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PHYSIOCHEMICAL CHARACTERIZATION OF COPTIC JESUS CHRIST ICON, CHURCH OF SAINT MERCURIUS, EGYPT

Neven Kamal

Conservation Department, Fayoum University, Egypt, nkf00@fayoum.edu.eg

Shaaban Abdelaal

Professor at Faculty of Archaeology, Fayoum University, Egypt., smm00@fayoum.edu.egs

Emil HENIN

Head of Coptic Painting Conservation at Monasteries and Churches in Haret Zuweila, Cairo, Ministry of Tourism and Antiquities, Egypt., omel2010@yahoo.com

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PHYSIOCHEMICAL CHARACTERIZATION OF COPTIC JESUS CHRIST ICON [CHURCH OF SAINT MERCURIUS, EGYPT]

BY

Neven Fahim, Shaaban Abdelaal & Emil Henin

Lecture at Faculty of Archaeology, the Conservation Department, Fayoum University, Egypt.

Professor at Faculty of Archaeology, Fayoum University, Egypt.

Head of Coptic Painting Conservation at Monasteries and Churches in Haret Zuweila, Cairo, Ministry of Tourism and Antiquities, Egypt.

ABSTRACT

[Ar] التوصيف الفيزيوكيميائي لأيقونة السيد المسيح ، كنيسة ابي سيفين ، جمهورية مصر العربية
قدم هذا البحث التوصيف الفيزيوكيميائي لأيقونة قبطية من القرن التاسع عشر من كنيسة القديس مرقوريوس حيث تمثل مثال فريد من هذه
الايقونات التي تعود الى العصر القبطي. ولكي يتحقق الغرض من الدراسة تم استخدام بعض الوسائل الفحص والتحليل لشرح وايضاح كل مظاهر
التلف مما يساعد في التوثيق الأثري للأيقونة، وقد يعطي ذلك تفسير عن ميكانيكية تلف الأيقونات الأثرية ، كما كشف الفحص البصري أن الأيقونة
القبطية تتكون من أربع طبقات: دعامة لوح خشبي ، وطبقة تحضير ، وطبقة طلاء ، وبقيّة طبقة الورنيش ، وقد عانت كل طبقة من ظروف البيئة
المحيطة، وظهر ذلك في العديد من علامات التلف. تم فحص وتحليل العينات المأخوذة من أماكن مختلفة عن طريق الفحص باستخدام الميكروسكوب
الإلكتروني الماسح المجهز بكاشف الأشعة السينية المشتت للطاقة (EDS) والتحليل الطيفي بالأشعة تحت الحمراء فورير «FTIR» وحيود الأشعة
السينية «XRD». أظهرت نتائج التحليل الطيفي بالأشعة فوق الحمراء للعينات الأثرية أن الطبقة الأرضية تتكون من الجبس والطباشير والغراء
الحيواني كمادة رابطة. كما أشارت أطيف «XRD» إلى مصادر الأصباغ المستخدمة في رسم الأيقونة. وتم تحديد المادة الملونة الحمراء من أحمر
الرصاص، وبالنسبة لمادة اللون الأزرق من الالترامارين ، و المادة الملونة البيضاء من الرصاص الأبيض ، والمادة الملونة البنية كانت من أكسيد الحديد،
بالإضافة إلى استخدام صفار البيض كمادة رابطة في طبقة الطلاء والورنيش المستخدم كان من راتنج الشيلاك. ثم تم الكشف عن حالة التدهور،
وفقاً لنتائج «SEM» ، والتي أظهرت بوضوح وجود بقع وسناج وشمع وتشققات وثقوب في الطبقة السفلية من الأيقونة، وتراكم الغبار وطبقات
الورنيش الداكنة على الطبقة العليا.

[EN] In this paper ,we present a physiochemical analysis of a Coptic icon from church of Saint Mercurius that back to the 19th century. Identified deterioration phenomena were illustrated using various examination and analysis tools in order to fulfill the study's objectives. So as to find an explanation for how ancient icons were damaged. The visual inspection revealed that the Coptic icon is made up of four layers: the wooden panel support, the preparation layer, the painting layer, and the remaining varnish layer. Each of these layers suffered from surrounding environment, appearing in various signs of damage in the visual assessment. Samples taken from different places were investigated and analyzed by using scanning electron microscope equipped with an energy dispersive X- ray detector (EDX), FTIR, and X- Ray diffraction. The results of FTIR analysis revealed that the ground layer consists of gypsum, chalk and animal glue. XRD spectra confirmed that the red pigment was red lead, blue pigment was ultramarine, white pigment was white lead, and brown pigment was iron oxide (burnt sienna), in addition to the use of the egg yolk as a binder in the painting layer and the varnish was shellac resin. The state of deterioration was detected, according to the SEM results, which show clearly the presence of stain, soot, wax, cracks and lacunae at the lower layer of the icon, and accumulation of dust and darkening of varnish layers on the upper layer.

KEYWORDS: Icon, St. Mercurius, SEM- EDX, FTIR, XRD, Coptic, church.

I. INTRODUCTION

Saint Mercurius was born to Christian parents during a time of intense persecution, he was an officer in the Mareusian army, and Saint Mercurius was titled «Abi Shefin» because he vanquished the entire Barbarian army with the sword given to him by the Lord's angel while serving as a commander in the Mareusian army. He also rejected the worship of other gods¹. Numerous churches across the entire nation bear the name of St. Mercurius, who is regarded as one of the most well-known martyrs in the history of the Coptic Church in Egypt's Orthodox churches². The church of St. Mercurius is situated in Tamouh, Giza, and its style components were influenced by the region's environment and climate³.

Art historians classify the Coptic period as a Coptic art produced by Egyptian Christians in the late Roman, early Byzantine, and early Arab eras⁴. Icon [ἱκονα] is a word that refers to a picture or a portrait. It included historical occurrences connected to Biblical history, saints, holy Christians, and figures from Christian theological conceptions⁵. They are usually placed in centers of worship such as monasteries and churches for the worshipers to honor them by touching or adoring gestures and are generally made out of painted wood⁶. Additionally, because it is holy and sanctifying, the icon plays a variety of roles in Christian worship and church⁷. Every conservator finds the archaeological studies of Coptic icons difficult since they are made up of numerous strata with various physical and chemical characteristics, conflicting behaviors, and environmental deterioration factors⁸.

The icon artwork can be damaged by a number of deterioration factors, including temperature, light, humidity, air pollution, and insects⁹. Therefore, to understand the technical process by which the artist created the icon¹⁰. It is required to examine and analyze the icon's constituent parts. Which serve as a reference for the researcher and conservator to determine the extent of damage, the way in which damage occurred, and the elements of icon layers. The technique for Coptic icons painting depends on painting on wooden panels made from one or several planks held together with wooden pegs and supported by wooden traverses, nailed on the reverse with iron nails¹¹. which is a well-known technique for Coptic icon painting. Support, ground, paint [pigment and binder] and varnish layers are the icon's fundamental layers.

Characterization of the icon materials gives us crucial knowledge about the condition of the icon and the impact of age and environmental factors.

¹ SHENOUDA 2019: 3.

² HENIN 2022: 128-143.

³ MORGAN 2016: 14-36.

⁴ MEDHAT et al 2015: 152.

⁵ SKALOVA & GABRA 2003: 124.

⁶ MALATY 1998: 1-10; PUICA 2006: 37-50

⁷ BALIGH & SHALABY 2014: 2; PRIESS 2019: 281.

⁸ ELSAYED 2019: 241.

⁹ YOUSRY et Al. 2015: 492; ISSA et Al. 2015: 72.

¹⁰ MAHMOUD 2018: 197.

¹¹ MOHAMED et Al. 2019: 4685.

There are many factors that make the process of identifying the icon layers difficult such as the use of small quantities and the lack of sample purity¹². The purpose of this study is to characterize the components of a Coptic icon painting that the conservator might use while choosing potential conservation methods.

II. MATERIAL AND METHOD

1. Sampling

Six samples from each of the four pigments: brown, red, blue, and white were taken, to determine the pigment and binder used in the icon. Samples were collected from the white ground. The previous samples were prepared before examination and analysis by visual examination, SEM, FTIR and XRD. In general Examination and analysis procedures should be done by non-invasive, non-destructive methods, because in most cases, only micro-samples [a milligram] are permitted for analysis¹³.

2. Visual Examination

To highlight the artwork and an icon's technical aspects and assess its degree of degradation, numerous photographs were taken of the object under study, to determine its state of damage. And the required restoration in order to keep it in a condition that matches the value and significance of such artifacts.

3. Scanning Electron Microscope [SEM] Equipped with [EDX]

Samples were examined by SEM [scanning electron microscope] using an EDX unit, to characterize the morphological appearance of the layers of the icon. And determine its chemical composition. The instrument was FEL QUANTA FEI Philips– Holland, 3D 200i, coupled with EDX unit Thermo Fisher path finder (conservation lab at the Grand Egyptian Museum in Giza, Egypt).

4. Fourier Transform Infrared Spectroscopy [FTIR]

The device, a Bruker optics model vertex 70 with a mid-infrared source, which has a crystal diamond and does not require sample preparation, measured the spectra of samples in the spectral rang of (4000-400 cm^{-1}). The results were compared to those from the standards data in the lab at the Grand Egyptian Museum in Giza, Egypt.

5. X-RAY Diffraction

XRD analysis was employed to determine the chemical components of the colored pigments (40kV, 30mA). Measurements were made in 25° with a 0.02° step (Projects Sector Ministry of Antiquities and Tourism, Cairo, Egypt). It was performed by a Philips X'Pert PW3195 Diffractometer, using Cu K α radiation (40kV, 30mA).

¹² ŞERIFAKIA et Al. 2009: 303.

¹³ BRANIA 2010: 1-39.

III. RESULTS AND DISCUSSION

1. Visual Examination

The icon has been fixed in the iconostasis's center. The icon was painted in the 19th century by the Jerusalem artist Hafez Shamndi¹⁴. And is composed of four layers (wooden support layer, ground layer, paint layer, and varnish layer). The varnish was applied in layers over the icon's surface. Traces of unprofessional restoration (over painting). There are also numerous losses in the preparation layer, paint layer, and gilding layer. in addition to strange dark spots covering the paint [FIGURE 1].



[FIGURE 1]: Overview of Jesus Christ icon, damage signs and missing parts all over the Coptic icon.

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2. Scanning Electron Microscope [SEM] Equipped with [Edx]

The SEM photomicrograph and microanalysis data were used to evaluate the damage mechanism, which reflects the Coptic icon's surroundings. The micrograph photo revealed very brittle, cracked, cleaved outer layer contaminated with dirt, and a wooden inner layer, appearing to be in a good state [FIGURE 1]. SEM-EDX microanalysis of a few samples revealed that Mg was present in most cases. Which, refer to Kaolinite and Chlorite (natural clay compounds accumulated on the pigment samples)¹⁵.

The microanalysis of brown pigment indicated the presence of 3.96 % lead, 0.79 % aluminum, 0.84 % sodium and 0.69 % silica and 39% oxygen. Numerous researchers have demonstrated, that the color may be created by combining ochre yellow, blue ultramarine, and red lead [FIGURE 2] & [TABLE 1] Some studies suggested that brown pigment is frequently derived from iron oxide, also known as «burnt sienna»¹⁶. And the observation of unexpected little percentage of lead may be explained due to impurities from the priming layer which was mainly composed of anglesite (PbSO₄)¹⁷.

¹⁴ HENIN et Al. 2022: 133

¹⁵ BARTON & KARATHANASIS 2016: 187-192.

¹⁶ CLARK 2002: 7-20.

¹⁷ ŞERIFAKIA et Al. 2009: 303.

The red pigment spectrum revealed the presence of 4.95 % lead, 2.99 % sodium, 1.01% calcium, 1.89% aluminum, and 6.61% chlorine, which are refer red lead (II) oxide (Pb_3O_4) and some impurities [FIGURE 3]& [TABLE 2]. It is noticeable that the lead red was converted to black shades, as a result of to the conversion of red lead (Pb_3O_4) into (Pb_2O_3) by the effect of deterioration factors such as «thermal ageing»¹⁸, and the presence of chlorine salts oxide¹⁹. The use red lead dates back to the Roman era in the first century AD when it was utilized to paint linen shrouds for burial purposes²⁰. Additionally, a wide range of artworks, such as wall paintings, manuscript illuminations, and canvas paintings, were painted with it²¹.

Microanalysis of blue pigment revealed the presence of ultramarine blue pigment $(Na,Ca)_8(AlSiO_4)_6(SO_4,S,Cl)_2$, which is proven by the presence of 10.55% sodium, 29.32% calcium, 8.32% aluminum and 10.99% silica, [FIGURE 4] & [TABLE 3].

Ultramarine was the finest and most expensive blue that could be used by painters²². Lapis lazuli has been used as a source of Ultramarine blue pigment in artwork since at least 3000 BC. Paintings were enriched in their artistic value when ultramarine blue was existed on the artist's palette²³. It was determined via micrographs that several areas of the surface of the pigment sample had accumulated blackness, black soot, and wax. Interact in some way with an oil binder, which affects how its chemistry, According to [FIGURE 4]²⁴.

The white pigment in [FIGURE 5] & [TABLE 4] was composed of 53,74% Pb, 20.37 % C, 21.92% O, 1.71% Al and 2% Fe, which are refer to white basic lead carbonate $2PbCO_3 \cdot Pb(OH)_2$. White lead is a naturally occurring mineral known by the name hydrocerussite²⁵. From antiquity until the 19th century, lead white manufacture remained mostly unchanged²⁶. On the surface of the white pigment, there were macro and micro cracks of different width and depth²⁷, Air pollution and the presence of hydrogen sulfate caused white lead pigment to turn into dark lead sulfate²⁸. In addition to superficial sandy deposits accompanied by loss of paint²⁹.

¹⁸ KUMAR et Al. 1986: 415–418.

¹⁹ ANGLOS 2001: 186-205.

²⁰ ABDEL-GHANI 2009: 355; WALTON & TRENTMAN 2008: 845 – 860.

²¹ AZE et Al. 2008: 145–154.

²² LOTUT 2018: 1-18.

²³ ACETO et al 2013: 4184

²⁴ FRISON & BRUN 2016: 41-55.

²⁵ GRANT 2001: 4439- 4442.

²⁶ MAKSOUUD et Al. 2020: 102085

²⁷ LUSSIER & SMITH 2007: 41-53.

²⁸ GLIOZZO & IONESCU 2022:14-35.

²⁹ KARYDIS 2006: 1-24; BUDU 2018: 1-9.

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Element	Wt%	At %
C k	51.51	61.80
O k	39.45	35.61
Na k	0.84	0.52
Al k	0.79	0.37
Si k	0.69	0.37
S L	0.81	0.37
Ca L	1.94	0.70
Pb M	3.96	0.28

[TABLE 1]: The SEM-EDX micro analysis of brown pigment from archaeological icon.

Element	Wt %	At %
C k	23.02	15.47
O k	63.70	57.01
Na k	2.99	2.33
Mg k	1.01	0.75
Al k	2.85	1.89
S k	2.24	3.51
Cl K	6.61	3.33
Ca k	1.89	4.23
Pb L	4.95	0.43
Fe L	1.37	0.44

[TABLE 2]: The SEM-Edx micro analysis of red pigment from archaeological icon.

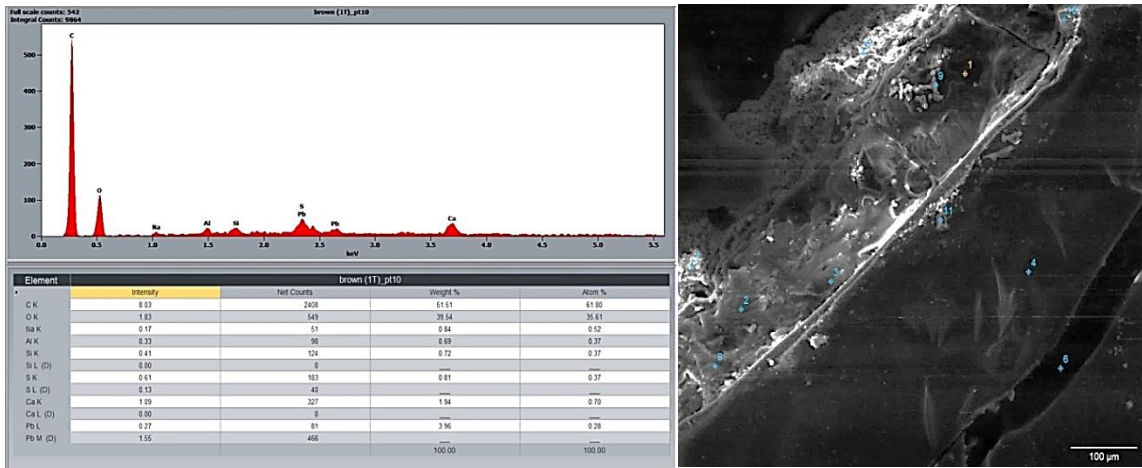
Element	Wt.%	At %
C k	29.32	39.76
O k	40.54	41.26
Na k	10.55	7.47
Al k	8.32	5.02
Si k	10.99	6.37
S L	0.28	0.12

[TABLE 3]: The SEM-EDX micro analysis of blue pigment from archaeological icon.

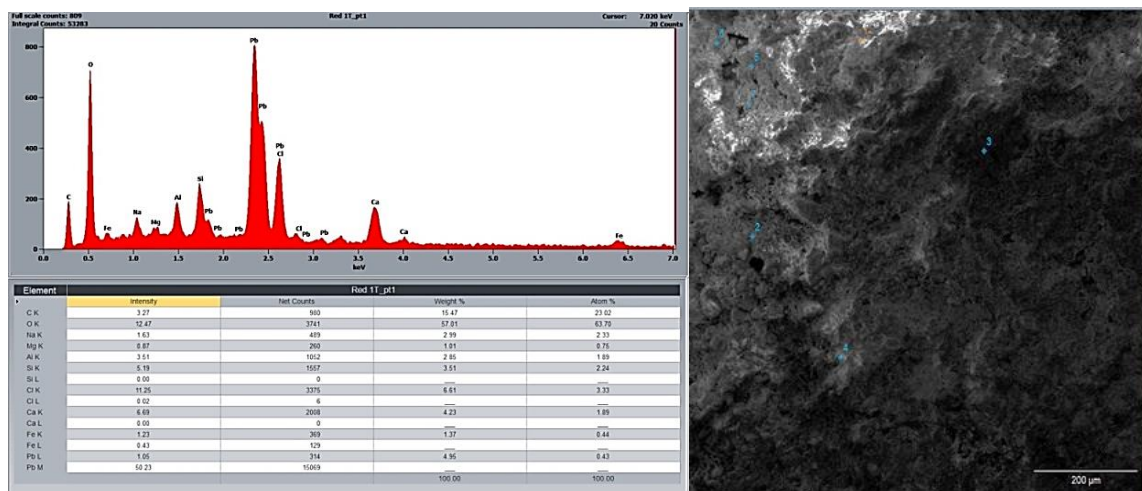
Element	Wt%	At %
C k	20.73	49.93
O k	21.92	39.64
Al k	1.90	2.04
Fe L	1.71	7.51
Pb L	53.74	0.09

[TABLE 4]: The SEM-EDX micro analysis of white pigment from archaeological icon

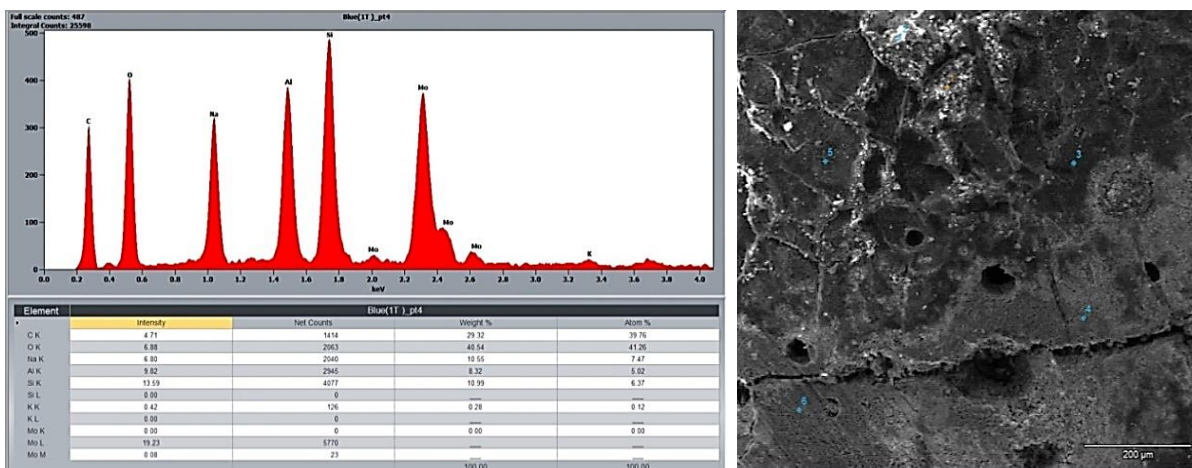
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[FIGURE 2]: SEM- EDX results of brown sample indicate the presence of lead oxides (Pb_2O_3).

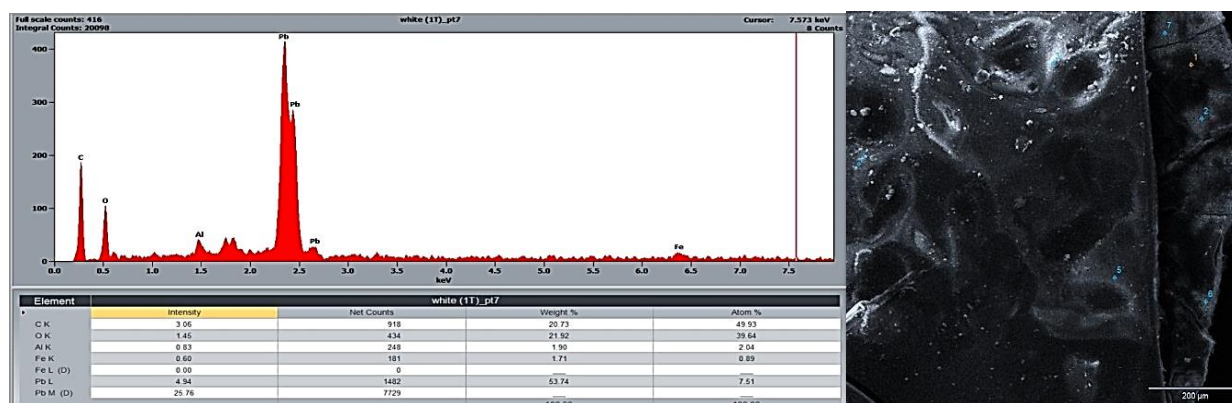


[FIGURE 3]: EDX micro-analysis results of red pigment indicate the presence of red lead oxides (Pb_3O_4).



[FIGURE 4]: SEM-EDX microanalysis of blue pigment indicate the presence of ultramarine $(Na, Ca)_8(Al SiO_4)_6(SO_4 S, Cl)_2$.

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[FIGURE 5]: SEM-EDX microanalysis results of white sample indicate the presence of basic lead carbonate $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$

3. FTIR Analysis

Infrared spectrometry is used for identification of coloring pigments, and binders. Yet there are difficulties, due to the identical chemical composition of many organic compounds, like glue, egg white and yolk, and comparable spectra in the IR range³⁰.

The spectrum of archaeological medium was compared with the proposed material's reference. The FTIR spectrum showed the presence of amide groups of proteins by observing the NH bending band (between $700\text{--}1500\text{ cm}^{-1}$) combined with the C=O bond at the band 1645 cm^{-1} , and CH₂ bond of the methylene group (1500 and 1700 cm^{-1}) and C=H methyl group band at the band 2920 cm^{-1} , This proved the use of egg yolk as a medium for pigments³¹ [FIGURE 6]. The band at 1171 cm^{-1} is suggested to be a characteristic sign of shellac resin³² [TABLE 5].

Calcium carbonate (CaCO_3) produced a strong signal, at the absorption band $1450\text{--}1400\text{ cm}^{-1}$, assigned to C=O stretch group, and gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) (calcium sulfate) gave a signal at absorption functional group at 983 cm^{-1} , which is assigned to (SO_4)³³.

Furthermore the presence of animal glue as a binder is highlighted by the strong absorption bands in the region of 3401 cm^{-1} , 2920 cm^{-1} , ascribed to amide I (C=O stretching) and amide II (C-N stretching and N-H bending), as well as the peaks connected to methylene groups at band 2929 and 2850 cm^{-1} [FIGURE 7].

Due to thermal ageing, calcium sulfate is thought to occur when preparation layers contain cracks and have missing pieces. The sulphation processes that cause the ground layer to deteriorate, due to the presence of sulphur dioxide (SO_2) gas acts as an oxidant and a catalyst, reacting with calcium carbonate to form calcium sulphate when there is a high relative humidity³⁴. The FTIR spectra of the Coptic icon's preparation layer showed that it is made of calcite, gypsum, and animal glue [TABLE 6].

³⁰ BERG et Al. 2002: 22-27.

³¹ VAHUR et Al. 2016: 3373-3379; KHASAWNEH & ELSEROGY 2019: 85-91.

³² DERRICK et Al. 1999: 190; GHANI 2015: 23 - 37.

³³ ANBALAGAN et Al. 2009: 226-230

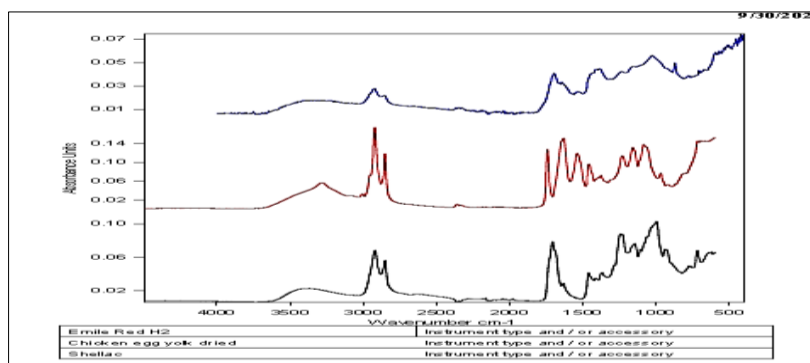
³⁴ PAVLIDOU et Al. 2006: 709-717

Functional group	Egg yolk	Shellac
-CH- stretch	2900cm ⁻¹	3100 cm ⁻¹
C=O stretch	1750cm ⁻¹	1650 cm ⁻¹
C-H bending	1380 cm ⁻¹	1480cm ⁻¹
C-O stretch	-	1171cm ⁻¹

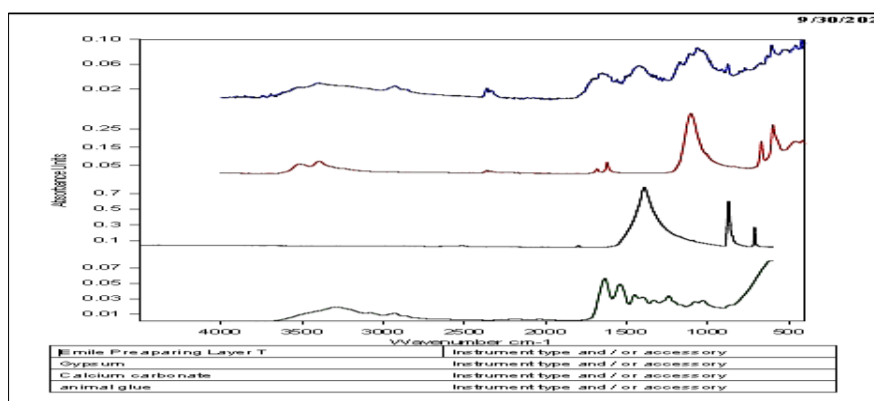
[TABLE 5]: FTIR spectra of a red pigment indicate the presence of egg yolk medium, and shellac varnish.

Functional group	Animal glue	Calcite	Gypsum
-CO- bending	-	875 cm ⁻¹	-
-SO ₄ - stretch	-	-	1140cm ⁻¹
S-O bending	-	-	669 cm ⁻¹
C=O stretch	1450 cm ⁻¹	1425 cm ⁻¹	-
-NH- stretch	3401 cm ⁻¹	-	-
-CH- Bending	2920 cm ⁻¹	-	-

[TABLE 6]: FTIR spectra of a ground layer and medium indicating the presence of gypsum, calcite and animal glue as a binder.



[FIGURE 6]: FTIR spectra of archaeological red sample which show egg yolk as a medium binder, shellac as a varnish.



[FIGURE 7]: FTIR spectra of ground layer sample indicate that it consists of gypsum, calcium carbonate, mixed with animal glue as a binder.

4. X-RAY Diffraction Analysis

Ground layer and pigmented samples were used to conduct this investigation; the outcomes of the XRD studied have demonstrated that the ground layer is composed entirely of calcite, gypsum, and animal glue as a binder³⁵. XRD pattern of a brown pigment reveals the presence of brown lead [FIGURE 8]³⁶. XRD spectrum of red pigment as seen in [FIGURE 9], shows that it consists of red lead. The use of ultramarine is seen in the XRD spectrum [FIGURE 10], refers to blue pigment, and the XRD spectra of white pigment showed, that it was composed of calcite [FIGURE 11]³⁷.

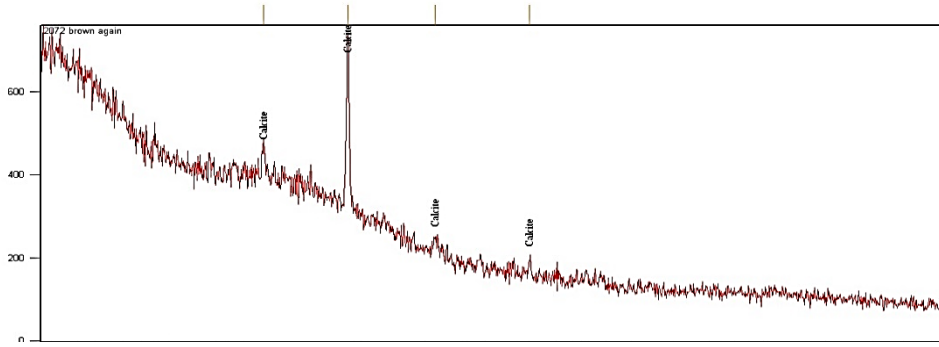
It is apparent that the preparation layers are abundant in calcite and quartz. The changes in temperature and relative humidity, mechanical stresses, and the physiochemical characteristics of the sample (mineral content) lead to the conversion of preparation layer components into anhydrite phase, which is weaker and brittle. It results in loss of the preparation layer components, contains large porous areas, flaking of the outer surface layer, and ultimately loss of the original painting structure. Dark areas and cracks can be noted on the surface³⁸.

³⁵ MEDHAT et Al. 2015: 151-161.

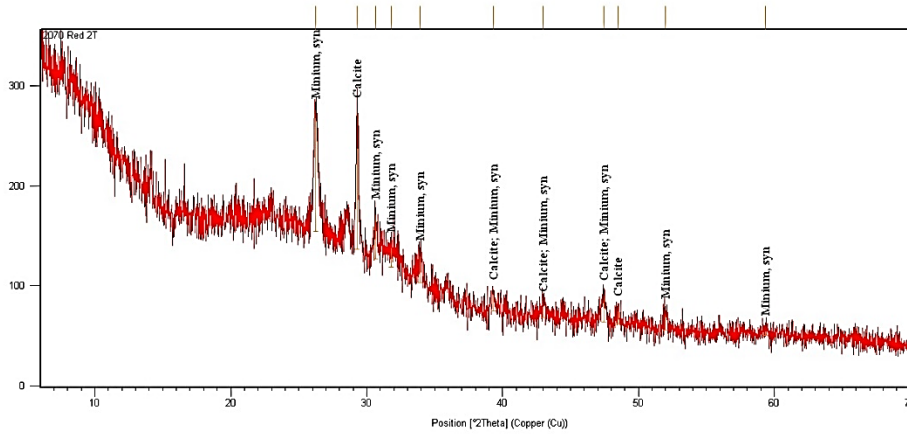
³⁶ HRADIL et Al. 2003: 223–236; HELWIG 2007: 38-109.

³⁷ BERNARD 1971: 33-121; MAHMOUD 2013: 82; GONZALEZ et Al. 2016: 43-49.

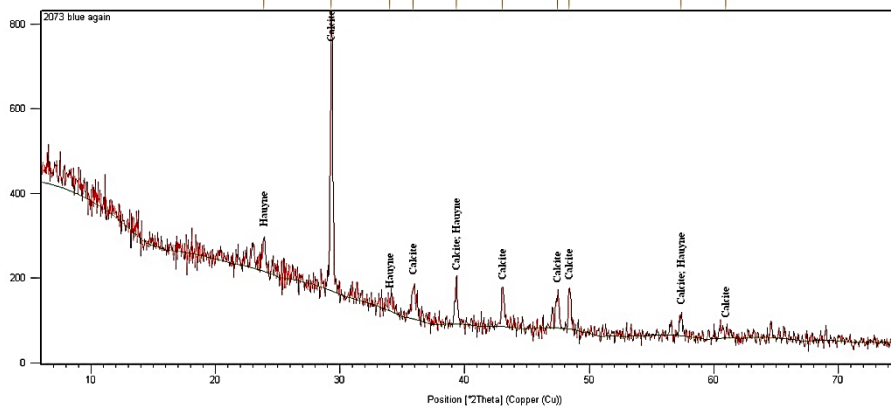
³⁸ SALLAM 2016: 493-500.



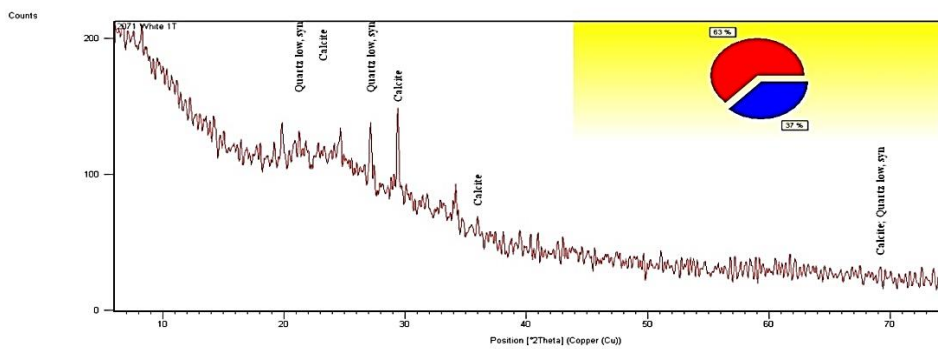
[FIGURE 8]: XRD results of brown pigment indicating the existence of calcite and iron oxide.



[FIGURE 9]: XRD results of red pigment indicating the existence of calcite and red lead.



[FIGURE 10]: XRD result of blue pigment indicated to the existence of calcite.



[FIGURE 11]: The spectra of archaeological white pigment, which prove the use of calcium carbonate, and in addition to quartz.

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Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-072-1937	34	Calcite	-0.100	0.728	CaCO ₃
*	00-041-1493	23	Iron oxide	-0.139	0.654	Fe ₂ O ₃

[TABLE 7]: Results of XRD spectra of brown pigment.

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-072-1937	34	Calcite	-0.100	0.728	CaCO ₃
*	00-041-1493	23	red lead	-0.139	0.654	Pb ₃ O ₄

[TABLE 8]: Results of XRD spectra of red pigment.

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-072-1937	58	Calcite	0.000	0.941	CaCO ₃
*	00-020-1087	16	ultramarine	0.000	0.126	Na ₆₋₈ Al ₆ Si ₆ O ₂₄ S ₄

[TABLE 9]: XRD analysis of blue pigment ,which revealed the presence of calcite in the ground layer and ultramarine pigment.

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-086-2334	17	Calcite	0.000	1.001	CaCO ₃
*	01-083-2472	19	Quartz low, syn	0.000	0.559	SiO ₂

[TABLE 10]: XRD analysis of white pigment.

IV. CONCLUSION

The physical and chemical analysis of a holy Jesus icon was described in this work. The icon is a part of the collection of Coptic icons, that can be found in the main sanctuary of the Mercurius church in Giza. Four pigments were found to have been employed to decorate the Coptic icon. SEM-Edx, FTIR, and XRD techniques were used for analysis.

SEM-EDX and XRD microanalysis results reveal that the brown color is made up of iron oxides, while the red, blue and white pigments are made up of red lead, ultramarine, and calcite, respectively.

SEM images revealed numerous deterioration features on the painted icon's surface, including fractures, flaking, blackness, and accumulating layers of soot and dust. According to the XRD analyses, the preparation layers contain gypsum and calcite [calcium carbonate] [calcium sulphate]. According to FTIR spectra, the preparation layer is made up of calcite, gypsum, and animal glue, and the medium used is egg yolk. The analysis of the coloring pigments revealed the existence of several minerals, which may have been the primary factor in the archaeological icon's ability to absorb water and exert pressure on its exterior layer³⁹.

³⁹ ALI & YOUSSEF 2020: 37-52

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**PHYSIOCHEMICAL CHARACTERIZATION OF COPTIC JESUS CHRIST ICON
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