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# ANALYTICAL STUDY OF THE BLUE PIGMENT USED IN OTTOMAN HOUSE MURAL PAINTINGS, CAIRO, EGYPT

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## ABSTRACT

The Al-Sadat House was established in 1070 – 1168 AH. / 1754 – 1659 AD. The architects used two degrees of blue pigments to decorate the house because blue was highly favoured by the Ottomans. Moreover, the Ottomans used this to colour clothes, believing that it could prevent envy.

Several methods of analysis were used, including the optical polarised light microscope (PLM), scanning electron microscope coupled with EDX, X-ray Diffraction (XRD), and Infrared spectroscopy (FTIR) to recognize the blue color used in two layers.

The results of the analysis indicated that there was a thick preparation layer of calcite, gypsum, and quartz. Moreover, the blue colors used are ultramarine in the first layer and cobalt in the upper layer and it was revealed a preparation layer of red lead minimum under the blue colours. Animal glue was used as a binding medium, which indicates using the mural painting in the tempera technique.

**KEYWORDS:** Osman, House, Ultramarine, Cobalt, Lead Minimum, Tempera, Stratigraphic, wall painting, FTIR, SEM-EDS, XRD, PLM

## 1. INTRODUCTION

The importance of the wall paintings is that they are not just a work of art, but that they bear and provide information about ancient civilization and customs of peoples and humanity (Garg *et al.*, 1995). Al-Sadat House is considered one of the important Ottoman houses in Cairo. The owner of the house, Sheikh Mohamed Abu al-Anwar al-Sadat, was a scholar of Al-Azhar.

This house consists of two floors. The ground floor includes the hall named the (Um Al- Afrah Hall). It was intended for religious ceremonies and chanting. It was decorated with mural painting, ceramic tiles, and a decorative wooden ceiling. The second floor contains a group of residential rooms decorated also with mural paintings. The house was subject to damage, vandalism, and destruction. The remaining parts still have very few painting layers, including two degrees of blue colours (Fig.1A, B, C, D) (Brania and El-Rashidy, 2009).

The blue colour was widely used in artistic works due to its importance. It was considered one of the sanctity colours. It symbolized the sky and expressed the divine truth and justice (Ragai, 1986). Various degrees of blue were used in the artworks during the Ottoman and Mohamed Ali periods in Egypt. Ultramarine blue was used in El-Gawhara Palace (Mahmoud *et al.*, 2021) and Al-Bimaristan Al-Muayyidi's mural painting (Ali *et al.*, 2021). Prussian blue was used in El-Safa Palace (Ali & Darwish, 2011), whereas cobalt blue was used in Elshinawy Palace (Ali *et al.*, 2018). Furthermore, papagonite blue was used in Sibile Abdel Rahman Katkhada and the Hall of Moheb El Dein (Ali *et al.*, 2019).

Egyptian blue ($CaCuSi_4O_{10}$) is the oldest synthetic pigment with multiple compositions (Marey Mahmoud, 2011). In the 16th and 18th centuries, Egyptian blue was used instead of azurite due to its quality. It appeared in some Coptic artworks in the 18th century (Sakr *et al.*, 2016).

Blue Smalt ($CoO.nSiO_2$) is considered a potassium glass with cobalt that was added to reach the required degree. It was used in the 15th century to decorate the paintings and drawings of the sky and other less heavenly characters (Schelvis *et al.*, 2006) (Cianchetta *et al.*, 2012).

Ultramarine (Lapis Lazuli) (Na8 ($Al_6SiO_{24}$)Sn) was used in the manufacturing of amulets, jewels, seals, and inlays as the result of the belief that it provided god protection to these collectibles and their owners (Alessandro *et al.*, 2009). It was not widely used for colouring as a result of the high costs of the natural color. The industrial colour was produced and used in the 19th century due to its low costs (Ragai, 1986; Abdel-Ghani, 2015; Favaro, *et al.*, 2012). Natural and synthetic ultramarine were almost identical. The synthetic was made by the French Jean Baptiste and the German Christian Gottlob (Darwish, 2013). Synthetic ultramarine was first reported in 1827 and was extensively used as an artistic pigment in the second half of the 19th century (Plesters, 1993).

Natural blue pigment ultramarine is obtained from the semi-precious stone lapis- lazuli, comprising a complex sulphur-containing aluminium silicate (Eleonora *et al.*, 2006). The most important mines of lapis lazuli are in Sar-e-Sang in Badakhshan, Eastern Afghanistan, while other sources are in Tajikistan and Russia (Sancho *et al.*, 2007). Aston *et al.* (2000) suggested that lapis lazuli occurs in the Uweinat Oasis in Southeast Egypt. The mineral that forms this color is found in heterogeneous rocks consisting of lazurite and other minerals, such as calcite and pyrite (Calligaro *et al.*, 2014).

Cobalt ($CoOAl_2O_3$) was used widely in the Amarna Period, especially in the decoration of pottery and building facades (Shortland *et al.*, 2006) for its features, such as resistance to adverse environmental condition, thermal stability, as well as lack of effect by acids and alkalis. Because it is characterized by a degree of toxicity to microorganisms, it is still in use (Yoshimura al., 2002). It was one of the industrial colors known to the ancient Egyptian after the Egyptian green and the Egyptian blue (Ali *et al.*, 2018).

Al-Sadat House was exposed to different deteriorated factors, which led to the collapse of wall paintings and the disappearance of most of the colored paintings. All these manifestations affected the painting's visibility (Nabil & Waeel, 2014). This house contained two degrees of blue, but they were rarely in two layers on the same wall. With the help of investigation and analysis methods, these materials were identified. The research aims to:

- Characterize the structure of the paint layers, containing blue pigments and media.
- Understand the composition of the layers.
- Identify the blue pigments used in the mural painting.
- Determine the factors causing damage and its forms.

The examinations indicated the presence of two layers of blue on the top of each other as new rare use. This finding was confirmed by the multiple analysis techniques.


*Figure 1: (A) Al-Sadat house Entrance level for the street, (B) The second floor, (C) The mural paintings interior of the house near to the windows (D) The remaining wall paintings around the door shows the extent of deterioration of the paint layers and missing parts.*

## 2. MATERIALS AND METHODS

### 2.1. *Sampling*

The samples collected from fallen edges, detached parts and worn-out parts and cracks scattered with the remnants of the preparations layer from the second floor, the samples were in the micro size (Fig.2), and 5 samples were used in the study which, visually, were suffering from color loss, salts, and fading.

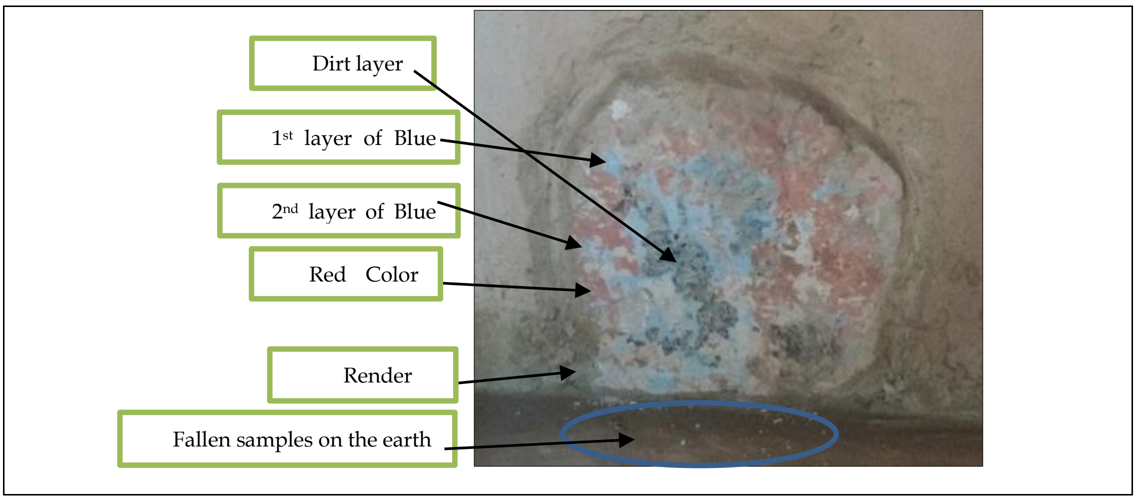



*Figure 2: The samples collected from detached parts fallen edges on the earth.*

## 2.2. *Methods*

Investigation and analysis techniques, including the polarised light microscope (PLM), scanning electron microscope coupled with EDX, X-ray Diffraction (XRD), and Infrared Spectroscopy (FTIR) were used to provide the necessary studies to support future restoration projects (Liritzis *et al.*, 2015).

### *2.2.1 Light optical microscopy*

The samples were examined with a digital microscope (USB Digital Microscope, model: Olympus) to identify the colour appearance pigments used in the colour layer, the alteration of these used pigments, the surface damage manifestations, as well as the microcracks with the following technical specifications: image sensor 1.3 Megapixels, magnification factor from 200X to 500X, photo capture resolution 640×480, 320×240, and LED illumination light resource adjustable by the control wheel.

### *2.2.2. Field-emission scanning electron microscope*

Two samples were examined by the scanning electron microscope coupled with EDX to identify the chemical composition and microstructural layers of the wall paintings. One sample was examined using a common imaging technique of different magnitudes to obtain more accurate information on the morphological properties of the coloured surfaces (Ali & Abd Elkawy, 2021). Microanalysis was carried out using an energy dispersive X-ray analyser (EDAX). The chemical analysis of the pigment samples was done by Quanta 250 scanning electron microscope (National Research Center, Dokky, Cairo).

### *2.2.3. X-ray diffraction analysis*

The XRD analysis was carried out on the collected samples to recognize the mineralogical composition of the painting layers. This method is used with crystalline materials. Samples are processed by grinding in dry form by using a mortar to obtain a fine powder (Ali & Abd Elkawy, 2021). Device type: Bruker d8 discover was used to do this analysis in the Petroleum Research Centre. These samples were examined with Radiation X-rays, diffraction meter filtered CU Kα (1.541874A) only directed at samples. The count statistics for the X-ray diffraction method Was, as follows size: 0.011, data range: 5.122 ° – 80.080 °, original data range 5.022 ° - 79.980 °, number of points 6916, 2theta correction 0.1 °, with a wavelength of 1.541874 A.

### *2.2.4. Fourier Transform Infra-Red spectroscopy (FTIR)*

This type is widely used in the archaeological field to identify the organic medium (Madejova, 2003) that binds the grains of colouring materials together and links them to the preparation layer (whitewash). Comparing these results with standard samples helps identify the type of the painting medium. The analysis was done in the National Research Centre, Dokky, and Cairo (FTIR Lab) using a device type (JASCO 6100, FT-IR) with a wavenumber range of 400:4000 ($cm^{-1}$). Samples were mixed with KBR.

## 3. RESULTS AND DISCUSSION

### *3.1. Optical microscope*

Through the examination, it was found that the stratigraphic installation of the wall painting consists of a preparation layer (coarse & fine renders) of gypsum, red lining, two layers of blue colour, and a superficial black layer (Fig.3a). The examination also showed several fine cracks in addition to lost large areas. Layers of accumulated black greenish dirt and dark brown spots appeared on the surface in some areas. There were different shades of blue in addition to the presence of a lining of red colour above the layer of plaster (Fig.3b, c, d, e, f).

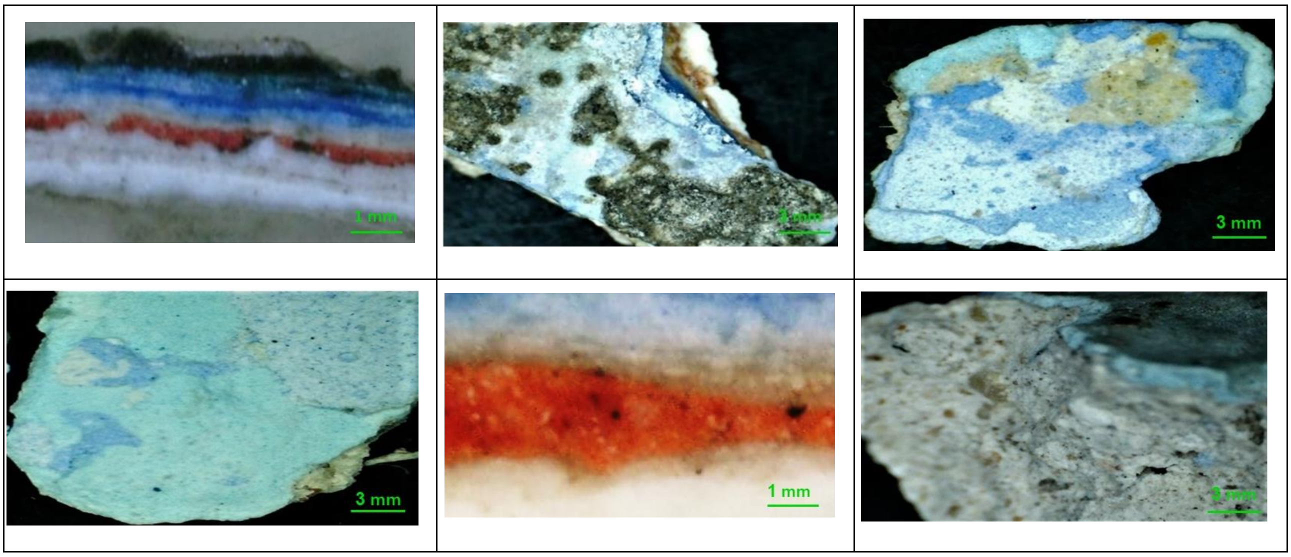



***Figure. 3: (a) Stratigraphic section of paintings layers in Al-Sadat house, (b) Dirt layer, (c) Upper layer of Blue color, (d) lower layer of Blue color, (e) Red Color lining, (f) Render.***

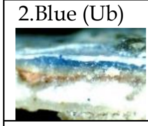
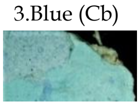
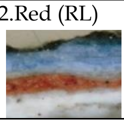
### *3.2. Field-emission scanning electron microscope*

The results of SEM – EDX examinations and the analysis of the studied samples in (Table.1) and (Fig.4, 5, 6, 7, 8, 9, 10) indicated that SEM_EDX examinations of the upper blue colour layer using enlargements (50 - 500x) revealed the presence of cobalt blue. These results were confirmed using XRD analysis (fig.10a). The strange thing, in this case, is that cobalt is present above ultramarine. This use may be due to the artist's desire to preserve the aesthetic appearance and hide the defective damage of ultramarine blue, so he used the second layer of cobalt blue. It was used for its thermal stability, as well as resistance to environmental conditions and biological damage.

The lower blue colour layer using different enlargements (3000x-6000x) revealed that the surface suffered severe damage, and there were some cavities. Halite crystals existed on the surface and between the layers. Moreover, the blue granules were unconnected, and the bonding material was lost. The elemental analysis indicated the presence of Na (1.23 & 1.56), Al (1.37&1.19), Si (2.9&2.51), and S (3.57&5.75) elements which lead to ultramarine blue pigment and the presence of sodium chloride salts that attacked the paintings and swept into it through the walls (Domenico *et al.*, 2021).

The SEM-EDX examinations of the red-orange layer under blue colour indicated the presence of a percentage of lead (0.83: 0.88%) that was used because it provides protection and stability of the blue colour. However, it was not achieved due to lead that interacted with the sulphur and caused fading and discoloration of blue pigments.

***Table 1. EDAX data of the studied pigment***

| Sample | Elements % | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | O | Na | Mg | Al | Si | S | Cl | K | Ca | CO | Pb |
| 2.Blue (Ub)  | 12.89 | 50.64 | 1.23 | 0.58 | 1.37 | 2.9 | 3.57 | 1.26 | 0.76 | 24.8 | — | — |
| | 12.55 | 48.29 | 1.56 | 0.59 | 1.19 | 2.51 | 5.75 | 2.09 | 0.8 | 24.68 | — | — |
| 3.Blue (Cb)  | 12.10 | 51.78 | 0.77 | 0.53 | 0.42 | 0.70 | 8.62 | 0.94 | 0.51 | 21.59 | 2.03 | — |
| | 12.17 | 51.33 | 0.90 | 0.61 | 0.49 | 0.63 | 6.13 | 1.10 | 0.57 | 25.43 | 0.64 | — |
| 2.Red (RL)  | 9.90 | 49.59 | 0.60 | 0.69 | — | 0.64 | 7.03 | 1.08 | — | 29.64 | — | 0.83 |
| | 10.90 | 58.94 | 0.33 | 0.37 | — | 0.22 | 11.93 | — | — | 16.44 | — | 0.88 |

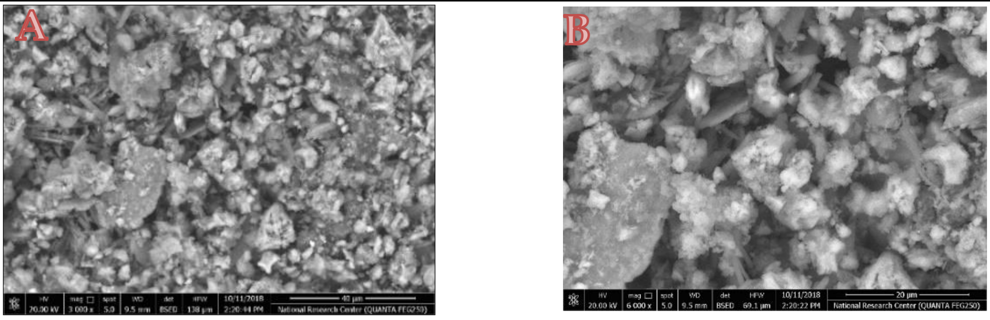



*Figure 4: SEM image thine section for paintings layers, a) shows the salts crystals, b) residual color and salts crystals*

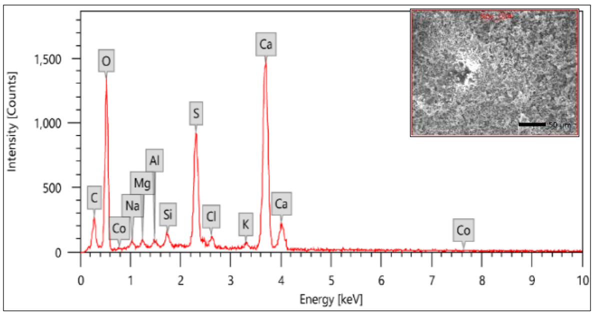



*Figure 5: SEM-EDX pattern of the upper blue color (50x), which indicated the presence of cobalt.*

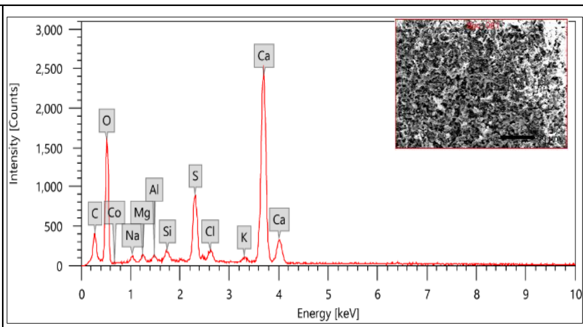



*Figure 6: SEM-EDX pattern of the upper blue color (500x), which indicated the present of cobalt.*

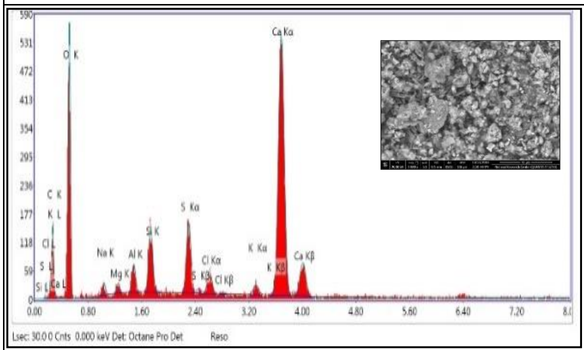



*Figure 7: SEM-EDX (3000x) pattern of the salts crystals spread in the layers and concentration on residual colors.*

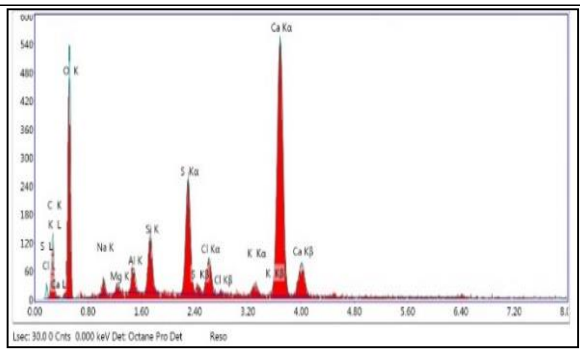



*Figure 8: SEM-EDX (6000x) pattern of the lower blue layer which indicated the present Ultramarine.*

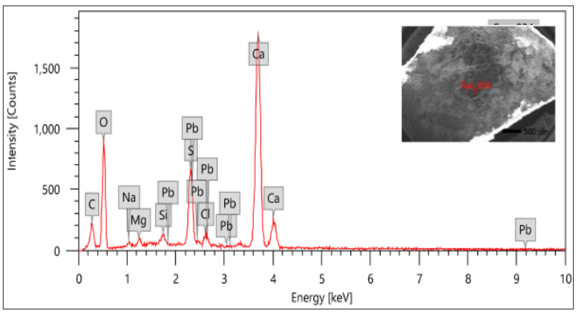



*Figure 9: SEM-EDX pattern of the red lining color, which indicated the presence of red lead.*

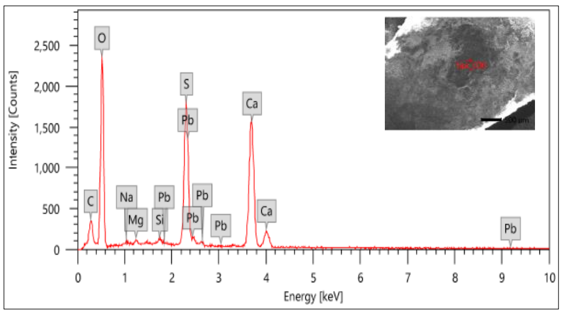



*Figure 10: SEM-EDX pattern of the red lining color, which indicated the presence of red lead.*

### 3.3. *X-ray diffraction analysis*

The results of the XRD analysis of the preparation and paint layers indicated that (Table 2) (Fig.11, 12):

- Calcite and quartz compounds existed in different proportions in the preparation layer. The coarse ground layer was composed of calcite $CaCO_3$ as a major component with quarts $SiO_2$, while the plaster layer was composed of gypsum $CaSO_4.2H_2O$. The whitewash layer consisted of calcite, and gypsum, and a small amount of halite.
- The result of the analysis of the blue-coloured substance indicated the use of cobalt blue ($[Co, Ni]_3 [As O_4]_2 .8H_2O$) in the upper layer (Shortland *et al.*, 2006) (Cianchetta, I *et al.*, 2012). It was one of the most popular colours between the 16th and 18th centuries. It is made from raw cobalt, a source of silica, and potash as the flux (Robinet, L *et al.*, 2011). The presence of the orange color was due to the use of the red lead ($Pb_3O_4$) (Coccato, A *et al.*, 2017).

Ultramarine was also found in the lower blue layer (Rosi *et al.*, 2004). Natural lapis lazuli ($Na_8 [Al_6Si_6O_{24}]Sn$) was found (Coccato *et al.*, 2017).

***Table 2: XRD results of the studied sample***

| Sample | The approximate mineralogical results | | | | | |
|---|---|---|---|---|---|---|
| | Calcite $CaCo_3$ | Quartz $SiO_2$ | Gypsum $CaSO_4.2H_2O$ | Halite NaCl | Cobalt Aluminate $CoAl_2O_4$ | Lazurite $_3Na_2O._3AL_2O_3._6SiO_2._2Na_2S$ |
| **G+CB** | **58.3** | **28.1** | **8.8** | **3.3** | **1.5** | ____ |
| **G +UB** | **69.3** | **24.5** | **24.5** | __ | ___ | **1.0** |

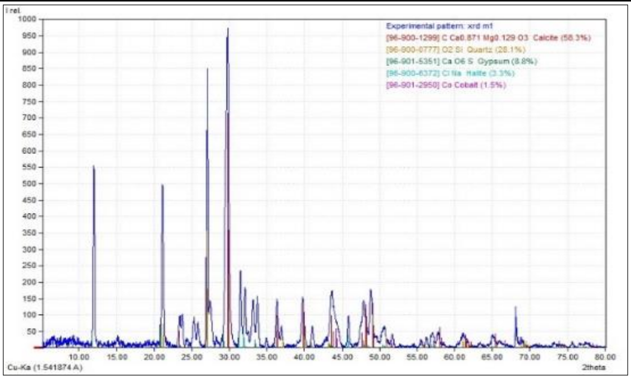



***Figure 11: X-ray diffraction pattern of ground layers + cobalt blue***

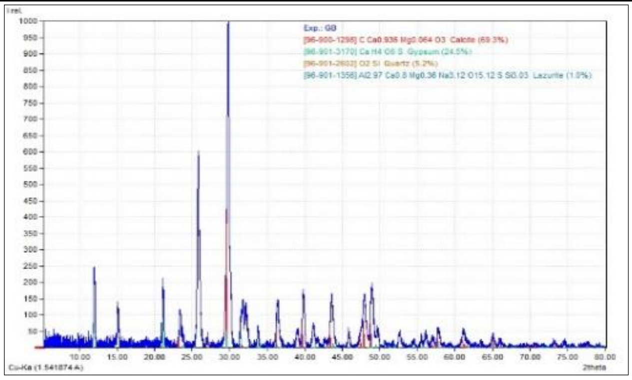
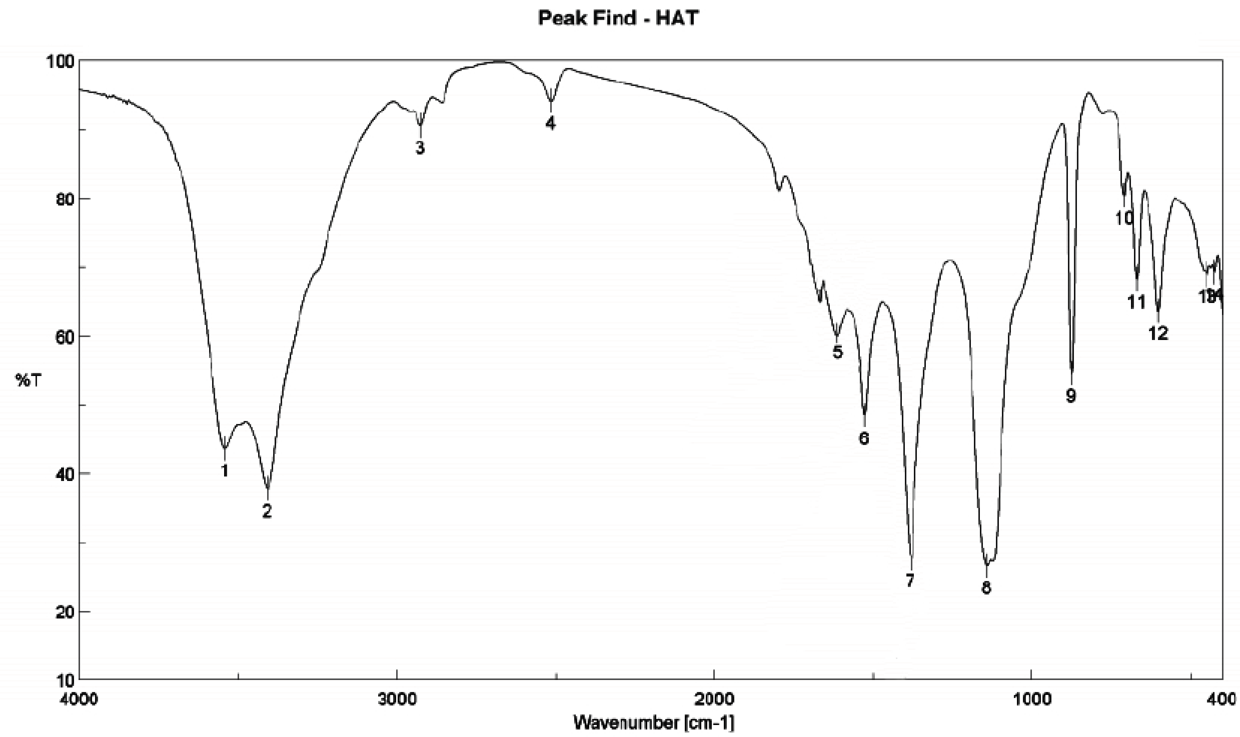



***Figure 12:X-ray diffraction pattern of ground layers + ultramarine blue.***

### 3.4. *Fourier Transform Infra-Red (FTIR)*

This section illustrates the results of the FTIR analysis of the archaeological samples taken from the color layer. Making a comparison with the standard sample showed that the active groups highlighted animal glue as a color medium. This result suggests that temper was the adopted method. Gypsum was found because it was used in the whitewash (Fig.13) (Table 3), as shown in Islamic wall paintings (Ali & Abd Elkawy, 2021; Ali *et al.*, 2019) whereas calcite existed because it was utilized in the preparation layer (Table 2). Gypsum was added to make the surface smooth and ready for coloring through the use of brushes and color oxides.




[ Result of Peak Picking ]

| No. | Position | Intensity | No. | Position | Intensity | No. | Position | Intensity | No. | Position | Intensity |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3541.63 | 43.65053 | 2 | 3406.64 | 37.62504 | 3 | 2925.48 | 90.51341 | 4 | 2514.72 | 94.09171 |
| 5 | 1648.33 | 81.12576 | 6 | 1522.8 | 50.70967 | 7 | 1428.03 | 14.36222 | 8 | 1142.62 | 26.56929 |
| 9 | 874.56 | 54.50123 | 10 | 708.712 | 80.40257 | 11 | 671.106 | 68.18056 | 12 | 603.61 | 63.54262 |
| 13 | 448.369 | 68.89661 | 14 | 425.227 | 69.25745 | | | | | | |

*Figure 13. FTIR spectra indicated the presence of animal glue.*

*Table 3: The main function groups from the color layer*

| N | Functional Groups bands | $cm^{-1}$ | Substance |
|---|---|---|---|
| 2 | OH + NH stretching | 3406 | Gypsum |
| 5 | Stretching S-O C=O (Amide I) | 1648 | Gypsum, Animal Glue |
| 6 | CN stretching (Amide II) | 1522 | Animal Glue |
| 7 | $CH_2$ bending | 1428 | Animal Glue |
| 8 | C-O Group | 1142 | Gypsum, Animal Glue |

## 4. CONCLUSION

The study illustrated that mural painting was applied as two layers: The first rough layer (Intonaco and Arriccio) consisted of calcite, gypsum, and sand, then the whitewash layer consisted of calcite and gypsum. It found red colour, as a lining layer, under the blue colour. The painting layer contained three different layers. The first layer was red of lead because the artist believed that it provided protection and stability for the Ultramarine (the second layer). The artist mostly used the second degree of blue, which is (the third layer) cobalt above the ultramarine, as a result of the degradation and discoloration of the Ultramarine. This finding was identified through the work of examination and analysis of the archaeological samples. It was made of tempera technique (the colour medium is animal glue).

It was found that the paintings layer lost adhesion due to the spread of fine cracks and the crystallization of salts in the building, which has suffered from a high level of groundwater.

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