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STUCCO DECORATIONS OF ZAWIYA AL-QADIRIYYA OF MAMLUK ERA IN CAIRO: MULTI-INSTRUMENTAL EXAMINATIONS AND ANALYSES

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Abstract

Investigation and analysis of falling part samples of the stucco decoration from different places of Zawiya al-Qadiriyya (the Zawiya of Zain al-Din Youssef) have been performed. Zawiya al-Qadiriyya in the Southern Cemetery was built in 1297–1298 A.D. and expanded in the early 14th century in Cairo, Egypt, dating to the 19th century, designed by Antonio La Schiac. X-ray fluorescence (XRF) analysis was done to detect the elemental chemical composition of the collected falling part or deciduous part samples. The main detected elements were Ca, S, Si, and C. X-ray diffraction analysis (XRD) was carried out for various samples from different places from the top surface and core to investigate the crystal structure and phase analysis of these samples. XRD patterns confirmed that the samples are mostly composed of gypsum, anhydrite, calcite, and quartz sand as the main constituents. The microscopic investigation was performed using a stereo optical microscope (OM) and scanning electron microscope (SEM) to examine the surface morphology and explore the core microstructure. In addition, physical characteristics such as density, porosity, and water absorption were carried out to complete the characterization profile of the stucco decoration of falling part or deciduous part samples. The complete image of the samples' characteristics will help us to select a proper restoration and maintenance route to clean, consolidate, and preserve the unique original structure and features of its stucco decoration.

Keywords: *Mamluk era; Zawiya al-Qadiriyya; XRF; XRD; Optical microscope; SEM.*

Introduction

The language of Islamic art is obviously reflected by the unique characteristics' decoration of the Arab-Islamic architecture [1]. The integration and progress inspired by the Islamic faith have been started with the Islamic civilization. The whole world has been glowed by time and illuminated by the light of knowledge after eras of darkness and ignorance. When the Islamic culture reached its richest era, the real features of the oriental Islamic arts were known. The main aim of Islamic arts was to keep the value of art for the service of life. This was clearly observed, especially in mosques with their architectural elements internally and externally. In the early stages of the Islamic arts, no artistic Islamic civilization and no artistic

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style of value in the emergence of Islam appeared due to the nature of the Arabian region. The overlap of the arts of the opened countries by the Arab Muslims with each other in the embodiment of Islamic art forms. This led to the development of decorative arts and the emergence of star shapes. "Star dishes" have been led by scientific and technological progress and economic and urban growth, which had a clear impact on the plastic creativity of Islamic arts [2]. The arts of Arab decorative writings began to flourish and possessed their engineering character in the decoration of palaces, mosques and domes in their geometrical artifacts, or mixed forms of arabesques.

The Mamluk period is considered one of the most significant periods in the history of Islamic architecture in Egypt [3]. The Mamluk Bahri and Burgi period (648-923 AH/1250-1517 AD) in Cairo [4]. Up till now, it is considered the golden era of the historical Islamic arts in Egypt. The extent of the influence of entrances and balconies of the ears and window openings in Mamluk architecture elements on the architectural elements has been studied. On the other hand, the influences of the Mamluk architectural and decorative elements on the same elements in the Ottoman period have been investigated through examples from Egypt and Turkey [5].

Since ancient times, from the Pharaonic periods till now, stucco has been utilized in Egypt. It has been extensively investigated using spectroscopic, typological, and archaeological methods to determine its purpose, make-up and age [6-14]. Many types of stucco decorations were widely used in buildings and mosques during Egypt's Islamic era and developed into significant architectural decorative components for wall friezes, geometric, floral, and inscriptive ornaments, medallions, mihrabs, and stained-glass windows made of stucco [15-16].

A detailed study on the analysis and characterization of the components and deterioration products of the stucco frieze of Zain al-Din Youssef Zawiya (Fig. 1) plays an important role in selecting the proper conservation materials and techniques. Zawiya of Zain al-Din Youssef belonged to the Bahari Mamluk Period. It was built (697-736 AH/1298-1335 A.B.) [17]. Chemical composition and physical characteristics of the deciduous part samples will help in performing a proper strategy for restoration and conservation of the antiquities. The epigraphic decoration was implemented in stucco friezes by the bas-relief carving method on a ground of floral and geometric bands located in the corners of friezes and stucco windows were decorated with perforations or hollowing out and formed with arabesque motifs. The stucco decorations in the corner suffer from many different manifestations of damage, such as the black calcified layers on the surfaces of the stucco decorations, the presence of various cracks and cracks, and the previous restoration using gypsum to install the windows. There are also many manifestations of loss in the stucco decorations, whether in the windows themselves or parts of them or in the stucco bands. Written, geometric, and vegetal and the corner is vaulted from the ions and the courtyard therefore rainwater accumulates on it, and the presence of moisture in the stucco decorations and human damage.

In this work, we report on the investigation and analysis of some collected deciduous part samples from stucco decorations in Zain al-Din Youssef Zawiya from the Mamluk era in Cairo. Chemical and phase composition of the top surface layer, bulk core, and backside samples were investigated through multi-instrumental examinations and analyses. Among them is the fluorescence of an X-ray and its diffraction, which is one of the non-destructive methods of analysis. It determines the chemical and phase composition without alteration of the material's identity through fluorescence and diffraction of X-ray beams. In addition, surface morphology and core structure have been investigated microscopically using stereo, polarising light, and scanning electron microscopes. Moreover, spectroscopic analysis with FTIR was carried out to detect the functional groups composing the collected samples. Physical and mechanical characteristics, including density, porosity, water absorption, and compressive strength, were made to complete the image of the collected samples for further better selection of the route of conservation and restoration of the monument.

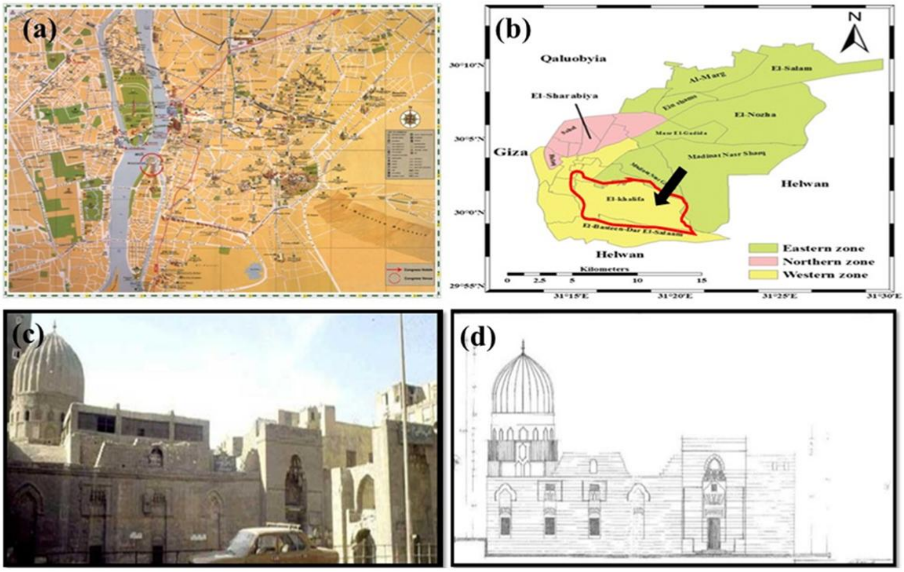

Fig. 1. (a) Site of Almadrasa Al-Qadiriyya in Historic Cairo by Satellite map; (b) The arrow and red-lined zone refer to the site of Zawiya of Zain al-Din Youssef (Al-Qadiriyya Zawiya) in Historic Cairo by Satellite map; (c) general view from Facade Zain al-Din Youssef Zawiya, founded in 1297–98 and expanded in the early 14th century in Cairo, Egypt, dating to the 19th century, designed by Antonio La Schia; and (d) image of Facade from Zain al-Din Youssef Zawiya.

Experimental part

Methodology

Stucco samples from outer decorations in the dome of Zain al-Din Youssef bn Yahia Zawiya from falling parts in different places were collected and coded according to its position in the stucco decoration (Fig. 2).

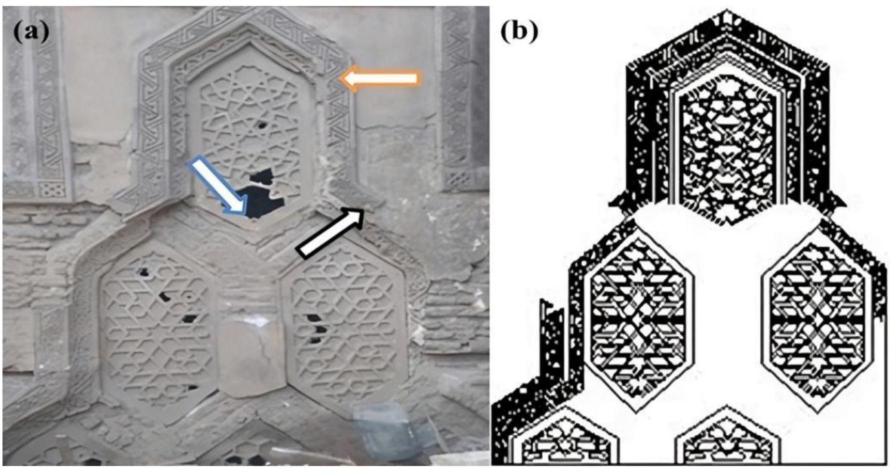

Fig. 2. (a) Photo of a part of stucco decoration and the places of sampling, (b) drawing of stucco decoration showing the falling parts and weak layers and damage of some parts.

KZ1 is the collected sample from the dusty layer from the outer surface to identify the accumulated dirt on the surface of the stucco frieze surrounding the dome. It is from the outside overlooking the street which contains the main entrance to the corner, which is from the southeast direction, and suffers from some missing parts, as is the case with the stucco windows surrounding the dome from the outside. Another sample of core (KZ2) was taken from the components of the archaeology to identify its components so that a simulation of the weather effect with the same basic components is made in further study. Mortar of weak layers that lead to the fall and damage of some parts (KZ3) of the stucco frieze and a sample of the stone support of the stucco and a sample of the bricks used in the construction of the dome. Table 1 represents the collected samples and their definitions.

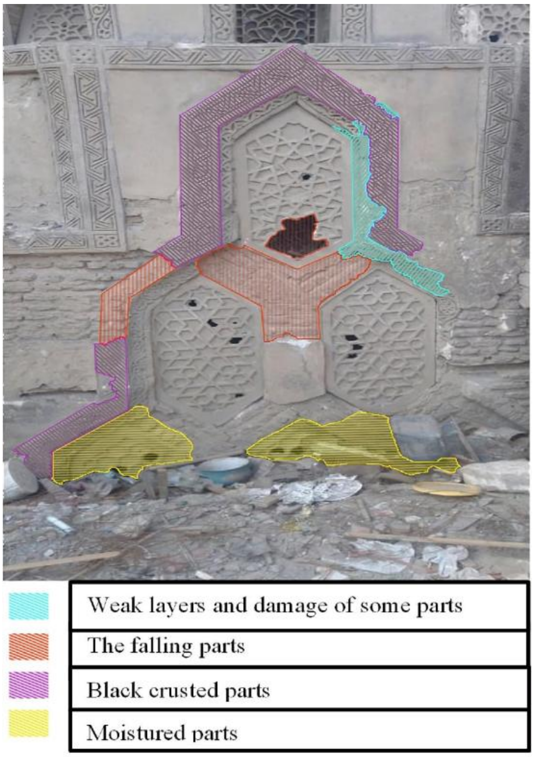

Fig. 3. Stucco ornamentation degradation map displaying the missing, frail and damaged components

Table 1. Collected samples and its code of definition

Definition of samples	Sample Code
Top surface layer sample	KZ1
Core sample	KZ2
Backside sample	KZ3

Characterization tools

Portable Energy-dispersive X-ray fluorescence analysis (p EDXRF)

The collected samples have been analysed using portable XRF-gun Thermo Scientific Niton. It is a non-destructive technique and requires no or very little powdered sample for measurement [18, 19]. A portable and handheld system for energy-dispersive X-ray fluorescence analysis has been used.

X-Ray diffraction analysis (XRD)

It is one of the methods used to identify the components of the collected samples of solid materials with crystal structure to identify the phase composition of inorganic antiquities such as stucco and cartonnage [8, 19, 20]. An X-ray diffraction (XRD) analysis was carried out using a PANalytical computer-certified program with the aid of the International Centre of Diffraction

Database (ICCD), PDF-2 Database-CD-Release 2005-Type No. 943050001611. X-Pert HighScore Software 2006 - Licensed modules: PW 3209 Tube anode; Cupper (Cu). The 2ϴ (theta) scan range varied from 5-50^o.

Fourier Transform Infra-Red analysis (FTIR)

About 150 mg of sample is mixed with potassium bromide (KBr) and then it is introduced into a circular mould and held under pressure to obtain a circular disc with a thickness ranging from 0.1 to 0.01mm. The infrared spectrum is then collected at a resolution of 4cm^{-1} and 20 scans were recorded per sample. Spectrum in the range 4000-400cm^{-1} is recorded, the baseline is corrected and atmospheric compensation is done. IR Prestige-21 FTIR Spectrometer for analysis and the IR solution software were used for data interpretation.

Stereo light microscope (LM)

It is a continuous zoom digital microscope with a magnification range of 1 up to 1200×. With the ability to control and adjust the level of illumination, the results in the form of images are easily obtained to a computer via a USB connection without installing any drivers. Through these results, preliminary images were produced, showing the shape of the grains of the surface and its cohesion, in addition to the shape of the various manifestations of damage on it. Using an optical microscope to examine the collected samples. The samples were placed on a slide, which was placed on a platform and secured with platform clamps. Move the slide if it is necessary that the sample is located directly below [21].

Polarizing light microscope (PLM)

A thin section of the collected samples is studied and analysed by a polarising light microscope. It was affixed to a transparent glass slide with Canada balsam [22]. It is polished so that its thickness becomes very thin, about 0.03mm (30 microns) and cold adhesives such as cyanoacrylate are used to fix the samples on the glass to prevent the sample from disintegrating during the lamination process. The scientific basis for this type of microscope depends on the use of polarised light (instead of ordinary light) to identify the minerals that constitute the different rocks by studying the optical properties of the minerals, such as the shape, colour, fracture, cleavage and refractive index. In addition, colour change, surface topography, morphology and details of the relationship of grains and crystals in the samples are also studied.

Scanning electron microscopy (SEM)

Surface structure and morphology of the collected samples are examined using the scanning electron microscope (FEL quante3D200i). It is recommended that the size of the sample to be examined must be very small in order not to absorb the electrons falling on it and must be chemically inert [23].

Physical characteristics

The compressive strength, density, water absorption and porosity of the collected samples are measured according to the corresponding standards.

Results and discussion

X-Ray fluorescence analysis

Samples for XRF analysis were collected as follows: KZ1 sample was taken from the surface, KZ2 sample from the core and KZ3 sample from the backside of the stucco strips that surround the dome from the outside from the southwestern side and more than one analysis was conducted to verify their components. The results of X-ray fluorescence analysis showed the presence of calcium oxide (CaO), sulphur oxide (SO_2), silicon oxide (SiO_2) and carbon in the collected samples with various amounts. As CaO and SO_2 are present in large amounts (47~57%) and C is expressed as CO_2 in small amounts (0.04~0.6%), this might be an indication of the presence of gypsum or anhydrite phases. SiO_2 may be present as silica sand, as will be shown from the XRD investigation. Table 2 shows the chemical composition and percentage of each component constituting the samples [15].

Table 2. Non-destructive chemical analysis by EDXRF-portable handheld system of the collected samples

Item	SO_3	CaO	SiO_2	CO_2
KZ1	50.55	35.83	13.8	0.11
KZ2	57.70	40.39	1.85	0.044
KZ3	47.31	32.65	17.3	0.63

X-Ray diffraction analysis

Figure 4 and Table 3 show the phase composition and its semi-quantitative percentage in the collected samples (KZ1, KZ2 and KZ3). It is clear that the sample is composed mainly of four phases: calcium sulphate hydrate (Gypsum, $CaSO_4.2H_2O$), calcium sulphate (Anhydrite, $CaSO_4$), silica (Quartz, SiO_2) and calcium carbonate (Calcite, $CaCO_3$). In addition, sodium chloride (Halite, NaCl) phases are also present in some collected samples. Semi-quantitative analysis of the detected phases is also represented in Table 3. In the first surface layer sample (KZ1), it is clear from the analysis of the sample the presence of sodium chloride salt (4%) and quartz (17%) and a large proportion of hydrated calcium sulphate (79%). This indicates the presence of deterioration of the surface stucco layer and the presence of calcifications from archaeology and dirt with the presence of chloride salts. Whereas in the second core sample (KZ2), the results of the analysis show the presence of calcium sulphate compound in a large proportion, as gypsum (63%), which is the main component for the elements of decoration stucco. In addition to the presence of anhydrite in an appreciable amount (30%). This is evidence of the interaction between the gypsum compound with the surrounding environment and the occurrence of deterioration and evidence of the passage of the impact for a long period of time to make phases change and the presence of calcium carbonate phase. While lime is added to the gypsum compound in order to prolong its solidification time and the quartz acts as a filler, the stucco components are mainly composed of gypsum, lime and quartz in certain proportions. These decorations are exposed to vibrations at various temperatures and moisturising weather, which might affect the water content. This might be the main sign of the turning of plaster from the gypsum to the bassanite phase [24]. The other affected component is the calcium oxide, where it turns to calcium hydroxide upon hydration.

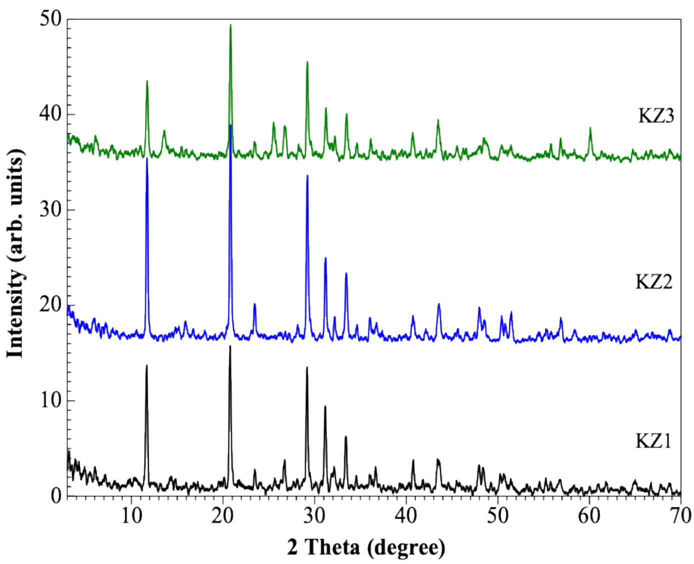

Fig. 4. XRD patterns of KZ1, KZ2 and KZ3 samples. Sample were taken from the stucco strips that surrounds the dome from the outside from the southwestern side, to verify their components.

Table 3. Semiquantitative analysis of phase composition for the three collected samples

Sample code	Calcium Sulphate Hydrate ($CaSO_4.2H_2O$), % [Gypsum, 01-086-8063]	Calcium Sulphate ($CaSO_4$), % [Anhydrite]	Sodium Chloride, % [Halite, 01-080-3939]	Silicon Oxide (SiO_2), % [Quartz, 01-085-0457]	Calcium Carbonate ($CaCO_3$), % [Calcite]
KZ1	79.0	--	4.0	17.0	--
KZ2	63.0	30.0	--	2.0	5.0
KZ3	47.0	--	13.0	32.0	--

It is well known that the air pollutants have an impact on stucco decorations [25]. Those pollutants together with the heat and moisture effects have a serious impact on the mechanical properties of stucco decoration and affect their weather resistance. Moisture will have a serious effect when the groundwater is rich in salts. In this case, the minerals that compose the stucco decoration will be the most damaged component in stucco decorations.

Analysis by FTIR

One sample (KZ2) from the core of the stucco strips that surround the dome from the outside from the southwestern side was selected to perform FTIR spectroscopic analysis. Infrared absorption spectroscopy is considered one of the most important spectral methods in the identification of intermediate and amorphous organic materials used in the archaeological field by identifying their functional groups [26]. Fig. 5 illustrates the FTIR spectrum obtained for the KZ2 sample. Table 4 shows the detected bands and the corresponding functional group for the core sample (KZ2) [27].

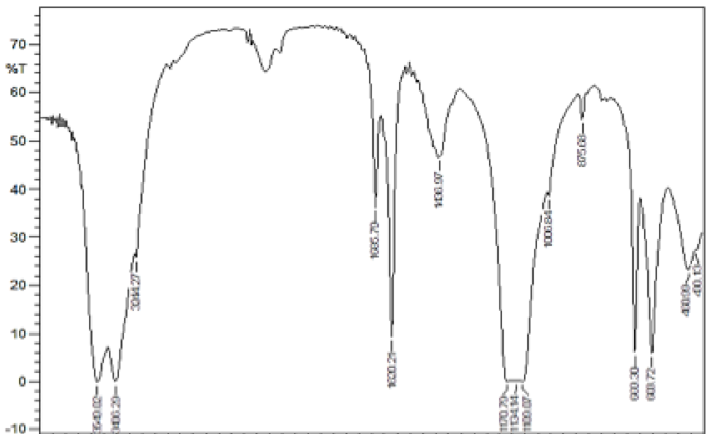

Fig. 5. FTIR spectra of the collected KZ2 sample

Table 4. FTIR analysis of core sample KZ2

	Bands	Functional group	Components
Sample of stucco decoration (KZ2)	460 - 603, 669	Si-O-Si	Silicates
	875 - 1006	Si - O	Quartz
	669 - 875	C - O	Calcite
	1436 -1620	CO3	Calcite
	2100 - 2500	Carbon triple bond	Carbonate
	1006 - 1109	SO4	Anhydrite
	1170 - 1436	SO4	Gypsum
	1620- 1685	O-H	Gypsum, water
	3244- 3500	O-H	Water, Gypsum hydroxide

Stereo-light microscope

One sample from the surface of the stucco strips that surround the dome from the outside from the southwestern side was selected for stereo-light microscopic investigation. Stereo light microscopy images of top surface samples showing rough structure of the outer surface layer, intensive dirty layer with spaces between the granules of the metal species (Fig. 6), porous and cracked surface layer [21].

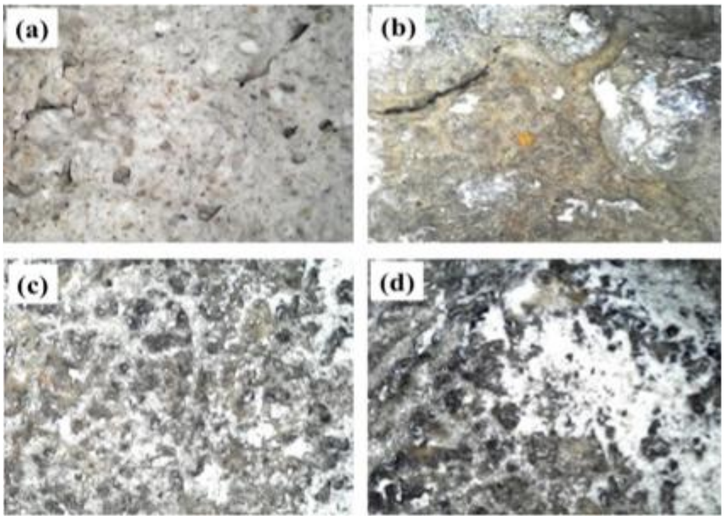

Fig. 6. Stereo-light microscopy images of top surface samples showing rough structure of the outer surface layer, intensive dirty layer with spaces between metal species granules, porous and cracked surface.

Polarizing light microscope

One sample from the surface of the stucco strips that surround the dome from the outside from the southwestern side was selected for polarising light microscopic investigation. Images of the polarising light microscope investigations show the shape of the outer surface layer (Fig. 7.).

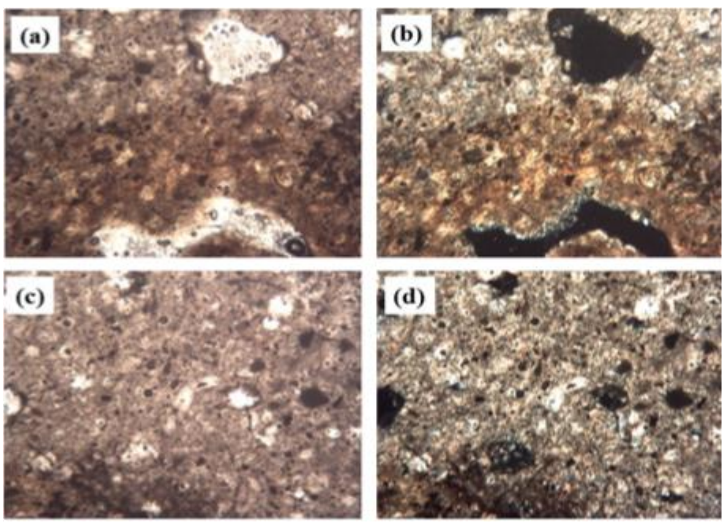

Fig. 7. Polarising light microscopy images show the shape of the outer surface layer. It has gypsum grains as well as diffuse grains of calcite and sharp-angle grains of quartz of small size in polarised light.

The petrographic structure of an archaeological optical sample in normal light and polarised light shows the sensory sample after being photographed with a microscope without the transducer. Through examination, it became clear that the main component, the gypsum grains, are well present, as diffused grains of calcite and sharp-angle, small-size grains of quartz are also obviously detected (Fig. 7).

Scanning electron microscope

One sample from the surface of the stucco strips that surround the dome from the outside from the southwestern side was selected for SEM investigation. The scanning electron microscope differs from other ordinary microscopes in that it does not contain lenses and uses electron beams as a light source. It is considered one of the most important modern technologies that have volunteered to serve the field of archaeology. By examining the sample, one can obtain adequate and accurate information from the mineral and crystalline structures of the surface of the sample and identify the extent of the change that occurred on the mineral crystals as a result of various damage factors, which gives a clear picture of the extent of damage that the impact may reach. It is characterised by high magnifications that show all the small details on the surface (Fig. 8).

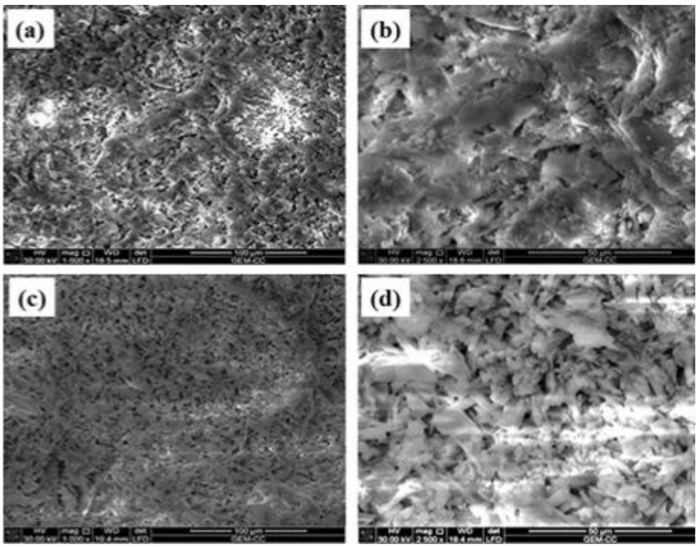

Fig. 8. SEM images of the collected samples: (a and b) give a clear picture of the extent of damage; (c and d) from the mineral and crystalline structures of the surface.

Physical characteristics

The compressive strength, water absorption, density and porosity of the collected samples were measured and evaluated as shown in table 5. It is clear that the surface layer sample has low compressive strength (11.59kg/cm^2) with high water absorption (78.64%) and high porosity (75%). On the other hand, the core sample (KZ2) has relatively higher compressive strength (1356kg/cm^2) with low water absorption (67.23) and low porosity (57%).

Table. 5. Compressive strength, water absorption, density and porosity of the collected samples

Sample Code	Compressive strength, (kg/cm^2)	Water absorption, (%)	Density, (g/cm^3)	Porosity, (%)
Top surface layer sample (KZ1)	11.59	78.64	2.738	75
Core sample (KZ2)	13.56	67.23	2.7976	57

Conclusion

In conclusion, one of the most important steps for the restoration and maintenance of cultural heritage is to conduct examinations and analyses to obtain the chemical composition and basic components of stucco decorations. Examination and analysis of falling part samples of the stucco decoration from different places of the Zawiya of Zainal-Din Youssef from the Mamluk era have been performed. The main detected elements by XRF analysis were Ca, S, Si and C. Investigate of the crystal structure and phase analysis by XRD confirmed that the samples are mostly composed of gypsum, anhydrite, calcite and quartz as the main constituents of stucco materials in the Islamic period [15]. The microscopic investigation reveals that the surface structure has clear voids and cracks. In addition, physical investigations such as density, porosity and water absorption were carried out to complete the characterisation profile of the stucco decoration of falling part or deciduous part samples. The surface layer has different physical characteristics from the core samples, which declare the effect of various environmental parameters on the stucco decoration. Through these analyses, experimental simulated samples will be made to conduct various experimental studies on those decorations after obsolescence, in order not to allow direct application of the effect with new materials and routes on the monuments.

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