ORIGINAL PAPER

MULTI-ANALYTICAL TECHNIQUES FOR THE ARCHAEOMETRY STUDY OF THE STAINED GLASS WINDOW FROM THE ROYAL PALACE AT EDFENA, EGYPT

RANIA ABDEL GWAD ELORIBY[1]

Manuscript received: 07.09.2023; Accepted paper: 16.12.2023;
Published online: 30.12.2023.

Abstract. *This work aims to study the case of the stained glass window at the Royal Palace in Edfena, Egypt. The research also aims to identify the chemical composition and diagnose the manifestations of damage on the stained glass window. Therefore, the examination and analytical study were conducted using several different techniques, including AutoCAD, digital and stereo microscopies, scanning electron microscope - X-ray energy dispersion, X-ray diffraction, and microbiological examination. The microscopic study revealed that the surface of the painted glass suffers from the presence of layers of damaged products, which obscure the decorations and lead to opacity. Light holes spread across the layers of different paints with varying sizes, and pits appear as a result of corrosion. Using EDX, the element responsible for the color in the painted glass layer was determined, as well as the composition of the lead came. XRD was used to identify the crystalline compounds in the painted glass layer and the putty composition. In the end, an appropriate treatment plan was drawn up for the condition of the lead-stained glass window to preserve it from loss and damage due to its archaeological and artistic value.*

Keywords: *Stained glass; deterioration; microscopy; XRD; SEM-EDX; microbiological examination.*

1. INTRODUCTION

Stained glass windows hold significant importance in both religious and civil architecture, serving multiple purposes, primarily acting as light filters, creating beauty and splendor, and also reducing the load on walls and arches [1, 2]. The magnificence of stained glass windows emerged during the Gothic period in Europe, particularly favored due to architectural advancements [3, 4]. In Egypt, stained glass windows became known in the 19th century and were utilized in the Pasha's and Royal palaces. The designs of these stained glass windows were primarily characterized by exquisite floral and geometrical patterns [5-7].

Stained glass windows are composed of small colored pieces of glass held together by a lead framework, which not only supports the glass pieces but also contributes to the artistic design [8-10]. Some glass pieces have paint applied to their surfaces that have been heat-treated to create decoration. Glass paint is made up of various amounts of silicon oxide, or sand (SiO_2), mixed with metal oxides (PbO, CuO, MnO, and Fe_2O_3). Usually, they are dissolved in water, acetic acid, or other solvents after being combined at room temperature with a bonding agent like sugar or arabic gum. Using a brush, apply this finely ground mixture to the glass surface. The paint must then be adhered to the glass surface by thermal treatment up to 500–600°C [11, 12]. Stained glass windows are exposed to deterioration due

[1] Conservation Department, Faculty of Archaeology, Cairo University, 12613 Giza, Egypt.
E-mail: rania88@cu.edu.eg.

https://doi.org/10.46939/J.Sci.Arts-23.4-b04

to the surrounding environmental conditions, such as humidity, temperature, air pollution, and microorganisms, either separately or in combination [13-15]. The main type of deterioration that affects stained glass is humidity. In the presence of humidity, glass corrosion will occur due to the formation of a hydrate layer and migration of alkaline ions of Na^{+} and Ca^{+2}. When there are pollution agents such as CO_2 and SO, corrosion crusts develop on the surface. These crusts are made of insoluble salts such as calcium carbonate, calcium sulphate, and others, leading to the irreversible degradation of the original glass once it has transformed into corrosion crusts. As the crust thickens, it changes the way light moves through the glass, diminishing the visual impact and beauty of the stained-glass window [16-19]. Humidity also affects the putty and causes it to decompose and lose shape. It also helps fungi grow on the surface of the glass and leads to damage to the glass pieces [20].

The paper aims to study a lead-stained glass window in the Royal Palace of Edfena, located in Rosetta on the western shore of the Nile. Rosetta is considered a coastal city, as it is the confluence of the Nile River with the Mediterranean Sea [21]. It is also an ancient archaeological city. The city is distinguished by its unique historical, architectural, and decorative heritage, as it is considered a museum of Islamic architecture. It is the second Egyptian city after Cairo in terms of the number of antiquities [22]. Edfena lies approximately 10 kilometers south of Rosetta (Fig. 1).

Figure 1. Location of Edfena Royal Palace

The royal palace was built in three stages. The first stage took place during the era of Khedive Abbas Hilmi II (1892–1914). The second stage occurred during the reign of King Fouad (1917–1936), and the third stage was carried out under the rule of King Farouk (1937–1952) [23]. The Edfena Palace was distinguished by its impeccable quality, elegant beauty, and harmonious exterior design. Its layout forms the letter F, which can be seen from every angle. The palace was constructed in the Italian style (Fig. 2).

Figure 2. Shows the royal palace. (a) façade; (b) entire palace from above.

The stained glass window under study is situated on the western wall of the pavilion of Khedive Abbas Hilmi II (1892–1914). The window comprises three panels adorned with floral designs in brown, red, and turquoise colors, showcasing European influences [24]. The upper portion of the wall is arched with semicircular arches (Fig. 3).

Figure 3. Stained glass window under study: (a) from inside; (b) from outside.

Furthermore, the Royal Palace's exploitation, as allocated to the College of Veterinary Medicine from 1974 until 2020, and the resulting misuse of human behavior have contributed to the deterioration of the heritage building's condition [25]. Even after the college vacated the premises, the palace has yet to undergo any restoration processes. Similarly, the previous restoration attempts made on the studied glass window were not conducted by specialized restorers, leading to the distortion of its overall appearance and the failure to preserve its aesthetic and functional values.

Therefore, this research seeks to study the current state of the stained glass window by photographing and documenting it using AutoCAD programs and following scientific methods for examination and analysis to identify the chemical composition and the various deterioration forms that affect the stained glass window. The paper also intends to develop a scientific plan to preserve and conserve the stained glass window.

2. MATERIALS AND METHODS

2.1. SAMPLING

The current study was conducted on samples of broken pieces of painted glass (turquoise, red, and brown) that fell from the window, and samples of the lead came and putty were used for examination and analysis.

2.2. VISUAL ASSESSMENT

The stained glass window was observed with a SONY camera. The camera features include a 25 mm wide-angle lens (35mm format), Full HD movie (1920 * 1080) with up to 16x Clear Image Zoom and a 16 mm CMOS sensor with 8X optical zoom.

2.3.AUTOCAD DOCUMENTATION

The documentation results obtained with the AutoCAD program revealed that the stained glass window had certain deteriorating features.

2.4. DIGITAL USB MICROSCOPY

Digital microscopy uses the PZ01 model (Shenzhen Supereyes Co. Ltd, China), uses a USB 2.0 interface, and is compatible with Linux and Mac OS versions higher than 10.5.5. It is a non-destructive testing technique. This technique provides a 10X–500X magnification range.

2.5. STEREO MICROSCOPY

A Leica MZ6 stereo zoom microscope (Leica Geosystems AG, Heerbrugg, Switzerland) with 10x eyepieces and a magnification range of 6.3 x to 40 x was employed. The examination was carried out in Egypt's Dokki District by the Egyptian Mineral Resources Authority.

2.6. SCANNING ELECTRON MICROSCOPY - ENERGY DISPERSIVE X-RAY SPECTROMETRY

Scanning electron microscopy-energy-dispersive X-ray spectrometry (SEM-EDX) was utilized to examine the external surfaces of stained glass. It provides several exact details, such as durability, scratches, surface deposition layers, pits, and fissures. A model SEM Quanta 250 FEG (FEI Corporation, USA) attached to the Energy Dispersive X-ray Analytics (EDX) unit was used, with a higher voltage of 30 kV, 14 x magnifications up to 1,000,000, and a gun resolution of 1n. This procedure was done in the SEM Laboratory of the Egyptian Mineral Resources Authority in Dokki, Egypt.

2.7. X-RAY DIFFRACTION

To investigate the presence of crystalline components and potential degradation in archaeological glass panels, X-ray diffraction (XRD) analysis was used. Glass, a non-crystalline substance, typically does not yield significant results in XRD analysis unless there is degradation or the presence of crystalline phases of metallic oxides used for coloring the glass. The XRD analysis was performed using the model D8 (Burker, Germany), which offers reflectometry, high-resolution diffraction, in-plane grazing incidence diffraction (IP-GID), and small-angle X-ray scattering (SAXS) capabilities. This analysis took place at the Egyptian Centre for Nanotechnology, located in Sheikh Zayed, Egypt, affiliated with Cairo University.

2.8. MICROBIOLOGICAL EXAMINATION

The Microbiological Examination involved assessing fungal degeneration by taking sterile cotton swabs and collecting samples from various areas of a painted glass window. These samples were then placed on Petri dishes containing Potato Dextrose Agar (PDA), which consisted of 200 g of potato extract, 20 g of dextrose, 20 g of agar, and one liter of distilled water. Subsequently, the plates were incubated for 5-7 days at a temperature of 28°C. Finally, the samples were assessed at Cairo University's Faculty of Science's Microanalysis Centre.

3. RESULTS AND DISCUSSION

3.1. VISUAL ASSESSMENT

Visual evaluation is considered the first step in the examination process and is used to assess the condition of stained-glass windows and the type of deterioration. It is observed that the window has deteriorated, and various aspects of damage have appeared due to neglect and lack of protection over many years. Among the significant forms of damage found are curvature, illumination, use of heterogeneous glass, limpness, darkening, cracks, fractures, and loss of glass pieces.

Curvature (Fig. 4a) is observed, indicating that the glass window is curved with a convex inward and concave outward curvature. This curvature arises from the weakened state of the lead strips, which compromises the structure and cohesion of the window. The combined effect of strong winds and temperature fluctuations contributes to the slackness and fragility of the lead strips, eventually leading to the subsequent loss of glass pieces [26].

Illumination (Fig. 4b) refers to voids that expel glass pieces from the lead cavity when the putty material is lost and the lead strips harden. These openings allow light to penetrate, distorting the window's aesthetic appearance [27]. This marks the onset of glass piece collapse, as increased pressure causes the voids to expand until the glass pieces dislodge and become lost.

The use of heterogeneous glass for the missing parts (Fig. 4c, d, e) implies that the window underwent restoration work some time ago, and the missing glass pieces were replaced with colorless or improperly colored glass, which distorts the overall aesthetic design. Additionally, a significant accumulation of dust and cobwebs is observed on the surface of the window glass (Fig. 4d, e).

Limpness (Fig. 4f) denotes a general weakness in the lead strips, causing a significant reduction in their ability to hold the glass pieces in an upright position. This results in a lack of cohesion within the lead cavity and the loss of the putty material [28]. Limpness is particularly noticeable in the lower part of the window due to increased loads in that area.

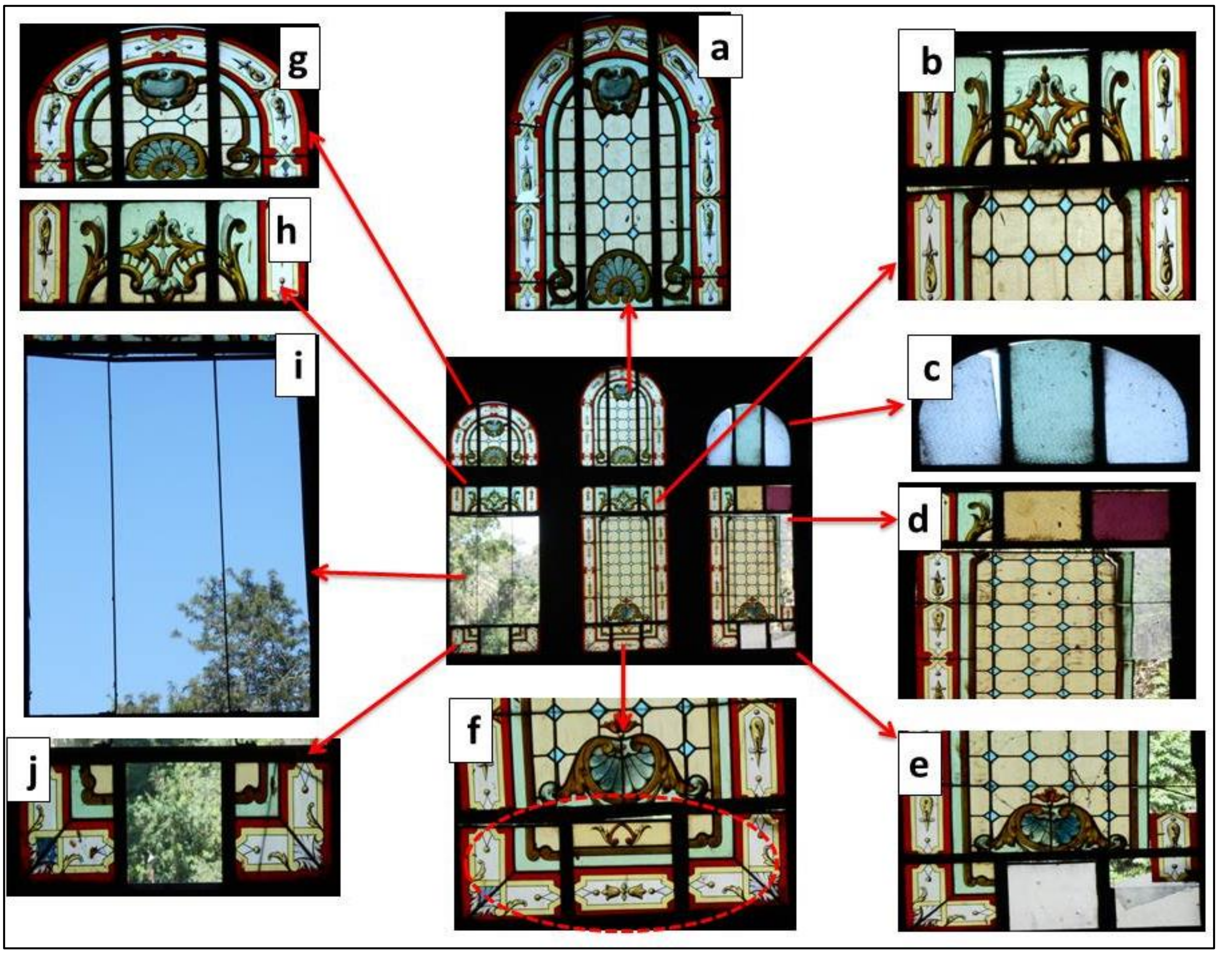


Figure 4. Various aspects of deterioration in stained glass window (a) curvature of stained glass; (b) illumination of the glass; (c, d, e) use of heterogeneous glass for the missing parts; (e, d) cobwebs and dirt stuck to the surface; (f) limpness of lead strips; (g, h) darkening of glass paintings; (i) loss of glass pieces; (j) cracks and loss of glass pieces.

Darkening (Fig. 4g, h) indicates the opacity or loss of transparency in the glass resulting from high humidity. The coastal environment with heavy rainfall severely affects the window, causing the glass to become opaque and lose its transparency [29].

Due to the fragile nature of glass, glass pieces are susceptible to cracking and breakage under pressure. This vulnerability becomes more pronounced when the lead strips lose their strength and flexibility, leading to twisting and exerting pressure on adjacent glass pieces [30], resulting in the formation of cracks (Fig. 4j). As this phenomenon worsens, it leads to the eventual loss of glass pieces (Fig. 4i, j).

3.2. AUTOCAD DOCUMENTATION

The dimensions of the stained glass window were confirmed and documented using the AutoCAD software. It is used to precisely record the many types of deterioration that are present in the glass window (Fig. 5).

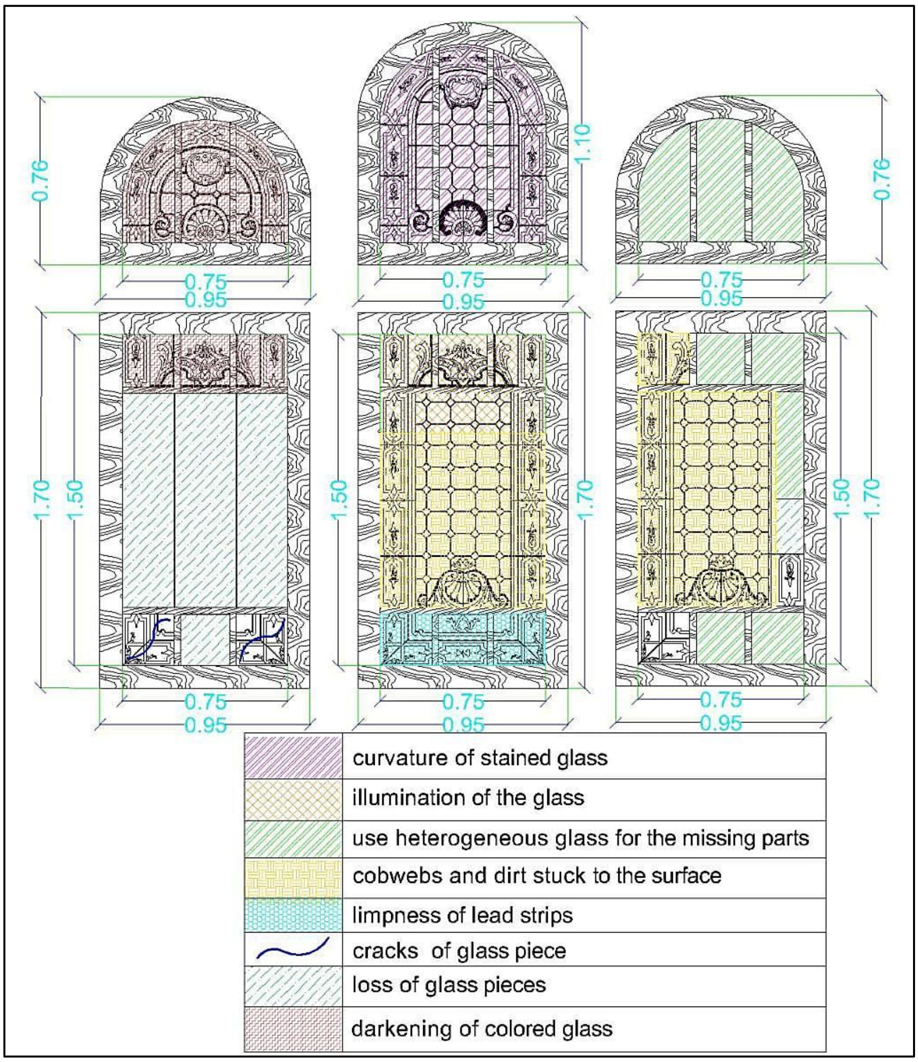


Figure 5. Documentation of the stained glass window using AutoCAD.

3.3. DIGITAL MICROSCOPY

Digital microscopy is regarded as one of the most important techniques available to restorers, and its images aid in assessing the aspects of deteriorated objects [31]. It is observed that the surface of the transparent glass bearing the paint layers is full of air bubbles (Fig. 6a). Air bubbles are a manifestation of manufacturing defects resulting from the glass not achieving a homogeneous mixture during the melting process, representing the weakness of the glass [32]. Additionally, pitting is observed on the surface of the red paint layer (Fig. 6b). Scratches are visible on the surface of the brown paint (Fig. 6c). The surface of the turquoise paint shows air bubbles and fine cracks (Fig. 6d), as well as the deposition of dust, dirt, and pits filled with corrosion products (Fig. 6e). The surface of the lead came exhibits a layer of oxidation and damage to the lead metal in the form of white calcifications on the surface (Fig. 6f).

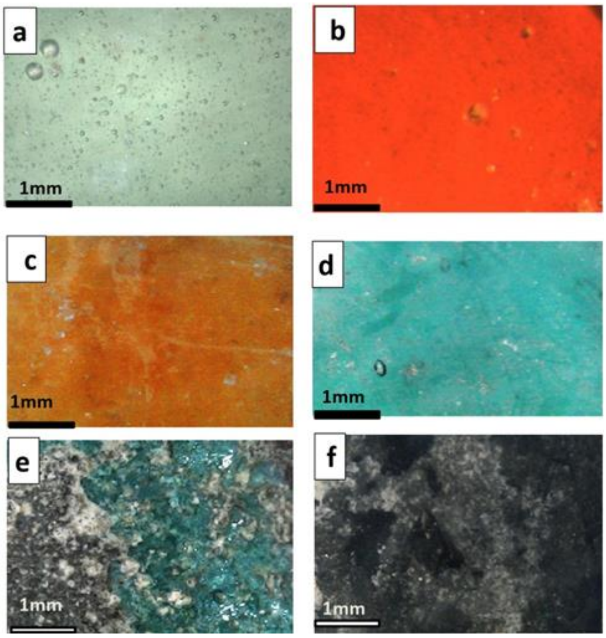


Figure 6. Digital microscope images showing the results of the samples examination (a) air bubbles on the transparent glass; (b) pits on red painted glass; (c) scratches on brown painted glass; (d) microcracks and air bubbles on turquoise painted glass; (e) accumulation of deposited layers of dust and dirt on the surface of the turquoise painted glass; (f) the lead came with an oxidized surface.

3.4. STEREO MICROSCOPY

Stereo microscopy is used to observe and analyze changes in the characteristics of glass surfaces, aiding in estimating potential damage. On the surface of the transparent glass, cracks, fissures, and opacity can appear (Fig. 7a).

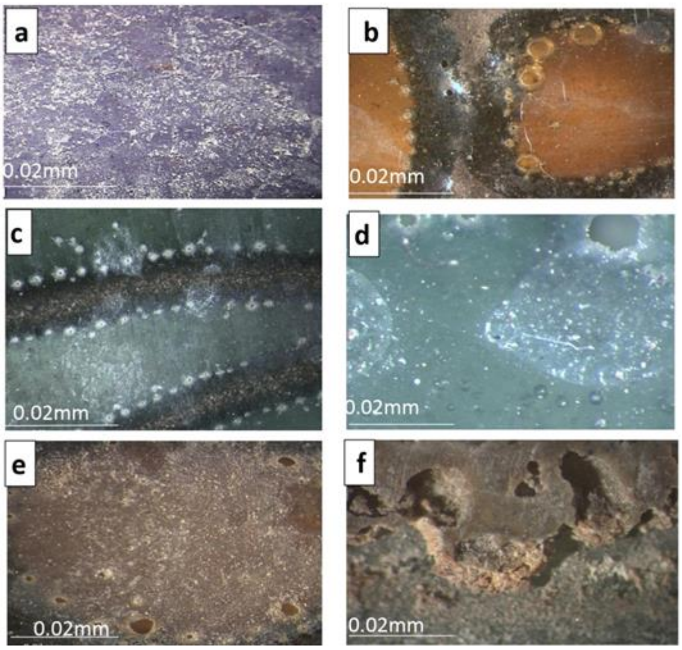
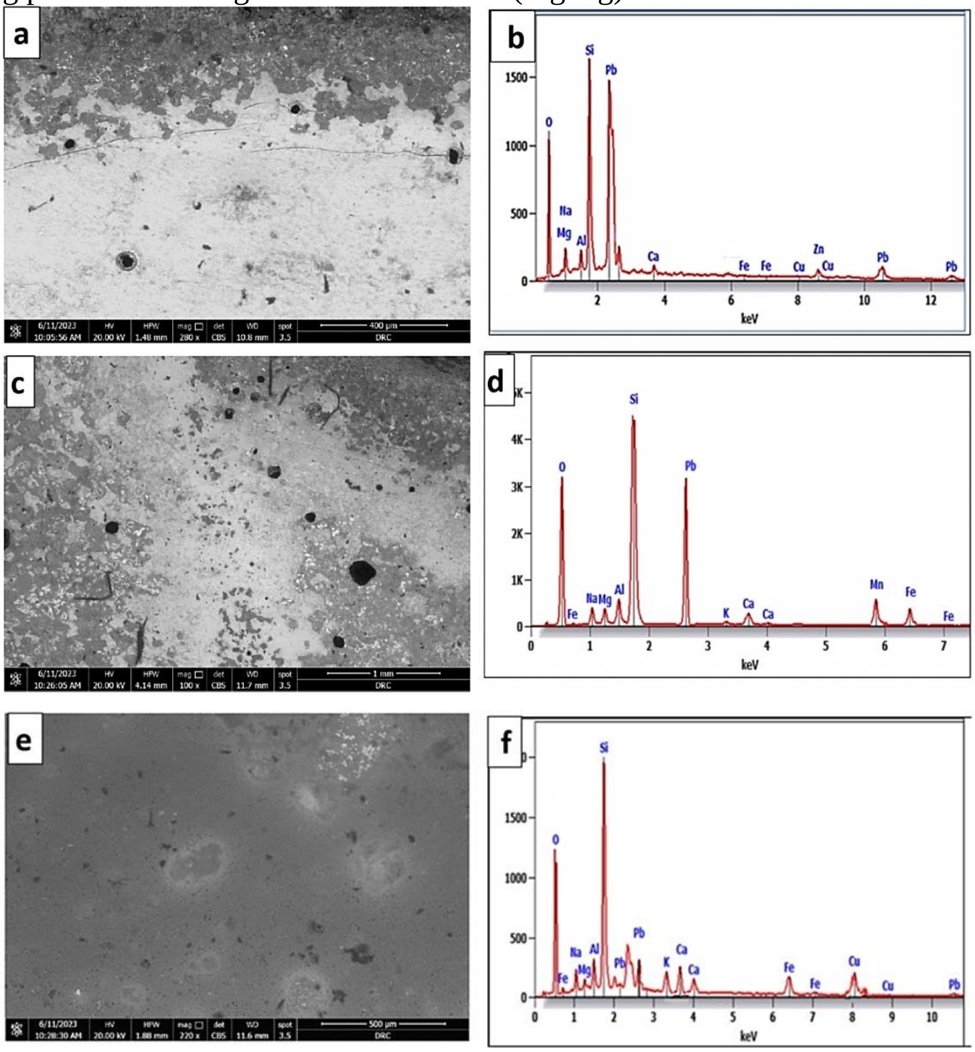


Figure 7. Stereomicroscope images showing the results of the samples examination (a) cracks, fissures, and opacity of the glass surface; (b) pits and blisters on the brown painted surface; (c) optical holes and pits parallel to the tracing line in the turquoise paint; (d) craters on the turquoise painted surface; (e) deposition of deterioration products on the surface of the brown painted glass; (f) lack of cohesion between the grains of the putty sample.

Pits and blisters are noticeable in the brown paint layer surrounded by black liner, leading to damage to the paint layer (Fig. 7b) [33]. Optical holes and pits appear parallel to the tracing line in the turquoise paint (Fig. 7c). Additionally, the surface of the turquoise paint exhibits craters resembling volcanic craters in the form of deep pits (Fig. 7d). Deposition of corrosion products in the form of a thin white layer on the surface of the brown painted glass, resulting in opacity, is observed (Fig. 7e) [34]. The examination of the putty used to cement the lead strips and the glass reveals a lack of cohesion between its grains, weakness, and deterioration (Fig. 7f).

3.5. SCANNING ELECTRON MICROSCOPY - ENERGY DISPERSIVE X-RAY SPECTROMETRY

Examining the glass samples using the SEM method, which is extensively used to evaluate the surface appearance of the painted glass sample, resulted in the detection of corroded surfaces and the clarification of deterioration. The red paint sample (Fig. 8a) shows an irregular distribution of painted oxides on the glass surface and cracks. The brown paint sample (Fig. 8c) exhibits a layer of superficial deposits resulting from corrosion processes, along with holes and pits on the surface. The turquoise paint appears stable but contains some pores (Fig. 8e). The lead sample shows apparent corrosion in the lead layer, with sunken and protruding parts and an irregular and flat surface (Fig. 8g).



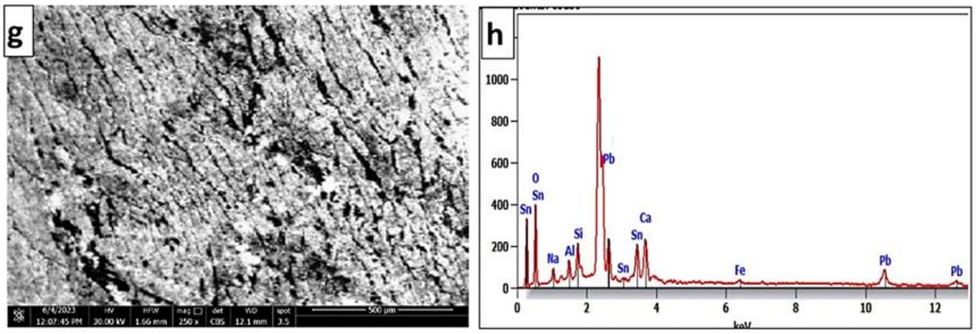


Figure 8. SEM images and EDX spectra of the analyzed samples (a, b) red paint; (c, d) brown paint; (e, f) turquoise paint; (g, h) the lead came sample.

EDX analysis was conducted to determine the percentage of elements in the samples under study, and the results are shown in Table 1. It is observed that the percentage of silica in the painted glass samples is less than the basic rate. The red-painted glass sample has a silica percentage of (35.5%), and the brown sample has (38.9%). However, the turquoise glass sample has a higher percentage of silica (49.4%), indicating relative stability. The percentage of alkaline substances in the painted glass is low, with sodium (avg. 4.2%) and potassium (avg. 1.7%). This indicates that the layers of different paints may have been damaged due to moisture or condensation, resulting in alkaline substances migrating to the surface and dissolving [35-43]. The results also indicate a higher percentage of sodium than potassium, indicating that this glass consists of the soda-lime-silica type. The results also show a high percentage of lead in painted glass samples (avg. 22.3%), indicating lead oxide as a fluxing agent for low-melting glass paint [12]. Calcium is also present in proportions (avg. 4.53%) and included in mixtures as a stabilizing agent [44].

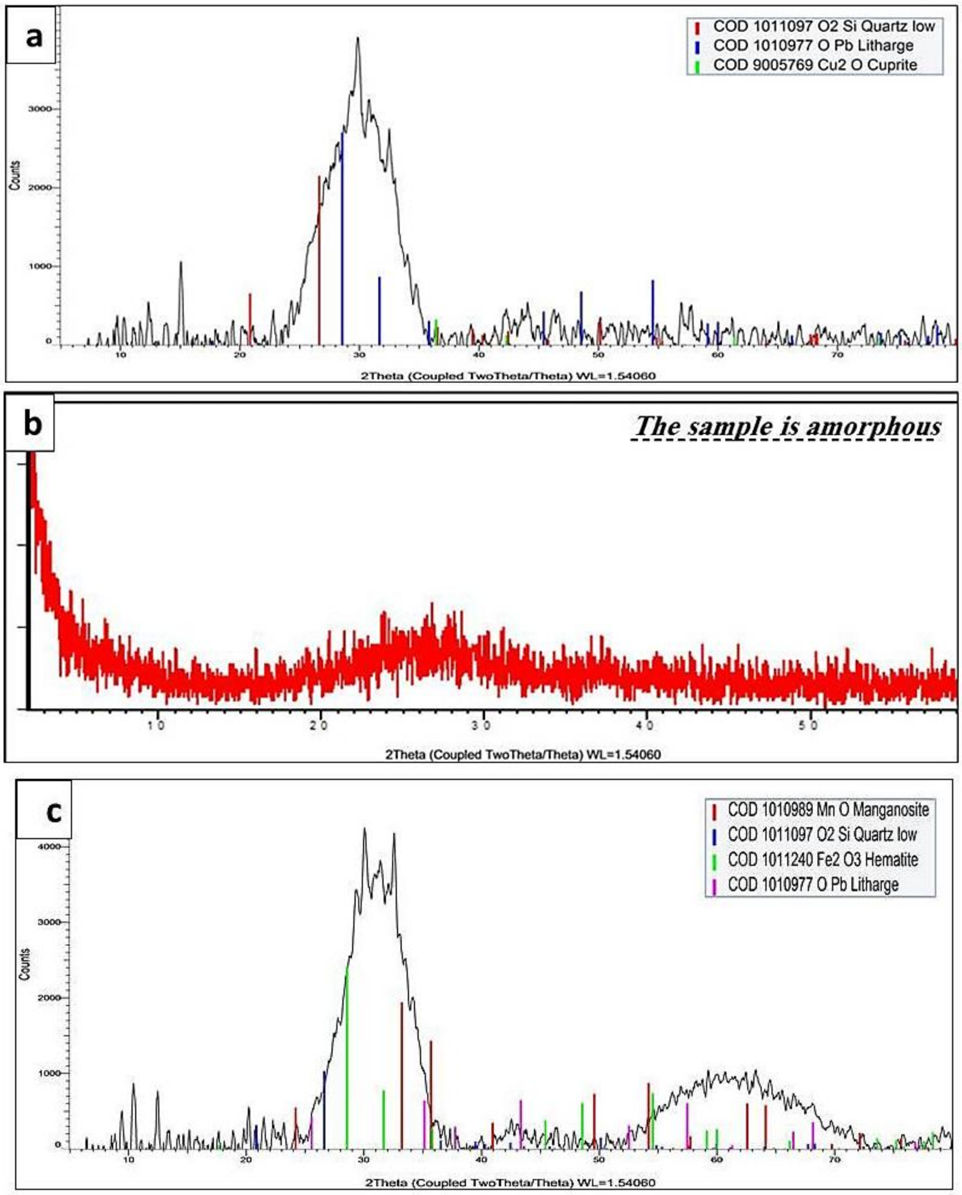
Table 1. The percentage of elements in painted glass samples and the lead came with EDX.

Sample	Elements [wt.%]												
	O	Si	Na	Ca	Mg	Al	Fe	Pb	K	Cu	Mn	Zn	Sn
Red paint	20.7	35.5	3.9	5.8	0.7	1.5	0.3	28.9	----	1.9	----	0.8	----
Brown paint	20.6	38.9	3.2	1.3	2.6	3.5	1.8	25.6	0.2	----	2.3	----	-----
Turquoise paint	10.7	49.4	5.6	6.5	2.1	5.7	1.6	12.4	3.2	2.8	----	----	----
Lead sample	14.0	1.6	1.0	3.2	----	1.0	1.1	68.3	----	----	----	----	9.8

The EDX results reveal the presence of a coloring agent in the painted glass, such as copper, at a percentage of (1.9%) in the red painted layer responsible for the red color [45-47]. Manganese and iron appear at a percentage of (2.3% and 1.8%, respectively) in the brown-painted glass sample, contributing to the brown color [48]. Copper and iron appear at a percentage of (2.8% and 1.6% respectively) in the turquoise-painted glass, responsible for the turquoise color [49]. Additionally, the EDX results show that the lead came sample is composed of a high percentage of lead oxide as the main component and a significant amount of tin, indicating that lead came is made of an alloy of lead and tin to improve its properties, strength, and durability [50].

3.6. X-RAY DIFFRACTION

This method is used to qualitatively determine compounds in crystalline materials since the rays interact with the crystalline structure [51]. In this study, XRD is employed to analyze painted glass samples to identify the crystalline patterns representing damage to the paint layers, as it has the same properties as glass in that it is an amorphous material [52]. The sample of the red-painted layer (Fig. 9a) consists of quartz (48%), cuprite (17.5%), and lead oxide (34.5%). Quartz is an essential component of the painted glass layer, while cuprite is responsible for the red color [53]. Lead oxide is a fluxing agent and an essential component in the color mixture [54]. The turquoise paint layer is amorphous (Fig. 9b), indicating that it has not been damaged, which aligns with the results obtained from the EDX. The sample of the brown paint layer consists of quartz (40.9%), manganese oxide (10.6%), hematite (18.5%), and lead oxide (30%) (Fig.9c). Quartz and lead oxide are the main components of the painted layer. In contrast, manganese oxide and hematite are responsible for the brown color [55]. The putty sample (Fig. 9d) consists of calcite (81.4%), halite (15.9%), and hematite (2.7%). The presence of halite salt indicates that the putty has been damaged and the salts have crystallized within it [56].



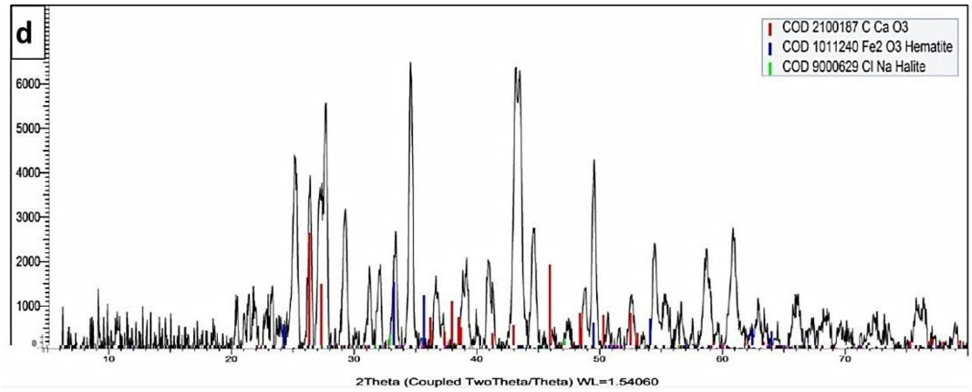


Figure 9. X-ray diffraction pattern showing the results of the studied samples. (a) red paint; (b) turquoise paint; (c) brown paint; (d) putty sample.

3.7. MICROBIOLOGICAL EXAMINATION

The study revealed the growth of the *Aspergillus flavus* fungus, depicted in (Fig. 10a), as a yellowish-green color in a petri dish and visible under the microscope in (Fig. 10b). Additionally, the presence of Aspergillus niger fungus was observed, appearing as a blackish-brown color (Fig. 10c), and visible under the microscope as shown in (Fig. 10d). These fungi are widespread. They produce spores known as ascospores, which contribute to the physical and chemical damage of the window components [57].

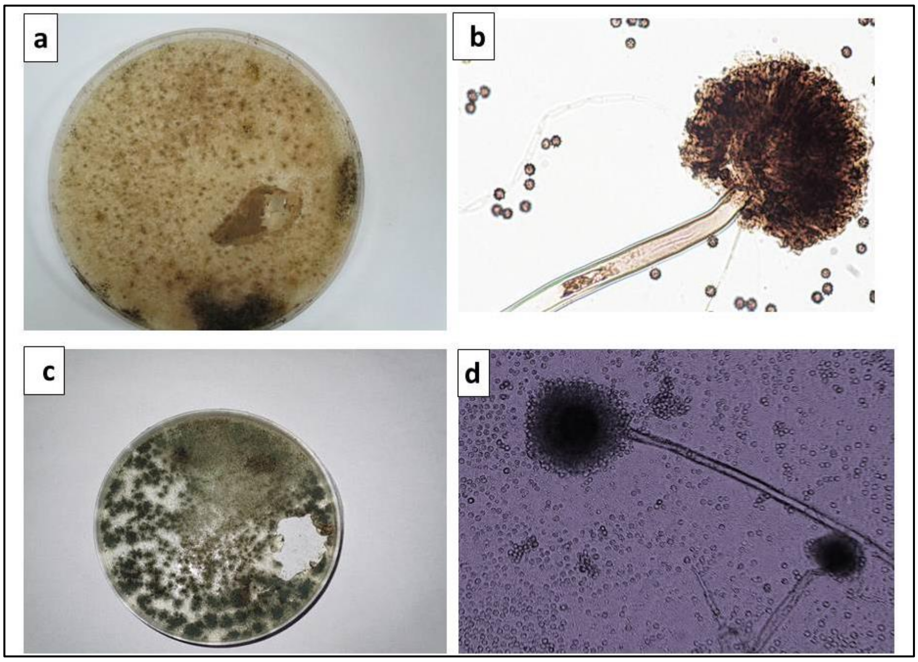


Figure 10. Identification of the fungi that infect the stained window glass (a, b) *Aspergillus flavus* fungus; (c, d) *Aspergillus niger* fungus.

3.8. PROPOSED CONSERVATION STAGES FOR THE STAINED GLASS WINDOW

Removing the window from its current location due to its height and transporting it to the workplace is preferable to facilitate maintenance and conservation operations. The initial stage involves mechanical cleaning using soft brushes and skilled restorers to remove dust and dirt. In cases where mechanical methods are insufficient, chemical cleaning solutions and solvents such as acetone and alcohol can be employed [58, 59]. Next, broken pieces are carefully assembled using Kemapoxy 101 SF with 3% nano-Al_2O_3 [31]. Afterward, any missing parts are completed, and thermal color treatments are applied. The examination of the window damage indicates a significant loss of glass panels. The original existing parts serve as a reference for studying the details of the missing glass pieces. Thermal color treatments involve oxides and color pigments, which are fixed by heating the glass pieces at temperatures between 500-600°C [12]. It is essential to replace any non-matching glass with colored glass that matches the original decoration in the window.

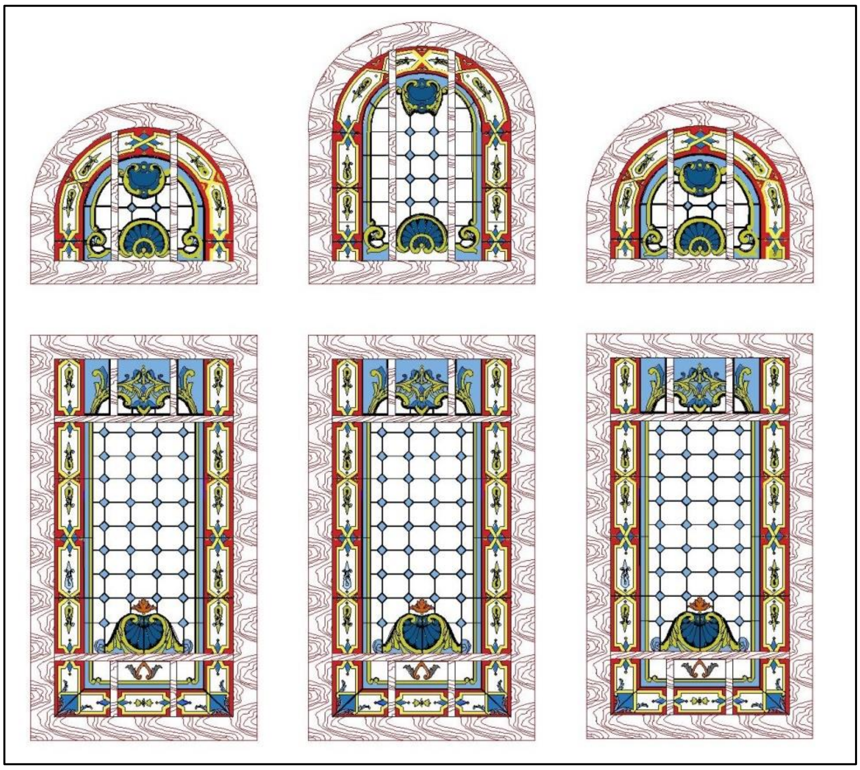


Figure 11. Shows the stained glass window after the imaginative conservation, with the missing parts completed using original decorations and colors

Furthermore, the weakened existing lead strips should be carefully reused by tightening them to restore their original shape and replacing damaged ones with modern equivalents. Soldering is performed to ensure the stability of the lead strips. Additionally, a putty mixture comprising calcium carbonate, linseed oil, and red lead oxide is applied to preserve and strengthen the glass fillings [60]. This putty mixture exhibits excellent adhesion to lead and glass and effectively withstands heat and humidity. Moreover, it is necessary to use natural oils as fungicides, as they can inhibit the growth of fungi, including clove and cumin oils, mixed in a small proportion (0.5%) with consolidation materials [61]. For consolidation purposes, it is preferable to utilize a combination of nano-alumina and nano-silica at 2% with Silres BS-290 at 7% [18]. Fig. 11 illustrates the window after the imaginative conservation, with the missing parts completed using original decorations and colors.

4. CONCLUSIONS

The stained glass window in the Royal Palace of Edfena has exhibited numerous signs of deterioration due to neglect and damage, which is consistent with the overall condition of the palace. The surrounding environment, particularly the coastal location, significantly affects the stained glass window by increasing humidity, the primary enemy of archaeological glass surfaces. High humidity leads to the loss of alkaline compounds and accelerates the corrosion process. Therefore, a diagnostic and analytical study using various techniques is crucial for understanding the different aspects and causes of deterioration and establishing the scientific basics for appropriate treatment methods.

Visual examination and documentation using AutoCAD revealed a substantial loss of decorative glass panels in the stained glass window. Moreover, non-homogeneous glass was observed, deviating from the original colors of the decorations. The appearance of illumination in the glass panels and the opacity of the glass surface were also observed. Through microscopic examination using digital, stereo, and scanning electron microscopes, it was observed that the layers of glass paint had numerous optical holes, scratches, cracks, and surface corrosion. Analysis using EDX noted a low percentage of silica and alkalis in the composition of glass paints, explaining the extent of weakness in their composition. The results also indicate that the glass used in the stained window is of the soda-lime-silica type. EDX analysis also observed the essential elements that cause colors in the glass paints, with copper appearing in red, manganese with iron in brown, and iron and copper in turquoise. Lead was also observed in all glass painting samples, indicating its use as a fluxing agent in all paint mixtures. The composition of the lead came was also identified, consisting of lead as an essential component along with tin, indicating it is manufactured from an alloy of lead and tin.

Through the analysis using XRD, crystallized compounds were identified in glass paints, indicating that they were subjected to damage. The presence of silica oxide with cuprite and lead oxide was observed in the red paint. In contrast, the brown paint showed lead oxide with silica oxide, manganese oxide, and hematite, which is consistent with the EDX analysis. No crystallized layers appeared in the turquoise paint layer, indicating its stability and lack of damage, consistent with the EDX analysis for the turquoise paint. The composition of the putty layer was identified as consisting of calcium carbonate with hematite, as well as the presence of halite salts. This indicates the crystallization of salts in the putty layer, exerting pressure on the glass window. Microbiological analysis revealed the presence of two types of *Aspergillus* fungi, namely *Aspergillus niger* and *Aspergillus flavus*, due to the high humidity in the environment surrounding the window. Finally, an appropriate treatment plan for the glass window is proposed based on the examination and analytical study results. The study recommends paying attention to the Royal Palace and the stained glass window under study due to their great archaeological and historical value.

REFERENCES

[1] García-Heras, M., Carmona, N., Gil, C., Villegas, M.A., *Journal of Cultural Heritage*, **6**, 91, 2005.

[2] Corrêa Pinto, A., Macedo, M., Vilarigues, M., *Journal of Cultural Heritage*, **34**, 172, 2018.
[3] Reynolds, E., *JCCC Honors Journal*, **4**, 2, 2013.
[4] Stanley, D., *Journal of the Society of Architectural Historians*, **65**, 334, 2006.
[5] Abdel Baky, N., Egyptian *Journal of Archaeological and Restoration Studies*, **7**, 103, 2017.
[6] Hunault, M., Bauchau, F., Boulanger, K., Hérold, M., Calas, G., Lemasson, Q., Pichon, L., Pacheco, C., Loisel, C, *Journal of Archaeological Science: Reports*, **35**, 3, 2021.
[7] Silvestri, A., Molin, G., Pomero, V., *Spectrochimica Acta Part B: Atomic Spectroscopy*, **66**, 81, 2011
[8] Carmona, N., Wittstadt, K., Römich, H., *Journal of Cultural Heritage*, **10**, 404, 2009.
[9] Domınguez Rodes, M.C., Canellas Martinez, S., *Journal of Cultural Heritage*, **9**, 86, 2008.
[10] Schibille, N., Marii, F., Rehren, T., *Archaeometry*, **50**, 628, 2008.
[11] Bassoa, E., Riccardia, M.P. Messigaa, B. Menderab, M., Gimenoc, D., Garcia-Vallesd, M., Fernandez-Turiele, J.L, Bazzocchic, F., Aulinasc, M., Tarozzif, C., *Material Characterization*, **60**, 1546, 2009.
[12] Schalm, O., Van der Linden, V., Frederickx, P., Luyten, S., Van der Snickt, G., Caen, J., Schreiner, M. *Spectrochimica Acta Part B: Atomic Spectroscopy*, **64**, 812, 2009.
[13] Pinto, A.M.C., Sanjad, T.A., Angélica, R.S., da Costa, M.L., Paiva, R.S., Palomar, T., *Boletín de la Sociedad Española de Cerámica y Vidrio*, **57**, 133, 2018.
[14] Bernardi, A., Becherini, F., Bassato, G., Bellio, M., *Journal of Cultural Heritage*, **7**, 71, 2006.
[15] Kontozova-Deutsch, V., Deutsch, F., Godoi, R.H., Van Grieken, R., De Wael, K., *Microchemical Journal*, **99**, 509, 2011.
[16] Eloriby, R., *Egyptian Journal of Archaeological and Restoration Studies*, **11**(2), 176, 2021.
[17] Shams, A.H., AlKaradawi, A.S., Eloriby, R.A, *Mediterranean Archaeology and Archaeometry*, **22,** 53, 2022.
[18] Eloriby, R.A., Mohamed, W.S., Alkaradawi, A.S. *Mediterranean Archaeology and Archaeometry*, **22**(1) 68, 2022.
[19] Robinet, L., Hall, C., Eremin, K., Fearn, S., Tate, J. *Journal of non-crystalline solids*, **355**(28-30), 1479-, 2009.
[20] Papadopoulos, N., Drosou, C.A, *Journal of the University of Chemical Technology & Metallurgy*, **47**, 429, 2012.
[21] Darwish, M.A., *International Journal of Innovation and Applied Studies*, **18**(4), 1239, 2016.
[22] Shaheen, B., Madkour, M., Sharaf El-deen, S., Rezk, A., *Mansoura Engineering Journal*, **46**(3), 56, 2021.
[23] Overton, J., *Master Thesis*, Simon Fraser University, **135**, 1971.
[24] Behrens-Abouseif, D., *Brill*, **3**, 89, 1992.
[25] Moussa, A., *Shedet*, **5**(5), 230, 2018.
[26] Gregorini, A., Paganoni, S., Zappa, E., Cigada, A., Canali, F., *Journal of Cultural Heritage*, ***56***, 65, 2022.
[27] Rambusch, V.B, *Technology and Conservation*, **8**(3), 46, 1984.
[28] Schalm, O., Caen, J., Janssens, K., *Studies in conservation*, **55**(3), 217, 2010.
[29] Gentaz, L., Lombardo, T., Chabas, A., Loisel, C., Neff, D., Verney-Carron, A., *Corrosion Science*, ***109*****,** 206, 2016.
[30] Becherini, F., Bernardi, A., Daneo, A., Geotti Bianchini, F., Nicola, C., Verità, M., *Studies in Conservation*, **53**, 238, 2008.
[31] Eloriby, R.A., Mohamed, H.M. *Pigment & Resin Technology*, 2023. https://doi.org/10.1108/PRT-07-2023-0064.
[32] Dawi, S.G.E., Mostafa, M.M., Eloriby, R.A.N. *Journal of Architecture, Arts and Humanistic Science*, **4**, 214, 2019.
[33] Perezy Jorba, M., Dallas, J.P., Bauer, C., Bahezre, C., Martin, J.C., *Journal of Materials Science*, **15**, 1640, 1980.

[34] Abd-Allah, R. *International Journal of Heritage in the Digital Era*, **1,** 277, 2012.
[35] Tournie, A., Ricciardi, P., Colomban, P., *Solid State Ionics*, **179**, 2142, 2008.
[36] Melcher, M., Wiesinger, R., Schreiner, M., *Accounts of Chemical Research*, **43**(6), 916, 2010.
[37] Abd-Allah, R., *Journal of Archaeological Science*, **37**(8), 1866, 2010.
[38] Van der Snickt, G., Schalm, O., Caen, J., Janssens, K., Schreiner, M., *Studies in Conservation*, **51**, 212, 2006.
[39] Ion, R.M., Bakirov, B.A., Kichanov, S.E., Kozlenko, D.P., Belushkin, A.V., Radulescu, C., Dulama, I.D., Bucurica, I.A., Gheboianu, A.I., Stirbescu, R.M., *Applied Science-Basel*, **10**(11), 3781, 2020.
[40] Bintintan, A., Gligor, M., Dulama, I.D., Teodorescu, S., Stirbescu, R.M., Radulescu, C., *Rev. Chimie*, **68**(4), 847, 2017.
[41] Bintintan A., Gligor M., Radulescu C., Dulama I.D., Olteanu R.L., Stirbescu R.M., Teodorescu S., Bucurica I.A., *Analytical Letters*, **52**(15), 2348, 2019
[42] Ion R.M., Iancu L., Vasilievici G., Grigore M.E., Andrei R.E., Radu G.I., Grigorescu R.M., Teodorescu S., Bucurica I.A., Ion M.L., Gheboianu A.I., Radulescu C., Dulama I.D., *Coatings*, **9**, 231, 2019.
[43] Tomus (Szabo), D.E., Gligor, M., Dulama, I.D., Radulescu, C., Bucurica, I.A., Stanescu, S.G., Stirbescu, R.M., *Journal of Science and Arts*, **21**(1), 285, 2021.
[44] Alberta, S., Gianmario, M., Valentina, P., *Spectrochimica Acta Part B: Atomic Spectroscopy*, **66**(1), 81, 2011.
[45] Mirti, P., Davit, P., Gulmini, M., *Analytical and Bioanalytical Chemistry*, **372**(1), 221, 2002.
[46] Farges, F., Etcheverry, M.P., Scheidegger, A., Grolimund, D., *Applied Geochemistry*, **21**(10), 1715, 2006.
[47] Colombian, P., Tournie, A., Ricciardi, P., *An International Journal for Original Work in all aspects of Raman Spectroscopy*, **40**(12), 1949, 2009
[48] Schalm, O., Janssens, K., Caen, J., *Spectrochimica Acta Part B: Atomic Spectroscopy*, **58**(4), 589, 2003.
[49] Bracci, S., Bartolozzi, G., Burnam, R.K., Corallini, A., Journal *of Cultural Heritage*, **44,** 307, 2020.
[50] García-Heras, M., Villegas, M.A., Caen, J.M.A., Domingo, C., García-Ramos, J.V., *Microchemical Journal*, **83**(2), 81, 2006.
[51] Mohamed, H.M., Journal *of Science and Arts*, **22**(3), 723, 2022.
[52] Ojovan, M. I., *Advances in Condensed Matter Physics*, **2008**, 1, 2008.
[53] Nakai, I., Numako, C., Hosono, H., Yamasaki, K., *Journal of the American Ceramic Society*, **82**(3), 689, 1999.
[54] Machado, C., Oujja, M., Cerqueira Alves, L., Martínez-Weinbaum, M., Maestro-Guijarro, L., Carmona-Quiroga, P. M., Palomar, T., *Heritage Science*, **11**(1), 1, 2023.
[55] Palomar, T., Agua, F., Gómez-Heras, M., *International Journal of Applied Glass Science*, **9**(4), 530, 2018.
[56] García-Heras, M., Gil, C., Carmona, N., Villegas, M.A., *Materiales de Construcción*, **53**(270), 21, 2003.
[57] Ali, M.F., Afifi, H.A., Lotfy, A.M., *Journal of Science and Arts*, **21**(3), 789, 2021.
[58] Altavilla, C., Ciliberto, E., La Delfa, S., Panarello, S., Scandurra, A., *Applied Physics A*, **92,** 251, 2008.
[59] Eloriby, R.A., Mohamed, H. A., *International Journal of Conservation Science*, **14**(4) 1279, 2023.
[60] Palomar, T., *Boletín de la Sociedad Española de Cerámica y Vidrio*, **57**(3), 101, 2018.
[61] Geweely, N. S., Afifi, H. A., Ibrahim, D. M., Soliman, M. M., *Geomicrobiology Journal*, **36**(2), 148, 2019.