SCIENTIFIC CULTURE
www.sci-cult.com

SCIENTIFIC CULTURE, Vol. 8, No. 2, (2022), pp. 1-15
Open Access. Online & Print

DOI: 10.5281/zenodo.6323156

CHARACTERIZATION OF THE OTTOMAN CERAMIC TILES IN THE FAÇADE OF MUSTAFA SINAN'S SAPIL (CAIRO, EGYPT)

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Received: 07/01/2022
Accepted: 28/02/2022

ABSTRACT

The Sapil (public fountain) and Kuttab (school) of Mustafa Sinan are important monuments dating back to the Ottoman Era (1630 AD). The main façade of the Sapil in the southeastern side on the Soq al-Silah Street has a set of ceramic tiles with floral decorations, but heavily deteriorated from environmental factors. In fact, the tiles suffered severe deterioration, causing some deterioration phenomena, such as crazing and large, macro cracks, peeling of the glaze layer, broken ceramic tiles and accumulated dust and soot on the surface, resulting in the deformation and obliteration of the decorations.

The present research paper is a characterization study (chemical composition and mineralogical structure of the body and the glaze layer) to determine their deterioration aspects. USB digital microscope, X-Ray Diffraction, and SEM-EDX were used to study ten various samples. The results of tiles analysis showed the use of local clay mixed with a small amount of lime. The firing temperature of these tiles was around 900-1000 ºC according to the presence of diopside. The mortar of the installation of the ceramic tiles was composed of a mixture of gypsum, lime, and sand. It was determined that the alkaline and lead oxides were the main components of the glaze layer, with rising lead oxide in all samples. It was also seen that the white glaze color was obtained with calcium, whereas the cobalt oxide was used for dark blue. At the same time, the copper oxide was used for light blue and green color. Iron oxide with manganese was used to get the coral red glaze. This helped to choose compatible materials for their future restoration.

KEYWORDS: Characterization, Ceramic tiles, Deterioration, Manufacture defects, Crazing, Peeling, Examination, Analysis.

1. INTRODUCTION

Ceramic tiles have received due attention in Egypt because they have been widely used inside and outside most archaeological and historical buildings, such as mosques, palaces, and churches. They are often attractive tiles, constituting an essential patrimony within cultural heritage to be maintained (Silva et al., 2013). Sapils are one of the most important buildings in Islamic architecture. The Waqf (endowment) system helped spread sapils as they were greatly welcomed because of their relation to charity and providing the passersby with water. Therefore, they were established between alleyways to offer cold water, especially in crowded areas; since ancient times, water has been a fundamental factor in most civilizations' emergence, continuation, and progress. (Mahmoud, 2019). Egypt contains many of these sapils, such as the Sapil and Kuttab of Mustafa Sinan "Monument No. 246" of the Ottoman era (1040 AH. / 1630 AD.) on the Soq al-Silah Street and the forepart of Alshamasherjy alleyways, Al-Darb Al-Ahmar Neighbourhood. Scholars differed on the founder of the sapil. While some called it the Sapil of Mohamed Agha and Mustafa Sinan, others knew it as the Sapil of Mustafa Sinan. However, the first name is more acceptable, especially when comparing the foundation text on the southeastern façade. The general layout of the sapil follows the local style of Ottoman sapils: A rectangular Tasbil (distributing water) compartment with two Tasbil windows and the Shazrawan in the front part with two openings to the Tasbil compartment on the sides.

The northeastern side opens on a rectangular opening raised from the Tasbil compartment. This sapil is the first Ottoman sapil with a prayer area attached to the Tasbil compartment with a simple hollow mihrab facing the entrance of the sapil directly (Fig. 1A). The sapil is separated. It was topped by a Kuttab that is currently demolished (Fig. 1B). It contains two Tasbil windows: One window is in the middle of the façade overlooking the Soq al-Silah Street. It is topped by a six-line foundation text and fillings divided by frets into rectangular and round areas covered with decorative tiles "understudy". There is an opening to provide the cistern with water to the right. The other window is in the middle of the façade overlooking Alshamasherjy alleyways next to the door of the entrance of the sapil (Al-hussieny, 1988).

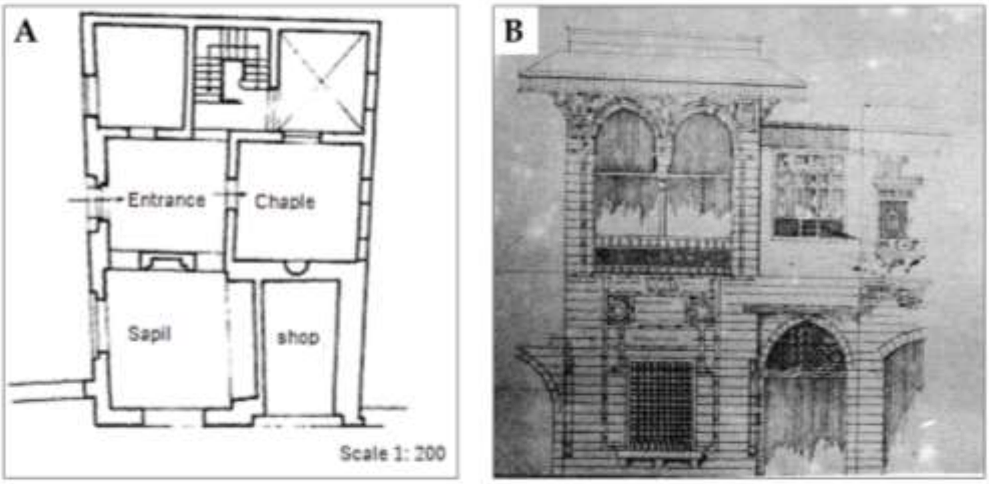


Figure 1. A. Plan of the Mustafa Sinan's Sapil, B. The façade of Mustafa Sinan's Sapil overlooking Soq al-Silah Street, monument 246, and shows the façade of the Kuttab "Archives of the Antiquities Authority" (Al-hussieny, 1988, 404-464)

The main façade on the southeastern side overlooking the Soq al-Silah Street has a set of decorative tiles (Fig. 2A) in the tympanum "Nafees" above the window and another set on the right, the left, and above the foundation text. These tiles were used in decorating the outer wall. They date back to the Ottoman era (date of establishment). They contain colorful floral ornaments on an opaque white background (Fig. 2B). These flowers constitute one of the most distinctive and familiar aspects of Ottoman-style art. Therefore, having these decorations relieves the boredom of having the stones on the entire façade of the sapil (Al-hussieny, 1988). Unfortunately, the field study revealed several deterioration aspects in these tiles because of the exposure to several deterioration factors, causing the collapse and loss of a tile and the long-term distortion and deterioration of the sapil and its decorative items.

The characterization of ancient materials has a great significance in protecting cultural heritage. As the information obtained from the characterization studies provides guidance in terms of the restoration interventions to be applied to the material. Moreover, it produces a significant data basis regarding the reintegration and re-manufacture that may be required in some cases (Dabanli et al. 2021). On the other hand, the archaeometry studies help archaeologists to investigate and understand the ancient monuments, objects, and the different human cultures (Liritzis et al., 2020). Also, the characterization study contributes to overcoming some of the basic problems relating to the chemical identification of different clay groups the main component of ceramic tiles (Krueger et al., 2021). Undoubtedly, the glaze is an early form of glass that could be coloured by the addition of coloring agents. The glazed material has been known in Egypt since the pre-dynastic periods through the material known as faience (Tite et al., 2007). The ceramics industry has greatly developed, during the Islamic period, ceramics were produced all over the middle east (Sadek, 2016). The ceramic industry developed significantly due to the trade exchanges between the Mediterranean Sea countries (Belfiore et al., 2021).

The glazing method requires various degrees of difficulty, which might involve a great variety of raw materials. Therefore, a simple glazing process consists of a single layer applied over the body's surface, becoming a glossy and transparent coat after firing. The addition of pigments would result in a coloured glaze that can be transparent or even opaque. The more complex sequence occurs in decorated glazed pottery, where opacifiers, pigments, and metals are added to produce an opaque white glaze to enhance the decoration. This glaze is applied to the bisque pottery and manufactured, generally, in a second or third firing (Fernández et al., 2021).

The major components of the glaze are silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3), in addition to flux agents (the most used lead and/or alkaline). Fluxes are substances, usually oxides, used in glazes, glasses, and ceramic bodies to lower the high melting point of the silica. These metallic oxides (Cu, Co, Mn, Fe, etc.) are added in a small amount to produce a suitable coloured glaze (Costa et al., 2014; Pradell and Molera 2020).

Currently, deterioration rates are high and fast, especially in congested urban areas, where building materials are constantly exposed to destructive environmental factors, such as high humidity levels, rainfall, and air pollutants (Grossi and Brimblecombe, 2007). Furthermore, the archaeological buildings of historical Cairo are suffering from high rates of humidity. The deterioration of these buildings closely relates to the high level of groundwater. Thus, many historical buildings and its decorative elements have deteriorated. Human factors and air pollutants also play a key role in deterioration (Coppola et al., 2020), resulting in breaking and macro cracks in the ceramic tiles of the Sapil's façade. The weakened sewage networks because of the population increase, and the expansion of unplanned urban growth in Cairo added another factor of deterioration (Elyamani et al., 2021).

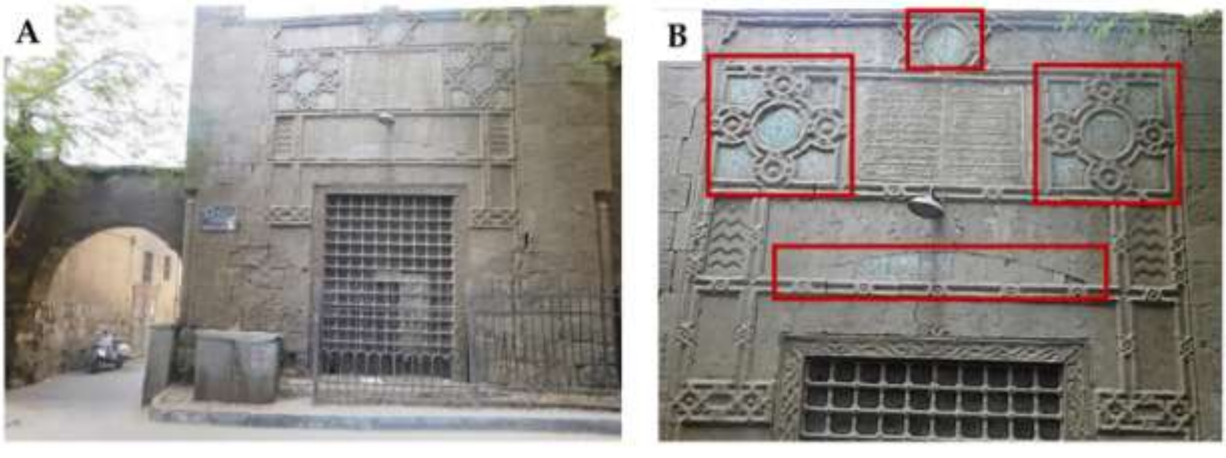


Figure 2, A. The main façade of Mustafa Sinan's Sapil overlooking Soq al-Silah Street, B. The locations of ceramic tiles in the Tympanum, right, left, and top of the foundation text on the façade of the Sapil

The present work aims to understand the techniques used in production of Ottoman ceramic tiles and its application on the façades of archaeological buildings. So, it can be recognized through the identification of the chemical composition, burning degrees of these tiles, and components of the mortar installation. The monitoring of the deterioration aspects that caused damage of these tiles will help in their future conservation.

2. DESCRIPTION AND DETERIORATION PHENOMENA OF THE CERAMIC TILES IN THE SAPIL'S FAÇADE

The southeastern façade of the sapil overlooking the Soq al-Silah Street is decorated with stone ornaments of frets with memes on both sides and above the foundation text. The inner space is covered with ceramic tiles on the four sides and the middle section of the fret. The decorations of the tiles featured a floral composition in the central area and duplicated on the four sides of the fret. The tile is square and measures (25× 25 cm) and thickness (1 cm). It is decorated with a modified pomegranate flower coloured dark blue, and green, with touches of coral red. Two branches implemented in palmette emerge from the four centers.

- A small branch of a serrated leaf in green and touches of red and white.
- A small branch coloured dark blue from which the flower of plumed cockscomb coloured dark blue and touches of coral red emerges. (Fig. 3A, B) illustrates the glazing colours on a ceramic tile with documenting the decorations using the AutoCAD program.

The tympanum "Nafees" part is covered with tiles of the same type. These decorations and colours characterize the ceramic tiles of the Ottoman era (the time of establishment).

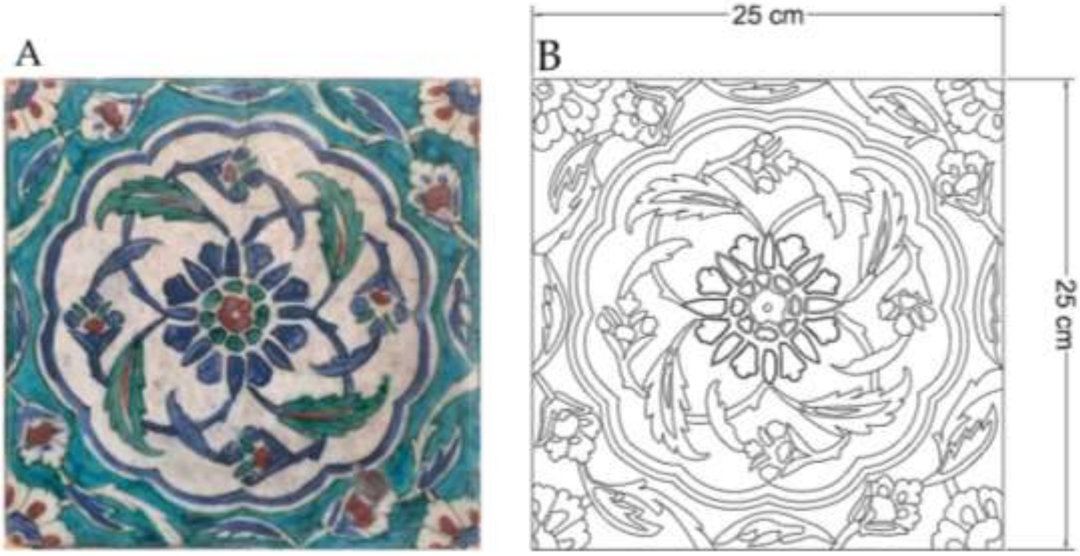


Figure 3. Decorated ceramic tiles, A. Glazing colours, B. Documentation of the ceramic tile decorations by AutoCAD program

2.1 Deterioration Factors related from the Manufacturing Process

The study showed that these tiles suffer from several manufacturing-related deterioration aspects, such as defects in the raw materials and glazing preparation method, the pre-burning process of the body, or post-glaze final burning. These factors cause internal deterioration and several deterioration aspects (Sadek, 2005). Crazing is a significant defect in glazing as it creates a network of fine cracks on the glaze surface. It results from several reasons, including mainly rapid cooling after burning. Fast and uneven burning causes internal pressures in the artifact as both the body and the glaze are subjected to slight expansion upon heating and contraction upon cooling. Expansion and contraction relate to the thermal expansion coefficient and happen unevenly (Buys and Oakely, 1999, 20). It was reported that the glaze with large amounts of sodium and potassium is more prone to crazing because of the high contraction rate resulting from the high thermal expansion (Sadek, 2005, 98). Crazing and shelling are mainly caused by the tensile strength and pressure in the glaze layer or the difference in expansion and contraction between the body and the glaze layer in use. When rising tensile, peeling occurs, causing total separation or fall of the glaze layer. Additionally, applying a thick glaze layer on the body is a cause of the difference in the expansion and contraction coefficients between the glazing layer and the body (Abd algwad, 2015). The poor industry also determined because of the presence of various pits, cracks, and non-melted hard impurities in the glaze layer (Fig. 4A).

2.2 Deterioration Features related from the external factors

2.2.1 Surface dirt and deposits on the ceramic tiles

Air pollutants and micro-deposits, including dust, soot, and carbon particles, are the major issue in the deterioration of archaeological materials. They interact directly with these materials and cause damage (Çiçek, et al., 2009). Ceramic tiles of the façade of the Sapil suffer from heavy layers of dust, dirt, and deposits that are firmly attached to the surface. It causes the blur and distortion of the tiles' decorations and playing an active role in the deterioration and damage, especially under high humidity that helps firmly attaching the dirt to the surface of the tiles (Abd-Allah, Al-Muheisen and Al-Howadi, 2010). It becomes a catalyst for the chemical reactions (Buys and Oakley, 1999, 84). Moreover, in the long run it causes the fading of colours or decreasing its brightness (Fig. 4B).

2.2.2 Peeling, breaking, and loss of some tiles

It was noted that most ceramic tiles of the façade suffer from severe deterioration and damage, such as peeling and breaking that causing falling and loss of them. These aspects resulted from environmental factors such as humidity and air pollution gases (Gulzar, 2017). It was also noted that a tile of the ceramic composition to the upper right of the foundation text and parts of the tympanum "Nafees" tiles were lost (Fig. 4C).

2.2.3 Tile Detachment

This phenomenon resulted from the weak and improper mortar of installing the tiles due to the high humidity rates that dissolved the lime mortar. The fluctuations between temperature and humidity accelerated mortar deterioration (Abd-Elrahim and Weshahy, 2017). Accordingly, mortar lost its properties and ability to stick and fix, causing gaps behind the tiles. This phenomenon is identified through the rattling sound when tapping gently on the tile (Buckling). The outer flat surface of the tiles raises. Finally, the mortar around the tiles falls, and some tiles break and lost (Fig. 4D).

Figure 4. Deterioration phenomena of the ceramic tiles, A. Manufacture defects such as pits, cracks, and hard impurities in the glaze layer, B. The accumulation of dirt and dust on the surface of ceramic tiles and the blurring and distortion of the decorations, C. Break in the ceramic tiles, large cracks with damage and falling off fixing mortar, D. Breakdown of a ceramic tile and parts of other tiles due to damage the adhesive mortar.

3. MATERIALS AND METHODS

Ten samples were studied in this work, two samples of the mortar, two samples of the ceramic tile body, another sample of ceramic tile for cross-section and five spots of the glaze colors from one tile that represent the whole colors (Fig. 5). Small fragments were taken to identify of the components and structure of the ceramic tiles. Also, technological features of texture, clay processing, and estimation of firing temperature were recognized (Măţău, et al., 2019).

The samples were studied using the portable optical digital microscope (Image sensor 1.3 Megapixels, magnification factor 10~1000 times, photo capture resolution 640×480, 320×240, and LED illumination light re-source adjustable by the control wheel). It is a non-destructive important method of examining and diagnosing the condition of the ceramic tiles, identifying the various deterioration aspects, manufacturing defects, crazing, etc., that are unobservable by the naked eye (Ali and Omar, 2021). Consequently, meaningful information about the manufacturing process, the clay type, and the burning temperature could be obtained.

X-Ray Diffraction Analysis (XRD) was used to identify the components and structure of the mortar of installing the tiles and the body of the ceramic tiles (Sánchez Ramos, et al., 2002; Francesco, et al., 2021). An XRD device with the following specifications was used: 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.

The scanning electron microscope with energy dispersive X-ray (SEM-EDX) was used to identify the glaze layer's physical properties, type, colored oxides, and elements through the attached analysis unit (Vecstaudža et al., 2013; Kaplan et al., 2017; Gelzo et al., 2021). A device with the following specifications was used: Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., using 14-x up to 1000000 magnification and resolution for Gun.1n.

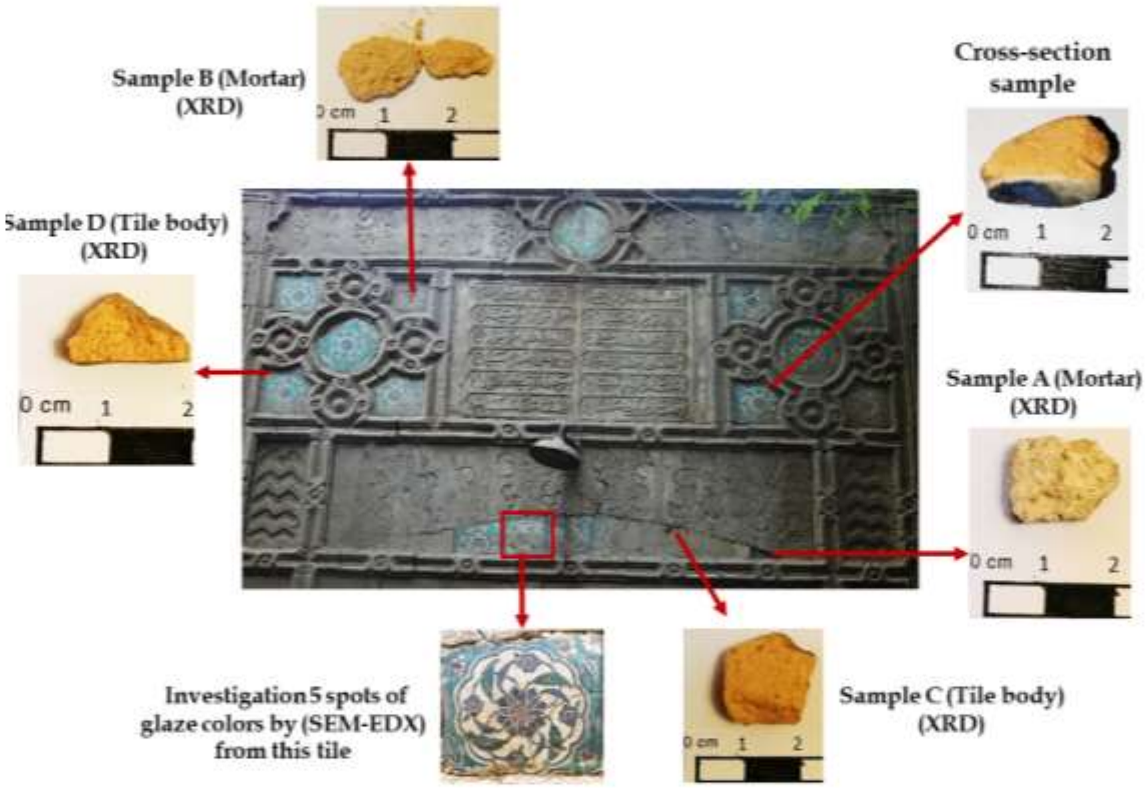


Figure 5. The samples of this study and their locations on the façades

4. RESULTS

4.1 *USB Portable Digital Microscope Results*

The examination using the portable optical digital microscope showed the deterioration of the ceramic tiles, as shown in (Fig. 6). It illustrates crazing and micro holes in the glaze layer and dust penetration in the cracks (Fig.6A). The distortion results from some hard impurities of the glaze (manufacturing defects) (Fig. 6B). (Fig. 6C) shows the highlights of small pits and tiny air bubbles in the glaze layer (also manufacturing defects). Cracks and the accumulation of dust and dirt blurring the decorations of the ceramic tiles (Fig. 6D). In addition to the Cracks in the glaze layer. Moreover, it highlights peeling and falling the glaze layer. (Fig. 6E-F).

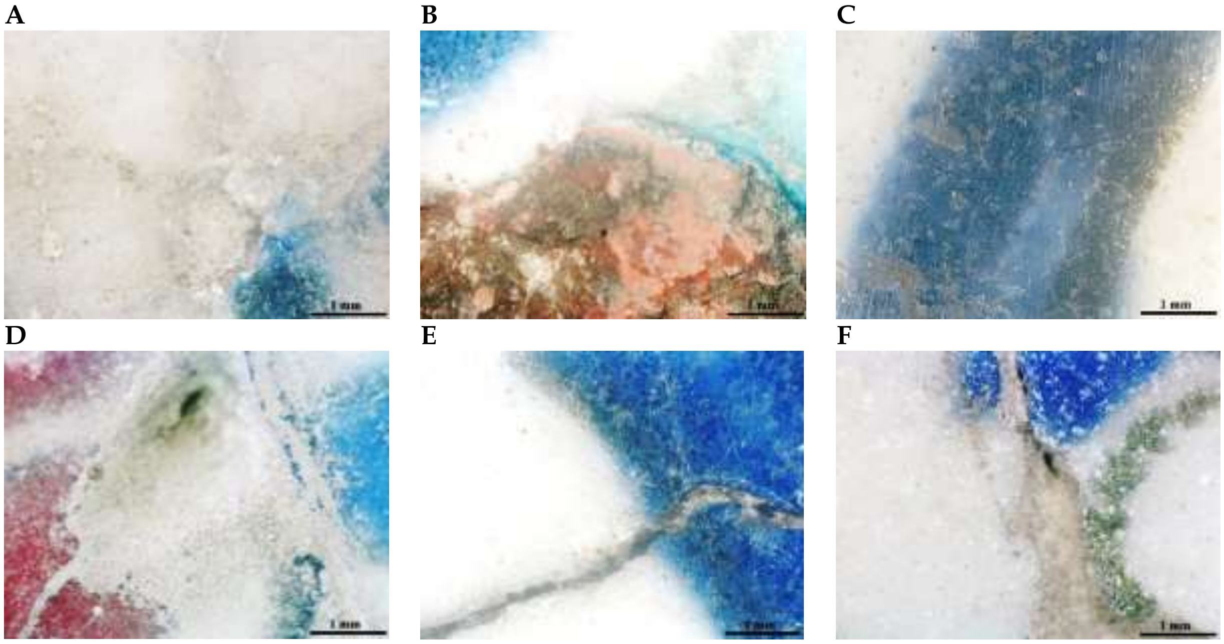


Figure 6. USB digital microscope images of the ceramic tiles, A. Microcracks and pits in the glaze layer, dust penetration in the cracks, B. Distortion resulting from some hard impurities of the glaze (manufacturing defects), C. Small pits and tiny air bubbles in the glaze layer (manufacturing defects), D. Cracks, accumulation of dust and dirt, and blurring the decorations of the ceramic tiles, E. Cracks, F. peeling and falling the glaze layer.

4.2 *X-ray Diffraction Results*

4.2.1 Mortar Samples

XRD results showed that the mortar of installing and pasting the ceramic tiles in the Nafees area (Sample A), consisted of anhydrite (50.6%), gypsum (33.3%), calcite (12.7%), quartz (2.2%), and halite (1.3%) (Fig. 7A). The (Sample B) of the mortar that was taken from behind the lost ceramic tile to the right of the foundation text showed that it consisted of anhydrite (77.6%), calcite (9.1%), gypsum (8.6%), and quartz (4.7%) (Fig. 7B).

4.2.2 The body of the ceramic tile samples

The results of XRD analysis of a ceramic tile's body decorating the "Nafees" (Sample C) showed that it consisted of quartz (61.1%), spinel (21.4%), diopside (6.1%), calcite (5.8%), and mullite (5.6%) (Fig. 7C). Analyzing the (Sample D) of a ceramic tile's body decorating the upper part of the façade showed that it had the same components of the Nafees's tile but with different percentages, as follows quartz (58.6%), spinel (18.7%), diopside (11.5%), calcite (5.6%), and mullite (5.6%) (Fig. 7D).

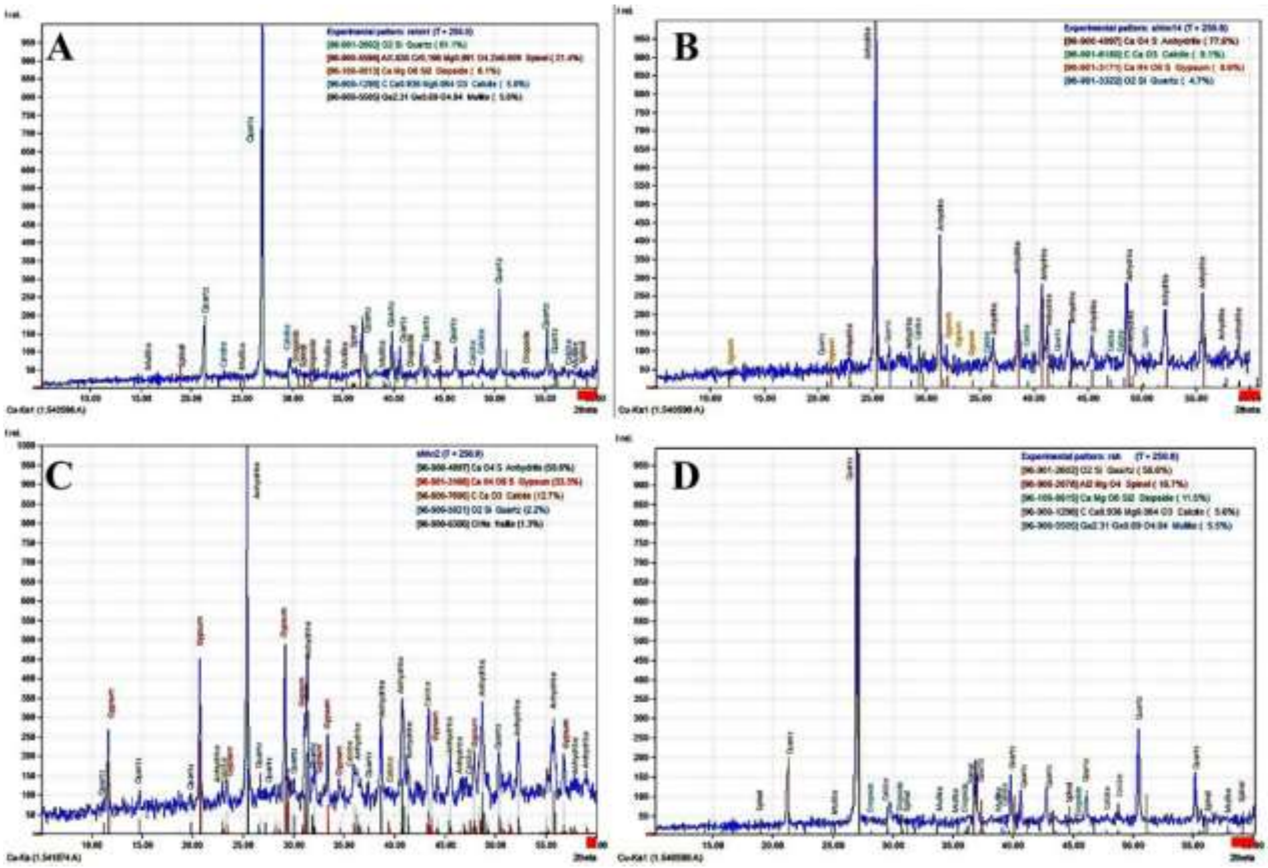


Figure 7. XRD pattern, A. Mortar sample used for fixing the Nafees tiles, B., The sample of the mortar used in fixing of the ceramic tile to the right of the foundation text, C. Sample from the body of the ceramic tile that decorates the Nafees, D. Sample of a ceramic tile's body decorating the upper part of the façade.

4.3 SEM-EDX results

SEM-EDX results of the colors on ceramic tiles' glaze showed that:

White glaze

There were many aspects of crazing, tiny holes, and hard particles that did not melt during burning. There was a large amount of Si and Pb, a small amount of Na and Ca, and a smaller amount of Al, as shown in Table 1 (white glaze) and (Fig. 8A).

Dark blue glaze

The SEM investigation showed cracks, fine scratches, holes, and peeling glaze. There were (Na, Al, Si, K, Ca, and Pb) in addition to (Co and Cu), as shown in (Fig.8B) and Table 1 (dark blue glaze).

Light blue glaze

The examination showed peeling, several holes, uneven surfaces, and crazing in the glaze layer. There were (Na, Al, Si, K, Ca, and Pb) in addition to (Cu), as shown in (Fig.8C) and Table 1(light blue glaze).

Green glaze

SEM investigation results of green glaze showed tiny holes and crazing. EDX results showed (Na, Al, Si, K, Ca, and Pb) and a large amount of (Cu), as shown in (Fig. 8D) and Table 1 (green glaze).

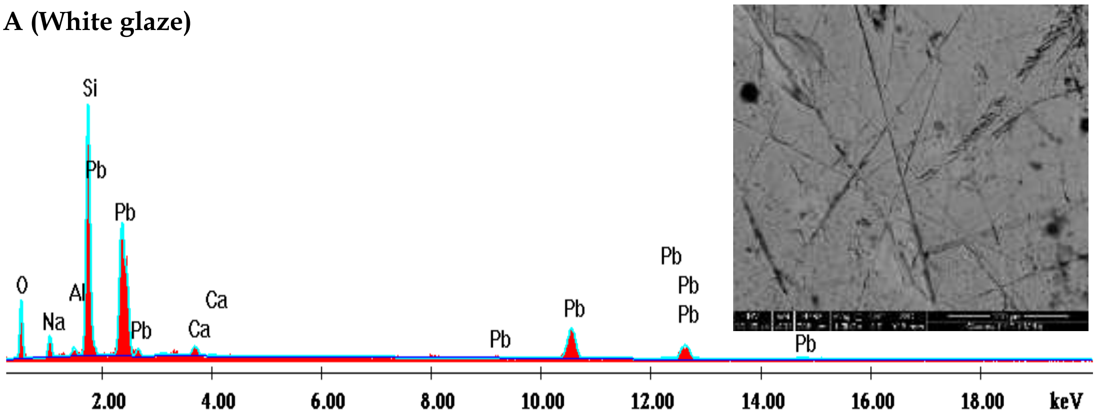
Coral red glaze

The examination of the coral red glaze illustrated some cracks, crazing, and uneven surface glaze. The results showed (Na, Al, Si, K, Ca, and Pb) and (Mn and Fe), as shown in (Fig. 8E) and Table 1 (Coral red glaze).

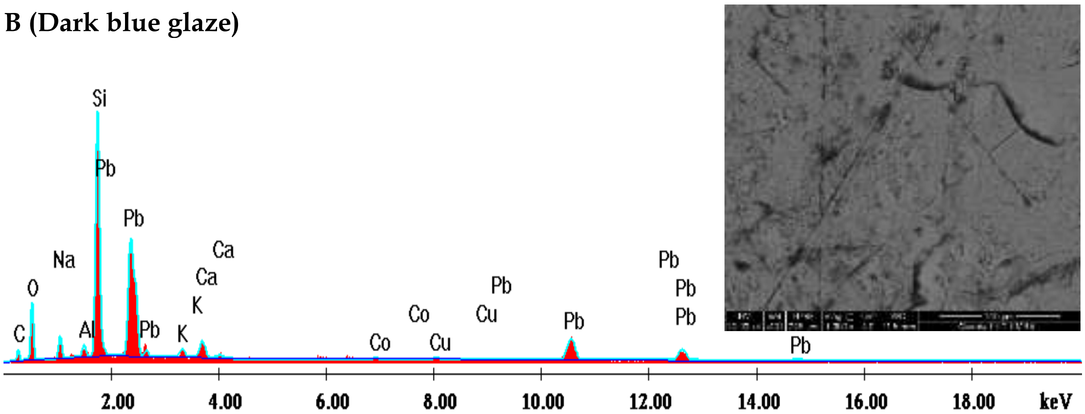
4.3.1 Cross-Section Study

A cross-section of a ceramic tile sample was prepared to identify the glaze interaction region with the body besides the glaze layer thickness (Pradel et al. 2010; Moussa and Ali, 2013). The prepared cross-section was examined using SEM to identify the varied thickness of the glaze layer. It revealed that the glaze layer has different thicknesses measuring 78.10 μm in one area and 120.1 μm in the other (Fig. 9).

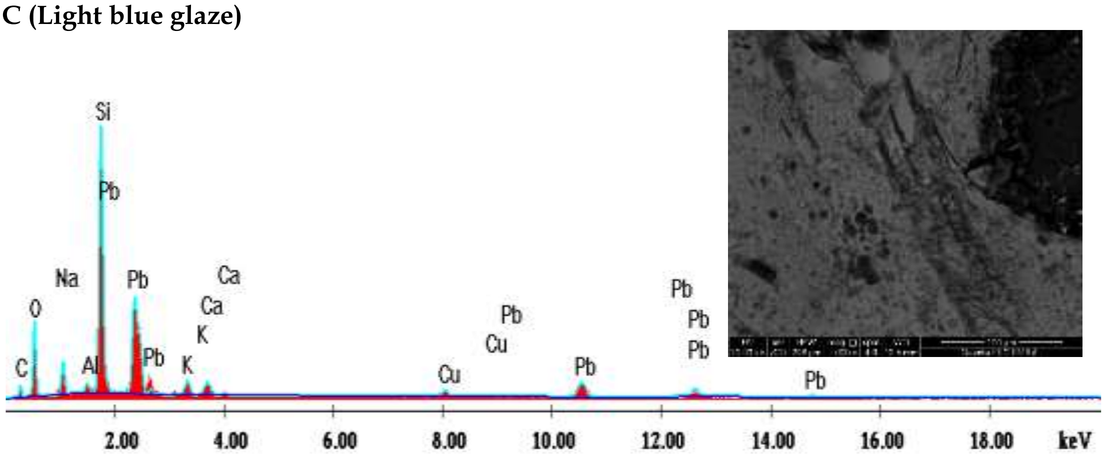
A (White glaze)



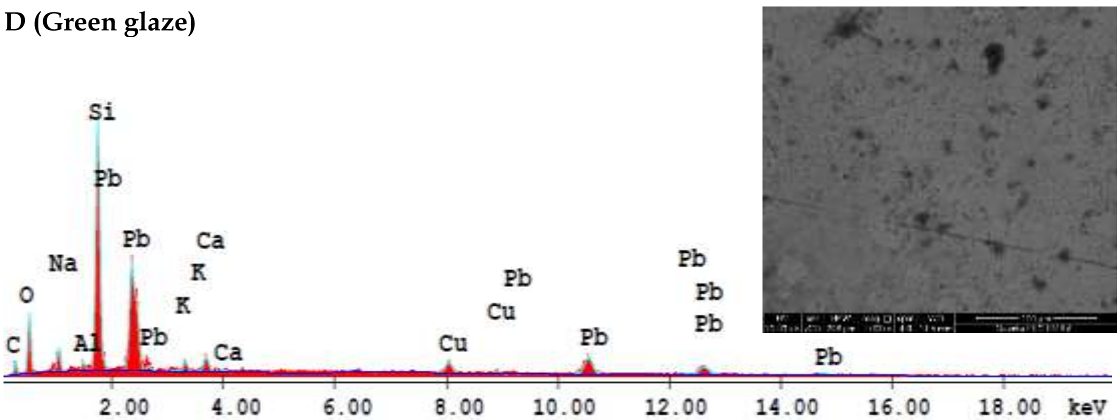
B (Dark blue glaze)



C (Light blue glaze)



D (Green glaze)



E(Coral red glaze)

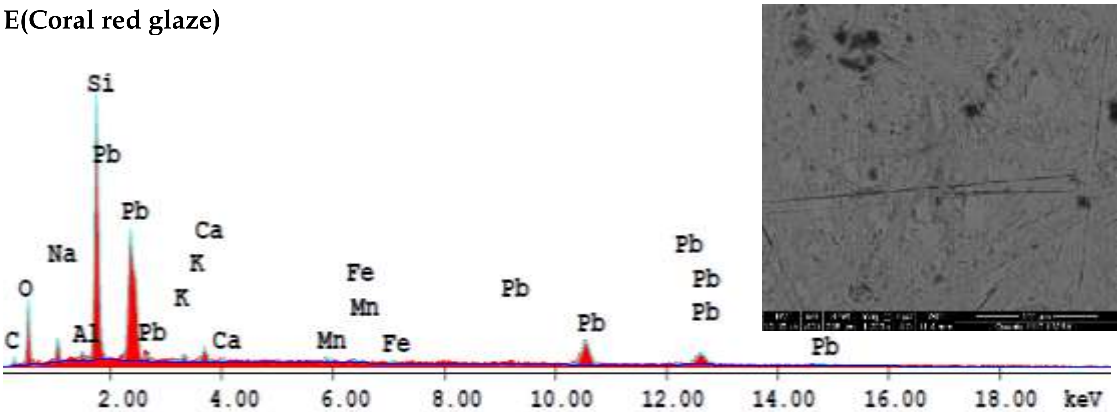


Figure 8. SEM-EDX results of the glaze colours, A. White glaze, B. Dark blue glaze, C. Light blue glaze, D. Green glaze, E. Coral red glaze

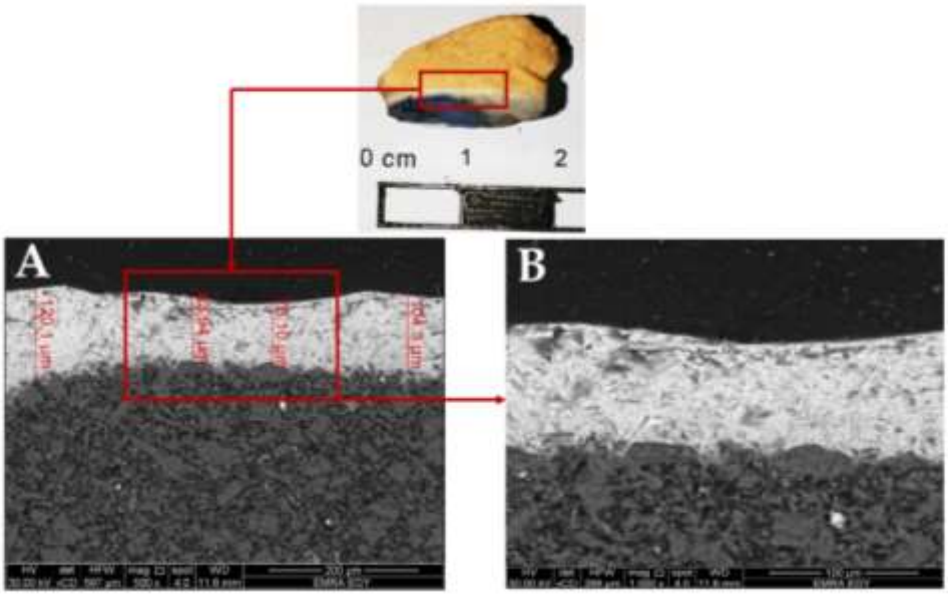


Figure 9. The Cross-section of a ceramic tile sample, A (500 X), B (1000 X)

Table 1. EDX wt% values of the chemical elements present in the glaze layer of the studied samples

Weight percentage of the elements in the samples %											
Analyzed Sample	**C**	**O**	**Na**	**Al**	**Si**	**K**	**Ca**	**Mn**	**Co**	**Cu**	**Fe**	**pb**
White glaze	--	20.83	5.29	0.96	21.17	--	**3.46**	--	--	--	--	**48.29**
Dark blue glaze	8.99	22.12	5.21	1.21	22.22	--	2.23	--	**0.73**	**0.36**	--	**35.77**
Light blue glaze	10.01	26.92	7.13	1.00	23.80	1.93	1.76	--	--	**2.01**	--	**25.44**
Green glaze	10.12	22.98	5.70	0.92	23.50	1.37	2.03	--	--	**3.78**	--	**29.59**
Coral red glaze	4.99	22.83	5.50	0.96	23.82	1.09	1.85	**0.76**	--	--	**0.62**	**37.58**

5. DISCUSSION

Ceramic tiles in the façade of the Sapil of Mostafa Sinan of the Ottoman era have many aspects of deterioration and degradation. Using the USB portable digital microscope, it showed some defects in the glaze layer resulting from the improper manufacturing of the tiles. Most glaze layers were crazing and peeling in some tiles, causing the glaze layer detachment from the ceramic body. These weak areas are the beginning of the complete deterioration of the tiles. The glaze surface may shrink or expand than the body of the ceramic tile, causing crazing.

Additionally, cracks may occur in the first burning of the biscuit (Mimoso et al., 2012). Crazing of the glaze can cause the loss of the layer's properties and permeability to liquids, resulting in severe damage to the ceramic tiles. Consequently, the tiles will be easily deteriorated because of external deterioration factors (Junior et al., 2021). Cracks and pinholes spread extensively in most ceramic tiles. Pinholes are tiny pin-like holes on the glaze surface, resulting from the explosion of air bubbles at the tile's top during burning (Rhodes, 1996, 264). The accumulation of dust and dirt and blurring the decorations resulted from human and industrial activities. The spread of dust and air pollutants, including sulfur dioxide, nitrogen oxides, and soot will spoil the ceramic structure. Although silicate materials are resistant to wear and corrosion, air pollutants of different sources can easily damage these materials if exposed for a long time. Dangerous chemical reactions can occur in cracks and crazing in the glazing surface transmitted to the ceramic tile body, causing severe damage and deterioration (Tournié, Ricciardi and Colomban, 2008). Additionally, distortion and blurring of the decorations of the ceramic tiles and opacity and darkness of the glaze occurred.

XRD results of the mortar illustrated that it consisted of gypsum, lime, and sand. It is commonly used these compounds of gypsum, calcite, and quartz as mortar at the time (Abd-Elrahim and Weshahy, 2017). Gypsum was transformed into anhydrite rated (50.6%), causing weak and fallen mortar and gaps behind the ceramic tiles. In the second mortar sample used in installing the lost tile, gypsum transformation into anhydrite was high (77.6%), and gypsum was (8.6%) only. This mortar change was caused by external deterioration factors, especially high humidity, temperature, and their fluctuations (Kamel, 2019, 50). Accordingly, the mortar lost its main characteristics for instance installing.

The halite in the first sample highlighted a simple salt infection in this part caused by the high rates of humidity and saline groundwater. Salts in most building materials can play a role as they move to the mortar and the ceramic tiles cause deterioration to both (Costa et al., 2014). Especially soluble salts (NaCl) that accumulate behind the glazed surface and crystallize (Costa et al., 1996). Salt crystallization in the pores of the material (crypto-florescence) can lead to severe damage in the form of e.g., sanding, crumbling, exfoliation, and spalling (Granneman et al., 2018). These results indicated of using gypsum, lime, and sand for this type of mortar. It is important stage to start the conservation process of the cultural heritage. The accurate raw materials should be carefully chosen and evaluated according to their characteristics to exploit their potential to make highly sustainable restoration mortars (Theologitis, et al., 2021).

The results of the XRD analysis of the ceramic tiles' body in the "Nafees" and to the right of the foundation text showed that they have the same structure. It is known that quartz mainly exists in the structure of all muds and was used as a modifying material in ceramics (Shepard, 1985). Some compounds, such as diopside and mullite, appeared in the high burning temperatures. Calcite interacts with silica forming diopside at 850 ^{o}C, and mullite (aluminum silicate) appears at 950 ^{o}C (Riederer, 2004, 150). Furthermore, there was spinel (21.4%) in the first sample and (18.7%) in the second sample, resulting from the transformation of metakaolin in the clay at (925-950 ^{o}C) (Rice, 1987, 90). The results also showed the presence of calcite, suggesting that the used clay had lime (a modifying ceramic material) (Trindade, 2009). XRD analysis of the body samples helped in identifying the approximate temperature of burning the tiles (Vecstaudža et al., 2013) rated (900-1000 ^{o}C). The clay used in manufacturing of the body was local and contained some lime. Whereas, Determining the type of clay and components through the different analysis methods is useful in the re-manufacturing process of these tiles in the case of restoration process.

SEM-EDX analysis of the glaze layer provided essential results on the ceramic tiles' glaze type and color oxides. Table 1 shows that the chemical composition of all samples was similar. They contained (Si, Al), indicating quartz and aluminum silicates are the base of glaze (Rhodes, 1996, 88). The samples also contained high percentages of lead (Pb) rating (25.44 - 48.29%), indicating the use of lead oxide (PbO) as a flux in burning to give a shiny glaze surface (Burleson, 2003, 23). Applying lead glaze to the ceramic body is more manageable than alkaline glaze because the lead glaze has low surface tension (Tite et al., 1998).

The appearance of sodium (Na) in approximate ratios in all samples rating (5.29%: 7.13%) with Pb indicated using sodium oxide (Na_2O) as another flux agent. It could be concluded that lead alkaline glaze was used to produce colorful ceramics over white glaze (Arantegui et al., 2004) introduced in the early Islamic era, rating 1:2%. Later, the lead percentage increased to 20-40% and alkaline to 5-12%. The compound was used to produce ceramics comparable to the Chinese ceramic in the Tang Dynasty. This glaze type was common in the Middle East and Europe to make ceramic with white glaze and cobalt blue decorations on an opaque glaze layer. Using large amounts of sodium oxide in the glaze layer raises the relative shrinkage rate of the glaze layer, making it prone to crazing. In this case, the shrinkage of the glaze layer is more significant than the expansion and shrinkage of the body. It also makes the glaze layer easy to scratch, erode, and be affected by different weathering factors (Tite et al. 1998; Abd algwad 2015).

- **White glaze:** SEM results showed the deterioration of the glaze layer due to crazing, tiny holes, and peeling because of glaze defects. EDX analysis of the white glaze illustrated (Si, Al, Na, and Pb) and Ca (3.46%), indicating using calcium to get matte white (Sadek, 2016).
- **Dark blue glaze:** SEM observation showed cracks, fine scratches, holes, and peeling in the glaze as manufacturing defects. There were (Si, Al, Na, and Pb) in addition to Co (0.73%) and Cu (0.36%), indicating the use of CoO to get blue glaze in the Islamic era (Pérez-Arantegui et al., 2009). Cobalt is the most strong and effective coloring material in the glaze, even if added in small amounts, resulting in dark blue with lead glaze (Britt, 2007, 23).
- **Light blue glaze:** SEM examination showed crazing, several holes, uneven surface, and crazing in the glaze layer. There were (Si, Al, Na, Ca, and Pb) in addition to K (1.93%) as a flux material that decreases the flow of the glaze layer in burning (Rhodes, 1996, 89). Light blue appeared due to copper oxide because Cu appeared in the sample (2.01%) (Dabanli et al., 2021).
- **Green glaze:** SEM study results of green glaze showed crazing and tiny holes. EDX results showed (Si, Al, Na, K, Ca, and Pb) and Cu (3.78%), indicating the use of copper oxide to get green in the lead glazes (Gerard and Porter, 2002, 17).
- **Coral red glaze:** The examination of the coral red glaze illustrated some cracks, crazing, and uneven surface glaze. EDX analysis results showed (Si, Al, Na, K, Ca, and Pb) and Fe (0.62%), Mn (0.76%), these results indicated that coral red color appeared due to using the iron oxide with manganese oxide (Rhodes, 1996; Muşkara and Kalayci, 2021).

The cross-section of the ceramic tile studied using SEM (Fig. 10) showed a difference in the thickness of the glaze layer that varies from 78.10 to 120.1 µm, suggesting the asymmetric glazing application. Commonly, glazing consisted of applying one or more layers of glaze with a total thickness ranged between 75 and 500 µm, covering the surface of the ceramic tile (Casasola et al., 2012).

Finally, the results of this study provide useful of information and data, that can be used in the process of restoration of these tiles, as well as in the manufacturing process of modern tiles to replace the lost tiles on the façade of the sapil. Based on the present analyses, the well-prepared mortar can be applied to reinstalling the ceramic tiles.

6. CONCLUSIONS

The present paper offered essential findings regarding the deterioration of ceramic tiles of the façade of Sapil Mustafa Sinan, Al-Darb Al-Ahmar, Cairo using non-destructive examination and analysis methods. It investigated and identified the components and structure of the ceramic tiles and the mortar of installation. It was reported that the poor manufacturing of the tiles and the external deterioration factors, including humidity, air pollutants, and neglect caused severe damage to the tiles, such as cracks, crazing, glaze detachment, cracking and mortar smashing, and loss of some tiles. The mortar of installation was composed of gypsum, lime, and sand. However, the gypsum turned into anhydrite due to the exposure to high temperatures, high humidity rates, and their fluctuation.

Additionally, the clay used in the body of the ceramic tiles was local; It contained a percentage of lime, and glazing was lead-alkaline. To obtain the glaze colors of the tiles, calcium oxide was used for the white glaze, cobalt oxide for dark blue, and copper oxide to get light blue. Copper oxide was used mainly with lead glaze for obtaining the green glaze. Iron oxide with manganese was used to get the coral red glaze. The ceramic tile was square and measured (25 cm ×25 cm) and 1cm thick. It was noted that the glaze layer was uneven and varied (78.10-120.1 µm), and the burning temperature of making the tiles ranged between 900-1000 ^{o}C. These significant findings could help in making a restoration and conservation plan of the ceramic tiles and producing new tiles instead of the lost ones to maintain the present tiles of the Sapil's façade and avoid their complete loss and contributes to the conservation programs of the similar historical buildings in Egypt.

ACKNOWLEDGMENTS

I extend my sincere thanks and gratitude to anonymous reviewers for their constructive comments. The researcher, also, would like to thank my professor Mohamed Moustafa Ibrahim, Department of Conservation, for his constant support, and Dr. Noha Gamil, Department of Islamic Archeology (Faculty of Archeology - Cairo University), for helping to describe the decoration of the tiles. I would like to thank Dr. Mokhtar El-Nady, Dr. Hany Gad-Alrab, Ms. Ghada Hashim, Mr. Ali Abdel Aal for helping in research accomplishment.

REFERENCES

Abd algwad, R., (2015) *Study of techniques, factors and aspects of deterioration of glazing layer on ceramic tiles in some Ottoman buildings at Rashid City, and methods of treatment and conservation*, MSc thesis, Faculty of Archaeology, Cairo University, Egypt. (In Arabic).

Abd-Allah, R., Al-Muheisen, Z., Al-Howadi, S., (2010) Cleaning strategies of pottery objects excavated from Khirbet Edh-Dharih and Hayyan Al-Mushref, Jordan: Four case studies, *Mediterranean Archaeology and Archaeometry* 10, 97-110.

Abd-Elrahim, E., and Weshahy, I., (2017) The mortar damage and its harmful effects on the glazed ceramic tiles in Terbana mosque – Alexandria, Egypt, *SHEDET* 4,155-166. Doi: 10.36816/shedet.004.10

Al-hussieny, M. H., (1988) *Ottoman sapils in Cairo 1517-1798AH., Cairo, Egypt*, Publisher: Madbouly Bookstore (In Arabic), ISBN 2-094-133-977.

Ali, M., Omar, Sh., (2021) Analytical investigation of deterioration aspects in the mihrab of Madrasa Gawhariyya of Al-azhar mosque, Egypt, *Egyptian Journal of Archaeological and Restoration Studies (EJARS)* 11, 9-17. doi:10.21608/EJARS.2021.179491

Arantegui, J., Larrea, A., Molera, J., Pradell, T., and Vendrell-Saz, M., (2004) Some aspects of characterization of decorations on ceramic glazes, *Applied Physics A* 79(2), 235-239. DOI:10.1007/s00339-004-2508-2

Belfiore, C. M., Mazzoleni, P., Manenti, A. M., Mastelloni, M.A., Corsale, V., Barone, G., (2021) Non-destructive XRF analysis of *Aegyptiaca* from Sicilian archaeological sites, *Mediterranean Archaeology and Archaeometry,* Vol. 21, No 1, 37-69: Doi: 10.5281/zenodo.4284407.

Britt, J., (2007) *The complete guide to high-fire glazes*, Sterling Publishing Company, Inc, New York.

Burleson, M., (2003) *The ceramic glaze handbook: Materials*, techniques, formulas, Lark Book Publication, New York.

Buys, S., Oakely, V., (1999) *Conservation and restoration of ceramics*, Butterworth – Heinemann, ltd., Oxford.

Casasola, R., Ma Rincón.J., Romero, M., (2012) Glass-ceramic glazes for ceramic tiles – a review, *Journal of Material Science* 47(2), 553-582. DOI:10.1007/s10853-011-5981-y

Çiçek, A., Aslan, A., Kenan, Y., and Ali, S.K., (2009) Effects of environmental conditions on historical buildings: lichens and NOx gases, *Environmental Monitoring and Assessment* 154, 187-195. DOI: 10.1007/s10661-008-0388-1.

Coppola, M., Benedetto, F., Garzonio, C., Pecchioni, E., Santo, AP., (2020) Groundwater damages on the historic buildings of Cairo: The case of the medieval walls of Mokattam limestone, *Materials Science and Engineering* 949, 1-9. DOI: 10.1088/1757-899X/949/1/012004.

Costa, J. P, Antunes, F. J.L., Figueiredo, M.O., Fortes, M. A. 1996. Removal and analysis of soluble salts from ancient tiles, *Studies in Conservation* 41:153-160. DOI:10.1179/sic.1996.41.3.153.

Costa, M., Cachim, P., Coroado, J., Rocha, F., Velosa, A.L., (2014) Ancient wall tiles – The importance of the glaze/ceramic interface in glaze detachment, *Materials Science* 20, 108-113. DOI: /10.5755/j01.ms.20.1.3815.

Dabanlı, Ö. Yıldız, D., Bayazit, M., (2021) Composition and phase analysis on glazed tiles of southeast Anatolia: production process identification, *Mediterranean Archaeology and Archaeometry,* Vol. 21, No 3, 1-22. DOI: 10.5281/zenodo.5545709 Doi: 10.5281/zenodo.3541108.

Elyamani, A., Bader, N., Algohary, M. and Hassan, R., (2021) Explanation of the Damage to the Royal Family's Cemetery in Historic Cairo and Examination of the Building Materials. *Open Journal of Civil Engineering* 11, 28-59. DOI: 10.4236/ojce.2021.111003.

Fernández, M. M., Visiedo, J. P., Garrigós, J.B., (2021) Exploring the technique of glazing used by the potters of Barcelona, *Mediterranean Archaeology and Archaeometry,* Vol. 21, No 3, 69-88. DOI: 10.5281/zenodo.5598243.

Francesco, A.M. De., Miriello, D., Forciniti D., Guido, A., (2021) Physicochemical analysis of original and restored carbonate material of the Romanic church bell tower in LONGOBUCCO (CALABRIA, ITALY), *Mediterranean Archaeology and Archaeometry*, Vol. 21, No 1, 121-132: DOI: 10.5281/zenodo.4394060.

Gelzo, M. Corso, G., Vergara, Al., Rossi, M., (2021) Archaeometric studies on a blue glass fragment from Pompeii: case study, *Mediterranean Archaeology and Archaeometry*, Vol. 21, No 3, 23-34. DOI: 10.5281/zenodo.5545711.

Gerard, D., Porter, Y., (2002) *The art of the Islamic tile*, Flommarion Publication, Paris.

Granneman, S.J.C., Lubelli, B., Van Hees, R.P.J., (2018) Characterization of lime mortar additivated with crystallization modifiers, *International Journal of Architectural Heritage* 12(5), 849-858. DOI: 10.1080/15583058.2017.1422570.

Grossi, C. M., Brimblecombe, P., (2007) Effect of long-term changes in air pollution and climate on the decay and blackening of European stone buildings, in: Přikryl, P. & Smith, B. J. (Eds.), Building stone decay: from diagnosis to conservation, *Geological Society, London, Special Publications* 271, 117-130. DOI: 10.1144/GSL.SP.2007.271.01.13.

Gulzar, S., (2017) Deterioration morphologies of historic ornamental panels from the Mughal Historic Monuments of Lahore (Pakistan), *International Journal of Advances in Mechanical and Civil Engineering* 4, 14-17.

Junior, G.S., Ferreira, J., Millán-Arias, C., Daniel, R., Junior, A.C., and Fernandes, B.J.T., (2021) Ceramic cracks segmentation with deep learning, *Applied Sciences*, 11, 6017, 1-13. DOI: 10.3390/app11136017.

Kamel, A. M.A., (2019) Dehydration of gypsum component of plasters and stuccos in some Egyptian archaeological buildings and evaluation of K_2So_4 activator as a consolidant, *SCIENTIFIC CULTURE*, Vol. 5, No. 2, 49-59. DOI:10.5281/zenodo.2649508.

Kaplan, Z., Ipekoglu, B., Boke, H., (2017) Physicochemical properties of glass tesserae in roman terrace house from ancient Antadros (Base glass, opacifiers and colorants), *Mediterranean Archaeology and Archaeometry*, Vol. 17, No 1, 141-157. DOI: 10.5281/zenodo.258103.

Krueger, M., Brandherm, D., Krueger, M., Niedzielski, P., (2021) Archaeometric analysis of late bronze age and early iron age pottery from SETEFILLA (SW SPAIN), *Mediterranean Archaeology and Archaeometry*, Vol. 21, No 1, 21-36. DOI: 10.5281/zenodo.4284405.

Liritzis, I., Laskaris, N., Vafiadou, A., Karapanagiotis, I., Volonakis, P., Papageorgopoulou, C., Bratitsi, M. (2020) Archaeometry: an overview, *Scientific Culture*, Vol. 6, No. 1, 49-98. DOI: 10.5281/zenodo.3625220.

Mahmoud, M. M., (2019) Water fountains in the islamic architecture: simplicity of techniques & fascinating performance, *International journal of architectural engineering and urban research* 2 (2), 34-39.

Măţău, F., Chişcan, O., Pintilei, M., Garvăn, D., Stancu, Al., (2019) Technological features of the chalcolithic pottery from Târpeşti (neamț county, eastern Romania), *Mediterranean Archaeology and Archaeometry*, Vol. 19, No 3, 93-104. DOI: 10.5281/zenodo.3541108.

Mimoso, J., Silva, A., Abreu, M., Costa, D., Goncalves, T., Coentro, S., (2009) Decay of historic Azulejos in Portugal: An assessment of research needs, International Seminar "*Conservation of Glazed Ceramic Tiles. Research and practice*", National Laboratory for Civil Engineering, Lisbon, April 15-16, 2009, 1-11.

Moussa,A. Ali,M.F., (2013) Color Alteration of Ancient Egyptian Blue Faience, *International Journal of Architectural Heritage* 7, 261–274. DOI: 10.1080/15583058.2011.634960.

Muşkara, Ü., Kalaycı, K., (2021) The feasibility of PXRF for discriminating attic black-figure painters using pigment analysis, *Mediterranean Archaeology and Archaeometry*, Vol. 21, No 1, 237-255. DOI: 10.5281/zenodo.4574639.

Pérez-Arantegui, J., Montull, B., Resano, M., Ortega, J.M., (2009) Materials and technological evolution of ancient cobalt-blue-decorated ceramics: Pigments and work patterns in tin-glazed objects from Aragon (Spain) from the 15th to the 18th century AD, *Journal of the European Ceramic Society* 29, 2499–2509. DOI:10.1016/J.JEURCERAMSOC.2009.03.004.

Pradel, T., Molera. J., Salvadó, N., Labrador, A., (2010) Synchrotron Radiation Micro-XRD in the Study of Glaze Technology, *Applied Physics A* 99(2), 407-417. DOI: 10.1007/s00339-010-5639-7.

Pradell,T., Molera, J., (2020), Ceramic technology. How to characterize ceramic glazes, *Archaeological and Anthropological Sciences 12*: 189, 1-28. DOI: 10.1007/s12520-020-01136-9.

Rhodes, D., (1996) *Clay and glazes for the potters*, London.

Rice, P.M., (1987) *Pottery analysis*, University of Chicago Press, USA.

Riederer, J., (2004) Thin section microscopy applied to the study of archaeological ceramics, *HyperFine Interactions* 154, 143-158. DOI: 10.1023/B:HYPE.0000032029.24557.b1.

Sadek, H., (2005) *Study of technology, treatment and conservation of archaeological Islamic ceramic lamps with application on some selected objects*, MSc thesis, Faculty of Archaeology, Cairo University, Egypt. (In Arabic).

Sadek, H., (2016) Multi-analytical approach for the study of glazed pottery from Al-Fustat, Egypt, *Mediterranean Archaeology and Archaeometry* 16(3), 65-71. DOI:10.5281/zenodo.160950.

Sánchez Ramos, S., Bosch Reig, F., Gimeno Adelantado, J. V., Yusá Marco, D. J., Doménech Carbó, A., (2002) Application of XRF, XRD, thermal analysis, and voltammetric techniques to the study of ancient ceramics, *Anal Bioanal Chem* 373, 893–900. DOI:10.1007/s00216-002-1376-x.

Shepard, A.O., (1985) *Ceramics for the archaeologists*, Carnegie Institution of Washington Publication 609, Washington.

Silva, T. P., Figueiredo, M. O., Prudêncio, M. I., (2013) Ascertaining the degradation state of ceramic tiles: A preliminary non-destructive step in view of conservation treatments, *Applied Clay Science* 82, 101-105. DOI: 10.1016/j.clay.2013.06.013.

Theologitis, A., Kapridaki, C., Kallithrakas-Kontos, N., Maravelaki-Kalaitzaki, P., Fotiou, A., (2021) Mortar and plaster analysis as a directive to the design of compatible restoration materials in frangokastello (Crete), *Mediterranean Archaeology and Archaeometry*, Vol. 21, No 1, 109-120: Doi: 10.5281/zenodo.4284427.

Tite, M.S., Freestone, I., Mason, R., Molera, J., Vendrell-Saz, M., Wood, N., (1998) Review article lead glazes in antiquity, methods of production and reasons for use, *Archaeometry* 40 (2), 241-260. DOI: 10.1111/j.1475-4754.1998.tb00836.x.

Tite, M.S., Manti, P., Shortland, A.J., (2007) A Technological Study of ancient faience from Egypt, *Journal of archaeological science* 34, 1568-1583.

Tournié. A., Ricciardi. P., Colomban. Ph., (2008) Glass corrosion mechanism: A multiscale analysis, *Solid State Ionics* 179, 2142-2154. DOI: 10.1016/j.ssi.2008.07.019.

Trindade, M.J., Dias, M.I., Coroado, J, Rocha, F., (2009) Mineralogical transformations of calcareous rich clays with firing: A comparative study between calcite and dolomite rich clays from Algarve, Portugal, *Applied Clay Science* 42(3/4), 345-355. DOI: 10.1016/j.clay.2008.02.008.

Vecstaudža, J., Jakovļevs, D., Bērziņa-Cimdiņa, L., Stikān, V., (2013) XRD and SEM studies of archaeological stove tile ceramics of Turaida Castle, *Material Science and Applied Chemistry* 29, 40-45. DOI: 10.7250/msac.2013.018.