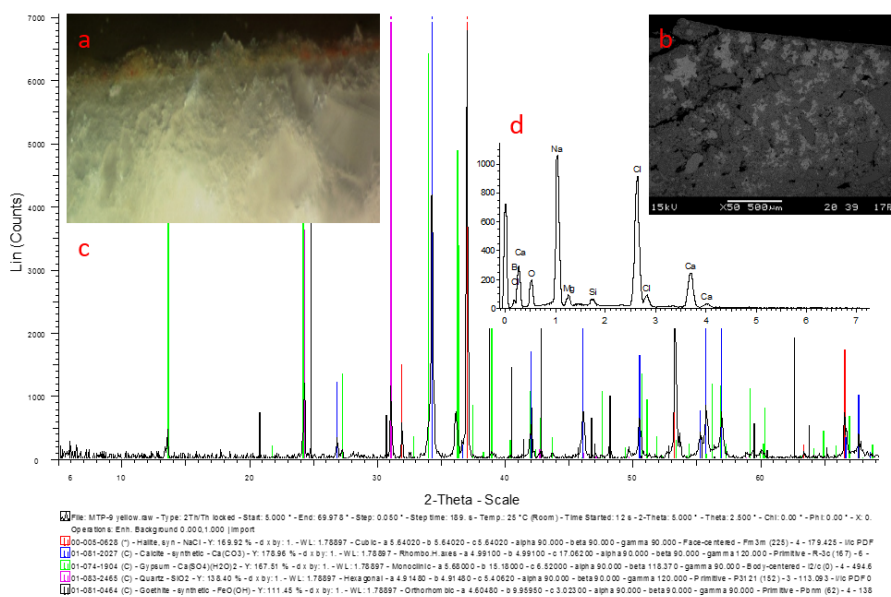


Fig. 6. a) Cross section image with optical microscopy of the yellow color sample showed a soft, light yellow coat with a mixture of red granules, there is a loss of color in some places and the image indicates a kind of drought and obvious damage to the surface of the sample. b) Scanning Electron Microscopy investigation of the sample indicate a loss in the color on the surface, many pools or calcinations or many white deposits, cracks in the ground layer extending to the colored surface. c) X-ray analysis pattern indicated the presence of goethite ($\text{FeO}(\text{OH})$) as a basic component of yellow color with calcite and gypsum forming the preparation material. Also, the results indicate sodium chloride salt is present. d) SEM/EDS analysis confirmed the presence of elements Fe, O as Goethite and hematite which were used for yellow pigment and the presence of Na, and Cl as halite salt.

The yellow painted sample was studied and scanned by scanning electron microscopy where the examination pointed to a lot of important observations such as the presence of a light and very thin painted surface which has been applied directly on preparation ground layer, there is no other layers. A loss in the painted tape, many pools or calcinations, many white deposits with the ground of preparation, presence of cracks in the ground layer extending to the painted

surface, dryness of the preparation layer and the separation of parts of the paint were also detected.

The study and analysis of the yellow painted sample was carried out by the X-ray analysis, which indicated the presence of goethite (Fe O(OH)) as a principal component of yellow pigment with calcite and gypsum. The results indicated present of sodium chloride salt.

Black Pigment Sample.

The black painted sample was performed by the optical microscope. which showed that painted layer is a mixture of black carbon and red pigment, they have been mixed together to obtain a certain tone of black color. This is one of the unique painting techniques in this tomb, where the ancient Egyptian artist preferred to mix together more than one pigment to obtain a distinctive, and characteristic pigment. From the examination can noticed black and red pigments granules mixed together to obtain a distinctive black pigment which was applied over the soft white preparation layer (Fig. 7).

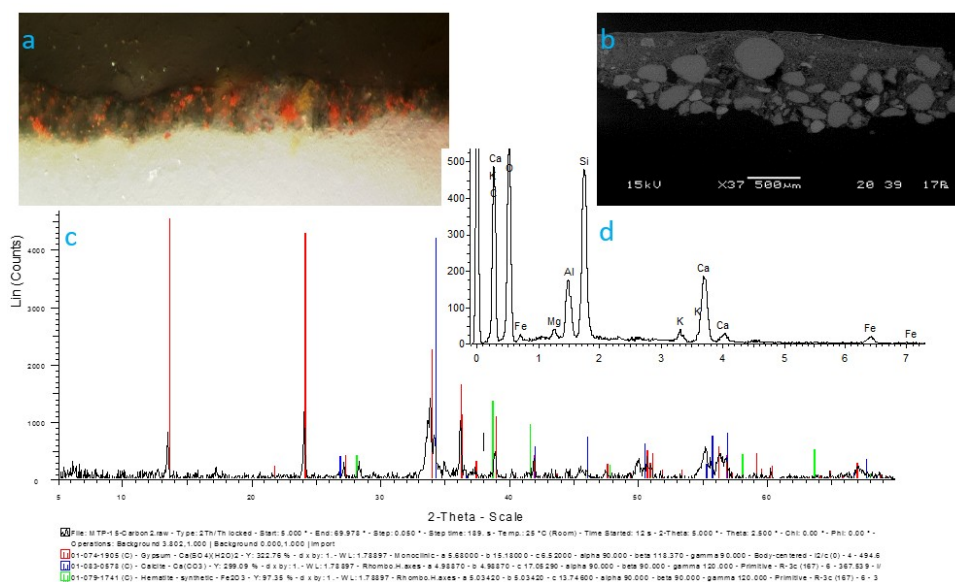


Fig.7. a) Optical microscope cross section image shows a color layer is a mixture of black and red, mixed together .b) scanning electron microscopy examination shows the presence of a superficial color bar is characterized as irregular, sample consisted of three layers, painted layer, followed by the soft and the smaller preparation ground layer followed by the preparation one which appears more rough and larger than the previous layers. c) X-ray analysis pattern showed hematite (Fe₂O₃) was the main component of the red color, as well as the presence of calcite and gypsum, which are the main constituents of the preparation. d) analysis by the Scanning electronic microscope equipped with the elemental analysis unit, the results indicated the presence of the following elements (Fe, O) a component of hematite the main component of the red color and also the presence of elements (Ca, S, O) the basic component of both Gypsum and calcite components of the preparation and element (C) carbon is the primary component of black color.

The black painted sample examined by the scanning electron microscopy showed the presence of a superficial painted bar which is characterized as irregular.

This sample consists of three layers, the painted layer, followed by the layer of the plaster- it looks white and is characterized by less thickness- followed by the ground one which appears more rough and larger than the previous layers.

XRD analysis: The black painted sample was analyzed by X-ray analysis, which indicated that hematite (Fe₂O₃) was the main component of the red pigment, as well as the presence of calcite and gypsum, which are the main constituents of the preparation ground layer.

SEM-EDS analysis: analysis by the scanning electron microscope equipped with the element's analysis unit, indicated the presence of the following elements (Fe and O) the main components of hematite (Fe_2O_3) and the red pigment respectively. also the presence of elements (Ca, S, and O) the basic component of both gypsum and calcite components of the ground preparation layer and element (C) carbon which is the primary component of black pigment.

Organic Medium

Samples were analyzed with FTIR to identify the organic medium which was used in painting process of painted reliefs.

The results indicated that, the Egyptian artist used egg yolk as organic medium (Fig. 8) in painting technique with pigments and the painting technique used is tempera method.

The results of coating material on the surfaces of painted relief referred to bee wax that maybe was used in previous treatments (Fig. 9).

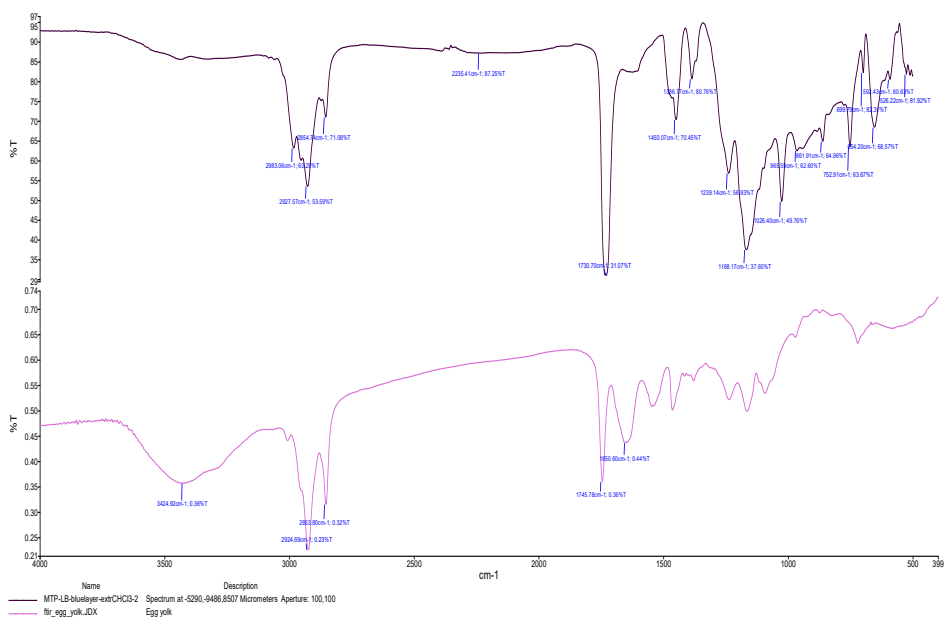


Fig.8. FTIR spectrum the result indicated that egg yolk was used as organic medium in the painting processes.

Ground layers

The results of painted samples examination using optical microscope and scanning electron microscope showed the use of a unique technique in the implementation of painted reliefs in the tomb such as applied a number of preparation layers ranging from two to four layers, different in number, grain size and thickness.

X-ray diffraction results of plaster layer indicated that, Gypsum was the basic component ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). Also, elements analysis unit results of SEM-EDS indicated that, the main elements are (Ca, C, O, and S) the main components of gypsum and calcite.

X-ray diffraction results of the second preparation layer indicated that, the main components were gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and calcite (CaCO_3). Also, elements analysis unit results of SEM-EDS indicated the existence of elements (Ca, O, S and C).

X-ray diffraction results of the third layer indicated that, the main components were Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and Calcite (CaCO_3). Also, elements analysis unit results of SEM-EDS indicated to the existence of elements (Ca, O, S and C).

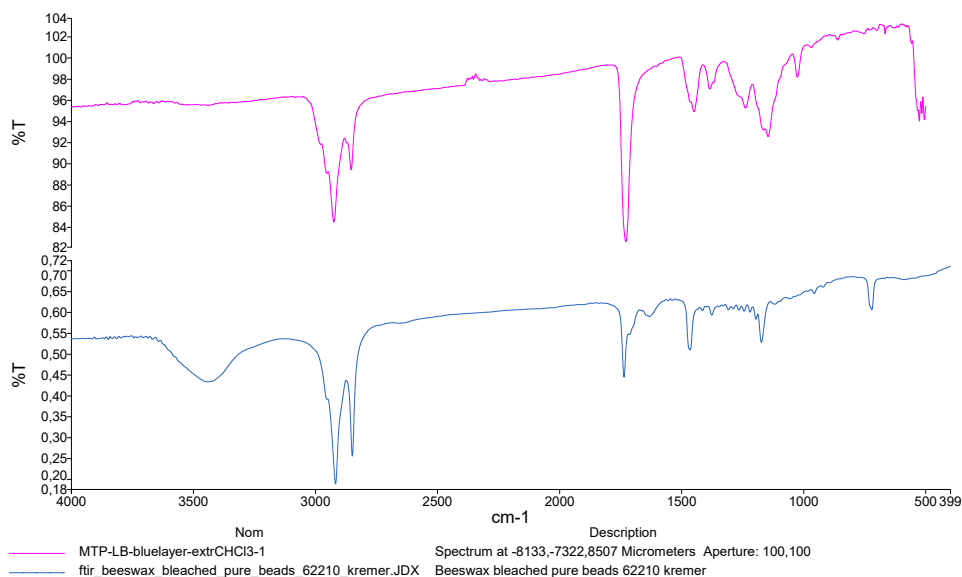


Fig.9. FTIR spectrum the results showed that there was a coating material was used as a coating in on the surfaces of painted relief it was bee wax and maybe it was used in previous treatments.

Discussions

Through the previous study of painted reliefs in the tomb of Pitosisiris, as well as the study of the current state, this study provided us with important information and results about the painted reliefs and its current state. These can help in drawing and developing a plan for the restoration and treatment for the Petosisiris tomb including inscriptions and painted surfaces.

The most important information gained is the identification of painting technique which was tempera style with egg yolk as organic medium. The study provided us with a set of data and very important results about the pigments materials used. These results showed some information about the techniques of implementation and painting techniques and the nature of these materials. The groups of tests and analyzes revealed the suffering and the current condition of these painted inscriptions in the tomb alongside with the structure of materials for painted reliefs. The results were as follows: The Egyptian artist used the style of tempera in the painting in the tomb and in its inscriptions, egg yolk medium was used as an adhesive and intermediary for pigments.

Moreover, the result showed that, hematite (Fe_2O_3) was used as a pigment for red paint. Hematite was available substance in the Egyptian environment. The results showed also that, the use of goethite (Fe OH) as painting material for the yellow paint, which can be added to hematite sometimes to give a clear hue of the color or bright yellow color. Goethite pigment materials was available in Egyptian environment. The blending method is distinctive style in Egyptian techniques in paintings.

The results indicated the use of Egyptian blue in painting even as a light and dark blue pigment. It is the first synthetic pigment in ancient and Egyptian history, where the Egyptian artist was able to prepare, milling and mix it with organic intermediary and paint. The size of granules ranging from large to small crystals.

The results pointed to using carbon in black color, with addition of hematite to carbon and well-mixed. This is a unique painting technique in the ancient Egyptian painting techniques in order to obtain a certain degree of black.

The results of green pigments study indicated the using of celadonite ferrous ($\text{K (Mg, Fe}_2\text{+) (Fe}_3\text{+, Al) [Si}_4\text{O}_{10}\text{] (OH)}_2$). It was found that, the green pigment is a mixture of a group

of metals and soil oxides, and not malachite or Egyptian green or green earth as it was known in the ancient Egyptian painting techniques.

Conclusions

Through the results of the examination and analysis of the painted reliefs components from the tomb of Pitosiris, as well as study the current state and condition of the painted inscriptions, it is clear that, the painted inscriptions in the tomb were made by Tempera technique using the yolk medium as an organic media for painting process.

The painted materials used are the same as those used throughout Egyptian history such as hematite for red pigment, goethite for yellow, carbon for black and Egyptian blue for blue color. But the Egyptian artist used Celadonite ferrous - which formed of a group of metals and soil oxides- to get green pigment. In this tomb the Egyptian artist preferred to use the technique of mixing pigments together to obtain different color grades.

The results of the study also confirmed the presence of sodium chloride salts in the components of tomb inscriptions. As for the current state of the tomb and its painted inscriptions, it is clear that, it is suffering from severe drought and the appearance of cracks, as well as loss in some areas and the separation of others.

The surface condition is very poor where weakness and debility that requiring rapid intervention for first aid and treatments, followed by a series of restoration and consolidation process.

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