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CHARACTERIZATION STUDY OF A ROMAN STUCCO DEATH MASK FROM THE NATIONAL MUSEUM OF EGYPTIAN CIVILIZATION Mohammed S. A. KHEDR*, Mona F. ALI¹, Abdullah M. A. KAMEL², Manal A. A. EL GHANAM³



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Abstract

This research aims to study a Roman Stucco death mask from the National Museum of Egyptian Civilization (the case study of the Stucco death mask dating back to the Greco-Roman era.). This mask consists of three different layers (a layer of linen threads, a layer of gesso, and a layer of painting and gilding. The mask has some deterioration aspects, such as separations, cracks, discoloration, brittleness in the layers and losses in some painted parts. Some examination and analytical methods, such as USB digital microscope, X-ray diffraction (XRPD), Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), portable x-ray fluorescence (XRF) and scanning electronic microscope with energy dispersive X-ray analysis (SEM.EDX) were used to identify the implementation techniques of the stucco mask, chemical composition, and deterioration aspects.

Some results were gypsum and calcite are the essential components of the studied stucco mask. Some pigments mixtures were used for painting the stucco mask such as pigments of Huntite, Realgar, Goethite, and Hematite were mixed to give the beige color degree of the face part of the studied stucco mask. As well, as the pigments mixture of Realgar, Hematite, Lazurite and Azurite to give the mixture of the violet color. Vine black pigment was used to give the dark color degree of the hair, and the stratigraphic sequence of the gilding layers which represents the following layers as the first layer contain calcite which used as a base layer for the gilding process, the second layer contains organic adhesives as this layer consists of red to yellow granules of iron oxide from hematite mixed with an organic medium and the third layer contains the gilding paper. It was also noted that animal glue was used as a medium for all pigments.

Keywords: Roman stucco masks, gypsum, Deterioration, pigment, violet color, Gilding, Raman spectroscopy, National Museum of Egyptian Civilization (NMEC)

1. Introduction

The ancient Egyptians thought that after death, people would come back to life and live forever. The body of its owner can be found by looking at its unique features. This is why funeral masks were so important since the soul was placed on the mummy and became a reflection of the owner's face [1-2-3]. Hence the importance of the funeral masks where it is placed on a mummy and become an expression of its owner's likeness, including their features [4]. The term 'mummy mask' has been coined by archaeological research in order to separate one kind of mummy adornment, a three-dimensional object that covers the face and some part of the mummy's

chest, from other objects with a different shape [5]. The main point of the mummy masks was to keep the dead person's head and face features [6]. Funerary masks were used on the heads of Egyptian mummies as an alternative to statues, since the first Intermediate Period (2160-2025 BC) [2], The first mummy masks appeared at the end of the Old Kingdom (about 2686-2181 BC). The Greeks in the Ptolemaic era followed the same Egyptian approach in using funerary masks, especially in the way of decorating their mummies without any significant change. Mummy masks and cartonnages began to be used on a wide scale by the Graeco-Roman times [7].A new custom arose in the early first century AD that replaced the Egyptian-style face mask for coffins and mummies and spread in Egypt during the Roman

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era, the phenomenon of providing mummies with a picture that shows the features of the deceased person, and sometimes this picture was a funeral mask, or drawings on wooden boards [8] and the funeral masks belonged to women, men and children of all ages [9-7], and it became expressive of its owners with their special features [10].Romanian art took citizens as a model of art, which expresses their features in an honest and realistic way [11]. When the Romans settled in Egypt, they did not understand to some extent the ambiguity of the belief Egyptians, but they were only affected by it, as they also became interested in the body in addition to the image that bears personal feature [8], The mummy masks were placed over the upper part of the deceased's body, and covered the head and chest. They were intended to depict the deceased in the best possible shape hoping that they could inspire them after death [9]. Roman death masks called "imagines" were actually wax models impressed directly on the face during life, and they bore a remarkable likeness to the person. Displayed during the funerals of the elite, they served as a link between the present and the past and were meant to inspire attendees to patriotic virtue [12-13]. The earliest masks date to the first century BC to the first century AD from Egypt. This practice spread through trade into the ports of Rome, simultaneously influencing the Roman plaster death masks with the elite images[14]. Roman mummy masks can consist of a whole variety of materials, the most common being cartonnage and plaster. Tongue masks are made of gypsum plaster, with varying additives. Helmet masks consist of cartonnage of several layers of linen that are glued together and receive a plaster finishing on the surface, stucco masks are created by pouring plaster into facial feature-matching moulds (either the same mould or very similar moulds) that are fashioned like faces. The plaster layer is removed and the panels are inserted after a brief drying period. Additional features, such as hair, ears, jewellery, clothing, and other accessories, are freely moulded and afterward painted or gilded. Only a small number of the pieces also have hair that has been shaped into a certain shape [15]. Masks of the dead played a distinct role in Egyptian art since the Old Kingdom and continued until the Greek and Roman eras [16]. Masks of Graeco-Egyptian type first came to use in later Ptolemaic times, but appearances could be strongly individualized and Roman fashions of hairstyle, dress and jewelry were followed to varying degrees[17].

Stucco is a material obtained by mixing binder, water and specified additives in different proportions depending on the available materials and the different techniques to obtain many stucco items [18]. Gypsum is one of the most important materials used in the

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implementation of stucco masks as well, lime (Ca (OH)₂) played an important role as a binding material in the implementation of stucco masks [3-16]. Through archaeological studies of the primary colors used in stucco masks, it became clear that the white color (either it is mostly calcium carbonate, which is limestone powder, or gypsum), which is used in a large proportion in painting the faces of masks [19]. Black color (a fine powder of soot that is left by the surfaces of vessels when food is cooked and on the walls of ovens), used in hair and human eyes. Alfred Lucas mentioned thatPink and light pink were made by mixing red and white colors as they wereused in coloring lips and making wreaths. Brown color was also used to color the skin [3].Gilding has been known in Egypt since ancient times (Naqada civilization. (3100-4000 BC) [20] As well, were affected by covered wooden artifacts such as coffins, statues, chests, and various pieces of furniture with gold leaves. Gilding continued throughout the Greek, Roman, Coptic, and Islamic eras, with different types of gilding such as water gilding and oil gilding [21, 22]. Some stucco masks were completely gilded as the gilding process had a religious, symbolic value and played a great role in the belief of the ancient Egyptians, especially for the deceased [33]. In other cases, stucco masks were partially gilded especially. On the face area using very thin gold leaves, it was known that the gilding layer preserve the underlying layers from corrosion and disappearance [23].Stucco masks were implemented and shaped by some methods [24] as will be listed: The first method: used a soft papyrus paste that was applied on the face of the deceased by gentile pressing to take the features of the face, then this layer was strengthened by applying a layer of plaster. Finally, the mask was attached to the face of the deceased. The second method: Stucco mask was implemented by applying a thin layer of damp cloth. This layer was reinforced by applying soft layers of stucco, after drying and finishing these layers, the mask was de-molded and fixed on the mummy. The third method: In this method, stucco masks were made by casting the masks in clay moulds by hand which was confirmed by the presence of fingerprints [16]. Then distinctive details were made in the Stucco using a spoon or a knife, and then the mask was covered with a thin layer of stucco so that all the details were visible. As for the rest of the other parts, they were made by hand separately and then added to the head, namely the ears and eyes. Eye grafting spread as an attempt by the artist to give the soul to the deceased, and it was a prevalent characteristic from the first century until the second century AD. At the end of the discussion about the parts of the stucco mask, it was necessary to point out the eye area, as it was distinct

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in the Greco-Roman as the manufacturer in this era would install it inside the eye socket and not color it in order for it to become real. So, the eye was implemented in two ways as follow: The first method: the eye was made of opaque white glass, while the human eyeballwas made of black glass, and this method was done in the Ptolemaic era. The second method: is drawing the human eye in black on a stucco base, and then covered with a thin layer of glass; this method dates back to the Roman era. Then the artist began to make ornaments, earrings, and hairdos separately and stick them on the mask after drying of plaster, then the mask has painted in different colors. Finally, after forming the mask, it is fixed on the mummy by adding a thin layer of plaster that allows the mask to be enclosed in the mummy rolls [3].

2. Object studied

It is a funerary mask that dates back to Roman Period, from Egypt. The mask was implemented of painted and gilded stucco. The height of the piece is 21 cm; the width is 20 cm as shown in Figure [1].



Figure (1) shows the studied stucco mask, point (A) shows the front of the piece, point (B) shows the backside of the stucco mask.

As shown in Figure [2] (A) point (A1 shows the stucco substrate which was strengthened by linen fibers and it is obvious that it was implemented in the next step as shown (point A2). Point (A3) and Figure (2B) show the hollow casting of the stucco head or mask. The figure (2 C and 2 D) shows the painting layers and the overlapping of them. The painting layer was expected to be implemented by inorganic pigments which are characterized by their stability towards many deterioration factors [25,26]. The reason for choosing inorganic oxides is likely precisely because of their stability and good resistance to UV rays and light, as well as not being easily affected by various weathering factors [26]. Colored materials are classified according to their uses, color grades, and degree of stability [27] and organic media are added to the color materials used in coloring stucco masks, including (glues-animal glue-white and egg yolks-wax), and the colored materials are placed in the form of an emulsion

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[28].The gilding is an ancient technique that uses a gold leaf or imitation of it made of silver or other metals applied over the preparation layer, called bole of red-brown or yellow ochre color, based on minimum lead or colored earth. The leaf is applied with a suitable adhesive based on animal glue or other natural resins, as shown in the various points in Figure (2) at the points (E-F)and based on the use of the least possible amount of lead or earth colorants.

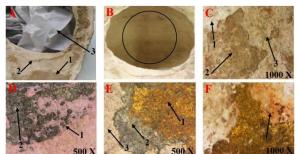


Figure (2) illustrates the manufacturing technique as shown in the following points, where points (A-B) show the technique of making the mask, points (D-C) show the phenomenon of colour overlap, and points (F-E) also show the technique of the gilding process.

3. Experimental methods.

3.1 Investigation and analysis of the case studies.

It is certain that the samples that were taken from the artifact were very small and collected from parts which suffer from deterioration and have been subjected to examination and analysis as shown in Figure [3] and Table 1, a detailed map of the samples.All the details and the deterioration aspects of the studied stucco mask were recorded using a digital camera: Nikon Digital camera D3200, and fitted with a Nikon Nikkor 18-55. The present study mainly aims to characterize and identify the layers of stucco, painting and gilding of the studied roman stucco mask. by many examination and analytical techniques, such as portable USB Microscope, X-ray Diffraction (XRD), Raman spectroscopy, Fourier Transform Infrared spectroscopy (FTIR), Scanning electron microscopy combined with energy dispersive X-ray (SEM-EDX), and X-ray Fluorescence (XRF).

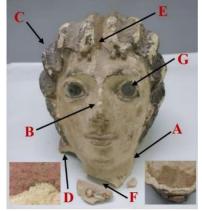


Figure (3) shows an illustrative map of the places of the selected samples from the studied stucco mask.

 Table (1) Illustrates the Examination and analysis of the selected samples.

symbol	Sample type	Examination
symbol	Sample type	Analysis
Α	The body of	USB, XRD,
	the mask	SEM-EDX
В	facial peels	USB, XRD, RMA,
		SEM-EDX
С	Black color	USB, XRD, RMA, FTIR
		SEM-EDX
D	Violet color	USB, XRD, RMA, FTIR,
		SEM-EDX
E	Gilding	USB, XRD, FTIR,
		SEM -EDX
F	fabric	USB, SEM-EDX
G	The inserted	USB, XRF
	eye	

3.2 USB Digital microscope.

Some samples of the studied stucco mask were examined by a portable digital microscope (model PZ01- made by Shenzhen supereyesco. Ltd, China) with a magnification ratio starting from (1000X: 500X) in order to examine and study the morphological features of the samples and also for separate areas of previous restorations. The observations made by the USB digital microscope revealed the following results resumed in Figure [4], and Table (2).

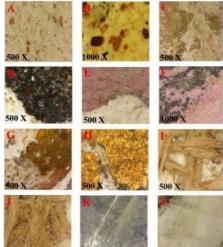


Figure (4). shows the observations made by USB digital microscope Table (2). shows the USB digital microscope examination results

results.	
Figure	USB digital microscope results
Fig.4(A)	Examining the mortar mixture that makes up the
(500X)	stucco mask and the fillers that it contains.
Fig.4(B)	An examination shows the size and percentage of
(1000X)	the granules added to the mortar mixture that
	makes up the stucco mask.
Fig.4(C)	Examination shows the yellow-colored substance
(500X)	and its appearance in the form of layers in yellow
	tones.
Fig.4(D)	Examination shows the black-colored substance,
(500X)	and it is noticeable that it has granules of different
	colors that tend to be blue, and the preparation
	floor below it, which is characterized by a degree
	of softness differs from the mixture that is the main
	component of the stucco mask.

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Fig.4(E)	An examination showing the colored substance,
(500X)	which is characterized by a violet color.
Fig.4(F)	An examination shows an overlap between the
(1000X)	colors, as the examination shows that there is a
. ,	black color layer on which a violet color layer has
	been placed.
Fig.4(G)	Examination shows the stratigraphic structure of
(500X)	gilding and what it contains from a white ground,
	then a dark layer, and then the gilding layer.
Fig.4(H)	Examination shows the layer of gilding and how it
(500X)	suffers from loss
Fig.4(I)	Examination shows a layer of threads under the
(500X)	stucco mask.
Fig.4(J)	Examination shows the severe damage and
(1000X)	splitting that these threads suffer from, and also
	confirmed that when examining them with SEM.
Fig.4(K)	Examination showing cracks and visible damage in
(500X)	the black graft in the inserted eye.
Fig.4(L)	Examination shows cracks and clear pitting in the
(500X)	white graft in the inserted eye.

Examining the samples of the object studied, it was found that the body of the mask contains a mortar mixture to give it the hardness. Also, the manufacturer used some pigments such as yellow to color the facial peels, black pigment to color the hair and revealed thelayer of threads under the stucco mask which suffer from deterioration. The Also the USB microscope revealed the gilding layer also showed some cracks which appeared in different places through the studied mask. Also, there were cracks and visible damage in the black graft in the inserted eye.

3.3 X-ray diffraction (XRD)

Five samples of the studied stucco mask were studied and X-ray diffraction patterns were obtained through the use of milled samples by using the diffractometer model: PW 1480 Netherland, operated at 35kv, using a Cu and Ka radiation wavelength of 1.54056Å. The reference database used for matching is PDF4. Scanning speed 0.04/sec., start position 2θ =50 to end position 2θ =700.

The semi-quantitative analysis of the mineralogical components resultsof the studied samples are resumed in Table 3 and The XRD patterns of the samples are shown in figure [5]. XRD analysis of sample Aindicated that gypsum (CaSO₄.2H₂O,74,5%) wt) is the main component of the mummy mask stucco. The widespread use of gypsum in the implementation of stucco masks may be related to its good technical properties such as; suitability for copying three-dimensional art works with great accuracy, validity, cheapness, good workability and its easy manufacturing process[16], The identified Anhydrite (CaSO₄, 1,6% wt) may relate to natural impurity in gypsum rocks, or it may have been added ground anhydrite to accelerate the setting process; or it may also have resulted from exposure gypsum component for varying temperatures and humidity

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[31]. So, the presence of gypsum indicates that it is the main component of the stucco mask.

-The presence of calcite (CaCO₃, 18,4% wt). It may be that slaked lime had converted to calcite after a reaction with carbon dioxide and water. Addition of lime to gypsum, as a matter of fact, enhances the durability of gypsum products towards cracking, separations and deterioration [32], also the presence of dolomite (CaMg (CO₃)₂, 1.6% wt) as it represents a form of calcite. The low ratios of quartz (SiO₂, 2.0% wt), periclase (MgO, 0.5% wt), (CaO, 0.7% wt) confirm that all these components were added to the mixture to give it the hardness. and the presence of halite (NaCl, 0.8% wt) may represent one of deterioration aspect.

Sample B, it became clear that the beige color which was applied on the mask face was made from a pigment mixture consisting of a white basic pigmenting substance of Huntite (Mg₃Ca (Co₃)₄81.5% wt) with a small percentage of red pigment realgar (As₂S₂, 4% wt), (Arsenic (Ii) sulfide) and hematite (Fe₂O₃, 1.2% wt) [33-34]; as shown in Table 3 which is as follows:

-The presence of Huntite as a white inorganic pigment, and its high appearance, which confirms that it was the main component of the color mixture, and also the presence of Calcite (CaCO₃, 8.8% wt) and gypsum (CaSO₄.2H₂O,4.5% wt), where their presence indicates that it is likely to be one of the components of the white color, as they were used to give the white color [33-34] or their presence in this sample as a residual from the mask stucco.

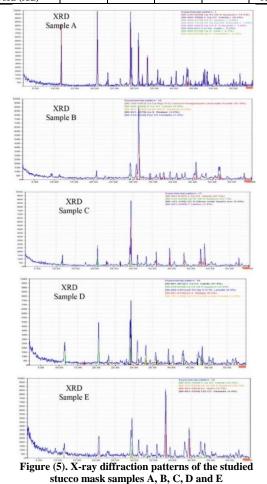
-The presence of realgar (As_2S_2) and hematite (Fe_2O_3) as hematite which is a mineral mixture that contains, in its pure form, about 95% of ferric oxides with other impurities such as silica SiO₂ and aluminum oxide Al₂O₃, so their presence indicates that they are red inorganic-colored materials pigments [33-34]. So, these results confirmed that the manufacturer mixed the white and red colors to obtain the beige color for coloring the face of the mask.

Sample C, revealed that the manufacturer made a color mixture consisting of a black basic of carbon which represented the vine black color (C,3.8% wt) which was used for hair painting. While the detection of gypsum (CaSO₄.2H₂O, 23.1% wt) and calcite (CaCO₃, 67.5% wt) may be a residual from the mask structure or mixed with the black color to lighten it, as shown in Table 3.

Sample D, of the purple color shows the detection of hematite (Fe₂O₃, 5.0% wt) and realgar (As₂S₂, 6.2% wt) as red pigments and lazurite (Na₈[Al₆Si₆O₂₄] Sn, 8.0% wt) as a blue pigment justifies the purple color.

Table (3). Shows the approximate XRD analysis results of the
studied roman stucce mask

studied roman stucco mask.											
Sample	D	Е									
Gypsum	74,5%	4.5%	23.1%	33.6%	38.1%						
$(CaSO_4.2H_2O)$											
Calcite (CaCo ₃)	18,4%	8.8%	67.5%	47.4%	43.3%						
Quartz (SiO ₂)	2,0%		5.6%								
Dolomite	1,6%										
(CaMg (CO ₃) ₂)											
Anhydrite	1,6%										
(CaSO ₄)											
Halite (NaCl)	0,8%										
Lime (CaO)	0,7%										
Periclase	0,5%										
(MgO)											
Huntite		81.5%									
$(Mg_3Ca(Co_3)_4)$											
Realgar (As ₂ S ₂)		4.0%		6.2%							
Hematite		1.2%		5.0%	4.9%						
(Fe ₂ O3)											
Vine Black (C)			3.8%								
Lazurite				8.0%							
(Na8[Al6Si6O24]											
Sn)											
Gold (Au)					13.7%						



Sample E, it turns out that the manufacturer made a stratigraphic sequence of the gilding layer, As shown in Fig. [6](Point 1 gilding paper, point 2 organic adhesives, point 3 calcite, point 4 stucco) the high ratio of calcite (CaCO₃, 43.3% wt) confirms its use as

a base layer for the gilding process. The presence of hematite (Fe₂O₃, 4.9 % wt) in the sample with a high percentage, refers to that the manufacturer applied a layer of red color over the prepared base layer to receive the gilding layer. This layer consists of red to yellow granules of iron oxide from hematite mixed with an organic medium [36]. One of the reasons for the damage of the gilding layer and the preparation layers is the heterogeneity between the organic and inorganic layers of preparation, which results in a difference in the rate of expansion and contraction between organic and inorganic materials [37].

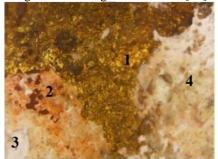


Figure (6) (USB 500 X) shows the stratigraphic sequence of the gilding layers. (point 1: gilding paper; point 2: organic adhesives; point 3: the preparation layer; point 4: stucco)

3.4 Raman spectroscopy.

Raman spectroscopy is widely regarded as one of the most effective techniques for analysing artefacts of cultural heritage. It enables the expeditious identification of compounds at the micrometre scale without the need for sample preparation. Three pigment samples beige, black, and violet colors were studied by BrukerSenterra II Raman confocal Microscope. The measurements were made with a 785 nm laser with a power of 10 microwatts. The analysis was performed in the Projects Sector at the Ministry of State for Tourism and Antiquities, Egypt. Figure [7] shows the samples which analyzed with Raman spectroscopy on the studied stucco mask.

By analyzing the samples (B-C-D) by using Raman, the following compounds were identified: organic Animal glue and some of the inorganic compounds as shown in Table 4.

Raman Shifts	Assignment	Sample B	Sample C	Sample D	References
1245	Animal glue (amid III)	1249	1245.30	1245.30	[38]
950-800	Animal glue (v(COC))	948.60-905.37-853.81			[38]
815	Animal glue (C-C) stretching)	825.02			[39]
853	Animal glue (C-S stretching)			849.06	
797	Animal glue (N-H wagging)	791.70			[39]
603	Animal glue (amide VI N-H deformation)			609	
1166-778	Quartz (SiO ₂)	1155-780.72	1158.97		[40]
	Quartz (Si-O-Si asymmetric stretching)		1065.64		[45]
839-808	Quartz (Si-O stretching)	841.97-802.16			[41-42]
1123-315-270	Huntite(Mg ₃ Ca (CO ₃) ₄)	1123-312-276			[43]
1085	Calcite (CaCO ₃) (CO ₃ stretching)	1083.41	1088.06	1088.06	[44]
712	Calcite (CO ₃ deformation)	709.55		702	[44]
998-676	Goethite (FeOOH)	1001.07			[45]
		688.12			[46]
1003-658-655	Gypsum (CaSO ₄ .2H ₂ O)	1001.07-645.38-			[45]
	(O-H bending out of plane)	630.52			
1016-627-150	anhydrite (CaSO ₄)	630.52-144.21	1024,93		[43]
610-410	Hematite (Fe ₂ O ₃)	616,43-412			[47]
	(Fe-O symmetric bend)				
191-182	Realgar (As_2S_2)	191.86-182.53			[48]
353-220-182				355-220-184	
1585-650:950	Vine Black (C)		1562.58-		[43]
			849.06		
919	Animal glue (C-C vibration)		925.21		[39]
1193	Gypsum (SO4 ⁻²)		1190.64	1190.64	[49]
1007	Gypsum (SO4 symmetric)		1006.16	1006.16	[44]
1565	Animal glue (v(COO)			1562,58	[50]
	asymmetric and symmetric				
1157-128	Quartz (O-Si-O bending)			1158.97 -124	[51]
1096-803	Lazurite (Na8[Al6Si6O24] Sn)			1105.03-805	[52]
1024-928-762-732	Azurite (Cu3 (OH)2 (CO3)2)			1024.93-	[52]
				925.21-764-729	_

Table (4). Shows the Raman shifts and assignments detected in the sample B, C and D of the object studied.

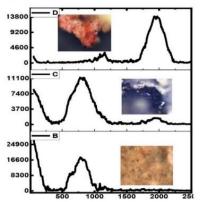


Figure (7). showed the samples which analyzed with Raman spectroscopy on the studied stucco mask, B Raman spectra of beige painted areas, C Raman spectra of black painted areas, D Spectra acquired on the purple painted areas.

All Raman analysis results confirmed XRD results that the three samples B, C and D contain calcite, gypsum that represent the white pigment as bands of gypsum as a white pigment and calcite from the under layer were detected also confirmed that the manufacturer mixed more than pigment to obtain mixture of pigments. But Raman's analysis added a new result that the manufacturer used an organic animal glue to all colors as well concerning sample B it revealed the presence of Goethite (FeOOH) in the color mixture to obtain the beige color for coloring the face of the mask and concerning sample D it revealed the presence of Azurite (Cu3 (OH)2 (CO3)2) to the color mixture to obtain the violet pigment.

3.5 Fourier Transform Infrared spectroscopy (FTIR)

Three samples were analyzed by using JASCO FT-IR-460 PLUS Spectrometer (Wave length Number Range: 4000 to 400 cm-1 at a resolution of 4 cm-1, Single Beam). Conducting that analysis aims to confirm and detect the organic medium used in the pigment layers and to determine the damage rate of these compounds [30].

The FTIR Analysis results of the studied samples of the roman stucco mask, allowed the characterization of samples taken from black hair sample C, the purple color sample D, and the bole layer of gilding sample E.

Figure. [8] and Table 5 showed the strong band centered around 1421 cm^{-1} , 1439 cm^{-1} , and 1443 cm^{-1} respectively refers to the characteristic of the C–O asymmetric stretching of carbonate, together with a weak band around 873 cm^{-1} , 878 cm^{-1} , 874 cm^{-1} of the bending vibration of the carbonate and the band at 716 cm^{-1} , 712 cm^{-1} , 714 cm^{-1} respectively, symmetric CO₃ deformation, demonstrated the presence of calcite (CaCO₃).

Gypsum (CaSO₄.2H₂O) was also observed in the spectrum. The strong band centered around 1121 cm^{-1} , 1121 cm^{-1} , and 1144 cm^{-1} which splits into two components at around 1139 cm^{-1} and 1118 cm^{-1} , and the weak peak at 670 cm^{-1} , 674 cm^{-1} , 671 cm^{-1} are assigned respectively to the stretching and bending vibrations of the SO₄, these results confirmed the results of XRD and Raman. The O-H stretching vibrations of the H₂O molecules in the gypsum occur at 3538 cm^{-1} and 3403 cm^{-1} for sample C, 3544 cm^{-1} and 3406 cm^{-1} for sample D 3556 cm^{-1} and 3404 cm^{-1} for sample E. The in-plane O-H bending vibration occurs at 1662 cm^{-1} , 1624 cm^{-1} .

The bands related to the organic binding medium indicate the presence of a proteinaceous binder animal glue that is widely used as a paint medium. Animal glue is characterized by the presence of (1600- 1650) cm-1, assignable to C=O stretching (Amide I) and a band in the region (1500- 1550) cm-1 associated with C–N stretching and the deformation vibration of the N–H (amide II).

This finding was confirmed by comparing the functional groups that appeared in the samples to the characteristic functional groups of animal glue that could be seen in the FTIR spectrum at wavelengths of $(1660-1600 \text{ cm}^{-1})$ due to the C=O stretching band (amide I) as it revealed at 1662 cm^{-1} , 1624 cm^{-1} , 1624 cm^{-1} for the samples C, D, E respectively, $(1500-1550\text{ cm}^{-1})$ due to the C–N–H bending band (amid 11), as it revealed at 1544 cm^{-1} , 1538 cm^{-1} , 1537 cm^{-1} for the samples C, D and E respectively [30, 49]

The medium in all of the studied samples was animal glue, according to FTIR spectroscopic analysis used to determine the organic medium of the pigment and gold samples, this has been confirmed by the Raman analysis.

Vibrational assignment	Sample C (cm ⁻¹)	Sample D (cm ⁻¹)	Sample E (cm ⁻¹)
C–O asymmetric stretching	1421	1439	1443
C–O bending vibration	873	878	874
symmetric CO ₃ deformation	716	712	714
stretching vibrations SO4	1121	1121	1144
bending vibrations SO4	670	674	671
The O-H	3538 and	3544 and	3556 and
stretching	3403	3406	3404
vibrations of the			
H2O molecules in			
the gypsum			
The in-plane O-H bending vibration	1662	1624	1624
C=O stretching (Amide I)	1662	1624	1624
C–N–H bending band (amid 11)	1544	1538	1537

Table 5. Shows FTIR peak wave number and vibrational assignments detected in the sample B, C and D of the object studied

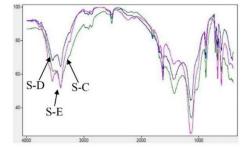


Figure (8). FTIR spectrum of pigments shows that animal glue was used as a binding medium.

3.6 Scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDX)

Six samples of gypsum, facial peels, black, and violet colors, gilding layer and fabric were analyzed by using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit, with accelerating voltage 30 K.V., magnification 14x up to 1000000 and resolution for Gun. 1n). FEI Company, Netherlands at the Grand Egyptian Museum.

The results of the EDX analysis of the samples (A1-A2-B-C-D1-D2-E-F) are shown in Table 6 and figure [9].

-The detection of the following elements (O)-(Ca)-(S)-(C)-(Mg) confirms the presence of the compounds of Gypsum, Calcite-Dolomite, and Anhydrite), which refer to the main components, of the stucco mask, and the presence of these elements

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also refers to the white inorganic pigment, which is represented by the compounds of Gypsum, Calcite and Huntite.

-The presence of the following elements (O)-(Si)-(Mg) confirms the presence of the compounds of quartz and periclase as a filler material such as sand used in preparing the stucco mask.

-The presence of the elements (O)-(Fe)-(As)-(S), confirms the presence of the (Realgar-Hematite-Goethite) which are used as red and yellow inorganic pigments.

-The presence of elements (O) and (C), confirms the presence of the compound vine black which refers to black inorganic pigments.

- The presence of the elements (O)-(Al)-(Si)-(Na), which confirms the presence of (Lazurite), While the detection of (Cu) confirms the presence of the compound (Azurite) as a blue inorganic pigment.

-The presence of the element (Au) n sample E confirms the presence of gold which is used in the gilding layer.

-The presence of the elements (Na)-(Cl).), confirms the presence of Halite, which is one of the sources of damage that affects the deterioration of the stucco mask.

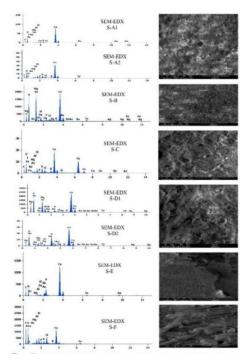


Figure (9). EDX spectrum and SEM image of stucco samples from the stucco mask, the detected elements confirm the results of XRD analysis and Raman.

CHARACTERIZATION STUDY OF A ROMAN STUCCO DEATH MASK FROM THE NATIONAL MUSEUM OF EGYPTIAN 117
CIVILIZATION

S	0	Ca	Mg	С	Si	Al	Fe	Cl	S	Ba	As	К	Na	Cu	Hg	Nb	Au	Р	Ti	Ν
S-A1	55,	32,	0,73	7,2	0,	0,	0,6	0,	1,		0,0		0,							
	55	89		9	62	49	8	29	12		9		25							
S-A2	52,	25,	1,20	8,3	2,	1,	1,5	1,	2,			0,	0,					0,		
	71	67		3	94	52	6	22	95			50	85					45		
S-B	54,	16,	14,	9,0	1,	1,	0,9	0,	0,	0,2	0,2	0,								
	82	22	25	5	90	08	9	71	41	4	0	13								
S-C	34,	17,	2,40	10,	3,	1,	22,	1,	2,		1,0	1,	0,	0,2				0,	0,4	
	50	28		80	22	45	83	50	50		0	13	83	0				43	5	
S-D1	51,	15,		15,	0.	3,	0,8	0.	1,		0,0	0,	0.	0,1						
	97	00		59	95	32	0	45	04		7	08	24	0						
S-D2	56,	19,	3,91	7,6	1,	1,	0,9	1,	5,	0,1	0,2	0,	0,		0,1					
	36	52		7	21	08	8	23	78	8	9	20	26		6					
S-E	41,	28,	0,38	13,	0,	0,	1,4		4,							0,5	7,5			
	40	99		62	61	65	4		82							7	3			
S-F	46,	7,	0,55	24,		1,	1,0	2,5	2,	1,0		0,	0,5					0,		10,
	07	49		83		17	1	5	20	0		30	0					76		87

 Table (6). The SEM-EDX analysis results of the samples of studied Roman stucco mask.

3.7 Portable X-ray Fluorescence

The purpose of this analysis is to identify the elements of the eye graft by using the portable X-ray fluorescence analyzer (XRF). Device Mode: (Head, Acquisition with mode: Manual, Acquisition Channels: 4096, Sample to Detector Material: Air). Measurement Time 40, 0 s, Tube Voltage: 40 kV, Tube Current: 20 μ A, Tube Target Material: Rh, Elio SN177 at the National Museum of Egyptian Civilization (NEMC).

The elemental analysis by XRF of some points of the white and black part of the eye are

shown in Table 7 which revealed that the inserted eye consisted of glass. As (Kóthay, 2014) confirmed the implanted eyes were confirmed in place of modeled and painted ones on masks. The most common variety has an opaque white piece of glass that is bent and set inside a smaller, circular piece of black glass; the eye is rimmed with either blue glass or plaster. A different method involves painting the eye from the inside out in black and white on a flat, translucent piece of glass [53].

Table (7).	The (XI	RF) analysis results	of the samples (Ey	ye) of studied roman	death mask.
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Sample	Al	Si	Р	S	Fe	Mg	Mn	Ti					
G1 Black	44,90%	38.80%	11.40%	3.71%	0.38%	0.18%	0.49%						
G2 Black	56,60%	32.10%	6.44%	4.11%	0.25%	0.11%	0.32%						
G3 Whit	62,50%	20.90%	3.25%	6.60%	0.29%			8.12%					

4. Conclusion

This study is an initial part of the conservation procedures to study a Greco-Roman stucco maskwhich preserved in the store of the National Museum of Egyptian Civilization of Cairo, Egypt (NMEC). Through the study of the painted and gilded stucco mask, some interesting information was provided about the methods of implementation the represented in the hollow casting of the stucco head or mask and the physical and chemical properties, and the stratigraphic sequence of the gilding floors was also known which represents the following layers as the first layer contain calcite which used as a base layer for the gilding process, the second layer contains organic adhesives as this layer consists of red to yellow granules of iron oxide from hematite mixed with an organic medium and the third layer contains the gilding paper. and it became clear that stucco masks include a mixture of

gypsum, calcite, and quartz, the analytical study confirmed the existence of a mixture of pigments consisting of Huntite, Realgar, Hematite and Goethite to give beige paint, which is the color of the face, as well as a color mixture of Realgar, Hematite Lazurite and Azurite to give the violet paint, while vine black pigment was used to give the dark color degree of the hair. It was also noted that animal glue was used as a medium for all pigments.When removing the halite salts from the examined mask, it is important to follow the guidelines for removing highly soluble salts from stucco artefacts. In the process of formulating the conservation strategy, it is critical to prioritise the regulation of the museum and store environment.

Conflicts of interest

There are no conflicts to declare.

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References

- [1] Carus, P., The Conception of the Soul and he belief in the Resurrection among the Egyptians, The Monist, July, Vol. 15, No. 3, 1905: 409-428.
- [2] Taylor, S.H., Death and After Life in Ancient Egypt, London: British Museum Press, 2001: 18.
- [3] Safaa, D. S., A Study of a Group of Funerary Masks in the Agricultural Museum in CairoJournal of the General Union of Arab Archaeologists, Volume 22, Issue 2, 2021: 125.
- [4] Svoboda, M., Cartwright, C.R., Mummy Portraits of Roman Egypt, Emerging Research from the APPEAR Project, J. Paul Getty Trust, 2020:159.
- [5] Müller, A., New Research on Roman Period Mummy Masks: The Case Example of the Budapest Museum of Fine Arts Collection, Burial and Mortuary Practices in Late Period and Graeco-Roman Egypt, Proceedings of the International Conference held at Museum of Fine Arts, Budapest, 17-19 July, Edited by Katalin Anna Kóthay, 2014:1-7.
- [6] Meeks, D., Dieu masque, dieu sans tete", Archeo-Nil. No.1, 1991: 5.
- [7] Torok, L., Mummy Masks from Roman Egypt Highlighted Works of http://www2.szepmuveszeti.hu/antik_gyujtemeny /evszak_mutargya/evszak_en.php?id=630.Access ed- May 3, 2022.
- [8] Vandenbeusch, M., O'Flynn, D., and Moreno, B., Layer by Layer: The Manufacture of Graeco-Roman Funerary Masks, The Journal of Egyptian Archaeology, Vol. 107(1-2), 2021: 281–298.
- [9] Riggs, C., Facing the Dead: Recent Research on the Funerary Art of Ptolemaic and Roman Egypt, of American Journal Archaeology, Archaeological Institute of America, Vol. 106, No. 1, 2002: 85-101.
- [10] Borowik, M., Funerary practices in ancient Alexandria in the Graeco-Roman Period (BC-AD syncretism Examples 642 - 332for in Alexandrian tombs and necropolises, University of Warsaw Faculty of Archaeology, Bachelor's thesis in the field of Archaeology, Warsaw, September, 2020: 1-142.
- [11] Crowley, P., Roman Death Masks and the Metaphorics of the Negative, Grey Room 64, Summer, Grey Room, Inc. and Massachusetts Institute of Technology, 2016: 4–103.
- [12] Laneri, N., Morris, E., Schwartz, G., Chapman, R., Cultraro, M., Chesson, M., Naso, A., Smith, A., Katz, D., Richardson, S., Pollock, S., Rutherford, I., Pollini, J., Robb, J., and Brown, J., Performing Death Social Analyses of Funerary Traditions in the Ancient Near East and Mediterranean, by The University of Chicago. All rights reserved. Published 2007. Printed in the United States of America. The Oriental Institute, Chicago, 2007.

[13] https://theimaginativeconservative.org/2020/07/r oman-imagines-death-masks-memory-emilykleinhenz.html Accessed- May 3, 2022

- [14] Walker, S. and Bierbrier, M., (2000) Ancient Faces: Mummy Portraits from Roman. Mummy Portraits from Faces: Roman. Egypt, Routledge, 1st Edition, p. 131.
- [15] Kóthay, K., A., Burial and Mortuary Practices in Late Period and Graeco-Roman Egypt", Proceedings of the International Conference held at Museum of Fine Arts, Budapest, 17-19 July 2014.
- [16] Frederiksen, R., and Marchand, E., Plaster Casts: Making, Collecting and Displaying from Classical Antiquity to the Present Hubert – Co.Gmbh and Co.KG, Gottingen, Germany, 2010.
- [17] Crimmon, M., Graeco-Egyptian Masks and Portraits in the Royal Ontario Museum, American Journal of Archaeology, 49(1), 1945: 52.
- [18] Abdullah, M., A., Hassan, A., H., Hala, A., Mona, F., Mineralogical characterization of Islamic stucco: Minaret of Shams El-Deen El-Wasty, Bulaq, Egypt, Construction and Building Materials 101, 2015: 692-701.
- [19] Gettens, R.G., Fitzhugh, E.W, and Feller, R.L., Calcium Carbonate Whites", Studies in Conservation Taylor & Francis, Ltd., Vol. 19, No. 3 (Aug., 1974), 1974:157-184.
- [20] Ceretti, E., Folder, E., and Aucouturier, M., Foil and Leaf Gilding on Cultural Art RevistaMifacts: Forming and Adhesion, RevistaMateria, V, 16, n.1, 2011: 540-559.
- [21] Dorge, V., and Howlett, C., Painted Wood: Conservation Williamsburg, History and Wooden Artifacts Group of the Virginia, American Institute for Conservation of Historic and Artistic Works, 1994.
- [22] Rivers, S., and Umney, N., Conservation of Furniture, Oxford: Butterworth-Heinemann Series in Conservation and Museology, 2003.
- [23] Colin, J., Karin, J., and Gillian, R., Ancient Faces, London, 1996: 80.
- [24] Adriani, A., Annuaire Du Musee Graeco-Romain, Alexandrie, 1950: 334.
- [25] Chabbi, A., Restoring a 20th Century Terrazzo Pavement: A Conservation Study of the Floor Map of the New York State Pavilion Queens, New York, Master Study, University Pennsylvania, 2004: 19. of
- [26] Comstock, M, C. Past, present, and future of high-performance inorganic pigments ", PhD, the Shepherd Color Company, Cincinnati, Ohio, 2006: 5.
- [27] Kumaril, S., Synthesis, Characterization and Optical Properties of Rare Earth - Transition Metal Based Environmentally Friendly Red and Yellow Pigments", PhD, Cochin University of Science and Technology, 2012: 5.
- [28] Hospodarova, V. Junak, J. Stevulova, N., Color Pigments in Concrete and Their Properties", An International Journal for Engineering and

Egypt. J. Chem.67, No. 8 (2024)

Information Sciences, Vol. 10, No. 3, 2015: 144-145.

- [29] Khedr, M., Ali, M., Kamel, A., and EL-Ghanam., M., Archaeometric study of historic terrazzo pavement of Prince Mohamed Ali Museum, Cairo, Egypt, An International Journal for Engineering and Information Sciences Vol. 15, No. 1, 2020: 221:232.
 [30] Derrick, R. M. et al., Infrared Spectroscopy in
- [30] Derrick, R. M. et al., Infrared Spectroscopy in Conservation Science", Scientific Tools for Conservation, the Getty Conservation Institute, Los Angeles, 1999.
- d Ahmed of the [31] Abdullah Mahmoud Kamel, mechanical study damage of archaeological plaster niches in Islamic religious structures and methods of treating them in application to one of the selected plaster niches in Master's Cairo. thesis, Department of Restoration, Faculty of Archeology, Cairo University, 2008.
- [32] Middendorf, B., Natural Stone, Weathering Phenomena, Conservation, Strategies and Case Studies, Geological Society, London, Special Publications, 205, 2010:173.
- [33] Edwards, H. G., Art Works Studies and Raman Spectroscopy", University of Bradford, Academic Press, 1999: 8-9.
- [34] Hospodarova, V. Junak, J. Stevulova, N., Color Pigments in Concrete and Their Properties", An International Journal for Engineering and Information Sciences, Vol. 10, No. 3, 2015:144-145.
- [35]Zalaffi, M., Karimian, N., and Review, P., Electrochemical and SERS Sensors for Cultural Heritage Diagnostics and Conservation: Recent Advances and Prospects, Journal of the Electrochemical Society,2020.
- [36] Murata, E., Sandu, E., Ferriera, S., Pereira, C., Stepanka, H., Valentina, V., and Antonio, E., A Comparative Multi-Technique Investigation on Material Identification of Gilding Layers and the Conservation State of 7 Portuguese Mannerist Altarpieces, A Comparative Multi-Technique Investigation on Material Identification Journal of Conservation Science, 2015: 439-459.
- Conservation Science, 2015: 439-459. [37] Cardoso, I., and Pye, E., Preparing the Foundation for Stable Gilding; Scientific Evaluation of the Durability of Baroque Gesso Gilding Grounds, Journal of Archaeological Science 79, 2017: 86-95.
- [38] Vandenabeele, P., Wehling, B., Moens, L., Edwards, H., De Reu, M., and Van, H.G., "Analysis with Micro-Raman Spectroscopy of Natural Organic Binding Media and Varnishes Used in Art", Analytica Chimica Acta, 407, 2000: 261–274.
- [39] Nevin, A., Osticioli, I., Anglos, D., Burnstock, A., Cather, S., and Castellucci, E., Raman Spectra of Proteinaceous Materials Used in Paintings: A Multivariate Analytical Approach for Classification and Identification", Journal of Analytical chemistry, 79, P. 2007.
- [40] Calza, C., Anjos, M.J., Mendonça, S., Brancaglion, J.A., and Lopes, R.T., X-ray microfluorescence analysis of pigments in decorative paintings from the sarcophagus cartonnage of an Egyptian mummy", Nuclear

Egypt. J. Chem. 67, No. 8 (2024)

Instruments and Methods in Physics Research B, 263, 2007: 249-252.

- [41] Raymond, K.M., and William, B.W., Vibrational Spectra of the Common Silicates: I. The Garnets", The American Mineralogist, 56, 1971: 54-71.
- [42] Kathleen, J.K., and Russell, J.H., Raman spectroscopic study of microcrystalline silica", American Mineralogist, 79, 1974: 269-273.
- [43] Guglielmi, V. Comite, M. Andreoli, F. Demartin, C.A. Lombardi and P. Fermo, Pigments on Roman Wall Painting and Stucco Fragments from the Monte d'Oro Area (Rome): A Multi-Technique Approach", Applied Science, 10, 2020: 7121.
- [44] Howell, G.M.E., and Dalva, L.A.F., "Comprehensive Analytical Chemistry: Non-Destructive Microanalysis of Cultural Heritage Materials", Wilson & Wilson's, 42, 2004: 1-80
- [45] Brian, C.S., Infrared Spectral Interpretation: A Systematic Approach", CRC Press, 1999:288.
- [46] Abrashev, M., Ivanov, V., Stefanov, B., N. Todorov, B., J. Rosell, B., and V. Skumryev, B., Raman Spectroscopy of Alpha-FeOOH (Goethite) Near Antiferromagnetic to Paramagnetic Phase Transition", Journal of Applied Physics 127, 2020. https://doi.org/10.1063/5.0006352.
- [47] Malebogo, A.L., Raman Spectroscopy Applied to Iron Oxide Pigments from Waste Materials and Earthenware Archaeological Objects", Ph.D. in chemistry, University of Pretoria etd – Legodi, M A, 2008:1-95.
- [48] David, A.R., Edwards, H.G.M., Farwell, D.W., De Faria, D.L.A., Raman Spectroscopic Analysis of Ancient Egyptian Pigments", Archaeometry, 43, 2001: 461-473.
- [49] Afifi, H.A.M., Analytical Investigation of Pigments, Ground Layer and Media of Cartonnage Fragments from Greek Roman Period", Mediterranean Archaeology and Archaeometry11,2011: 91-98.
- [50] Sophia, S., Žoi, E.P., and Lisa, V., Micro FTIR Imaging for the Investigation of Deteriorated Organic Binders in Wall Painting Stratigraphies of Different Techniques and Periods", Microchemical Journal, 2016: 124 1-23.
- [51] Kathleen, J.K., and Russell, J.H., Raman spectroscopic study of microcrystalline silica", American Mineralogist, 79, 1994: 269-273.
- [52] Bicchieri, M., Nardone, M., Russo, P.A., Sodo, A., Corsi, M., Cristoforetti,G., Palleschi, V., Salvetti, A., Tognoni, E., Characterization of Azurite and Lazurite Based Pigments by Laser Induced Breakdown Spectroscopy and Micro-Raman Spectroscopy", Journal of Spectrochimica Acta Part B: Atomic Spectroscopy, 56, 2001: 915-922.
- [53] Kóthay, K., A."Burial and Mortuary Practices in Late Period and Graeco-Roman Egypt", Proceedings of the International Conference held at Museum of Fine Arts, Budapest, 17–19 July 2014:1-10.