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Diagnostic Investigation and Conservation Methods of the Wall Ceramic Mosaic in The Historical Church of the Blessed Virgin Mary, Mostorod, Egypt

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ABSTRACT

This paper presents a diagnostic examination and conservation of a ceramic mosaic in the Church of the Blessed Virgin Mary in Mostorod, Egypt. The panel, located in the cave of the church, depicts the Holy Family and is named "Cave Mosaic Panel" However, various deterioration factors and previous restoration works have negatively affected the mosaic panel. The panel has suffered from many aspects of deterioration, particularly buckling, soot, dirt, salts, and the loss of some pieces. The study aims to identify the type and structure of the ceramic mosaic, determine and diagnose aspects of deterioration, and carry out the necessary conservation works on the panel to protect it in the future. A diagnostic study was carried out using Scanning Electron Microscopy with Energy Dispersive X-ray, X-Ray Diffraction and Fourier-Transform Infrared Spectroscopy. The results showed the chemical composition and the type of colouring oxides of the ceramics. Cobalt oxide was used to obtain a dark blue glaze, with alkali and lead alkaline as fluxes in the glaze layer, and the degree of firing of the ceramic pieces higher than 1250 °C. Halite was detected between the tesserae. The results confirmed the use of animal glue as an adhesive for fixing some mosaic pieces. The treatment and conservation was carried out using mechanical and then chemical cleaning methods, the dissolved salts were extracted with a cotton poultice, and the buckling of mosaics were treated by injection with Primal AC33. The lost parts of the mosaics were completed. Consolidation and protection with pF4 nanocomposite was performed.

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INTRODUCTION:

The wall mosaic, known as the *cave ceramic mosaic mural portrait* (this name does not make sense: probably, Cave Mosaic Mural), is located in the historical Church of the Blessed Virgin Mary in Mostorod, about 10 km from Cairo, Egypt (Fig. 1 A). The church was built in the 12th century and its cave was discovered by St. Gabriel I in 1481. When it was still a synagogue and during their three-day stay in Egypt, the Holy Family used this cave. The Church of the Blessed Virgin Mary in Mostorod was an important stop for the Holy Family during their visit to Egypt. In Mostorod, the Virgin Mary, Christ, and St Joseph stayed in a small cave that could accommodate no more than two people and drank from a small well nearby. The Church of the Blessed Virgin Mary was built on this site (Habib, 3). It has become a favourite destination for blessings, traditions, and pilgrimages. It was named *Al Mahamaah* because Mary named the city where she showered Christ and washed his clothes (Abouzied and Aziz Abed, 2021, 198-215) (Fig. 1 B, C).

Figure 1. A. Location of the historical Church of the Blessed Virgin Mary in Mostorod, Egypt (Google Earth 15-9-2022); B. The staircase leading to the site of the wall mosaic; C. The cave mosaic panel of the Virgin Mary

Ceramic mosaic is one of the techniques used to glaze pottery or cut ceramic tiles into regular (1-2 cm) or irregular geometric shapes (Abdallah, 2017, 329-343). It is an art form used to create decorative patterns from small pieces. Its application requires a lot of time, effort, and craftsmanship. It is a form of artistic creativity and has various forms and types (Benyoussef , and Derrode 2011, 453-519). In Greek, the word *mouseios*, i.e mosaic, means the art and craft of making murals from small cubes that are incorporated into a mortar or adhesive mixture to decorate floors or walls with various geometric, floral, human, or animal shapes. Numerousmaterials can be used to create these murals, including stone, glass, shells, ceramics, and pottery. The word mosaic was later used by the Romans, who called it *vermiculatum opus musivum*.

In art, a mosaic is the decoration of a surface with patterns of carefully placed, usually differently coloured small pieces , which are arranged next to each other and form landscapes, geometric shapes, and human or animal figures (Hemdan, 2012, 14). The Greeks called it the *tessera technique* (Dunbabin, Katherine, 1999, 5). The earliest mosaic materials were small clay cones that were pressed into moist gypsum. Semi-precious stones such as lapis lazuli and agate, shells, and terracotta were also used. With the development of art, glass, ceramics, stone, and pebbles became the most commonly used materials. In the modern era, any small piece can be used individually, including traditional materials, moulded or chipped glass or ceramics, plastic, polymer dough,

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beads, buttons, bottle stoppers, and pearls (Fiori, Vandini, Prati, and Chiavari, 2009, 248–257). In the Mediterranean, the wealthy used mosaics to decorate certain parts of their homes during the Roman era. Later, the Byzantine Empire used mosaics extensively to permanently emphasise Christianity, especially in the decoration of churches (Andreescu, 1977, 20).

However, the deterioration of different types of mosaics is due to various factors. The deterioration of mosaics includes all forms of weathering forms and all processes affecting some or all components, including the primitive use of mosaics, natural disasters causing severe mechanical or thermal stress, and the deterioration of buildings causing severe mechanical damage to mural or floor mosaics. In addition, deterioration is caused by human factors such as improper use, negligence and previous restoration, which can lead to complete destruction (Chlouveraki, 2018, Inc,1-4) and alteration of the colour and texture of the mosaic pieces (Corradi, Leonelli, Veronesi, Fabbri, Macchiarola, Ruffini, Boschetti, and Santoro, 2005,402-405). Several signs of deterioration were observed in the ceramic mosaic on the wall (Fig. 2). It is located in a cramped place with high levels of various (soil and relative) moisture sources in the well / cave. Most historical and archaeological buildings in Egypt with their artistic and decorative elements are susceptible to deterioration due to of the high humidity (Ali, Omar, 2021, 9-17).

, We have therefore identified the efflorescence of the filling material under the mosaics. Dissolved salts are an important cause of deterioration and cause severe damage when they effloresce under the glaze layer of the mosaic pieces (Sara, N, Mona, F., 2015, 163-173). One of the deterioration phenomena of the mosaic is the calcification of wax and soot on the floor due to the use of candles below the mosaic during prayers, hymns and praises (Chabin, M., Preserving Christ's Birthplace: Restoration of Church of the Nativity Nears Completion, Posted on December 28, 2021, in: General News. https://www.holynameradio.org/Catholic-Community/News/articleType/ArticleView/articleId/220023/Preserving-Christs-Birthplace(24-3-2022)), causing damage, blurring, deformation, and colour changes (Skálová Z., Oerter W, B., 2020, 277-302). In addition, previous restorations and restorations by non- professionals in 2010, soot, dirt and improper actions by visitors have led to deterioration and loss of some mosaics of the mural.

The buckling of some parts is one of the main signs of deterioration of the mosaic. It is the separation of the mosaic layer from the ground or the separation, but not the falling off both layers from the primary support in the weak areas due to changes in the size of the mosaics or the materials forming the supports, the pressure of crystal growth of salts under the mosaic layer with cohesion at the edges, or the erosion of the iron used in the consolidation. In addition, the constant uneven expansion and shrinkage due to temperature and humidity variations (Cooper, 2008, 262) from day to night or seasonally in summer and winter are the main causes of pressure and weakness, leading to deterioration in the form of cracks, fractures, and buckling (Torraca, 1988, 25-26). The treatment and conservation of the wall mosaic was planned in accordance with its methodological principles. These include prior full documentation of the work (before, during and after conservation), fixing in place without separating the mosaic, minimum intervention, compatibility between the original materials and the conservation products, and maximum dissemination and discussion of the information gathered during the treatment.

Technically, the main objective of the present treatment to repair the mosaic and its parts that are susceptible to various deterioration factors(Nardi R., Zizola C., 2006, 18). The non-destructive examination and analysis of the mosaic parts has helped us to understand the manufacturing technique, the components, and the consequences of deterioration for appropriate restoration and conservation (Abd El Salam S., Mosaic in Libya, 2010, 99-151). In addition, it is important to take a conservation approach of the mosaic in its original location rather than moving it to another site and installing it there. Restoration and conservation procedures include mechanical and chemical cleaning, salt extraction, injection to fix the detached parts and complete the missing parts, and other procedures that ensure the conservation and protection of the mosaics (Ruby T, 2019, 239-288). The present study aims to identify and document the type and structure of the mosaic, the colour oxides, and the deterioration of the mosaic mural in the Church of the Blessed Virgin Mary in Mostorod. Scientifically, treatment and conservation methods were applied to overcome the deterioration and previous restorations. In this way, it helped to protect the studied mural as an artwork that should be preserved for future generations.

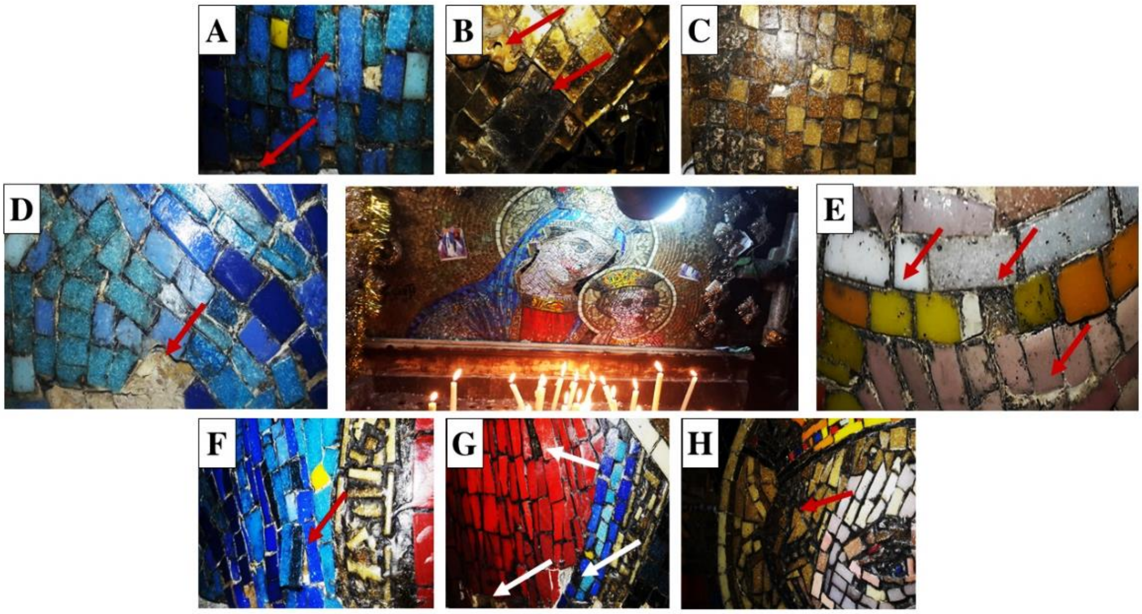

Figure 2 The deterioration aspects of the ceramic mosaic of the Virgin Mary in the cave; A. Dirt and missing parts, B. Missing mosaic and wax blocks, C. Darkness with discolouration and dirt, D. Missing parts, E. Efflorescence of salts and darkening of the filling mortar, missing parts with dirt, F. Buckling with detachment and soot, G. Buckling and wax, H. Local changes in the colour of the mosaic surface

2. MATERIALS AND METHODS

2.1. Sampling

Several samples of small mosaics, adhesives and salts were obtained and analysed using non-destructive examination and analysis methods.

2.2. Scanning electron microscopy (SEM) attached with EDX

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) was used to identify the physical properties, the type of glaze, the colours, and the components of the glaze layer of mosaic the using the connected device (Vecstaudža J., 2013 ,29,40-45). The characteristics of the device used were as follows: *Quanta 250 FEG (Field Emission Gun) model, connected to an EDX (Energy Dispersive X-ray Analysis)*

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unit, with an accelerating voltage of 30 K.V., with 14x up to 100000x magnification and resolution for the gun.

2.3. X-ray diffraction (XRD)

X-ray diffraction (XRD) was carried out to analyse the ceramic body and the filling material between the mosaic pieces. In addition, the type of efflorescent salts on the filling material was identified using a device with the following characteristics: *Diffractometer type: PW1840, Tube anode: Cu, Generator tension (KV): 40, Generator current (mA): 25, Wavelength Alpha1 (Å): 1.54056, Wavelength alpha2 (Å): 1.54439, Intensity ratio (Alpha2/Alpha1): 0.500, Receiving slit: 0.2, Monochromator used: NO.*

2.4. Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was performed to identify the adhesive of the mosaics using a device with the following f characteristics: FTIR spectrophotometer *(Bruker, ATR. Version 1.2.4).*

3. RESULTS AND DICTATION

3.1. SEM–EDX analysis

SEM- EDX analysis was carried out on some coloured samples of the glaze layers of ceramic mosaics (Fig. 3). The SEM images showed defects in the glaze layer resulting from the manufacturing process of the tesserae (Omar SH., 2022, 1-15), such as irregularities on the surface (Fig. 3A), flaking of the dark pink glaze and the red mosaic pieces (Fig. 3B), flaking of the glaze in some mosaic pieces, and weak parts, causing deterioration of some mosaic pieces. Crazing occur mainly due to the tensile strength in the glaze material and the differential in the expansion and contraction between the body and the glaze layer in use. In addition, the crazing and falling off the glaze layer or parts of it after burning during production, known as tensile stress, is caused by the differential contraction between the glaze layer and the body. Increasing tensile stress leads to flaking of the glaze layer, resulting in complete separation and falling off. Furthermore, the application of a thick glaze layer on the body is a reason for the different expansion and contraction coefficients between the glaze layer and the body of the ceramic (Abd algwadm 2015, 110-111). The pits, gaps, and irregularities in the glaze surface in the light pink mosaic pieces (Fig. 3 C) were created during the preparation of the glaze layer. The manufacturer did not remove the air bubbles in the glaze layer during preparation and application, which exploded during burning. They played a major role in the holes in the glaze layer (Rhodes, 1996, 264). Some silica particles, which make up the glaze, did not melt in the beige mosaic pieces (Fig. 3D). Moreover, the dark blue glaze showed surface irregularities (Fig. 3E). The surface of the white glaze was in good condition, but showed black lead stains (Fig. 3F). These phenomena in the glaze layer of mosaic pieces caused the loss of some properties and permeability to liquids into the ceramic body. They could cause serious damage to ceramic pieces , which are easy to be damaged and decomposed by external damage factors (Junior G.S., Ferreira J., Millán-Arias C., Daniel R., Junior A.C., and Fernandes B.J.T., 2021, 1-13), such as the accumulation of dust, dirt and air pollution gases, including sulphur dioxide, nitrogen oxides, soot and dust. Silicates are resistant to wear and corrosion. However, the phenomena caused by various sources can easily damage these materials if they are exposed to them over a long period of time. In addition, dangerous chemical reactions occur with fissures and cracks in the glaze, which can transfer to the body of the ceramic pieces and severely damage them (Baricza Á., Bajnóczi B., Tóth M., Szabó C., 2012, 7-14). The decorations of the mosaic generally had an opaque and dark glaze.

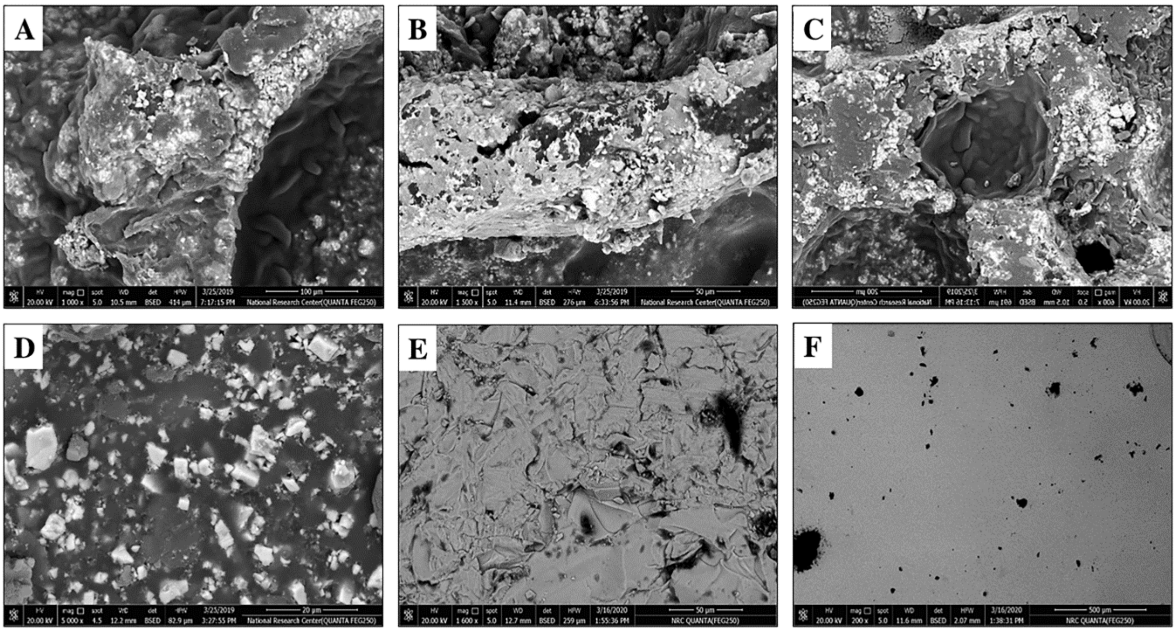

Figure 3 Electro-micrograph of the analysed ceramic fracture of the mosaics using SEM; A. Dark red glaze, B. Dark pink glaze, C. Light pink glaze, D. Beige glaze, E. Dark blue glaze, F., White glaze

The results of the chemical analysis by EDX of the ceramic mosaic pieces and the glaze layer showed significant results about the type of the glaze and the colour oxides used. The chemical composition of all samples was almost similar as shown in (Fig. 4) and (Table 1). Si and Al, indicating quartz and aluminosilicates, the basic elements of the glaze layer (Alawneh F., Balaawi F., Alghazaw R., 2019, 283-306). The percentage of Na was comparable in the dark red glaze (12.39%), the dark pink glaze (13.29%), the light pink glaze (16.58%), and the beige glaze (9.64%), indicating the use of sodium oxide as a melting material. The type of glaze used was therefore alkaline.

The dark blue glaze contained Na (6.23%) and a high proportion of Pb (41.74%). The white glaze also contained Na (7.85%) and Pb (37.55%). It can therefore be concluded that the glaze of the dark blue and white ceramic pieces was alkaline and contained lead as a melting material. This type prevailed in the Islamic era. At the beginning, the lead content in the simple alkaline glaze was 1: 2%. Then the lead content increased to 20-40%, and the alkali content to 5-12%, which was used to produce ceramics such as the Chinese porcelain of the Tang Dynasty. This type of glaze was well-known in the Middle East and Europe for the production of matt white glaze and blue cobalt on an opaque glaze layer. However, the use of large amounts of sodium oxide increased the rate of glaze shrinkage due to a high rate of thermal expansion, making the layers more susceptible to cracking. The contraction rate of the glaze layer was higher than the expansion and contraction of the body. In addition, the glaze layer was easy to scratch and erode due to its existence and was affected by various damage factors (Hamiliton, 1978, 161). As for the glaze colours of mosaic pieces, the dark red glaze contained Na (12.39%), Al (1.84%),

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Si (26.69%), S (1.1%), Cl (1.33%), K (1.7%), and Fe (8.69%), suggesting the use of iron oxides to achieve the dark red. It is one of the most common oxides used to obtain bright red and red-brown colour. It was added in the form of ferric oxide (Fe_2O_3) or ferrous oxide (Fe_3O_4). The latter had a higher percentage of iron in relation to oxygen than haematite, which made its colour darker (Gianni L., Renel H., Kremenovi A., Colomban PH., 2022, 61, 13-26). The result of the analysis of the dark pink glaze colour showed Na (13.29%), Al (1.64%), Si (26.14%), S (1.49%), Cl (0.52%), K (1.37%), Fe (3.95%) and Ca (1.58%), suggesting that the manufacturer reduced iron and added calcium to obtain this colour.

The analysis of the light pink glaze also revealed that the elements of this glaze correspond to those of the dark pink glaze, albeit indifferent proportions. The Fe decreased to (2.66%), the Mg to (1.05%) to obtain the light pink glaze, and the F to (3.48%). The elements of the beige glaze were Na (9.64%), Al (2.8%), Si (26.85%), Cl (0.82%), K (0.78%), Ca (7.46%), and F (5.18%). The content of fluorine (F) in the beige and light pink glaze suggests that it is used as an opacifying agent in the ceramics industry (Gazulla M. F., Rodrigo M., Orduña M., and Ventura M. J., 2015, 95-101). The dark blue glaze contained Na (6.23%), Al (1.39%), Si (19.74%), Ca (1.75%), and Pb (41.2%). As already mentioned, the glaze used was a lead-alkaline glaze, as the sample contained sodium and potassium. The blue glaze resulted from the addition of cobalt oxide (CoO), as cobalt (0.91%), the strongest and most effective colourant, even when added in a small percentage, is dark blue in lead glazes (Britt, 2007, 23). The use of cobalt in mass-produced glass dates back to the oldest Egyptian dynasties. At that time, it was used in various blue-coloured handicrafts, including glazed pottery, glass objects, and stained and lustre glass (Colomban Ph., Kırmızı B., and Franci G.S., 2021, 1-42). EDX analysis of the white glaze revealed Na (7.85%), Al (2.02%), Si (19.35%), Pb (35.55%) and Ca (3.24%), suggesting the use of calcium to achieve opaque white (Sadek, 2016, 65-71).

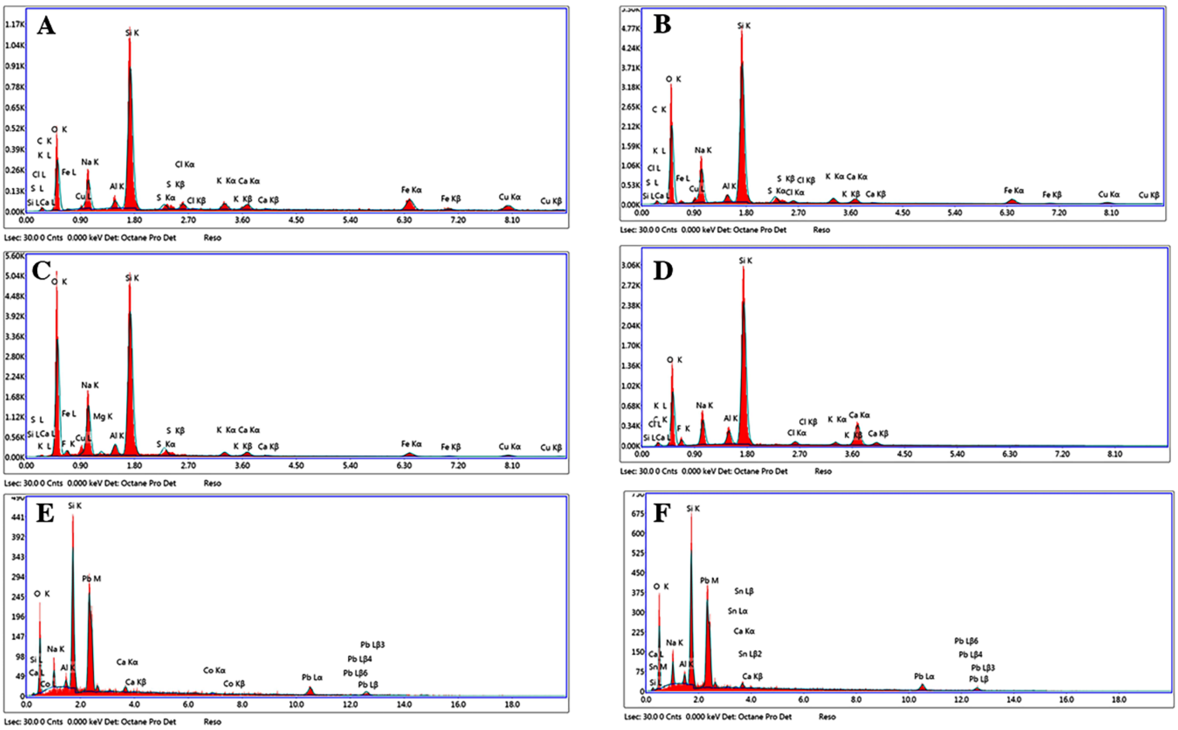

Figure 4. EDX results of the colour of the glaze layer of the mosaics; A. Dark red glaze, B. Dark pink glaze, C. Light pink glaze, D. Beige glaze, E. Dark blue glaze, F. White glaze

Table 1. EDX weight percentagesof the chemical elements in the colour of the glaze layer of the mosaics

Weight percentage of the elements in the samples %														
Analyzed Sample	**C**	**O**	**F**	**Na**	**Mg**	**Al**	**Si**	**S**	**Pb**	**Cl**	**K**	**Ca**	**Co**	**Fe**
Dark red glaze	6.88	29.89	--	12.39	--	1.84	26.69	1.1	--	1.33	1.7	--	--	8.69
Dark pink glaze	5.16	42.45	--	13.29	--	1.64	26.14	1.49		0.52	1.37	1.58	--	3.95
Light pink glaze	--	44.27	3.48	16.58	1.05	2.24	23.98	1.68	--	--	0.97	1.18	--	2.66
Beige glaze	7.31	39.15	5.18	9.64	--	2.8	26.85	--	--	0.82	0.78	7.46	--	--
Dark blue glaze	--	28.78	--	6.23	--	1.39	19.74	--	41.2	--	--	1.75	0.91	--
White glaze	--	31.49	--	7.85	--	2.02	19.35	--	37.55	0.51	--	1.24	--	--

3.2. XRD analysis

The results of the XRD analysis of the ceramic sherd sample of the dark red mosaic pieces showed that it is composed of compounds (Fig 5 A); tridymite (38.4%), mullite (23.2%), cristobalite (14.9%), quartz (9%), spinel (7.4%), halite (5.6%) and diopside (1.5%), which are known as firing compounds and whose presence indicates that the ceramics used were fired at high temperatures. At 1250 °C quartz transforms, and tridymite forms from beta-quartz to cristobalite at high temperatures. Kaolinite, the main component of most clays, also transforms into metakaolin at 500 °C. At 950 °C, metakaolin decomposed and spinel was formed alongside free silica. At 1050-1275 °C the spinel transformed into mullite (Sadek, 2012, 9-10). This result indicates that the ceramic pieces were fired at temperatures of around 1250 °C. In addition to the presence of a certain percentage of sodium chloride (halite) in the ceramic body, this is one of the degradation products detected in some mosaic pieces of the panel.

Furthermore, the results of the mortar sample used to fill the spaces between mosaic pieces showed that the mortar consisted mainly of calcite (99%) (Fig. 5 B). This indicates the use of lime mortar as a filling material between the ceramic pieces. The presence of halite in the mortar at a low percentage (1%) indicates the presence of sodium chloride salt, as confirmedin the sample of efflorescent salts on the mortar, which showed halite (100%) (Fig. 5 C). NaCl showed salt damage in this part. Chlorides are the most dangerous salts as they are very soluble in water and can easily spread to different areas. These salts can be caused by high humidity and salty groundwater . In addition, most building materials contained salts that migrated into themortar and caused damage. These salts could transfer to ceramic pieces and cause damage (Costa M., Cachim P., Coroado J., Rocha F., and Velosa A.L., 2014, 108-113) such as cracks or flaking of the glaze layer (Pilz M., McCarthy B. 1995, 29-39).

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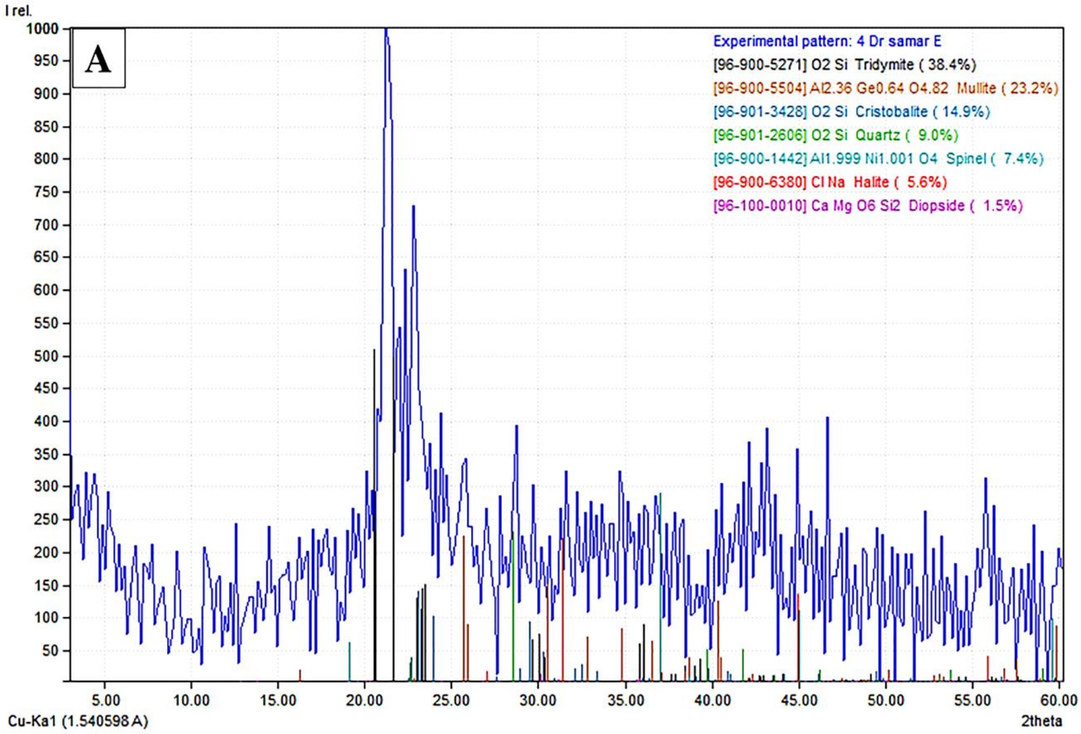
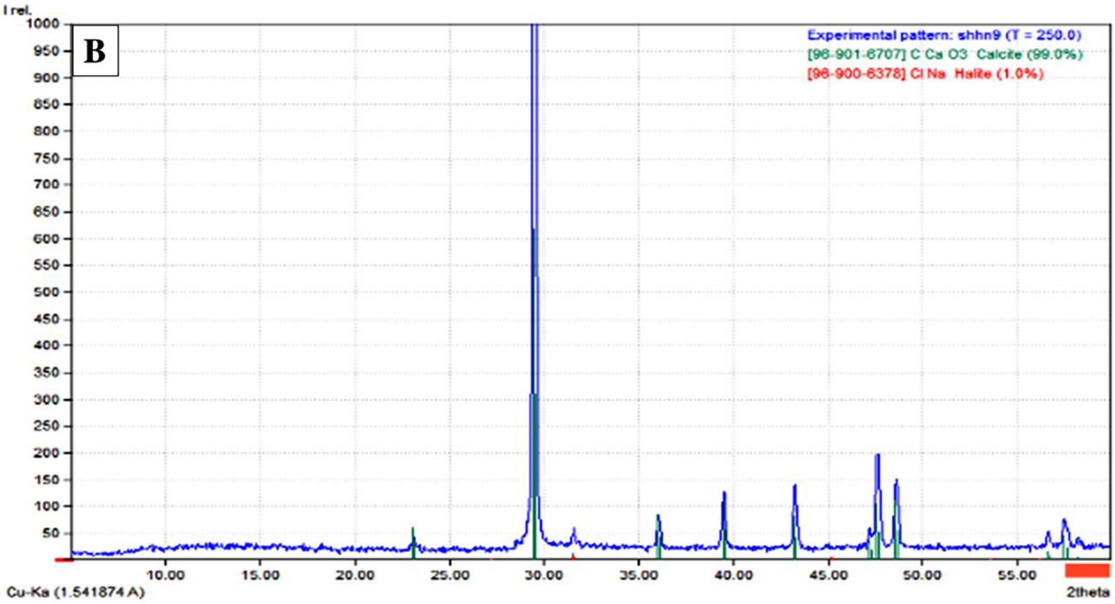

A
I rel.
Experimental pattern: 4 Dr samar E
[96-900-5271] O2 Si Tridymite (38.4%)
[96-900-5504] Al2.36 Ge0.64 O4.82 Mullite (23.2%)
[96-901-3428] O2 Si Cristobalite (14.9%)
[96-901-2606] O2 Si Quartz (9.0%)
[96-900-1442] Al1.999 Ni1.001 O4 Spinel (7.4%)
[96-900-6380] Cl Na Halite (5.6%)
[96-100-0010] Ca Mg O6 Si2 Diopside (1.5%)
Cu-Ka1 (1.540598 A)
2theta


B
I rel.
Experimental pattern: shhn9 (T = 250.0)
[96-901-6707] C Ca O3 Calcite (99.0%)
[96-900-6378] Cl Na Halite (1.0%)
Cu-Ka (1.541874 A)
2theta

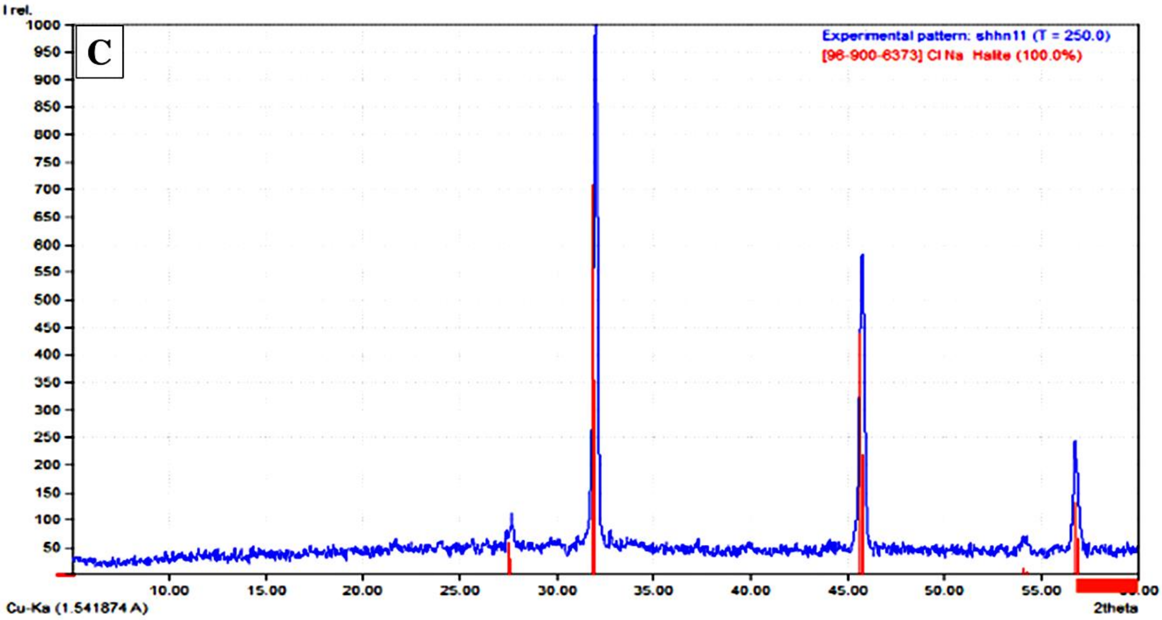


Figure 5. XRD pattern; A. Sample of the ceramic body, B. Sample of the filling mortar between mosaic pieces, C. Salt efflorescence

3.3. FTIR spectroscopy analysis

FTIR analysis of the adhesive and filler of some mosaic pieces revealed that it is animal glue, as it has the characteristic functional groups: OH stretching at 3428, C=O stretching at 1646, C=O stretching+ NH bending at 1526, and the characteristic spectrum of calcium carbonate CO_3 stretching at 1403, suggesting the use of animal glue as adhesive and lime as filler (Derrick M.R., Stulik D., Landry J.M., 1999, 181-194). (Fig.6, Table 2).

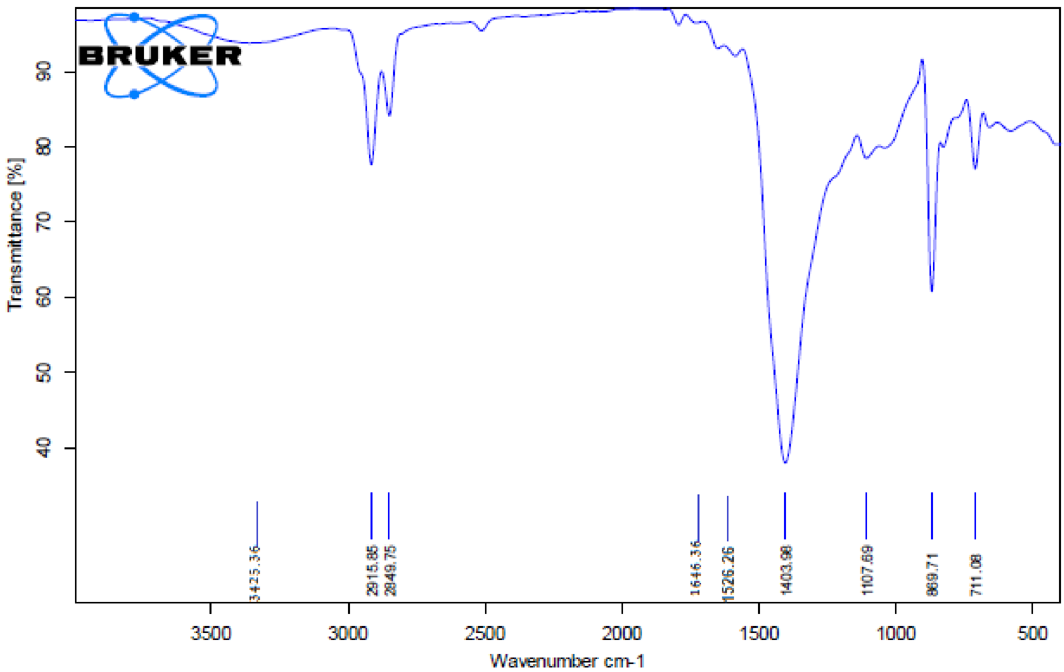


Figure 6. Pattern of the FTIR analysis for the mosaic fixing material

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Table 2. FTIR analysis of the adhesive and filler of mosaic

Wavenumber (cm^{-1})	Function group bands	Assignment
3423	OH stretching	Animal glue
2915-2849	stretching CH	Animal glue
1646	Stretching (Amide I) C=O	Animal glue
1526	stretching CN + bending NH (Amide II)	Animal glue
1403	stretching CO_3 + bending CH	Animal glue+ Calcium Carbonate
1107	stretching CO	Animal glue
889	bending O-C-O	Calcium Carbonate

4. CONSERVATION OF THE CERAMIC MOSAIC PANEL

4.1. Documentation

AutoCAD was used to document the mosaic mural panel (67 cm H.* 47 cm W.). It is a decorative mosaic depicting the Blessed Virgin Mary and Christ, using (dark and light blue, dark red, light and dark pink, yellow and white) mosaics on a (dark and light brown, beige and glass) mosaic background. In addition, various signs of deterioration were documented, including soot, wax, dirt, loss of some pieces, colour changes, and buckling in some parts (Mohamed M. I., Ayat A. H., and Sherif O, 2022, 1-14) (Fig. 7).

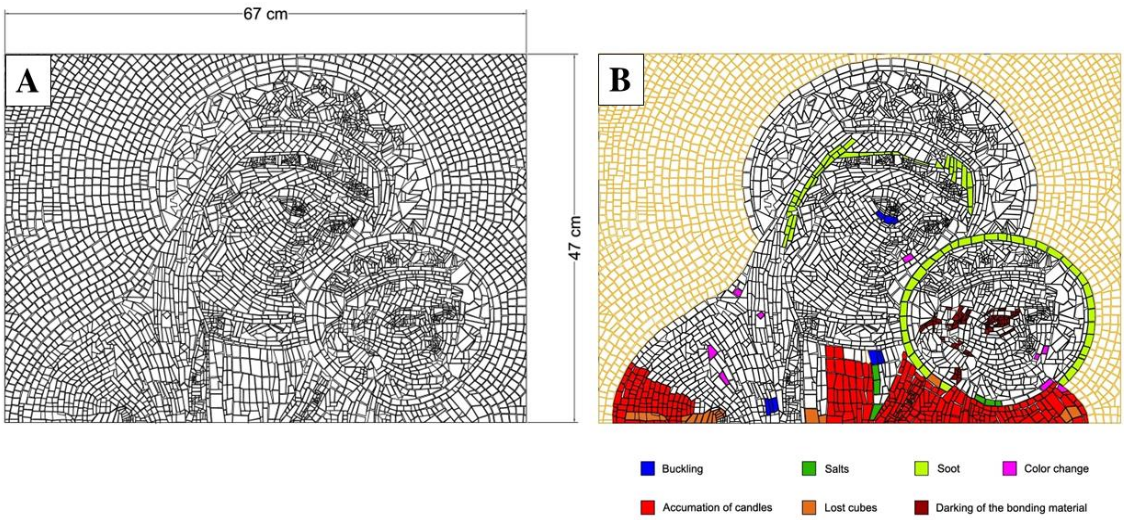

Figure 7 Autocade documentation; A. Dimensions, B. Deterioration map

4.2. Mechanical and chemical cleaning

The restoration of ceramic mosaics began with mechanical cleaning to remove dust and dirt from the surface of the ceramic pieces using mechanical methods that were easy to control compared to chemical cleaning. The cleaning methods depend on the nature and condition of the mosaic pieces as well as the type and nature of the dirt (Abd-Allah R., Al-Muheisen Z., and Al-Howadi S., 2010, 97-110). Mechanical cleaning mainly aims to remove or reduce the hard materials on the surface of the monuments in order to largely avoid chemical cleaning. Soft and hard brushes and other tools are used to remove undesirable material from the surface of the mosaic (Faulding R., and Thomas S., 2000, 38-55).

In the chemical cleaning of the mosaic portrait, various solvents and solutions were used to interact with the materials and dirt that were easily removed by chemical methods.

The first solvent was distilled water to remove dust deposits. It has many advantages, such as being cheap and available without any negative effects on the condition of the pieces (Abd-Allah R., 2013, 14-97). In addition, acetone and alcohol were diluted with distilled water to avoid the effects of the high concentration of these solvents on the surface (Adrian G., and. Paez M. C., (2007, 180-188). Gasoline delivered good results in removing candle residues. A mixture of distilled water, soap and ammonia also produced good results in removing the soot residues that had been left by the use of candles in front of the mosaic portrait. In this case, before cleaning the ceramic mosaic, it was ensured that the surface was not brittle and had no coloured crusts. Mechanical cleaning began with the removal of dust and dirt with soft and hard brushes (Alberti L., Bourguiguon E., and Roby Th., 2013, 89-90). Wooden sticks and scalpels were used to remove calcified parts (Fig 8 A, B), and the air pump was used to remove dust and dirt (Fig 8 C). Cleaning was carried out gradually and carefully from top to bottom (Arinat, 2019, 334-344). The heavy layers of wax on the surface were removed with different types of scalpels and a magnifying glass until the wax and dirt were completely removed. Then a chemical cleaning with distilled water was performed to remove some calcifications that were difficult to remove mechanically. Some organic dissolvents, such as ethyl alcohol, acetone and toluene, were then used. Acetone was diluted with distilled water, ethyl alcohol was used with distilled water (1:1) and neutral soap with water (1:1) (Casaletto M.P., Ingo G.M., Riccucci C., De caro T., Bultrini G., Fragala I., Leoni M., 2008, 35-42). Benzene was then used to remove wax residues. To clean wax soot , 1000 ml of a solution of water, 100 g soap, and 20 ml ammonia was used (Ricci S.S., Graham F., 2000, 37-56) (Fig 8 E). These treatments gave satisfactory results in removing dirt, dust, and adhesives from the surface.

4.3. Removal and extraction of soluble salt

The efflorescence salts were removed from the surface using mechanical tools and extracted. Cotton poultices and distilled water were then used and applied several times to achieve the best results. The type of salt, i.e. halite (sodium chloride), soluble salt (Abd-Allah R., Al-Muheisen, Z. , Al-Howadi, S., 2010, 97-110), was then identified (Fig 8 F). The NaCl salts were extracted and removed using cotton poultices applied several times, giving good results. To extract salts from the surface of monuments in small areas, it is preferable to use paper and cotton fibres in the poultice as they are easily to remove (Woolfitt C., Abrey G.,Poutltices, The true or plain poultice and the desalination of historic Masonry and Sculpture, The Building Conservation Directory, https://www.buildingconservation.com/articles/poultices/poultice.htm).

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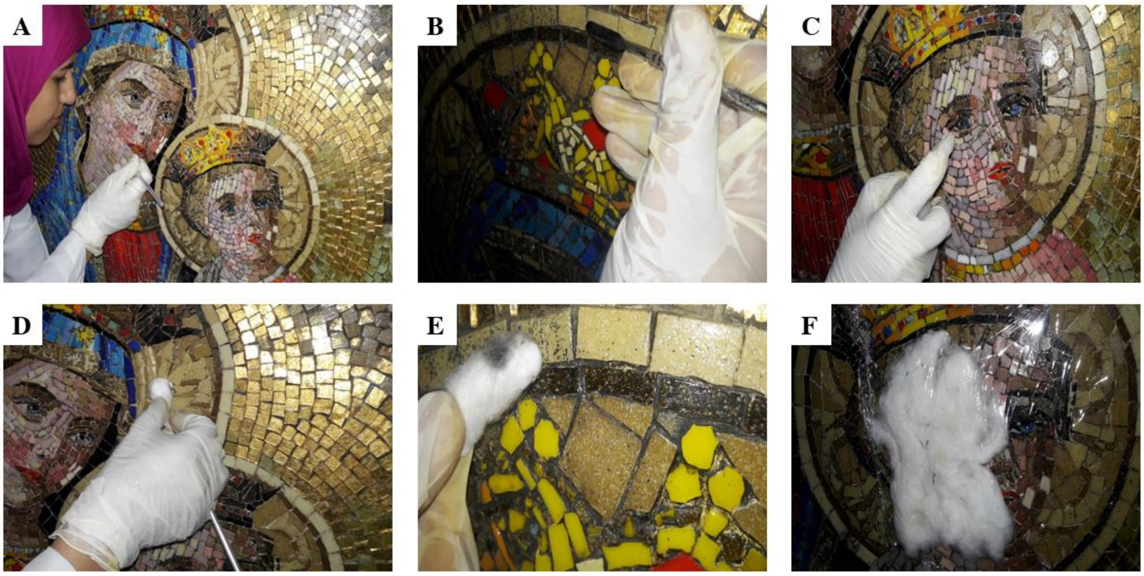

Figure 8 Mechanical and chemical cleaning and salt removal from the mosaic; A, B. Use of mechanical tools, C. Use of air pump, D, E. Chemical cleaning of the mosaic parts, F. Use of a cotton poultice for salt removal

4.4. Buckling treatment of some mosaic parts

The buckling of the mosaic was treated by injecting Primal AC33, which gave good results. Injecting binders behind the mosaic pieces with light pressure allowed the weak, damaged, or deteriorated mortar to reattach and strengthen (Rezzik A., Cherif H., 2021, 274-293). Researchers from the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM) conducted numerous experiments to find suitable injection materials that met the requirements and specifications for this problem. For instance, the mechanical strength of the new mortar should not exceed that of the old mortar; the injected mortar should have sufficient porosity to allow water evaporation; the mortar should be suitable for wet and dry conditions in ventilated or non-ventilated areas; the shrinkage of the mortar during the transition from the wet to the dry, hard state should not exceed the maximum of 4%. The ICCROM researchers arrived at a mixture with these specifications: 7 parts hydrated lime, 5 parts marble powder, 2 parts agar powder, and 1 part Primal AC33) (Tantawy, 2000, 143-145). As for as the implementation is concerned, a hole with a diameter of 2 cm was drilled in a case of one cube or more. If there were no falling cubes, the cube was dismantled, and a hole was drilled to penetrate the detached tiles. The dust was vacuumed and the internal cracks were rinsed out with a mixture of water and alcohol (3:1). A pretreatment was then carried out by injecting the emulsion primal AC33 (1:9) diluted with water. The previous mixture was then prepared and through cracks and holes in the surface using large syringes. After injection, light pressure was applied to the injected area for about 30 minutes. Then the disassembled cubes were fixed with the injection mixture, increasing the density of the mixture if necessary (Ferragni D., Forti, M., Malliet J., Teutonico J.M., and Torraca,G, 1983, 83-99). The work steps can be explained as follows:

- Remove a small cube from the dented mosaic pieces, noting its orientation in relation to the mosaic
- Remove dust and dirt with an air pump

- Moisten the area behind the mosaic cube by injecting distilled water and ethyl alcohol
- Inject primal AC33 into the area behind the dented parts
- Press the treated area with hand to ensure it sticks and put the dented piece back in place
- Replace the removed mosaic cube with a mortar (lime, sand, white cement and limestone powder), (Fig 9)

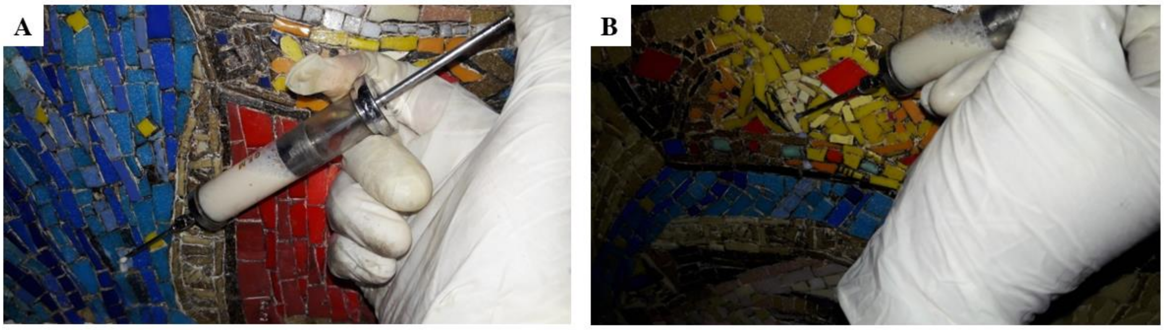

Figure 9. A, B. Treatment of the buckling by injection with Primal AC33

4.5. Completion of the missing parts of the mosaic pieces

The completion of the missing mosaic pieces was done with mortar (lime, sand, white cement and limestone powder) and the addition of nano-silica to the mortar components, as the use of nanoparticles in the mortar drastically changes its behaviour and improves the physical and mechanical structure (Senff L., Labrincha J. A., Ferreira V. M., Hotza D., and Repette W. L, 2009, 2487-2491). The use of nano-silica gel improved the compressive strength of the mortar compared to the structures without addition (Mondal P., Shah S. P., Marks L. D., and Gaitero J. J., 2010, 69).

In addition, nano-silica is the most commonly used material because of its effect on filling the pores. In the study of the adhesive pressure resistance and flexural strength, an increase in the strength of the paste was observed, which showed a significantly better mechanical potential and higher in compressive strength (Stefanidou M., 2012, 2706–2710). In terms of durability properties, e.g. permeability, pore structure, and particle size distribution, studies with nano-silica showed that the addition of nano-silica decreased water absorption and permeability (Zhang M. H., Islam J., 2012, 573-580). Several opinions were expressed to complete the missing mosaics. Majewski, for example, claimed that the gaps were filled with lime mortar. Then the surface was polished. After that, the surface was organized like the existing mosaic. The surface was painted with colours that corresponded to the original design of the mosaic (Majewski, 1977, 57). Another opinion argued that the gaps were filled with the appropriate mortar. It was then painted and coloured like the original. Hydraulic lime mortar is also used in emergencies when dismantled cubes have become detached from the mortar, requiring rapid intervention for fear of losing the cubes, especially at the edges (Nardi R., Schneider K., 2013, 55-70). The ceramic mosaic of *Sainte-Colombe-lès-Vienne* was finished with the same mortar used as a preparatory base with charcoal powder to obtain the black colour by filling in the lines of the missing with colorued mortar (Macchiarola M., Fiorella G., 2005). The process was completed here with the mortar (lime, sand, white cement, limestone powder and nano-silica) in a ratio of 3:2:2:1:1, which was applied underneath

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the cubes with a scalpel. It was then left to dry and painted with oil paints according to the colours specific to each area of the painting (Fig. 10).

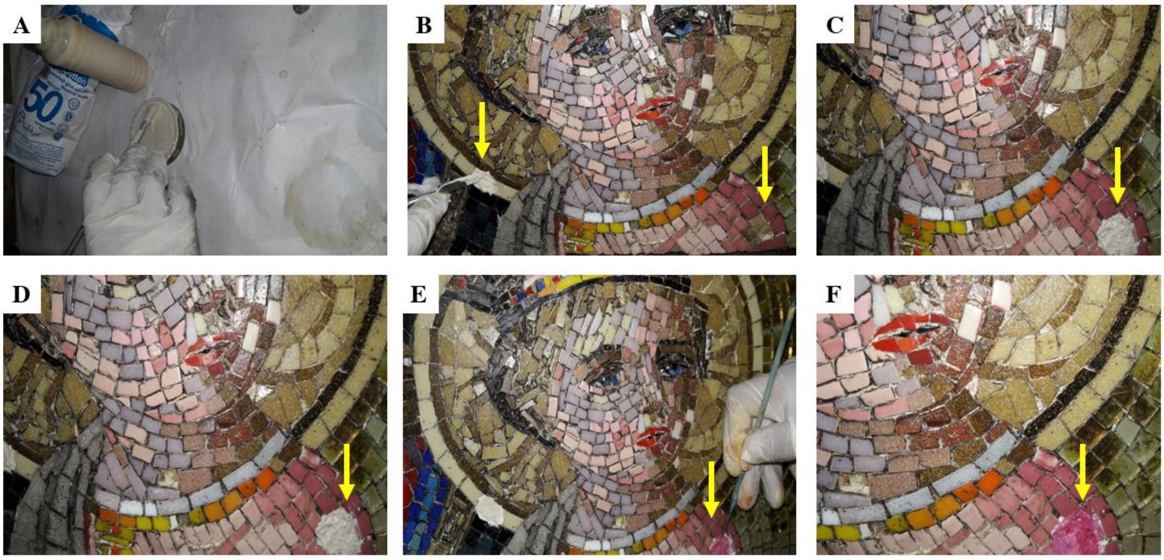

Figure 10. Completion of the missing parts of the mosaic pieces; A. Preparation of the completion material, B. Application with the metal spatula, C. D. After application of the completion material and drying, E. Colouring of the completed part and final shape of the completed part

4.6. Consolidation and protection

The final phase of the conservation of the mosaic panel consisted of finishing and chemical insulation to consolidate the weak mosaic pieces and isolate the panel from deterioration factors, especially moisture, asthe panel is located in a well in the church. The aim of this process was to consolidate and reinforce the weak mosaic parts caused by the damage to the glaze layer (the protective layer of the body) and to isolate the mosaic from the high humidity, as the portrait is located in a well. It was carried out with the nanocomposite PF4. The protection was carried out with the nanocomposite PF4, which consists of titanium dioxide nanoparticles dispersed in an alkylalkoxysilane polymer produced by Chem Spec Company, Italy (Helmi F.M., Hefni Y.K. 2016, 87-96). It is one of the multifunctional nanocomposites produced by adding one or more nanocomposite to a polymer. These materials were well blended to obtain a nanocomposite with good properties that cannot be achieved by polymers alone. It also has a high hydrophobic property. It is homogeneous with silicates and can improve their physical, chemical, and mechanical properties. It is highly resistant to ultraviolet rays and microorganisms (Omar Sh., 2018, 222-223). It was applied after cleaning the surface and removing dust and surface residues by impregnation with a brush. Six successive coats of the consolidant were applied, waiting between each coat until the consolidation and isolation coating was complete (Ibrahim M. M., Mohamed S. O., Hefni Y. K. and Ahmed A. I., 2019, 39-48) to protect the mosaic from future damage. Figure (11) shows the ceramic mosaic after completion of the consolidation and protection.

Figure 11 Ceramic mosaic after completion of restoration and protection

5. CONCLUSION

The study of the cave ceramic mosaic in the Church of the Blessed Virgin Mary in Mostorod brought to light many important findings about the components and colours of the ceramic mosaic and their structures. It helped to recognize the consequences of deterioration and to carry out the restoration and conservation of the panel using appropriate modern methods and materials. The study was conducted by accurately registering and documenting of the condition and diagnosing of the various aspects of deterioration, including soot, wax, salt, dirt, loss of some cubes and buckling in some areas. These results helped to carry out the restoration of the mosaic appropriately.

The SEM-EDX examination of some mosaic colours and samples revealed important results about the production, the type of glaze, and the oxides of the colorants. It showed an irregular glaze surface, pitting, lack of good melting behaviour of the silica (the main component of the glaze of some samples), and various fluxes that helped identify the glaze type. Two types of glaze were found (alkaline - lead alkaline). As for the colouring oxides, iron oxide was used in dark red mosaic pieces, calcium and iron oxides were used in different proportions to obtain a dark and light pink glaze, cobalt oxide was used to get dark blue glaze, and calcium was used to obtain an opaque white glaze. Fluorine was used as an opaque agent in some samples. The XRD examination showed that the ceramic pieces were produced at temperatures above 1250 °C as cristobalite, tridymite, mullite and spinel compounds appeared. Lime mortar was also used to fill the pieces. The efflorescent salt on the surface was sodium chloride (halite). The FTIR investigation also showed that animal glue was used to attach the mosaic pieces.

The conservation work began with the use of mechanical brushes to carefully remove dust and candles from the surface. Chemical cleaning with gasoline gave a good result in removing waxes. The mixture of distilled water, neutral soap and ammonia also gave good results in removing soot. The cotton poultice gave good results in the extraction of sodium chloride salt. As for the treatment of dents, the primal AC33 gave good results by injection behind mosaic pieces. The finishing was done with mortar, lime, sand, white cement, limestone powder and nano-silica. The mosaic was painted with oil paints in the respective colours of the individual areas to distinguish the restoration work and the original mosaic pieces and to show the completed areas. PF4 nanocomposite was used to

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reinforce and protect the mosaic portrait from damaging factors, especially moisture.. This study can be used for the restoration of similar ceramic mosaic objects.

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التحقيق التشخيصي وطرق الحفاظ على الفسيفساء الخزفية الجدارية في كنيسة السيدة العذراء مريم التاريخية، مسطرد، مصر

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بيانات المقال
تاريخ المقال
تم الاستلام في ١٢ فبراير ٢٠٢٣
تم استلام النسخة المنقحة في ٢٠ اكتوبر ٢٠٢٣
تم قبول البحث في ٢٨ اكتوبر ٢٠٢٣
متاح على الإنترنت في ٢١ يناير ٢٠٢٤

الكلمات الدالة
الفسيفساء الخزفية،
تحقيق،
الميكروسكوب الإلكتروني الماسح المزود بوحدة تحليل العناصر،
تلف الفسيفساء،
الانبعاج،
صيانة الفسيفساء.

الملخص

يتناول هذا البحث دراسة تشخيصية، وترميم وصيانة لوحة من الفسيفساء الخزفية بكنيسة السيدة العذراء بمنطقة مسطرد - مصر. تقع هذه اللوحة بالمغارة أو البئر الموجودة بالكنيسة، وهي تصور العائلة المقدسة، وتعرف بـ" لوحة فسيفساء المغارة". ولكن نتيجة لعوامل التلف المختلفة التي تعرضت لها، وكذلك بعض أعمال الترميم السابقة التي تمت لهذه الفسيفساء، ظهرت العديد من نواتج التدهور، والتي من أهمها ظاهرة الانبعاج، بالإضافة إلى الاتساخات والسناج والأملاح، مع فقدان بعض القطع. تهدف هذه الدراسة إلى التعرف على نوع وتركيب الخزف المستخدم في الفسيفساء، والتعرف على مظاهر التلف، وتشخيص الحالة، وإجراء عمليات الترميم والصيانة اللازمة من أجل حمايتها مستقبلاً. أجريت الدراسة التشخيصية باستخدام الميكروسكوب الإلكتروني الماسح المزود بوحدة تشتت طاقة الأشعة السينية (SEM-EDX)، والتحليل باستخدام حيود الأشعة السينية ((XRD، وطيف الأشعة تحت الحمراء (.(FTIRومن خلال النتائج، تم التعرف على التركيب الكيميائي والأكاسيد الملونة للقطع الخزفية المكونة للفسيفساء. تم استخدم أكسيد الكوبالت للحصول على اللون الأزرق الداكن مع استخدام القلوي فقط والرصاص والقلوي معاً كمواد مصهرة في التزجيج، ودرجات حرق قطع الخزف أعلى من ١٢٥٠٠ م تقريباً. أيضاً وجود تزهرات ملحية من كلوريد الصوديوم بين قطع الفسيفساء، والتعرف على الغراء الحيواني الذي أستخدم كمادة لاصقة في تثبيت بعض قطع الفسيفساء. هذا، وقد تمت عملية الترميم والصيانة من خلال استخدام طرق التنظيف الميكانيكي ثم الكيميائي، مع استخلاص الأملاح الذائبة باستخدام كمادة القطن، كما تم علاج مشكلة الانبعاج عن طريق الحقن باستخدام البريمال AC33 مع استكمال للأجزاء المفقودة من قطع الفسيفساء وصولاً إلى مرحلة التقوية والعزل باستخدام مادة pF4 النانوية المركبة.

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