Restoration and conservation of the archaeological Islamic sandstone tombstones applied on a tombstone from the second Abbasid era in Egypt

علاج وصيانة شواهد القبور الأثرية الإسلامية من الحجر الرملي تطبيقا على شاهد قبر من العصر العباسي الثاني في مصر

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Abstract

The stone Islamic tombstones are one of the most important sources of dating, with their inscriptions in various Arabic calligraphies. This research aims to study Islamic sandstone tombstones with an application to a tombstone from the second Abbasid era in the Islamic history which is dated through the inscriptions on its surface and written in simple Kufic calligraphy, where this tombstone dates back to 272 AH and bears the name of its owner Ibrahim bin Suhaib. Sandstone samples from the tombstone were studied and examined by stereomicroscope, scanning electron microscope (SEM) and analyzed by X-ray diffraction (XRD) and elemental analysis by the EDX unit, It was found through examination by microscopes that there is a weakness in the sandstone, it was noted through the analysis by X-ray diffraction that the main compound is silicon dioxide (SiO_2), and the presence of iron compounds was not clearly shown. As for the elemental analysis by EDX, it showed the presence of iron oxides within the sandstone components, which are highly susceptible to the presence of moisture and may have a partial dissolution. The restoration works were carried out for the tombstone, which bears No. 117 in the museumstore's register (Seven rooms museumstore – Al Fustat) , where the mechanical cleaning of the thick layers of dirt was carried out, as well as the chemical cleaning processes with a solution of ethyl alcohol and water to remove the mud stains ,consolidation processes were also carried out for the deteriorated sandstone by Wacker (OH) 100 at a concentration of 3%, and the final isolation was done by the same material, but with a concentration of 10%, with the aim of restoring and preserving it.

Keywords: Tombstone; Second Abbasid Era; Restoration; Sandstone; Examination and analysis; Cleaning; Consolidation.

الملخص

تعد شواهد القبور الحجرية الأثرية الإسلامية واحدة من أهم مصادر التأريخ بما تحويه من كتابات بخطوط عربية متنوعة ، يهدف هذا البحث إلى دراسة شواهد القبور الإسلامية من الحجر الرملي مع التطبيق على شاهد قبر من العصر العباسي الثاني في التاريخ الإسلامي ، حيث تم تأريخه من خلال الكتابات الموجودة على السطح والمكتوبة بالخط الكوفي البسيط والذي يعود إلى 272 هجريه ويحمل إسم صاحبه (إبراهيم بن صهيب)، تم دراسة عينات وفحصها بواسطة الميكروسكوب المجسم ، الميكروسكوب الإلكتروني الماسح ، التحليل بواسطة حيود الأشعة السينية والتحليل العناصري بواسطة وحدة الإدكس ، وقد وجد من خلال الفحص بواسطة الميكروسكوبات وجود ضعف بالحجر الرملي ، ومن خلال التحليل بواسطة حيود الأشعة

السينية تبين أن ثاني أكسيد السيليكون هو المركب الأساسي بالعينة ولم تظهر مركبات الحديد بوضوح ، بينما تبين وجود مركبات الحديد ضمن مكونات العينة من خلال التحليل العناصري بواسطة الإدكس والتي قد يحدث لها ذوبان جزئي في وجود الرطوبة ، تم إجراء عمليات الترميم لشاهد القبر المختار والذي يحمل رقم 117 بسجل المخزن المتحفي (مخزن السبع حجرات بالفسطاط) حيث تم التنظيف الميكانيكي لإزالة الطبقات الكثيفة من الإتساخات والتنظيف الكيميائي بمحلول مكون من الكحول الإيثيلي والماء لإزالة الإتساخات الطينية ، تمت أيضا عمليات التقوية للحجر الرملي المتدهور بواسطة مادة الفاكر (OH) 100 بتركيز 3% وتم العزل بواسطة نفس مادة التقوية بتركيز 10% بهدف ترميمها وصيانتها.

الكلمات الدالة: شاهد قبر، العصر العباسي الثاني ، ترميم ، حجر رملي ، فحص وتحليل ، تنظيف ، تقوية ، عزل.

1. Introduction

Tombstones are considered one of the most important methods of dating in different eras; they are pieces of stone, whether limestone, sandstone, or marble. These tombstones are placed over the graves to indicate the writings on them about their owner[1] . There are many forms of tombstones, some of them are rectangular, and some are in the form of cylinders or small columns around which inscriptions revolve. In the Ottoman era, the tombstones of men were distinguished from women; there are many inscriptions and decorations on the tombstones, some of which are inscriptions (Low- relief and high - relief) and the others are decorated with plant and geometric decorations.

Sandstone is widely used in carving tombstones due to its availability and ease of operation. As for the calligraphies used in these tombstones, the Kufic calligraphy was used, which is one of the oldest and most beautiful Arabic calligraphies in writings, especially the sunken Kufic. The Kufic calligraphy was spread in the Abbasid era, and this calligraphy relies on geometric rules[2][3].

The selected tombstone is from sandstone and bears number 117 in the museumstore record , it is preserved in seven rooms museumstore – Al Fustat museumstores – Ministry of Tourism and Antiquities – Egypt , its description in the museumstore record is (Tombstone with inscriptions in Kufic calligraphy in Low- Relief and bears the date of the year 272 AH (Abbasid era)) , the type of Calligraphy is Arabic Calligraphy - Simple Kufic (Kufic well- knit) , the dimensions is 40×30 cm , there is erosion in some of the writings.

[1]Hemida, H.H.A. , *etal*., An Artistic Archaeological study of a group of Coptic tombstones written in Arabic in the Church of Prince Tadros in Deir al-Sinqureya in Bani Mazar in Minya , Journal of the Association of Arab Universities for Tourism and Hospitality , 14, 2 , 2017, P.1-26 .

[2] Mohamed, A.S., Arab tombstones with a Coptic calendar from the Aswan Cemetery, CGUAA, 15, 157, 2012, P.1413-1437.

[3]Ahmed, S.A.I., Tombstones in Islamic Egypt since the Islamic conquest until the end of the era of rulers - 21- 254 AH/ 641-868 AD , unpublished master thesis, Islamic Archaeology division , Archaeology department , Faculty of Arts , Ain Shams University , Egypt , 2015.

The texts on the tombstone consist of nine lines written in Arabic (Kufic script) as follows:

1. بسم الله الرحمن الرحيم
2. هذا قبر مسلم فتى
3. ابرهيم بن وهيب
4. بن يحيى رحمت الله
5. عليه توفى يوم الأ
6. ربعا ليلتين خلون
7. من شعبان سنة
8. اثنين وسبعين وما
9. يتين

The texts show the name of the owner of the tombstone (Ibrahim bin Suhaib) and the date of death 272 AH (in the Hijri calendar -The second Abbasid era in the Islamic history in Egypt).

There are many deterioration aspects like: thick layers of dust, mud stains and erosion of the writings due to the weakness of sandstone - (Fig.1).

Fig.1. Tombstone no.117 before restoration

1.1. Restoration and conservation strategy of sandstone tombstones:

1.1.1. Mechanical Cleaning:

Mechanical cleaning is the first step in various cleaning operations, where scalpels and brushes are used in the addition of wooden and metal spatulas and chisels, mechanical cleaning is a very safe method if it is used under control [4].

1.1.2. Chemical Cleaning:

Chemical cleaning is the next step of chemical cleaning, it is used when mechanical cleaning is unable to completely remove dirt and stains on the surface where acids, alkalis, neutral soaps and gels are used, as well as organic solvents such as ethyl alcohol, toluene, trichlorethylene , xylene and acetone[5] . The mud dirt is removed with a solution of ethyl alcohol and water at a ratio of 2:1[6]. In advanced approach of cleaning of stones and from various deterioration factors such as extreme black crusts which have been formed on stone antiquities, different coatings (surfactants) have been invented for preservation and self-cleaning of stone monuments surfaces, Titanium nano particles (TiO_2) or Titania technique have been used as a semi-conductor photocatalysts in a lot of fields in stone restoration, due to its unique properties. It is used for cleaning and consolidation of building materials and lime mortars to enhance the self-cleaning and mechanical properties [7].There is another modern method for removing calcareous crusts from the surfaces of stones, the method is based on the property of aqueous solutions of CO_2 to dissolve and remove the carbonate components , found on the surface of porous materials , making it easier to remove from the surface , Calcium carbonate $CaCO_3$ - characterized by a low solubility (0,015 g/l) put in contact with a solution of H_2CO_3 is transformed into calcium bicarbonate $Ca(HCO_3)_2$, which is approximately 100 times more soluble (1,4 g/l) [8].

Besides mechanical and chemical cleaning, there are other methods of cleaning using enzymes, enzymes are used in the preservation of culture heritage to help remove dirt, stains and old adhesives, the most common preserving enzymes are hydrolases, protease,

[4] Khallaf, M.K., & Mohamed, R.A., Treatment and Conservation of Six Egyptian Archaeological Stone Sarcophagi , Shedet, 1, 2014, P.37.

[5]Abdel-Halim, M.A.H., & Al- Saad , Z., Deterioration and Damage Evaluation of the Monument of Qasr al-Bint , Petra-Jordan and a Suggested Conservation Program , Jordan Journal of Civil Engineering, 12, 3, 2018, P.424-434 .

[6] Doehne , E., & Price , C.A., Stone Conservation – An Overview of Current Research , Second edition , The Getty Conservation Institute , Los Angeles, 2010, P.29-30.

[7]Abdel –ElHady, M.M., *etal*., Nanotechnology in surface of archaeological stones, Academia Journal of Agricultural Research , 6, 5, 2018,P.173.

[8]Coladonato , M., *etal*., Removal of calcareous concretions from natural and manufactured stone archaeological artefacts through the use of CO2 water solutions, Procedia Chemistry, 8, 2013, P.65-66.

lipase, and amylase, the lipase enzyme for example works on lipids and fats[9], enzyme-based treatments are applied on building stones to remove biocolonisation and there are commercially available enzyme-based cleaning products[10].

Laser cleaning is also considered one of the important methods in cleaning; The Conservators used laser technology in the removal of many deteriorated layers, because it's safe, fast and easy operation[11].

1.1.3. Consolidation Process:

Consolidation process is used to improve the mechanical properties of the monuments to prevent the future damage from impact on the monuments[12], there are various consolidation materials used to strengthen sandstone, Silicate consolidants are among the best sandstone consolidants, such as Tetraethoxysilane[13] , and there are many sandstone consolidation materials: including: Paraloid B72, Addicon, Wacker (OH) 100, Kemtekt and nanosilica. Nano silica can also be added to conventional consolidation materials in order to improve its consolidation properties, such as two materials: Wacker (OH) 100 reinforced with silica nano particles and Paraloid B72 reinforced with silica nano particles[14] , another example of nanomaterials used is nano lime[15]. The nano-lime of 'CaLoSiL®', which is $Ca(OH)_2$ particles with sub-micrometric dimensions (nanoparticles) , is one of the modern consolidants that are applied in many countries for the treatment of heritage calcareous objects , nano-lime particle size ranges between 1 to 100 nm, it is smaller than the pore size range of porous and carbonated stones. There are

[9]Hamad, R., & Badway, M., Using lipase enzyme in cleaning of archaeological glass: an experimental applied study, Egyptian Journal of Archaeological and Restoration Studies (EJARS), 11, 1, 2021,P.20 .

[10]Schröer ,L.,*etal*., Examining the Potential of Enzyme-Based Detergents to Remove Biofouling from Limestone Heritage,Coatings 12,375,2022, P.2.

[11]Elhagrassy, A.F., & Hakeem, A., Comparative study of biological cleaning and laser techniques for conservation of weathered stone in Failaka island, Kuwait, Scientific culture, 4, 2, 2018, P.43-50.

[12]Saleh , I. , *etal*., Experimental study for the consolidation and protection of sandstone petroglyphs at Sarabit El Khadem (Sinai, Egypt), Scientific culture , 5,.1 ,2019, P.44.

[13]Gupta, S.P., Conservation of Stone Structure by Impregnation with Tetraethoxysilane, International Journal of Chemical and Analytical Science, 2, 8, 2011, P.115 .

[14]El-Sayed, S.S.M., & Maky, A.R.Y., Archaeometric investigation to evaluate acrylic, silicon materials and nano-additives as consolidation material to sandstone monuments of the sphinxes avenue (Luxor, Egypt), Scientific culture , 8 , 1, 2022, P.52 .

[15]Al-Omary, R.A., *etal*., Laboratory evaluation of nanolime consolidation of limestone structures in the roman site of Jerash, Jordan , Mediterranean Archaeology and Archaeometry, 18, 3, 2018, P.35-43.

two commercially available brands of nano-lime, the CaLoSiL® that was introduced to the market in 2006 and the Nano-restore that appeared in 2008[16].

Another example of nano consolidants is nanosilica-based consolidant more diffused is Nano Estel® (nanosilica), an aqueous colloidal dispersion of silicon dioxide with dimension of 10-20 nm, and the water suspension Syton X30 [17].

1.1.4. Isolation process:

The isolation process is carried out in order to preserve the stone monuments after treatment and restoration, a lot of synthetic coatings can be applied to preserve stone surface , some of the applied coatings are commercially available like :siloxane-, siloxane/acrylate- and perfluoroether-based polymers and oligomers and one is a recently synthesized fluoro-organosilane[18], Paraloid B72, polyvinyl acetate emulsion (PVAC), and PrimalAC-33 are still employed nowadays for coating the surface of stone material ,despite general approval, these resins are demonstrated as not being the best choice because of their rapid tendency to undergo photo-oxidation reactions, which is principally induced by the sunlight action , enhanced by temperature and atmospheric pollution[19].Some nanomaterials can be used in the protection of stone monuments surface like: TiO_2 (Titanium dioxide), TiO_2 nanoparticles solution mixed in a commercial silane resin (Alpha®® SI30)[20]. ZnO nanoparticles can be applied also as a protective layer of stone surfaces beside their role in fungal inhibiting, The photocatalytic inorganic nanoparticles of ZnO have been applied for the purpose of long-term protection of marble columns by inhibiting microbial-fungal attack and forming a protective surface layer [21].

This research aims to restore and preserve the Archaeological Islamic sandstone tombstones with their inscriptions in different Arabic calligraphies, the most important of

[16]Abdel–Aty, Y.Y. , *etal*., Experimental Evaluation of Consolidation Techniques of Fossiliferous Limestone in Masonry Walls of Heritage Buildings at Historic Jeddah, Kingdom of Saudi Arabia, Advanced Research in Conservation Science, 1, 1, 2020, P.16-33.

[17]Stucchi, N.M.E., *etal*., Synthesis and characterization of nanosilica products for the consolidation of stones, 2019 IMEKO TC-4 International Conference on Metrology for Archaeology and Cultural Heritage , Florence, Italy, December 4-6, 2019, P.299.

[18]Tsakalof,A. , *etal*., Assessment of synthetic polymeric coatings for the protection and preservation of stone monuments , Journal of Culture Heritage , 8, 2007, P.69.

[19]Artesani, A., *etal*., Recent Advances in Protective Coatings for Cultural Heritage–An Overview, Coatings 10, 217, 2020, P.3.

[20]Fierascu, R.C., *etal*., Selected Aspects Regarding the Restoration/Conservation of Traditional Wood and Masonry Building Materials: A Short Overview of the Last Decade Finding , Applied Sciences 10,1164,2020, P.18.

[21]Aldosari , M.A. , *etal*., Using ZnO nanoparticles in fungal inhibition and self-protection of exposed marble columns in historic sites, Archaeological and Anthropological Sciences, 11, 2019, P.3407.

which is the Kufic as it is a cultural heritage that must be preserved and one of the most important methods of dating, with an application on the selected sandstone tombstone no.117 from the second Abbasid era in the Islamic history in Egypt.

2. Materials and Methods

Samples of sandstone were taken to study its composition and the extent of damage by various deterioration factors in the surrounding environment, as they were examined by stereomicroscope, scanning electron microscope (SEM), and analyzed by X-ray diffraction (XRD) and elemental analysis with the EDX unit attached to the scanning electron microscope.

2.1. Stereomicroscope examination:

The stereomicroscope (S9i) with camera (Leica S9I Stereozoom) (Format: 720p (1280 x 720) 16:9, Exposure: 151.0 ms , Gain: 5.0 x ,Gamma: 0.45) at Centre of Research and Conservation of Antiquities (CRCA) – Faculty of Archaeology – Fayoum University was used to examine the surface of the sample with high resolution. Stereomicroscope is one of the most important examination methods of the sandstone morphology, the examination shows the samples deterioration, as well as the shape and size of the granules and their distribution[22].

2.2. Scanning Electron Microscope (SEM):

The Scanning Electron Microscope (SEM) (JEOL JSM 6510 1v) Central laboratories of the Egyptian General Authority for Mineral Resources – Dokki - Giza – Egypt was used to examine the texture of the surface with high magnification power[23].

2.3. X- Ray Diffraction (XRD):

The sample of sandstone was well- crushed and analyzed with x-ray diffraction device (PW1710) Central laboratories of the Egyptian General Authority for Mineral Resources – Dokki- Giza- Egypt, to identify the mineralogical compositions of the stone sample based on studying the crystalline structure of the materials, their proportion and its damage[24].

[22]Kamel, W.K., Characterization and Treatment of Rock Inscriptions in Wadi Nasib in South Sinai, Egypt, Case Study , Middle East Journal of Applied Sciences , 8, 1, 2018, P.67.

[23]Kamh, G., Klitzsch, N., Testing single and mixed resins for consolidating moderately weathered limestone of archaeological sites, new applied techniques, International Journal of Conservation Science, 9, 3, 2018, P.600 .

[24]Al-Bawab, A., *etal* ., Consolidation of archaeological basalt stone: A new experimental protocol by using different dispersions formulation, International Journal of Conservation Science , 8, 2, 2017,P.189.

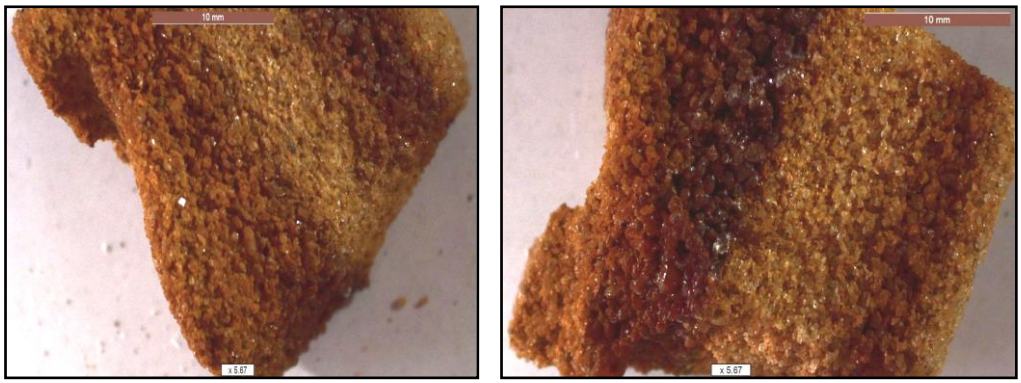
2.4. EDX (Energy-Dispersive X-Ray Spectroscopy):

Another sample was analyzed with EDX unit (ENERGY-DISPERSIVE X-RAY SPECTROSCOPY) attached with scanning electron microscope (SEM) to identify the elements of the sample and their proportions.

3. Results and discussion

3.1. Stereomicroscope examination:

Through the examination by the stereoscopic microscope of the surface of the sample, it was found that there were some iron compounds within the composition of the sample, and it was also noted that there were some voids between the quartz grains as a result of the weakness of the stone and the loss of the cement material - (Fig.2).



(A) (B)

Fig.2. (A-B). Stereo Microscope examination of the sandstone sample, it is clear that there is a percentage of iron compounds in the sample, (dark parts).

3.2. Scanning Electron Microscope (SEM):

As for the scanning electron microscope (SEM), it is found some holes in the sample's tissue as a result of a loss in the binding material as mentioned previously, which is the reason for the sandstone weakness, also the presence of crystals for some iron compounds appeared beside the presence of quartz - (Fig.3-4).

3.3. X- Ray Diffraction (XRD):

It was found through the analysis by x-ray diffraction analysis (XRD) that Quartz is the main component of sandstone by 95%, and iron compounds did not appear clearly - (Fig.5) , (Table.1)

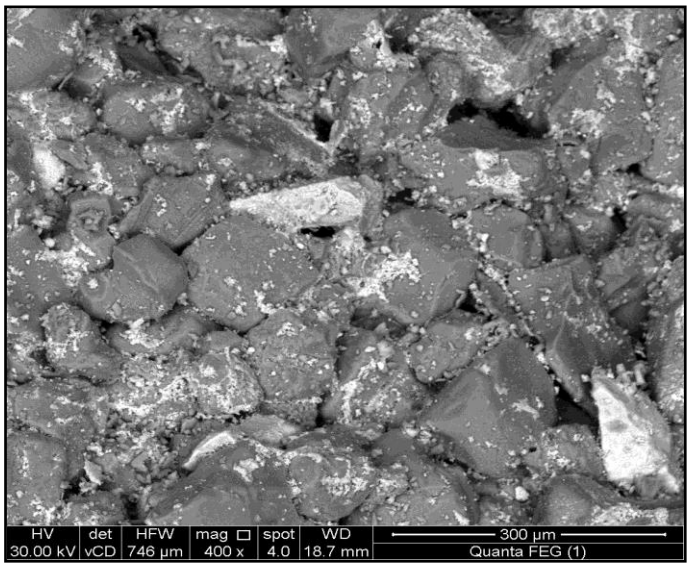


Fig.3. Scanning Electron Microscope (SEM) examination of the sandstone sample (mag 400×)

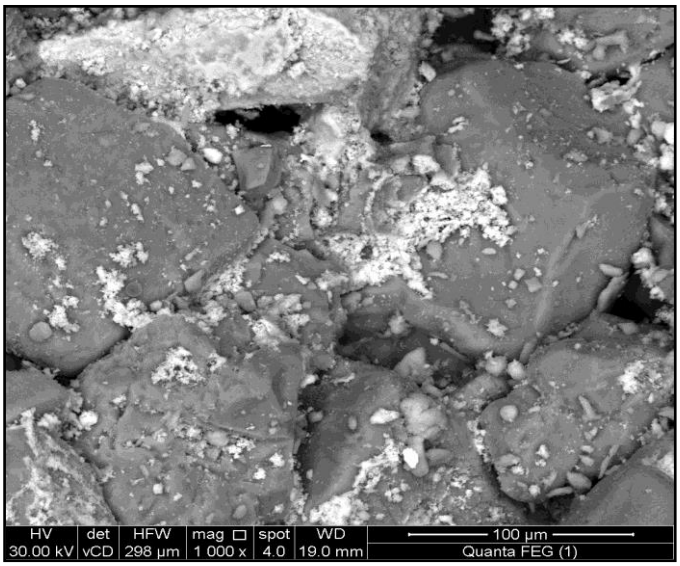


Fig.4. Scanning Electron Microscope (SEM) examination in another magnification power (mag 1000×)

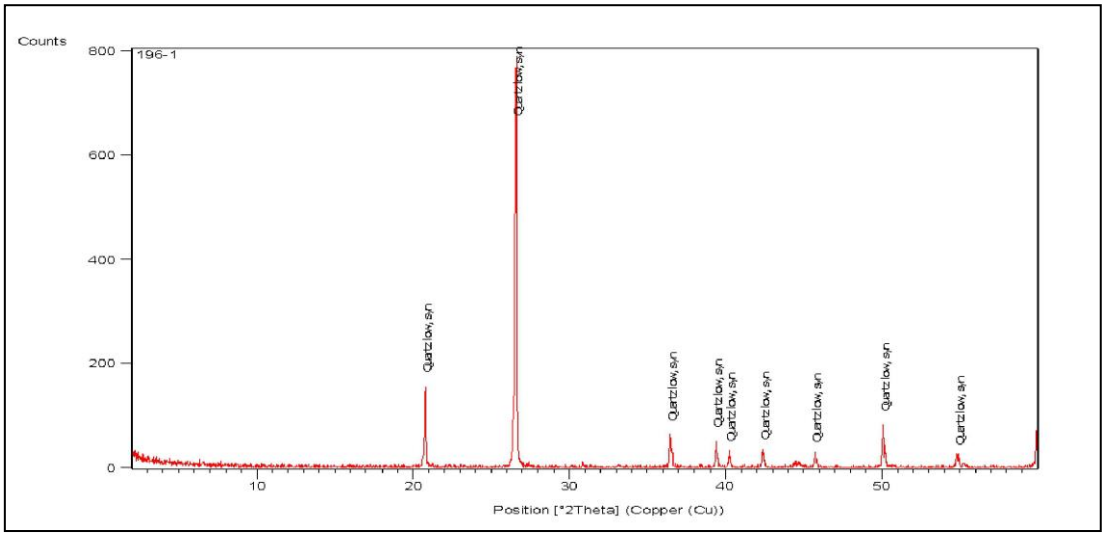


Fig.5. X-Ray Diffraction pattern of the sandstone's sample

Table .1. The Components of sandstone's sample (XRD analysis)

Component	Percentage %
Quartz	95

3.4. EDX (Energy-Dispersive X-Ray Spectroscopy):

Through the elemental analysis by EDX (energy-dispersive x-ray spectroscopy), it was found that the main element is Silica (Si) with percentage of 41.51% , Oxygen (O) 39.47% , Iron (Fe) 13.27% , Which means the presence of iron oxides inside the sandstone, even if a small percentage which was not shown clearly in the analysis by x-ray diffraction (XRD) - (Fig.6), (Table.2).

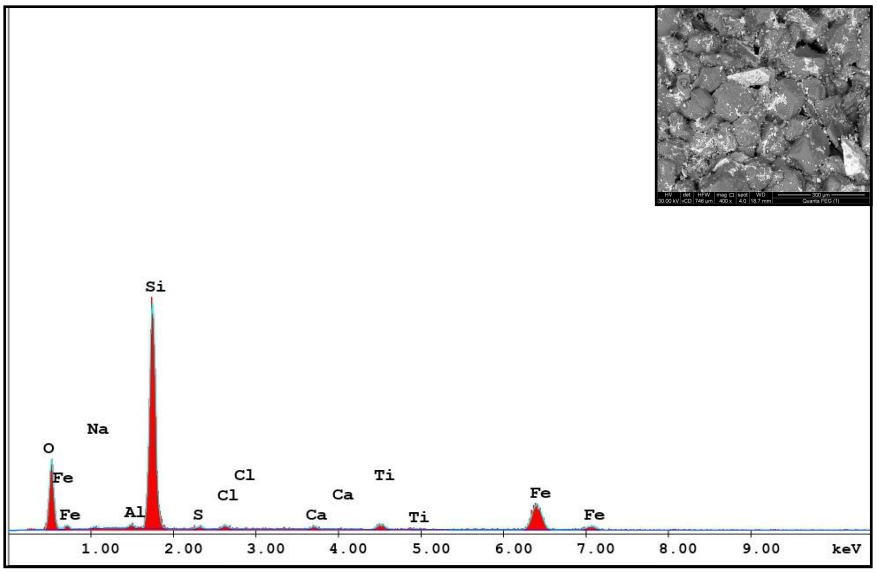


Fig.6. EDX's elementary analysis of the sandstone's sample

Table.2. The elements of the sandstone's sample and their proportions (EDX) - elements paired with potassium (K).

Element	Wt %	At %
O K	39.47	56.65
NaK	0.99	0.98
AlK	0.79	0.67
SiK	41.51	33.94
S K	0.69	0.50
ClK	0.98	0.63
CaK	0.71	0.41
TiK	1.60	0.77
FeK	13.27	5.46
Total	100.00	100.00

3.5. Restoration processes of the selected tombstone no.117:

3.5.1. Mechanical Cleaning:

Mechanical cleaning of the selected tombstone was carried out with soft brushes to remove the dense dirt and thick layer of dust on the surface, after which a scalpel was used to remove the mud stains – (Fig.7).

Fig.7. Mechanical cleaning of the tombstone by soft brushes and a scalpel

3.5.2. Chemical Cleaning:

Mud stains were removed with a solution of ethyl alcohol and distilled water in a ratio of 2:1 with swabs to show the writings- (Fig.8).

Fig.8. Chemical cleaning of the tombstone with a solution of ethyl alcohol and distilled water (2:1) by using swabs.

3.5.3. Consolidation processes:

The Consolidation of the tombstone was carried out in order to increase the mechanical properties of the degraded sandstone as there is erosion in the inscriptions due to the sandstone weakness.

The Consolidation was carried out with Wacker (OH)100 diluted with ethyl alcohol in the concentration of 3%[25] , in five cycles and with the brush so that the next cycle is not applied until after the previous cycle of consolidation has dried – (Fig.9).

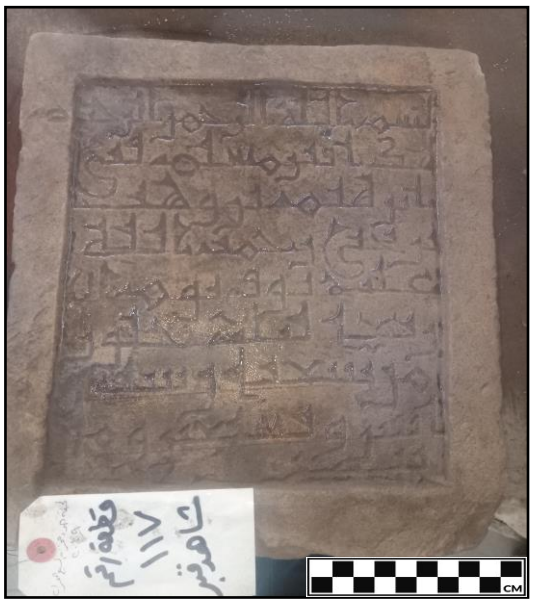


Fig.9. Consolidation process with Wacker (OH) 100 in the concentration of 3% by brushing

3.5.4. Isolation Process:

The isolation process was carried out using the same consolidation solution (Wacker (OH)100 diluted with ethyl alcohol , but with a different percentage, where it was 10% [26], and with only two cycles, taking into account the dryness of each cycle before applying the next cycle.

The aim of the isolation process is to preserve the tombstone and the valuable writings it carries from the deterioration factors in the surrounding environment – (Fig 10-11).

[25]Öztürk, I., Alkoxysilanes Consolidation of Stone and Earthen Building Materials , Master Thesis , University of Pennsylvania, Philadelphia, PA, 1992 .

[26] Grove, R.I., A Holistic Approach to Evaluating the Performance of Consolidants on Sandstone , PhD Thesis, St Cross College , University of Oxford, 2021.

(10) (11)

Fig.10-11. The Tombstone no.117 before and after the restoration processes

4. Conclusions

The study of tombstones is one of the important studies in Archeology, as it is an archaeological and cultural record, as it includes valuable information on genealogy, names, surnames, races, dates and the styles of Arabic calligraphy. Therefore, this research aims to preserve one of the Islamic tombstones from the Abbasid era and bearing the number 117 in seven rooms museumstore in Al Fustat, where it was noted the presence of dense layers of dust and mud stains in addition to the occurrence of erosion at the edges of the writings. It was found through the examination with the stereoscopic microscope and the scanning electron microscope that there is a weakness in the surface as a result of a loss of the binding material between the quartz grains in addition to the presence of iron compounds, through the analysis by X-ray diffraction and the elemental analysis by the EDX unit, it was found that silicon dioxide is the main compound in addition to the presence of a percentage of iron oxides , they are highly susceptible to moisture and may partially dissolve. The restoration processes were carried out with the aim of preserving this tombstone through mechanical and chemical cleaning processes, in addition to consolidation with Wacker (OH) 100 material at 3% and isolation with the same material at 10%.

The research recommends the necessity of good packaging for this tombstone after its restoration by placing in a foam support, wrapping it with polyethylene and keeping at a relative humidity not exceeding 40% , a temperature degree of 18-22 ^{0}C , away from direct lighting and atmospheric pollutants to preserve this important cultural heritage.

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