www.maajournal.com

Mediterranean Archaeology and Archaeometry
Vol. 20, No 2, (2020), pp. 1-16
Open Access. Online & Print.

**DOI: 10.5281/zenodo.3746944**

# MULTISCIENTIFIC-BASED APPROACH TO DIAGNOSIS AND CHARACTERIZATION OF HISTORIC STONE-MASONRY WALLS: THE MAUSOLEUM OF AL-IMAM AL-SHAFI'I, CAIRO (EGYPT)

**Osama Amer[1], Yaser Abdel-Aty[1], Mohamed Abd El-Hady[1], Danila Aita[2], Akram Torky[3], Yehia M. Hussein[4]**

*[1]Conservation Department, Architectural heritage conservation Division, Faculty of Archaeology, Cairo University, Egypt*
*[2]Department of Civil and Industrial Engineering - University of Pisa, Italy*
*[3]Department of Civil Engineering, Faculty of Engineering – Cairo University*
*[4]Concrete Construction Research Institute - Housing and Building National Research Center*

**Received: 16/11/2019**
**Accepted: 10/04/2020**
**Corresponding author: Osama Amer (Osama_amer@cu.edu.eg)*

## ABSTRACT

The purpose of this research is to carry out a scientific-based characterization of one of the most important historic masonry building in Egypt, the mausoleum of al-Imam al-Shafi'I, which is considered one of the few buildings still surviving dated to Ayyubid period (13th century). The study aims at determining not only the characteristics of the building materials' components, their relative proportions and morphological features, but also the causes of damage, decay and alteration phenomena occurred, in order to relate the structural and mechanical behavior of the construction materials depending on their compositional and microstructural nature and finally to set-up proper restorations protocols. Comprehensive studies have been implemented on stone masonry walls through an interdisciplinary procedure including in-situ investigations, and experimental testing on extracted core-samples together with the updated measurements and documentations of the current state of the mausoleum. The followed integrated diagnostic approach based upon field survey data allowed the identification of the mineralogical characteristics, fabric morphology, physical and mechanical properties of the construction materials including limestone and lime-based mortars of the inner and outer leaves of the stone-masonry walls, which give very important information for conservation and restoration strategies. Methods employed included X-Ray diffraction (XRD), scanning electron microscopy (SEM-EDX), petrographic microscopy and thin sections.

**KEYWORDS**: Architectural heritage; Stone-masonry walls; Diagnosis; Material characteristics; Conservation

## 1. INTRODUCTION

Masonry is one of the primitive building materials being used all over the world in the purposes of construction since the beginning of earliest civilizations due to the simplicity of building technique and the features that characterize these materials (Drysdale, Hamid, & Baker, 1999). In addition to these advantages, masonry materials have been used in the construction of the most long-lasting exciting ancient monuments and present in the most impressive historical structures as an evidence of spirit of enterprise of ancient cultures (Vasconcelos, Lourenco, Alves & Pamplona, 2008).

Throughout ancient times, historic masonry walls were commonly used for retaining earth, fortifying communities, and enclosing buildings (Drysdale, Hamid, & Baker, 1999). They were erected by the time-honored method of trial and error. The traditional methods and rules-of-thumb were passed, sometimes in secrecy, from one generation to another. Without using mathematical or predictive methods, but depending on experience and great skills they gained, an impressive empirical wisdom was obtained (Giamundo, 2014). Each geographical region and period in history has had its own characteristic way of building walls (Feilden, 2003). Therefore, each type of wall has different characteristics and behaviour that depend upon its construction technique, typology and the strength of the primary and secondary materials.

Regarding the building materials used in the construction of masonry walls, a general classification can be made between stone, brick, adobe and rubble masonry (Mazzon, 2010; Kržan, 2015). Furthermore, under each item, a second order of classification considers the assembling modality of these structural materials (Mazzon, 2010), which mainly determines its structural behaviour (Kržan, 2015).

The preservation and conservation of cultural architectural heritage, through active and integrated interventions, is nowadays seen as a cultural matter; since historic masonry buildings of various types constructed in different eras provide a significant world architectural heritage. Consequently, a great number of scientific researches in the structural engineering fields (especially structural analysis and testing works), have been carried out to understand the structural behavior of unreinforced masonry (URM) structures in general, and historical masonry buildings in particular (Lourenço, Milani, Tralli, & Zucchini, 2007; Agüera, Tornello, & Frau, 2016; Bennati, et al., 2020; Zade, Sarkar, & Davis, 2020), as the interpretation of the mechanical behaviour of these structures needs an accurate characterization of their construction materials. Additionally, since there is a reasoned tendency upon the preservation and further use of such buildings, the availability of knowledge, building techniques, precise documentation of dimensions and elevations, analysis of structural stability, and characterization of materials (Brosnan, 2014) are required for their assessment, conservation and providing guidelines for their safe use. Structural evaluation of buildings has been reported elsewhere (Yaser Abdel-Aty 2019), as well as, on stone conservation techniques and strategies (Elhagrassy et al., 2018; Abd El-Hakim et al., 2019; Sanfilippo and Aquilia, 2018; El-Gohary 2010).

As it is well known, the conservation and restoration works of architectural heritage require an interdisciplinary approach based on scientific assessment of the current condition. Comprehensive experimental study should be performed in order to determine material characteristics. Not only basic esthetical, physical and mechanical compatibilities, but also the mineralogical and chemical compatibilities must be investigated for the characterization works to judge overall performance (Ulukaya et al., 2017; Thomas et al., 2013). Therefore, a detailed diagnosis and construction material characterization are considered an initial demand. The preliminary evaluation and diagnostic phase are mainly a data acquisition phase. Analysis of these data is essential in determining and establishing the methodology (i.e. deciding and determining the procedures, techniques, materials and details of required work) for the conservation of the studied historical building. Consequently, the investigation of the masonry characteristics (composition, mineralogical characteristics, mechanical and physical properties, etc.) is very useful for the assessment of its structural behaviour and also for providing the most proper conservation and restoration strategies (Abdel-Aty, 2004). From this point of view, our objective is a robust implementation of characterization of the main building materials used in the construction of one of the most important historic masonry building in Egypt, the mausoleum of al-Imam al-Shafi'I. Testing plan and methodology were described comprising integrated approaches for providing a characterization of micro structures and the mechanical properties of main construction materials; describing the test results and determining the values of the basic mechanical properties of the building materials.

### 1.1. *RESEARCH AIMS AND SIGNIFICANCE*

This paper presents a scientific integrated methodology for the assessment and material diagnostic procedures of a complex historic building which combines field survey, microstructure analysis of the brick and stone-masonry walls and materials characterization along with historic information. The combination of results obtained from different approaches produces more precise information and allows the identification of construction materials and masonry patterns in the studied building. This information is essential for improving an integrated multidisciplinary assessment strategy to investigate the safety margins and deterioration rates of historic buildings, under their present conditions, against environmental (i.e. weathering) using complementary analysis techniques. The present comprehensive methodology was conducted by utilizing an integrated study aiming at the selection of the most suitable repairing material and techniques for the intended intervention process.

## 1.2. HISTORICAL BACKGROUND

al-Imam al-Shafi'i mausoleum was chosen as a case study because of its distinctive architectural importance. It is among the very few buildings still existing dating back to Ayyubid period (13th century A. D). In addition to its ideological significance, there are at least two reasons the al-Shafi'i mausoleum is important for our broader understanding of the architecture of medieval popular piety. First, its initial construction by Saladin reflecting the creation of a new ritual center by sparking a general shift northward in cemetery construction. Thus, this building alone transformed the urban landscape of the city of Cairo in a profound and lasting way. The mausoleum of al-Shafi'i was crowned by one of the largest domes in the Islamic world, an issue that is perhaps also connected with the positioning of the cemetery northward (Mulder, 2006).

The history of the building has been studied in the integrity of all its components as a unique architectural product of a specific building technology at its time and place. The historic studies and updated documentations of the present state of the building aim to recognize the original construction techniques, building materials, the deterioration processes that it has suffered through the time and the historical interventions carried out in the past.

The mausoleum of *al-Imam al-Shafi'i* now forms part of an eighteenth-century mosque complex constructed by *Abd al-Rahman Katkhudah* (AD 1762), Figure 1. It is one of the largest mausoleums in the Muslim world and its height of 29 meters is almost that of the Dome of the Rock in *Jerusalem* (Yeomans, 2006). The mausoleum is mainly composed of almost square plane whose inner width measures about 15.3m (Figure 2). It has a circular mihrab in the front centre, proceeded by a recess with two mihrabs (prayer niches), one on each side, which are also preceded by a recess (Figure 3). The three mihrabs are only symbolic and ornamental (OICC, 1992). They are lined with polychromed marble frames and their peaks are adorned with wooden carvings. Sultan *Qaytbay* (14th century) added a fourth mihrab in the eastern corner of the same wall to correct the direction of the qibla. The dome covering the mausoleum is considered one of the most palatial timber domes remaining in Egypt (Figure 3). Despite its exposure to many deterioration factors from the surrounding environment, it is still remaining in a good condition. Its overall height is about 29 meters over the floor of the mausoleum. Three rows of muqarnas squinches were formed at the four corners of the square ceiling as a transition zone from the square to the dome drum.

***Figure 1. Old photos of al-Imam al-Shafi'i mausoleum, R.N: 121 (Mulder, 2006; SCA-E; Behrens-Abouseif, 1985; Creswell, 1978), respectively.***

Considering the masonry walls of the mausoleum, the walls are about 2.75m (av.) thick and about 20m in height from the ground level. The lower part of the masonry walls consists of massive stone walls (Multiple-leave stone masonry walls, Figure 4), which support only the wooden dome which is covered with lead sheets. The upper part of the walls was constructed using brick-masonry.

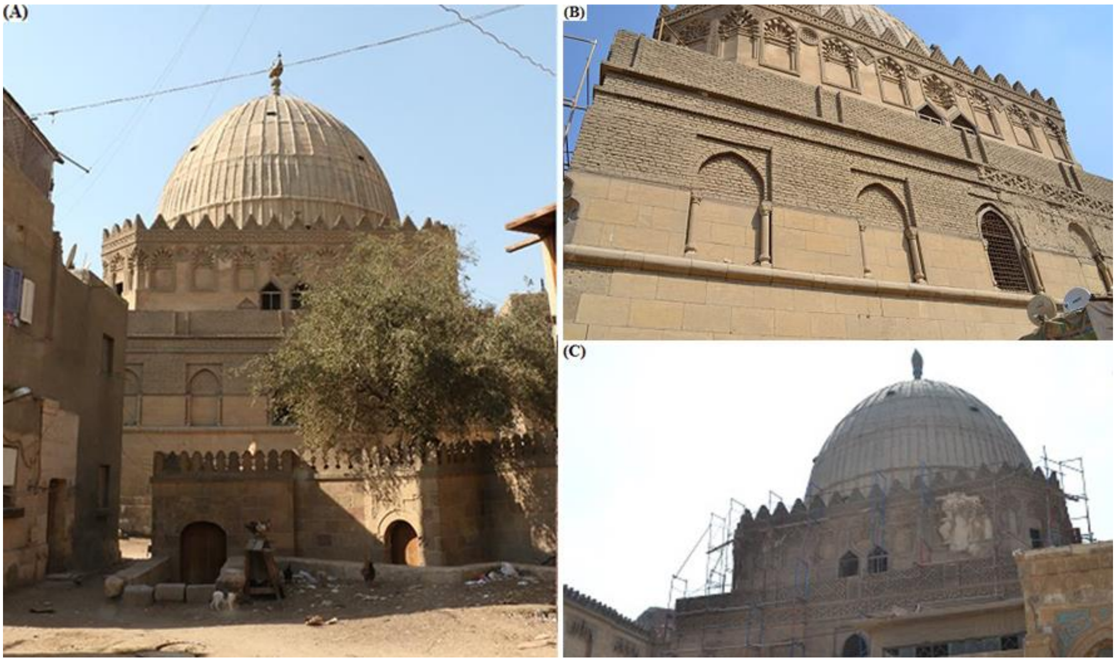


*Figure 2. The mausoleum al-Imam al-Shafi'i, (A, B) South-eastern, and (C) north-western façades*

## 2. MATERIALS AND METHODS

In order to identify the components of the building materials used in constructing the mausoleum and their physio-chemical and mechanical characteristics, in-site and laboratory testing works were carried out on representative samples of the main construction materials of the mausoleum (i.e. limestone, bricks, mortar at bed joints, and rubble infill), which were extracted from various locations of the building. The conducted investigations on the constitutive materials of the extracted samples aimed at having a detailed characterization of the microstructure and the physical-mechanical properties of the main construction materials, as well as assessing the durability of construction materials for providing recommendations regarding the most appropriate intervention techniques and conservation materials. The interpretation of the results obtained from these tests, in combination with the visual inspection and the architectural survey, were very crucial for the knowledge of the overall behaviour of the structure, for identifying the brick and stone-masonry typologies, and also for the evaluation of the effectiveness of the intended intervention techniques.

The research methodology is divided into: (I) Mineralogical analyses carried out by qualitative and quantitative analysis using XRD and EDX. They were conducted on various specimens of limestone, bricks and lime-based mortars in order to determine their chemical composition, constituting minerals and compounds. (II) Petrographic and morphological examinations using polarizing and environmental scanning electron microscopes in order to investigate the mineralogical composition, interlocking textures and microstructure. (III) Characterization of physical and mechanical properties through laboratory tests conducted on extracted core samples from masonry walls.

### 2.1. SAMPLING

The testing program includes extracting four cylindrical cores from various locations of the mausoleum walls for identifying the physical and mechanical characterization of the building materials used in the multiple-leaf stone-masonry walls. The cores were extracted using rotary cylindrical diamond blade coring machine (Figure 5). Cylindrical specimens with a diameter of 94 - 95 mm and height of 80 – 150 mm were prepared with flat circular surfaces perpendicular to the longitudinal axis of the core, while the physical and mechanical characterization of the brick-masonry were conducted on collected brick and mortar samples. Moreover, a set of twelve representative samples of limestone, bricks, mortar and core-infill (derived from the investigated multiple-leaf stone masonry walls) were collected for mineralogical analyses, petrographic and morphological examination.

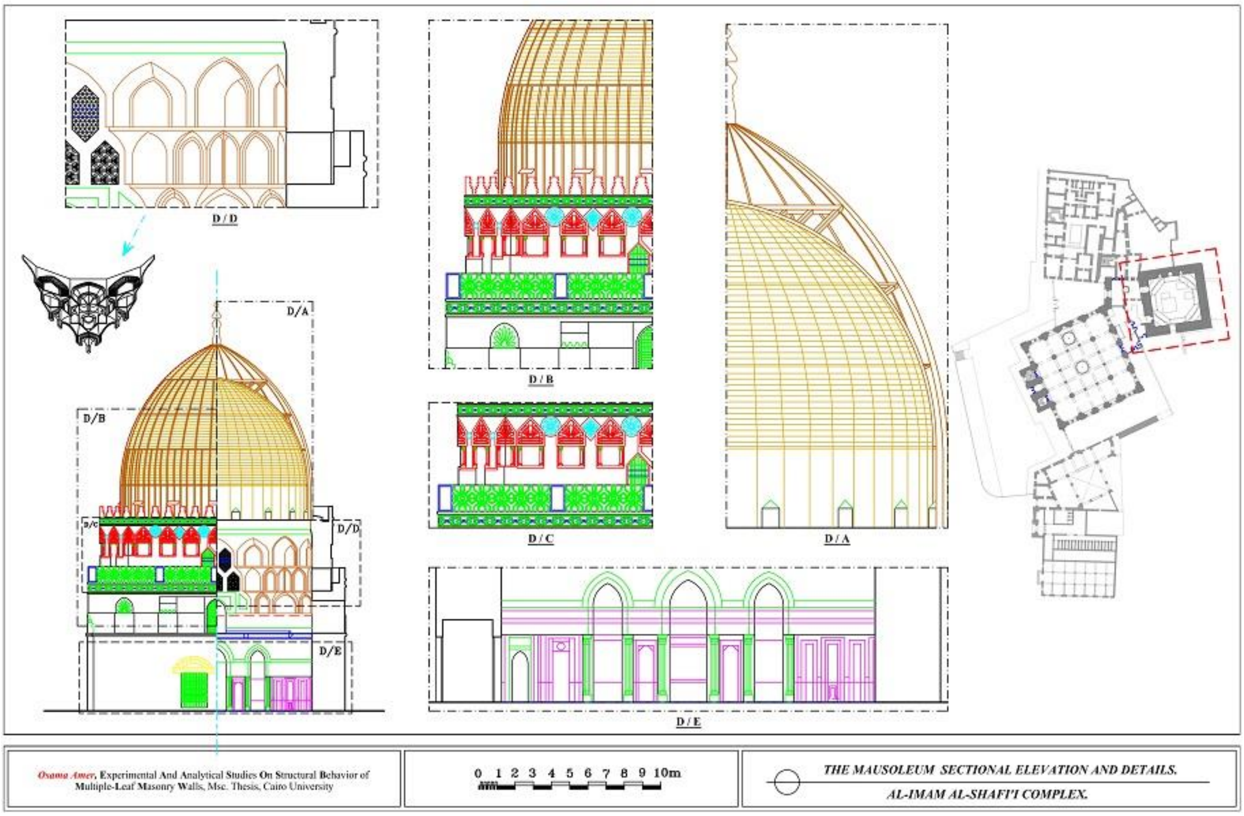


***Figure 3. Sectional elevation and details of the mausoleum***

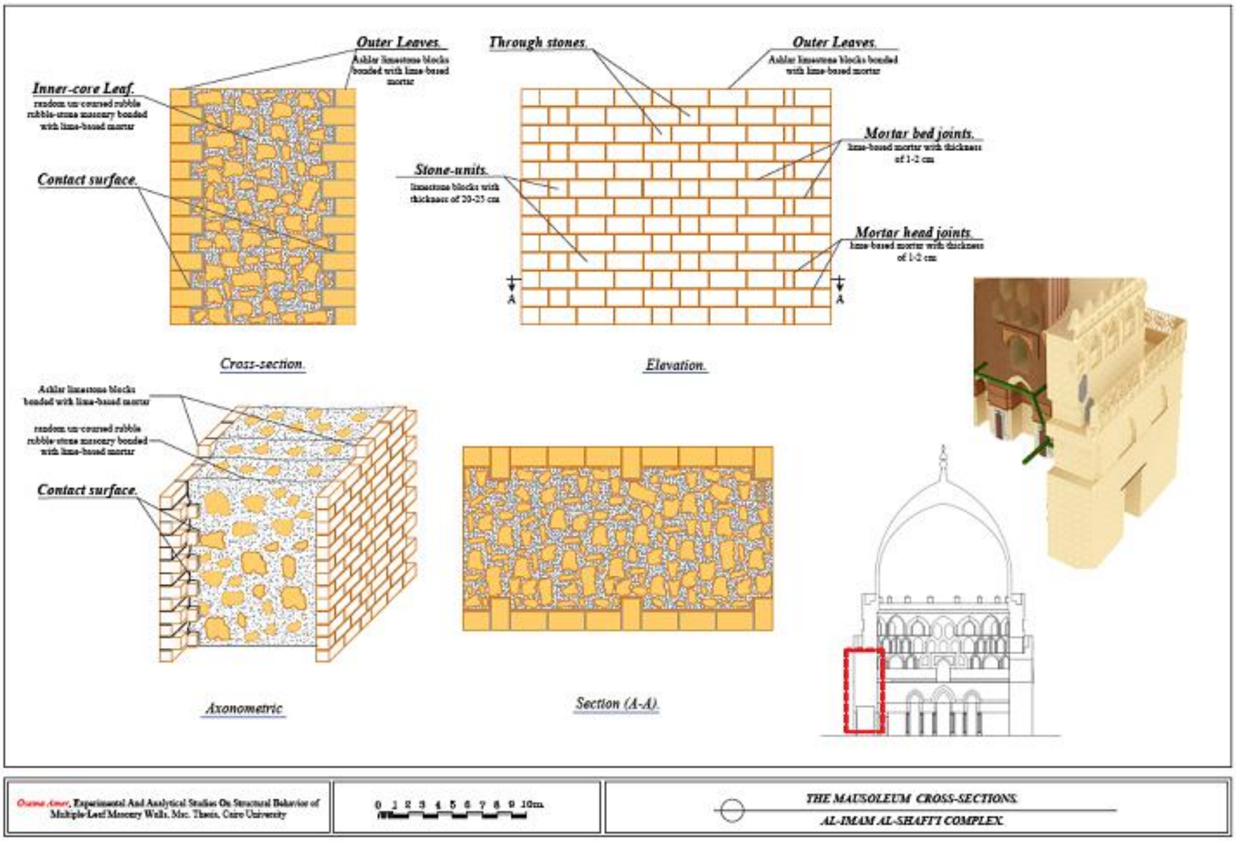


***Figure 4. Details of the multiple-leaf rubble stone masonry walls at the lower level of the mausoleum***

## 2.2. CHARACTERIZATION TECHNIQUES

### 2.2.1. Qualitative and quantitative analysis using XRD and EDX

The basic principles of quantitative analysis using X–ray diffraction technique (XRD) have been described in detail by (Nuffield, 1966; Warren, 1969; Suryanarayana & Grant Norton, 1998; Williams, May, & Guinier, 1999; Waseda, Matsubara, & Shinoda, 2011) among others.

Minerals composition of the collected samples were inducted by X-ray diffraction patterns, using PANanalytical X-Ray Diffraction equipment model X′Pert PRO with Secondary Monochromator. The examined samples were prepared for analysis and dried at 110˚ C and ground to less than 75mm. The analysis was run using Ni-filter and Cu-Kα radiation (λ=1.542Å) at 45 K.V., 35 M.A. and scanning speed 0.02˚ (2θ)/sec. The diffraction peaks between 2θ =0˚ and 60˚, corresponding spacing (d, Å) and relative intensities (I/I˚) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files.

EDS (energy dispersive X-ray spectrometry) attached to scanning electron microscopy (SEM) was used to conduct the quantitative analysis of the collected samples. Energy dispersive X-ray analysis (EDS) was used to determine the elemental composition where necessary, which proved complementary information to support the previous techniques employed. The analysis was performed on uncoated samples to avoid overlap of gold peaks with beaks of interest, using a higher accelerating voltage of 20 KV and a large spot size than the imaging. EDS spectra were recorded in the spot-profile mode by focusing the electron beam onto specific regions of the sample. EDS spectra were obtained between 0 to 10KeV.

*Figure 5. Extracting of the investigated core samples*

### 2.2.2. Microstructure and morphological examinations

Scanning electron microscopy (SEM) was used to identify the structural morphology and microstructure of the collected brick, stone and mortar samples, and to determine their forming minerals, and observe the weathering status. SEM images were taken using a SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDS Unit (energy dispersive X-ray spectrometry), with accelerating voltage 30K.V, magnification 14x up to 1,000,000x and resolution for Gun.1n.

### 2.2.3. Petrographic investigations

The petrographic investigation of stone and mortar samples was conducted for the determination of the mineral content, grain size, micro fracturing and interlocking texture using polarizing petrographic microscope on thin sections of the collected samples. Air–dried samples (at ~ 40ºC to avoid dehydration of components, especially Gypsum if present, and physical damage due to thermal shock) were subjected to impregnation with warmed low viscosity colour dyed epoxy resin, to aid in the visualization of pores, cracks and air voids. Very thin slices of collected samples (cut perpendicular to the bedding planes) were mounted on clear, flat glass slides. The reduction of thickness (commonly to 20-30/am) permits light to pass through crystalline or amorphous materials and for the detailed analysis and recognition of the stone and mortar's components. Morphological examination of the prepared thin sections of the stone and mortar samples was carried out using polarized transmitted light microscopy model NIKON OPTI PHOTO $x_{23}$ equipped with photo camera S23, under cross-polarized light XPL.

#### 2.2.4. Physical properties

The physical properties of the stone, mortar, and core-infill specimens, including porosity ($\eta$), dry density ($\rho_d$), bulk density ($\rho_{bulk}$), water absorption (*WA*) were determined in accordance with the procedures outlined in ISRM suggested methods (ISRM, 1981a). Oven dried specimens were dipped in deionized water and weighed constantly at prefixed intervals of time until a constant weight was attained; and its saturated-surface-dry mass ($M_{sat}$) was also determined.

#### 2.2.5. Mechanical tests

The mechanical tests were conducted on the extracted core samples derived from the investigated multiple-leaf stone masonry walls aiming to provide reliable and statistically representative data for the mechanical behavior of this type of walls. Uniaxial compression tests and indirect tension (splitting) tests were carried out on the dry and saturated specimens. A uniaxial testing machine with a hydraulic actuator was used. Uniaxial compressive tests were carried out according to (ISRM, 1981c; CEN, 2000; BS EN 772-1, 2011). Indirect tensile test (splitting tension, Brazilian test) was adopted in this work for the characterization of the tensile behavior of the extracted samples in accordance with (ISRM, 1981b; RILEM, 1994).

## 3. RESULTS AND DISCUSSION

### *3.1. Mineralogical analyses*

#### 3.1.1. Stone samples

According to the obtained analysis results, limestone is mainly composed of Calcite ($CaCO_3$) of about 95%. Sometimes Dolomite [Ca Mg $(CO_3)_2$] is found in a very low percentage. The detached gypsum ($CaSO_4{\cdot}2H_2O$) in limestone samples indicates a salt decay in a certain amount proportion to its percentage. Gypsum is commonly created from the reaction of Calcite ($CaCO_3$) with the sulphuric acid ($H_2SO_4$) that may result from the polluted air containing Sulphur dioxide ($SO_2$) and trioxides ($SO_3$). Additionally, the presence of Halite (NaCl) in limestone samples indicates salt weathering of limestone by chloride salts. Ferric oxide, Hematite ($Fe_2O_3$), is also found in limestone samples as a trace mineral (Table 1).

#### 3.1.2. Mortar samples

The obtained results from XRD analysis of mortar samples confirmed that, the mortar used in the stone-masonry walls is lime-based mortar; as it is mainly composed of lime as the major binders with sand as the aggregate and some additives such as red-brick powder (i.e. Hommra) or fly ash (i.e. Qusrmil) as pozzolanic materials. According to XRD results, the major component of mortar samples is Calcite ($CaCO_3$) that indicates the binder used is lime which is formed by the reaction of hydrated lime Ca $(OH)_2$ with carbon dioxide ($CO_2$) from the air. Quartz ($SiO_2$) in mortar samples indicates the use of sand as the aggregate. The presence of Plagioclase ($CaAl_2Si_2O_8$), as a clay mineral, in mortar samples indicates the use of Hommra as an additive for enhancing the setting and final strength of the lime-based mortar. Gypsum ($CaSO_4{\cdot}2H_2O$) in mortar samples may indicates a salt decay or it could be added to the mortar mixture with a small proportion to improve its strength (Table 2).

Figure 6, 7 present the results of the XRD analysis for limestone and mortar samples derived from the inner-core layer of the investigated multiple-leaf stone-masonry walls.

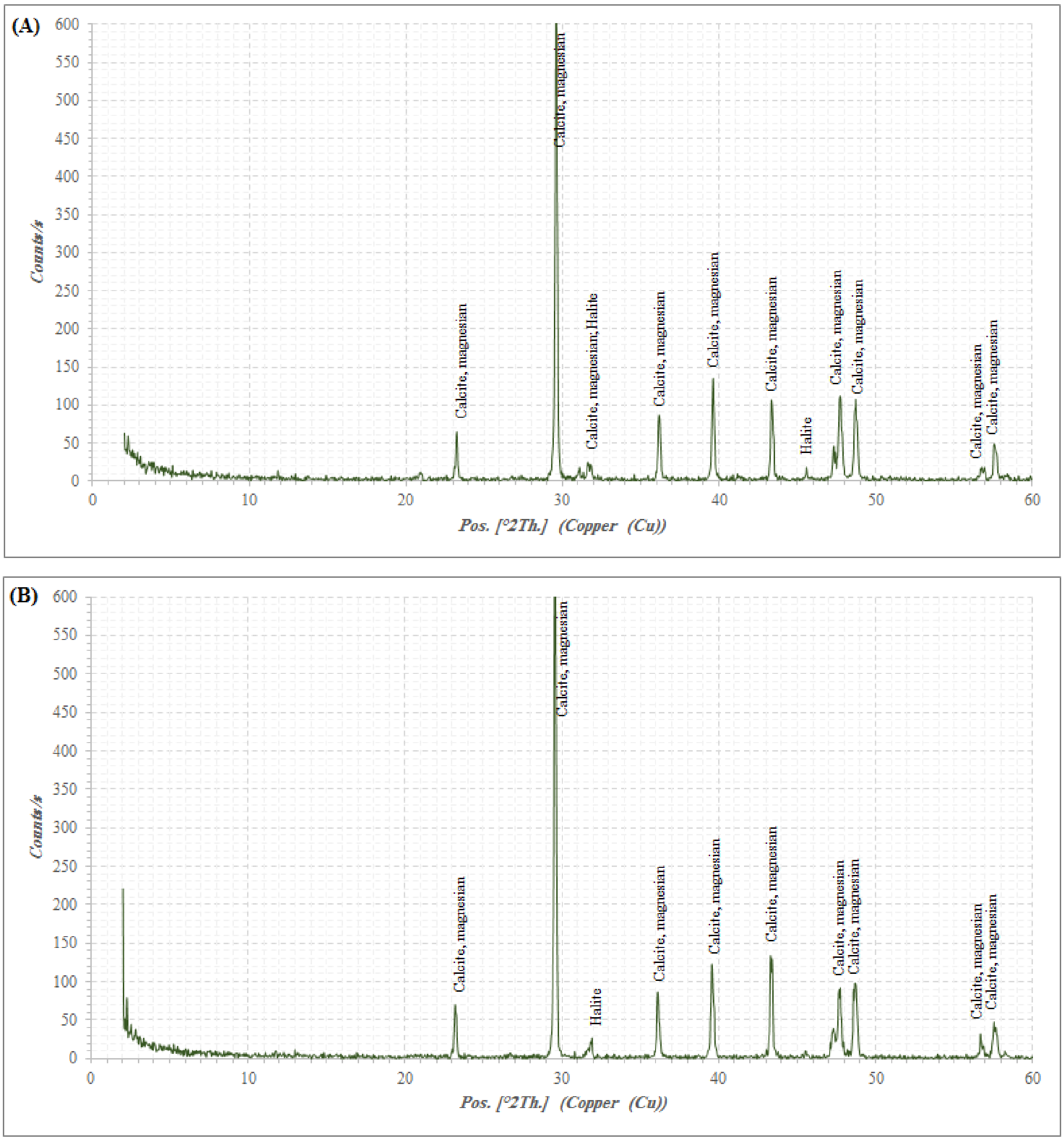


*Figure 6. X-ray diffraction pattern of representative limestone sample extracted from: (A) the external layer, $S_1$; (B) the inner-core layer, $S_2$*

## 3.2. *Petrographic and morphological examinations*

### 3.2.1. Stone samples

Limestone samples were collected from the external layer of the multiple-leaf rubble stone-masonry walls of the mausoleum, $S_1$. Generally, the samples are very fine-grained and composed of carbonate minerals, most probably calcite as a major component with minor amount of dolomite, quartz, gypsum, and iron oxides. Carbonate minerals occur as very fine-grained and represent the whole matrix of the stone. Some microfossils and shells of different shapes and sizes are scattered in very fine-grained matrix of carbonate minerals and filled with recrystallized carbonate minerals. Quartz is presented of minute grains and scattered in the carbonate matrix. The stone is relatively porous, and various micro fracturing are detected.

On the other hand, the limestone samples obtained from the inner layer of the multiple-leaf rubble stone-masonry walls, $S_2$, were very fine-grained and composed essentially of carbonate minerals, most probably calcite. The carbonate minerals are associated with minor amount of dolomite, rare amount of quartz, and iron oxides. Carbonate minerals are very fine-grained, representing the whole matrix of limestone enclosing other constituents. A considerable amount of microfossils and shells of different shapes and sizes are scattered in the very fine-grained matrix of carbonate minerals and filled with recrystallized carbonate minerals. Additionally, calcium sulphates and sodium chloride salts were detached in different concentrations and crystalline forms which play a dominant role in the deterioration of limestone samples. The stone is porous and some pore spaces are clearly detected. The interlocking texture and microstructure of the stone samples are shown in Figure 8.

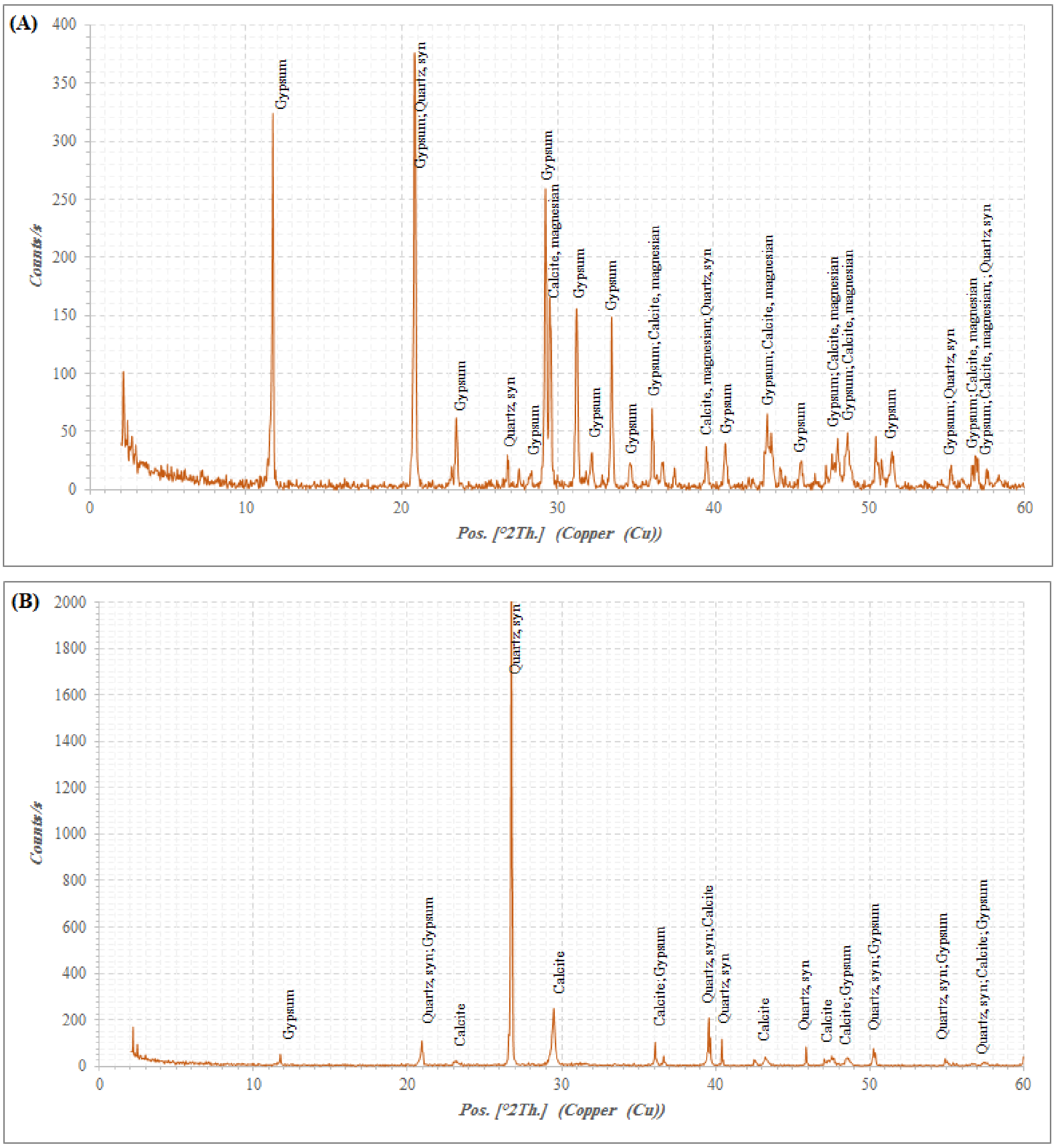


*Figure 7. X-ray diffraction pattern of representative limestone sample extracted from: (A) the bed joints of the external layer, $M_1$; (B) the inner-core layer, $M_2$*

### 3.2.2. Mortar samples

Sample $M_1$ represents mortar sample derived from the inner-core layer of the northeastern multiple-leaf rubble stone-masonry wall of the mausoleum. The sample varies from very fine to fine-grained and is composed mainly of gypsum as the major constituent in addition to minor amount of quartz, carbonate minerals, opaque minerals, clay minerals and iron oxides. Gypsum occurs as very fine to fine-grained minerals and fibrous aggregates representing the essential component of the sample. Quartz (sand grains) occurs as medium to fine-grained and has sub-rounded to sub-angular outlines scattered in the sample. Opaque minerals present as fine-grained of rounded to sub-rounded outlines scattered in the sample. Some microfossils and shells are observed. Carbonates present as very fine-grained and admixed with gypsum. The sample is stained by iron oxides and clay minerals in different parts. Pore spaces are detected as irregular cavities scattered in the sample.

*Table 1. Average composition of the minerals for the representative limestone samples.*

| Sample ID | Minerals | Chemical Formula | Semi-Quant [%] |
|---|---|---|---|
| | Calcite, magnesian | $(Mg_{.064} Ca_{.936}) (C O_3)$ | 91 |
| $S_1$ | Halite | NaCl | 5 |
| | Gypsum | $CaSO_4 (H_2O)_2$ | 4 |
| $S_2$ | Calcite, magnesian | $(Mg_{.064} Ca_{.936}) (C O_3)$ | 96 |
| | Halite | NaCl | 4 |

*Table 2. Average composition of the minerals for the representative mortar samples.*

| Sample ID | Minerals | Chemical Formula | Semi-Quant [%] |
|---|---|---|---|
| | Gypsum | $CaSO_4 (H2O)_2$ | 67 |
| $M_1$ | Calcite, magnesian | $(Mg_{.064} Ca_{.936}) (C O_3)$ | 25 |
| | Quartz, syn | $SiO_2$ | 8 |
| | Quartz, syn | $SiO_2$ | 63 |
| $M_2$ | Gypsum | $CaSO_4 (H2O)_2$ | 21 |
| | Calcite | $CaCO_3$ | 16 |

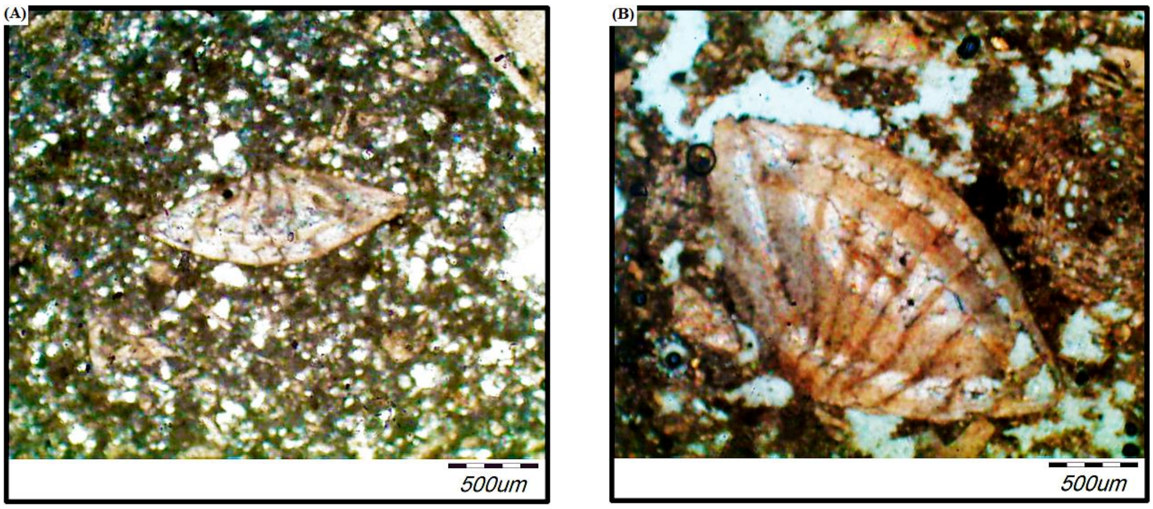


*Figure 8. Photomicrograph of limestone samples, under cross-polarized light (XPL) (A) Sample $S_1$, (B) Sample $S_2$*

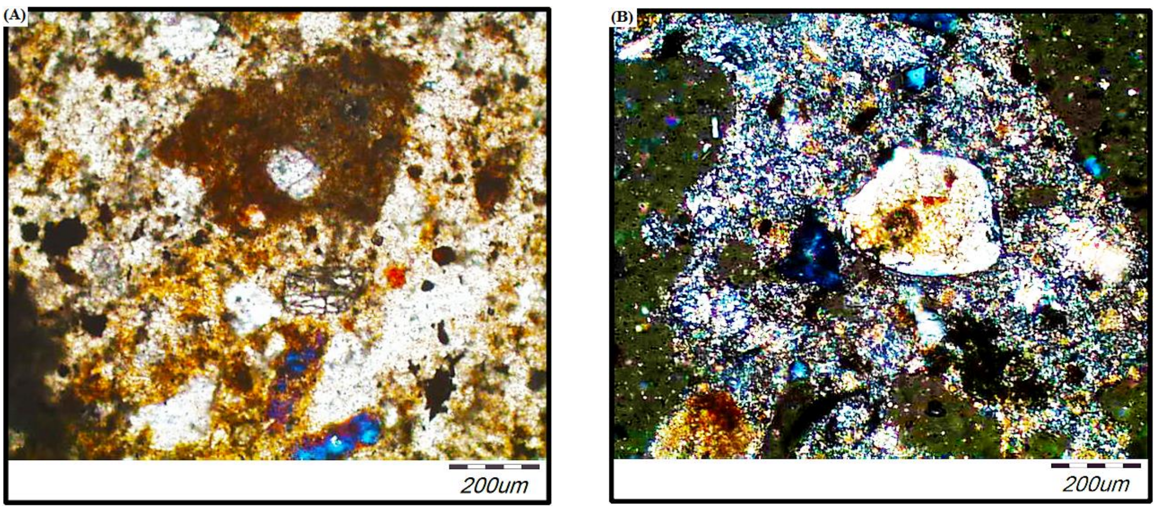


*Figure 9. Photomicrograph of lime-based mortar, under cross-polarized light (XPL) (A) Sample $M_1$, (B) Sample $M_2$*

Sample $M_2$ represents mortar collected from the external layer of the northwestern multiple-leaf rubble stone-masonry wall of the mausoleum. The sample is very fine to fine-grained and composed mainly of carbonate minerals, gypsum and quartz, with minor amounts of iron oxides. Some microfossils and shells are observed in the sample. Cement material is mainly composed of a mixture of carbonates. Carbonate minerals occur as very fine-grained aggregates scattered in the matrix of the sample. Quartz is fine-grained which has sub-rounded to sub-angular edges and is scattered in matrix of carbonates. Iron oxides present as very fine-grained aggregates and staining different parts in the sample. Some pore spaces are detected in the mortar. The interlocking texture, identified minerals and microstructure of the mortar sample are shown in Figure 9 under cross-polarized light (XPL), while Figure 10 and Figure 11 illustrate the morphological examinations and EDX microanalyses of thin section of limestone sample.

The morphological examinations of mortar samples (Figure 12 and 13) show the crystalline formation and identified mineral phases of mortar components, mainly calcite crystals, lime, quartz, and few gypsum formations. In the case of mortar samples extracted from the inner-core layer, organic fibers are present as additives in the mortar mixture (i.e. chopped straw or wood fibres, see Figure 13).

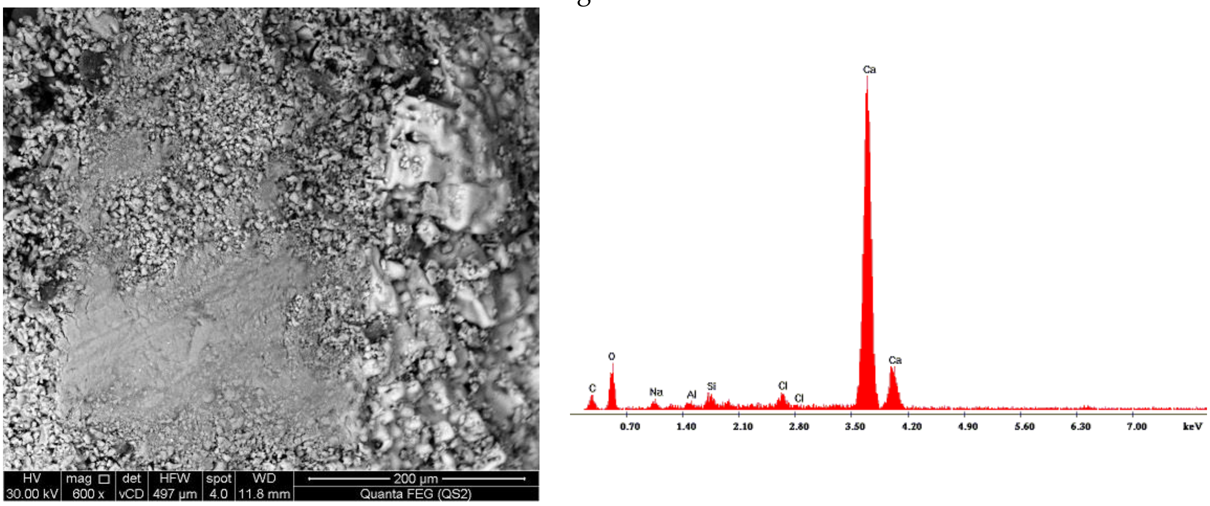


*Figure 10. SEM photomicrographs and EDX microanalysis of thin section of limestone sample ($QS_1$) derived from the external layer of the investigated multiple-leaf masonry walls of the mausoleum shows that calcite is the main constituent of the stone with a minor amount of fine rounded quartz crystals. Quartz grains are very fine and scattered in the stone, inhomogeneous distribution of gaps through the stone texture were noticed*

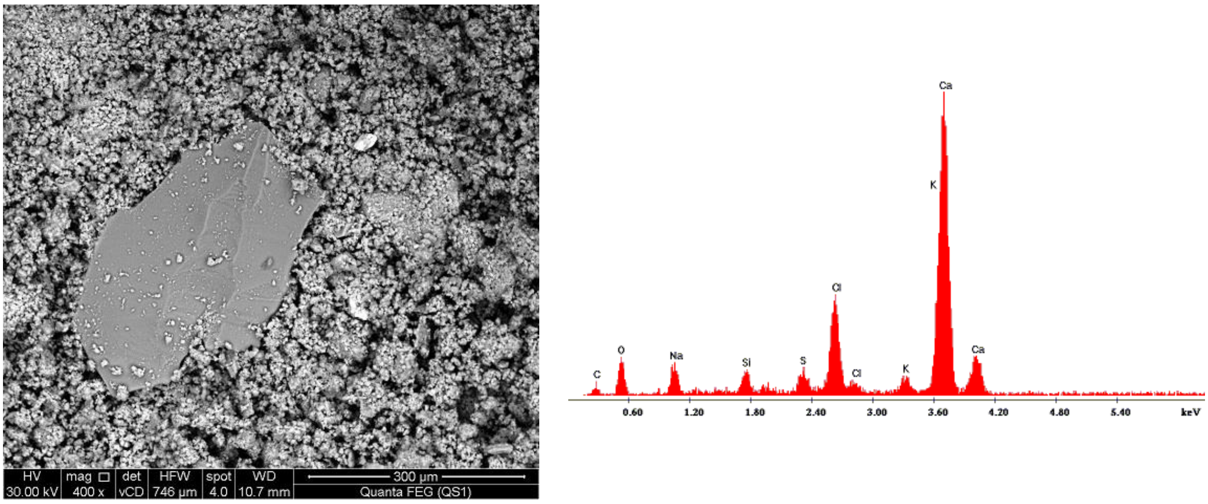


*Figure 11. SEM photomicrographs and EDX microanalysis of thin section of limestone sample ($QS_2$) derived from the inner-core layer of the investigated multiple-leaf masonry walls of the mausoleum shows that calcite is the main constituent of the stone with minor amounts of gypsum and fine rounded quartz crystals. Gypsum and quartz grains are very fine and scattered in the stone; additionally, inhomogeneous distribution of gaps and crystalline salts (Halite, NaCl) through the stone were noticed*

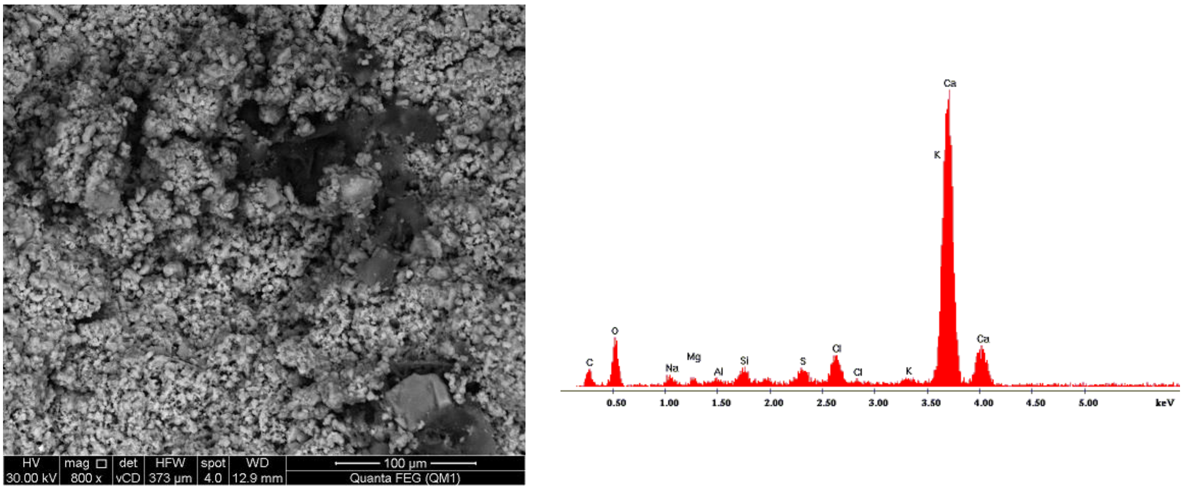


*Figure 12. SEM photomicrographs and EDX microanalysis of thin section of lime-based mortar sample ($QM_1$) derived from the external layer of the investigated multiple-leaf masonry walls of the mausoleum shows that the dominant constituent is calcite with fine rounded quartz crystals; decomposition of calcite and disintegration of the surface were noticed through the sample; it may be related to the surrounding deterioration factors*

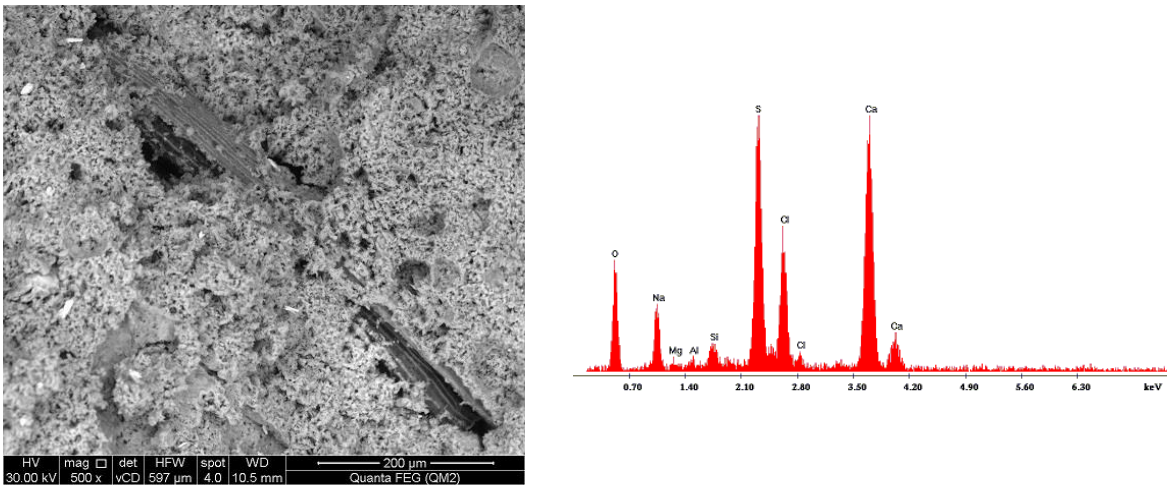


*Figure 13. SEM photomicrographs and EDX microanalysis of thin section of lime-based mortar sample ($QM_2$) derived from the inner-core layer of the investigated multiple-leaf masonry walls of the mausoleum shows that calcite and gypsum with fine rounded quartz crystals are the main constitutes. Organic additives (e.g. chopped straw) were found in the mortar, which were used to strengthen the mortar.*

## 3.3. Characterization of physical and mechanical properties

Physical properties of the core samples derived from the investigated multiple-leaf stone-masonry walls are shown in the Table 3, which provides porosity, $\eta$, bulk density, $\rho_{bulk}$, water absorption, *WA*, of samples of limestone, mortar and rubble-infill specimens.

Table 4 and Figure 14 describe the mechanical tests conducted on core samples derived from investigated multiple-leaf stone masonry walls. According to the obtained results, the average modified value for the compressive strength of limestone samples, derived from the inner layer, is 16.4 $N/mm^2$. While the average compressive strength of core-infill samples that represent the inner-core layer of the walls is 4.56 $N/mm^2$. Furthermore, the average splitting tensile strength of limestone samples, derived from the inner layer, is 1.49 $N/mm^2$.

***Table 3. Physical properties for the extracted core specimens from the investigated walls.***

| Core samples | $\rho_{Bulk}$(gr/cm³) | *WA%* | *η %* |
|---|---|---|---|
| *S3-14*[1] | 2.52 | 12.12 | 8.12 |
| *S4-15*[1] | 2.39 | 11.11 | 10.45 |
| *S3*[1] | 2.31 | 11.02 | 12.38 |
| *S4*[2] | 1.31 | 18.18 | 13.01 |
| *S1*[3] | 1.94 | 57.14 | 22.77 |
| *S3-M*[3] | 1.96 | 25.00 | 16.24 |
| *S4-M*[3] | 1.78 | 60.00 | 18.42 |

[1] Specimens represent limestone samples from outer and core layer
[2] Specimens represent core infill samples
[3] Specimen represents lime-based mortar samples

***Table 4. Results of the mechanical tests for the extracted core specimens from the investigated walls.***

| Core samples | Dimensions (cm) | | | Failure Load | Strength |
|---|---|---|---|---|---|
| | *D* | $h_{max.}$ | $h_{min.}$ | *KN* | *N/mm²* |
| *S1-10*[1] | 9.4 | 13.4 | 13.3 | 39.2266 | 5.64 |
| *S2-20*[1] | 9.4 | 12.3 | 12.2 | 27.4586 | 3.95 |
| *S4-10*[2] | 9.4 | 9.2 | 9.0 | 28.4393 | 4.09 |
| *S3-14*[2] | 9.4 | 8.1 | 8.08 | 106.124 | 15.29 |
| *S4-5*[3] | 9.4 | 9.8 | 9.75 | 121.602 | 17.52 |
| Av. value [1] | | | | | 4.56 |
| Av. value [2] | | | | | 16.40 |
| *S4-15*[4] | 9.4 | 9.5 | 9.38 | 69.39 | 1.49 |

[1] Specimens represent core Layer, core infill samples
[2] Specimens represent outer Layer and limestone core samples used in the inner-core layer
[3] Specimen represents saturated limestone core samples
[4] Brazilian test of core Layer specimen


***Figure 14. Mechanical tests conducted on the cylindrical core specimens extracted from various locations of the investigated multiple-leaves stone-masonry walls of the mausoleum; (A, B) compression tests of rubble-infill and cored stone specimens, respectively; (E) indirect tension test on cored stone specimens; (D, F and G) examples of compression failure of the investigated specimens***

## 4. CONCLUSION

The microstructure and morphological examinations provide qualitative information about orientation and size of particles, pores and cracks. Aggregates were identified using a petrographic investigation coupled with XRD Analyses. SEM coupled with EDS allows a qualitative determination of the chemical elements of the investigated sample. X-ray diffraction (XRD) analysis characterized crystalline or semi crystalline phases of the materials composition and identified the binder (lime, gypsum, cement, etc.), the aggregate (siliceous, calcareous, etc.) and other additions. Moreover, the organic additions were determined by SEM examination.

The conducted petrographic examinations, using polarizing petrographic microscope on thin sections of the collected stone and mortar samples, proved that the limestone samples mainly consist of very fine-grained and composed of carbonate minerals, most probably calcite as a major component with minor amount of dolomite, quartz, gypsum, and iron oxides. On the other hand, the lime-based mortar samples mainly consist of carbonate minerals, gypsum, quartz, minor amounts of iron oxides and clay minerals.

Regarding the conducted XRD Analyses on the collected limestone and lime-based mortar samples, the limestone is mainly composed of either Calcite ($CaCO_3$), or Calcite-magnesian ($Mg_{.064}$ $Ca_{.936}$) (C $O_3$). The detected Gypsum ($CaSO_4{\cdot}2H_2O$) in limestone samples indicates a salt decay infection in a certain amount proportion to the Calcite percentage. Halite (NaCl) mineral was found in limestone samples, which indicates a salt decay infection by chloride salts. Ferric oxide, Hematite ($Fe_2O_3$), was also found in limestone samples as a trace element. On the other hand, the mortar used in the stone-masonry and brick-masonry walls is lime-based mortar. It is mainly composed of lime as the major binders with sand as the aggregate and some additives such as red-brick powder (i.e. Hommra) or fly ash (i.e. Qusrmil) as pozzolanic materials.

The microstructure and morphological examinations using SEM with EDS conducted on the collected limestone and mortar samples, which derived from the external and inner-core layer of the investigated multiple-leaf stone-masonry walls, showed that:

- The crystal formation for limestone minerals were composed mainly of calcite crystals. Other crystals of calcium sulphates and sodium chloride were appeared, in different concentrations, and indicated the deteriorated condition of limestone samples.
- The morphological examinations of mortar samples showed the crystal formation of mortar components, which were composed mainly of calcite crystals, lime, quartz, and few gypsum formations. In some cases, organic fibers appear as additives to mortar mixture (i.e. straw or wood fibers).

Concerning the conducted in-site and laboratory testing investigations, the following conclusions could be summarized:

- The conducted mechanical tests on the collected samples of the main construction materials proved that the average value for the compressive strength of limestone samples derived from the inner layer is about 16.4 N/mm$^2$, while the average compressive strength of core-infill samples that represent the inner-core layer of the walls is about 4.56 N/mm$^2$. Furthermore, the average splitting tensile strength of limestone samples derived from the inner layer is about 1.49 N/mm$^2$.
- Physical tests of the investigated core-samples derived from the investigated multiple-leaf stone-masonry walls proved that the average water absorption, *WA*, of the limestone samples is about 11.42%, and the average *WA* of mortar samples and core-infill samples are 47.38% and 18.18%, respectively. The average bulk density, $\rho_{bulk}$, of limestone, mortar and core infill samples are 2.41, 1.31, 1.89 g/cm$^3$, respectively, while the average porosity, $\eta$, of limestone, mortar and core infill samples are 10.31, 13.01, and 19.12%, respectively.

## ACKNOWLEDGMENTS

The authors would like to thank the financial support from Cairo University. Moreover, the authors are grateful to Prof. Dr. May Al-Ibrashy and Prof. Dr. Osama Talat for the on-site collaboration and Prof. Dr. Bassam Saad for the collaboration in laboratory testing.

## REFERENCES

Abdel-Aty, Y. Y. (2004). Analysis and assessment of structural deficiencies in historical Islamic religious buildings from BahriMamluk period and the possible scientific methods for conservation and restoration with application on the Madrasa of Umm Al-sultan Sha'ban in Cairo. Ph.D. thesis, Cairo University, Faculty of Archaeology, Conservation Dept.

Abeer F. Elhagrassy and Amira Hakeem (2018) Comparative study of biological cleaning and laser techniques for conservation of weathered stone in Failaka island, Kuwait. *SCIENTIFIC CULTURE*, Vol. 4, No 2, pp. 43-50 DOI: 10.5281/zenodo.1214561.

Abd El-Hakim A. El-Badry, Mona F.Ali, Badawi M.Ismail (2019) Assessment of the aging treatments of sandstone greywacke rock art (Wadi Hammamat) by petrography, SEM, XRD, EDX . *SCIENTIFIC CULTURE*, Vol. 5, No. 2, pp. 37-48. DOI: 10.5281/zenodo.2649505.

Agüera, N. D., Tornello, M. E., & Frau, C. D. (2016). Structural Response of Unreinforced Masonry Walls. *Journal of Civil Engineering and Architecture,* 10, 219-231. doi:10.17265/1934-7359/2016.02.011

Behrens-Abouseif, D. (1985). Change in Function and Form of Mamluk Religious Institutions. Annales Islamologiques 21.

Bennati S., Aita D., Barsotti R., Caroti G., Chellini G., Piemonte A., Barsi F., Traverso C. (2020). Survey, experimental tests and mechanical modelling of the dome of Pisa Cathedral: a multidisciplinary study. *International Journal of Masonry Research and Innovation,* 5(1), 142 – 165. doi:10.1504/IJMRI.2020.104850

Brosnan, D. A. (2014). Characterization and Degradation of Masonry Mortar in Historic Brick Structures. *Journal of Structures,* 2014, 1-7. doi: http:/dx.doi.org/10.1155/2014/859879

BS EN 772-1. (2011). Methods of test for masonry units. Determination of compressive strength. BSI. doi: ISBN: 978 0 580 845840

CEN. (2000). Methods of test for masonry units: Determination of compressive strength.

Creswell, K. (1978). The Muslim Architecture of Egypt (Vol. 1). New York: Oxford: Clarendon Press. Hacker Art Books.

Drysdale, R. G., Hamid, A. A., & Baker, L. R. (1999). Masonry structures: behavior and design. (B. M. leon, P. Zurite, & S. Handy, Eds.) Masonry Society.

El Gohary, M (2010) Investigations on limestone weathering of El-Tuba minaret El Mehalla, Egypt: a case study. *Mediterranean Archaeology and Archaeometry*, Vol. 10, No. 1, pp. 61-79

Feilden, B. M. (2003). Conservation of Historic Buildings. Rome: International Centre for the Study of the Preservation and the Restoration of Cultural Property (ICCROM).

Giamundo, V. (2014). Seismic assessment and retrofit of historical masonry barrel vaults. Ph.D. Thesis, University of Naples Federico II.

ISRM. (1981a). ISRM Suggested methods for determining water content, porosity, density, absorption and related properties and swelling and slake-durability index properties, In: Brown E.T. Pergamon, Oxford: ISRM Suggested Methods.

ISRM. (1981b). ISRM Suggested Methods, suggested method for determining tensile strength of rock materials, in: Brown E.T. Pergamon, Oxford: ISRM Suggested Methods.

ISRM. (1981c). ISRM Suggested Methods, suggested method for determining uniaxial compressive strength and deformability of rock materials, in: Brown E.T. Pergamon, Oxford: ISRM Suggested Methods.

Kržan, M. (2015). Performance based experimental and numerical assessment of multi-leaf stone masonry walls. PhD thesis, Ljubljana, Univerza v Ljubljani, Fakulteta za gradbeništvo in geodezijo.

Lourenço, P. B., Milani, G., Tralli, A., & Zucchini, A. (2007). Analysis of Masonry Structures: Review of and Recent Trends of Homogenization Techniques. *Canadian Journal of Civil Engineering*, 34, 1443-57. doi:https://doi.org/10.1139/L07-097

Mazzon, N. (2010). Influence of Grout Injection on the Dynamic Behaviour of Stone Masonry Buildings. PhD Thesis, University of Padova, Italy.

Mulder, S. (2006). The mausoleum of Imam al-Shafi'i. Koninklijke Brill NV., ISBN 978 90 04 15492 6.

Nuffield, E. W. (1966). X-ray diffraction methods. Wiley.

OICC, O. (1992). Principles of architectural design and urban planning during different Islamic eras: analytical study for Cairo city. Jeddah, Saudi Arabia: Markaz al-Dirāsāt al-Takhṭīṭīyah wa-al-Miʻmārīyah, Center for Revival of Islamic Architectural Heritage.

RILEM. (1994). Tension by splitting of concrete specimens.

Sanfilippo, G and Aquilia, E (2018) Multidisciplinary process aimed at the diagnosis and treatment of damages in stony cultural heritage: the balustrade of Villa Cerami (Catania). *Mediterranean Archaeology and Archaeometry*, Vol. 18, No 5, pp. 191-205, DOI: 10.5281/zenodo.1285912

SCA-E. (n.d.). Archive data center of Supreme Council of Antiquities - Egypt.

Suryanarayana, C., & Grant Norton, M. (1998). X-Ray Diffraction: A Practical Approach. New York: Springer Science, Business Media.

Thomas, C., Lombillo, I., Setien, J., Polanco, J., & Villegas, L. (2013). Characterization of materials with repellents and consolidates from a historic building. *Journal of Materials in Civil Engineering*, 25(11), 1742–1751. doi:10.1061/(ASCE)MT.1943-5533.0000747

Ulukaya, S., Afife, Y. B., & Yüzer, N. (2017). Material Characterization of Byzantine Period Brick Masonry Walls Revealed in Istanbul (Turkey). Periodica Polytechnica Civil Engineering, 61(2), 209–215. doi: https://doi.org/10.3311/PPci.8868

Vasconcelos, G., Lourenco, P., Alves, C., & Pamplona, J. (2008). Ultrasonic evaluation of the physical and mechanical properties of granites. Ultrasonics, 48, 453–466. doi:https://doi.org/10.1016/j.ultras.2008.03.008

Warren, B. E. (1969). X-ray Diffraction. Courier Corporation.

Waseda, Y., Matsubara, E., & Shinoda, K. (2011). *X-Ray Diffraction Crystallography: Introduction, Examples and Solved Problems*. Berlin Heidelberg: Springer-Verlag.

Williams, C., May, R., & Guinier, A. (1999). *Characterization og materials*. (E. Lifshin, Ed.) Weinheim: Wiley-VCH.

Yeomans, R. (2006). The art and architecture of Islamic Cairo. (E. Hawker, & A. Hines, Eds.) Garnet Publishing Limited.

Yaser Yehya Amin Abdel-Aty (2019) Structural evaluation of façade projections and their supporting cambered corbels at historical masonry buildings in Cairo from medieval periods with a proposal for strengthening and retrofitting *SCIENTIFIC CULTURE*, Vol. 5, No. 3 pp. 21-33. DOI: 10.5281/zenodo.3340105.

Zade, N. P., Sarkar, P., & Davis, P. R. (2020). Seismic behaviour of unreinforced masonry. In: S. Chakraverty, & P. Biswas (Eds.), Recent Trends in Wave Mechanics and Vibrations. Lecture Notes in Mechanical Engineering (pp. 151-164). Springer, Singapore. doi: https://doi.org/10.1007/978-981-15-0287-3_13.