Restoration and conservation of an archaeological marble gravestone from the Greek era - Al-Fustat - Egypt

Dr. Shaimaa Sayed Mohamed El-Sayed
Conservation Department – Faculty of Archaeology – Luxor University – Egypt
Shaimaa.Sayed@farch.luxor.edu.eg

Abstract

The archaeological and historical gravestones are one of the important sources of dating with their inscriptions and writings, so they must be preserved as an important tangible cultural heritage. This research aims to restore and conserve an archaeological marble gravestone which bears No. 142 and stored in the seven rooms museum store in Al-Fustat- Egypt , this object dates back to the Greek era , samples were taken from the selected gravestone and examined by stereomicroscope and scanning electron microscope (SEM), it was also analyzed by X-ray diffraction (XRD) and elemental analysis by the EDAX unit attached to (SEM) Microscope, through the results of the examinations and analysis, it was found that there were some cracks and gaps on the surface of the sample as a result of the deterioration factors in the surrounding environment. The treatment plan included mechanical and chemical cleaning of dust and dirt on the surface of the gravestone, which hide the writings on the surface, as well as consolidation processes using Paraloid B-72 dissolved in acetone at a concentration of 2%, and in the end, the surface was isolated to preserve the writings by Paraloid B-72 at a concentration of 5%, in order to preserve this important cultural heritage.

Keywords

Restoration and Conservation, Gravestone, Marble, Greek era, Examinations and Analysis

الملخص

تعد شواهد القبور الأثرية والتاريخية واحدة من أهم مصادر التأريخ بما تحمله من نقوش وكتابات والتي يجب الحفاظ عليها وصيانتها كتراث مادي هام، يهدف هذا البحث إلى ترميم وصيانة شاهد قبر أثري من الرخام يحمل رقم ١٤٢ ومحفوظ بمخزن السبع حجرات بالفسطاط – جمهورية مصر العربية ، يعود هذا الشاهد إلى العصر اليوناني ، تم أخذ عينات من شاهد القبر المختار حيث تم فحصها بواسطة الميكروسكوب المجسم والميكروسكوب الإلكتروني الماسح ، بالإضافة إلى تحليلها بواسطة حيود الأشعة السينية والتحليل العنصري بواسطة وحدة EDAX الملحقة بالميكروسكوب الإلكتروني الماسح ، وقد تبين من خلال الفحوص والتحاليل وجود بعض الشروخ والفجوات على سطح العينة والتي نتجت عن عوامل التلف بالبيئة المحيطة. تضمنت خطة العلاج التنظيف الميكانيكي والكيميائي للأتربة والإتساخات الموجودة على سطح شاهد القبر والتي تخفي الكتابات على هذا السطح ، بالإضافة إلى عمليات التقوية بإستخدام الباراليويد ب ٧٢ المذاب في الأسيتون بتركيز ٢% ، وفي النهاية تم عزل السطح للحفاظ على هذه الكتابات بواسطة الباراليويد ب٧٢ بتركيز ٥% وذلك حفاظاً على هذا التراث الثقافي الهام.

الكلمات المفتاحية

ترميم وصيانة ، شاهد قبر ، الرخام، العصر اليوناني، الفحوص والتحاليل

1. Introduction

Gravestones or tombstones are an important and unique cultural heritage that must be taken care of and preserved, as they contain some important inscriptions and writings that are used in dating (A.A.E.Kabil., 2022), they have a great importance in Ancient Egyptian beliefs, where they represent a gate that separate between the burial room and the world outside, gifts and sacrifices are also presented to the dead through them, this belief continued in the Greek, Roman, Coptic and Islamic eras. Inscriptions or writings of them contain the name of the dead, the death date, invocations and prayers for the dead(A.A.A.Hashad.,2021), they are also one of the funerary customs that has been preserved throughout the ages, where some information about the dead is placed either in the form of an inscription, a picture, symbolic signs, writings, or depictions of all these elements with the aim of obtaining the Paradise **(D.M.Bahi El din., 2021),** men gravestones differed from women in the Ottoman era, where the gravestones of men adorned a hat or turban, while the gravestones of women adorned a wreath of flowers**(T.Ogreloil., 2008).**

There are many stones from which these gravestones or tombstones were carved, the most famous of which are limestone, sandstone and marble. Marble is a metamorphic rock which was transformed from limestone by heat, and it consists mainly of calcium carbonate ($CaCO_3$) in addition to some other components, during metamorphosis the crystals interlock , clay, sand, and chert impurities sometimes produce distinct veins and swirls within the marble stone, giving it a distinct and sought-after veining .Marble has many types and colors , these colors are due to the presence of some mineral compounds as impurities, where there are white, red, gray, green and others **(H.M.Gamil., *etal*., 2022).**

The selected gravestone bears no.142 in the museum store record and stored in seven rooms museum store – Al Fustat museum stores – Ministry of Tourism and Antiquities – Egypt, it's made of from marble and dates back to Greek era, its description in the museum store record is a complete tombstone with a text of ten lines written in Greek in low- relief, the type of calligraphy is Greek alphabet and the dimensions are 94 × 56 cm. It has dust, dirt; and abrasion on the edges.

A photographic documentation of the selected tombstone was made to show its condition and deterioration phenomena, as well as a drawing documentation or a deterioration map to show the most important inscriptions found in it which are considered very valuable yet a phenomena in its deterioration, explained in the following:

1-The presence of thick dust on the surface obscuring the writings below.

2- Mud stains adhered to the surface and hide the writings.

3- Writings in black ink on the object explain its number recorded in the excavation record.

4- The Latin letters edges abrasion caused by weakness of marble. (Fig 1, 2).

This research aims to restore and conserve a Greek marble gravestone no.142 that is preserved in the seven rooms museum store, Al- Fustat museum stores - Ministry of Tourism and Antiquities - Egypt.

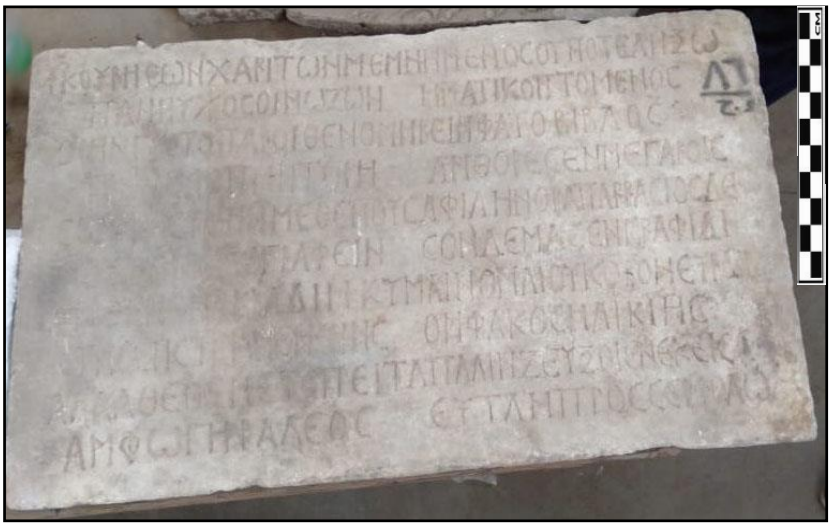

Fig.1. The dust and dirt that obscure the writings on the gravestone no.142

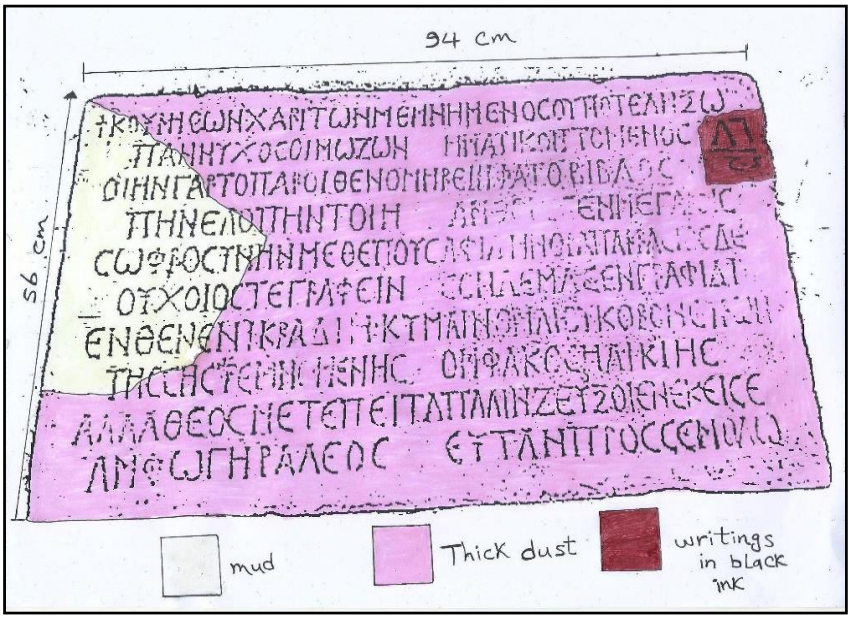

Fig.2. Drawing documentation or deterioration map of the selected gravestone

2. Materials and Methods

Samples were taken from the invisible side of the selected gravestone in order to identify its components and diagnose the status of damage to
determine the appropriate treatment plan.

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 marble samples with high resolution. Stereomicroscope is one of the most important examination methods of the marble morphology, the examination shows the samples deterioration, as well as the shape and size of the granules and their distribution (W.K. Kamel., 2018).

2.2. Scanning Electron Microscope (SEM):

The Scanning Electron Microscope (SEM) (ZEISS – Gemini, Sigma 500 VP) Faculty of Science – Fayoum university - Egypt was used to examine the texture of the marble sample's surface with high magnification power (G. Kamh., and N. Klitzsch., 2018).

2.3. X-Ray Diffraction analysis (XRD):

The marble sample 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 (A. Al-Bawab., *etal*., 2017).

2.4. EDAX analysis (ENERGY-DISPERSIVE X-RAY SPECTROSCOPY):

The marble sample was analyzed with EDAX unit (ENERGY-DISPERSIVE X-RAY SPECTROSCOPY) attached to scanning electron microscope (SEM) (ZEISS – Gemini, Sigma 500 VP) Faculty of Science – Fayoum university, to identify the elements of the sample and their percentages.

3. Results and Discussion

Through examinations and analysis of marble samples, the following results can be observed:

3.1. Stereomicroscope examination:

It was found by examining the surface of the sample with a stereo microscope, the clarity of the characteristic texture of marble, which is the sugary tissue. It was also noted that there were gaps and erosion on the sample's surface as a result of the gravestone's exposure to damage and deterioration while it was in the archaeological site or while being stored in the museum store - (Fig .3,4).

Fig.3. Examination of the marble sample with stereomicroscope

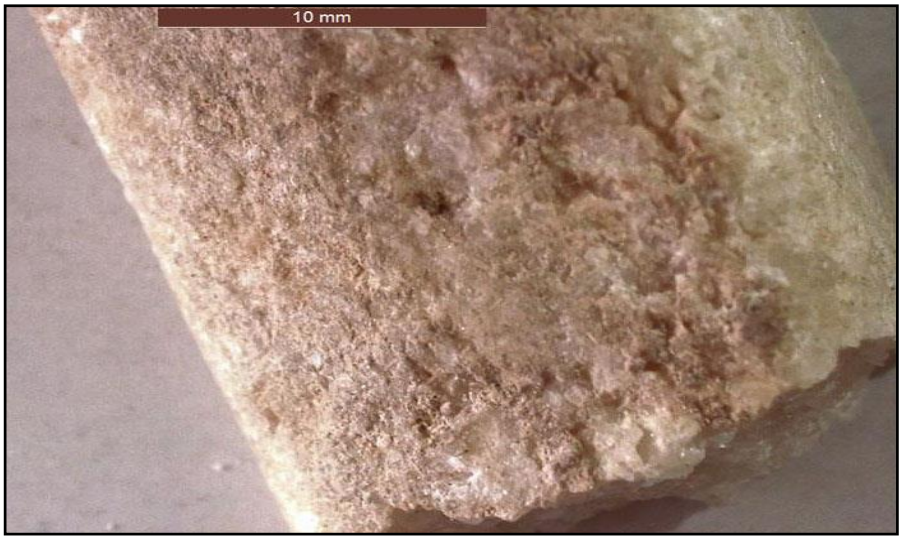

Fig.4. Stereomicroscopic examination of the sample, it shows gaps and erosion on the surface

3.2. Scanning Electron Microscope (SEM):

As for the examination by scanning electron microscope (SEM) of the sample, it was found that there are cracks, gaps and crystals fragmentation inside the sample caused by the weakness of the marble-(Fig.5).

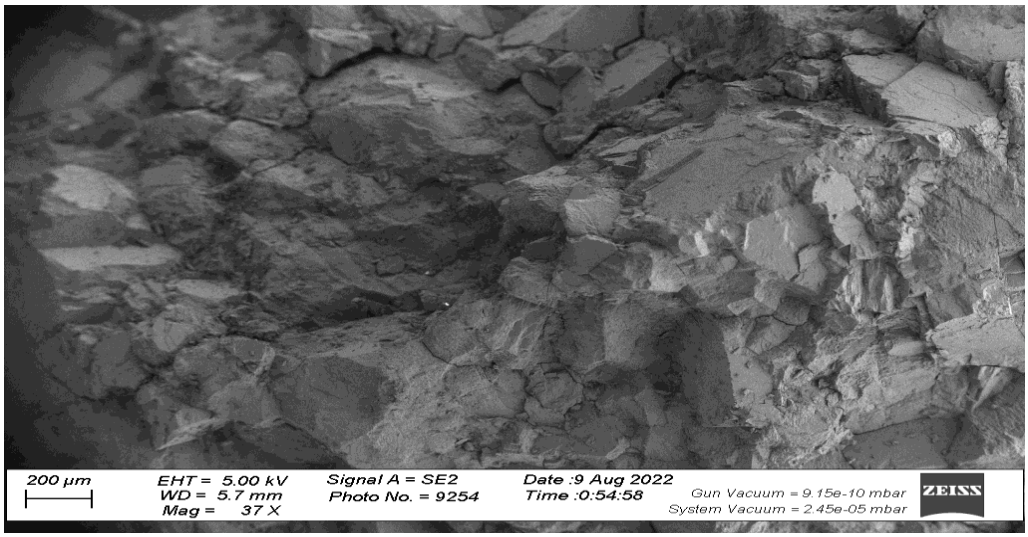

Fig.5. Scanning Electron Microscope (SEM) examination of the sample, it shows many cracks and gaps (Mag 37×)

3.3. X-Ray Diffraction analysis (XRD):

It was found from the analysis by X-ray diffraction (XRD) that the main compound in the sample is calcium and magnesium carbonate Ca Mg $(CO_3)_2$ (Dolomite) with a percentage of 100%, it is a qualitative and semi-quantitative analysis. Magnesium element represents the most important element in the formation of metamorphic marble - (Fig.6).

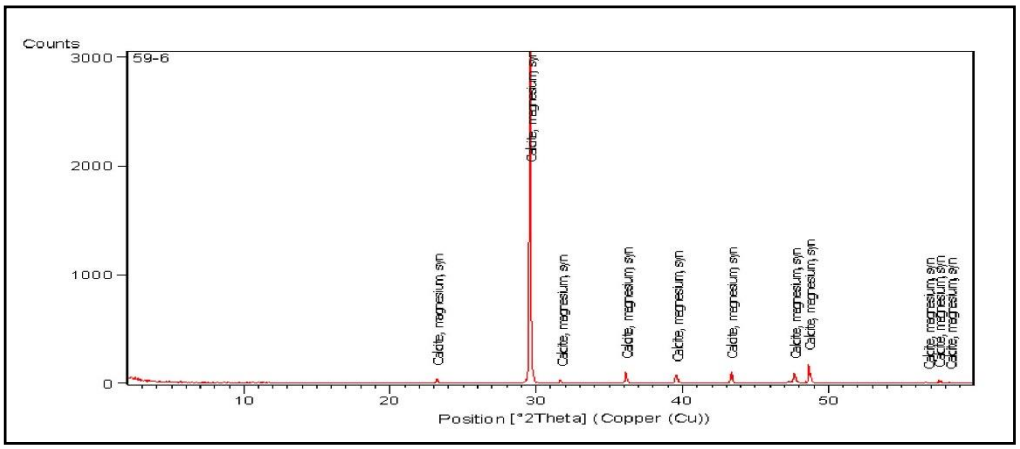

Fig.6. X-Ray diffraction pattern of the marble sample

3.4. EDAX analysis (ENERGY-DISPERSIVE X-RAY SPECTROSCOPY):

Several elements of the sample and their percentages were identified through the elemental analysis by EDAX unit , such as : Oxygen (O) 41,81% , Calcium (Ca) 30.65%, Carbon (C) 12.62% , Niobium (Nb) 3.88% , Magnesium (Mg) 0.04% , and Iron (Fe) 0.03% , it is noted that the results of the elemental analysis by EDAX are confirming the result of x-ray diffraction analysis (XRD).The marble color may be due to the presence of impurities, which is Niobium metal (Nb) - (Fig.7,8), (Table.1).

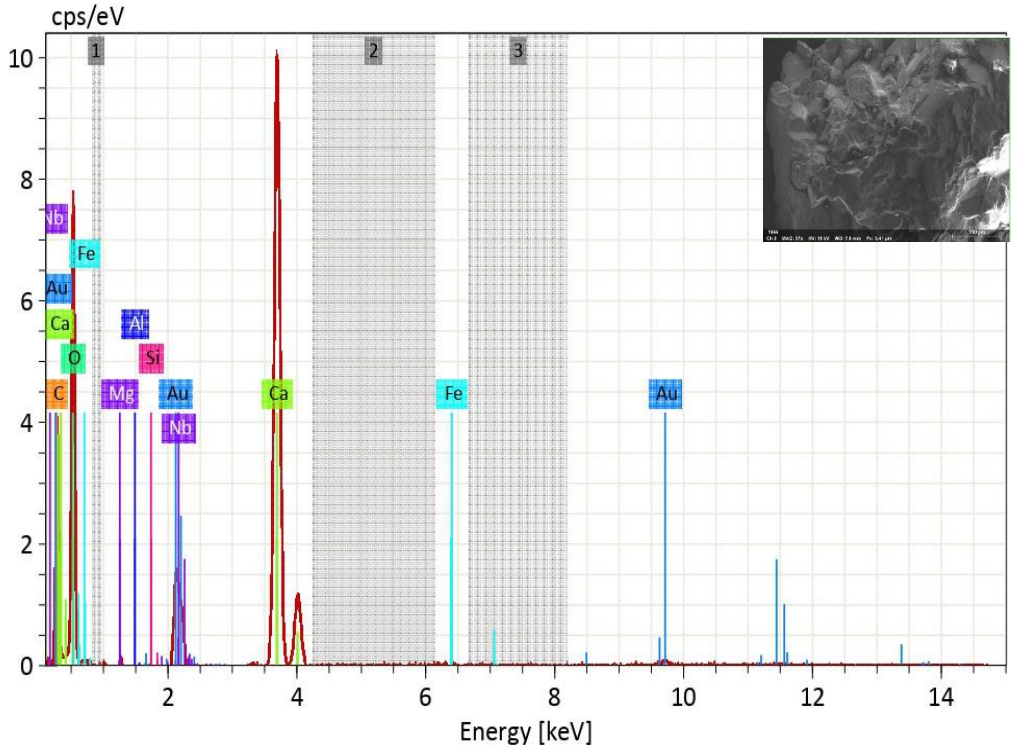

Fig.7. EDAX Spectrum of the sample

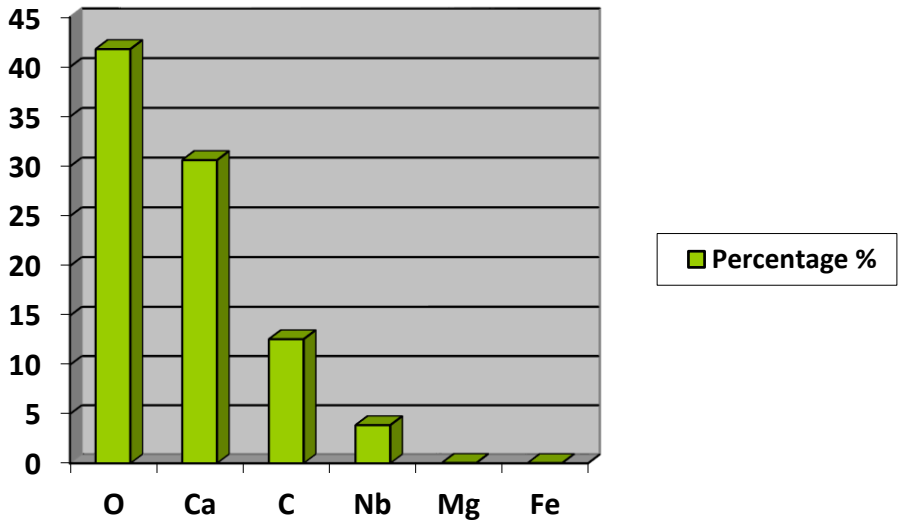

Fig.8. Flowchart of the marble elemental analysis by EDAX and their percentage

Map

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]
Carbon	6	9574	12.62	14.17	23.49
Oxygen	8	21759	41.81	46.97	58.43
Magnesium	12	144	0.04	0.05	0.04
Silicon	14	0	0.00	0.00	0.00
Calcium	20	61957	30.65	34.42	17.10
Aluminium	13	20	0.00	0.01	0.00
Iron	26	22	0.03	0.03	0.01
Niobium	41	10033	3.88	4.35	0.93
		Sum	**89.03**	**100.00**	**100.00**

Table.1. Elements of the sample and their percentage (EDAX analysis)

4. Restoration and Conservation of the selected marble gravestone no. 142

4.1 Cleaning processes:

Cleaning processes are considered one of the initial steps in the treatment plan for marble gravestones, as there are thick dirt, dust and stains on the surface of them, and therefore in this case, cleaning operations are considered the first step in treatment unless preceded by initial consolidation process. Mechanical cleaning is the first choice using some tools like: scalpels, brushes, chisels, spatulas to remove dust and dirt mechanically without using any chemicals after that the role of chemical cleaning to remove the remains of dirt which can't be removed mechanically, most people do not realize that marble is chemically reactive and fairly porous, conditions that allow alteration of its character by surface contaminants. The types of stains fall into several groups: dirt and grit, oils, inherent discoloration from oxidized metallic ions, and external contact with metals or glues and other adhesives. Usually, stains are a combination of

these factors. Organic solvents like: ethyl alcohol, toluene, acetone and trichloroethylene beside neutral soaps can be used to remove several types of stains (D.Rinne., 1976).

There are recent methods in cleaning such as Laser cleaning; it relies on the ablation effect as a result of intense and short pulse irradiation at wavelengths that are strongly absorbed by the materials. The laser induced removal of unwanted material from unique cultural heritage objects and monuments, it is a complex process closely dependent on the material properties and laser parameters (P. Pouli., *etal*., 2016), the type of laser beam which is used in cleaning is a high-purity laser (Nd: YAG), it is producing one wavelength in a very narrow beam and it is more selective, one of the advantages of the laser is that it is fast, easy to use, effective in removing layers of damage, and it stops automatically after removing these layers (A.F.Elhagrassy., and A.Hakeem., 2018). Nanomaterials can be used in cleaning and have good results , for example zinc oxide (ZnO), which is a substance that prevents the formation of biological damage on marble surfaces, in addition to its role in removing dirt and stains (E. Schifano., *etal*., 2020)(M.A. Aldosari., *etal*.,2019). Another nanomaterial can be used in self – cleaning for stone monuments is Titanium nano particles (TiO_2) or titania technique, it is used also for consolidation of building materials and lime mortars to enhance the self-cleaning and mechanical properties (M.M. Abdel-Hady., *etal*.,2018) (M.A. Aldoasri., *etal*.,2017) (F. Gherardi., *etal*., 2017).

The dust on the surface of the gravestone was removed with a soft brush; the sticky dirt was mud and was removed mechanically by wooden spatulas so as not to scratch the surface. As for the remaining dirt, it was chemically removed with a solution of acetone and distilled water in a 1:1 ratio by swabs in order to remove dirt from inside the writings, as for the writing in black ink on the surface of the object, it was difficult to remove and it is recommended not to write on the surface of the monument in any way so that it cannot be distorted, (Fig .9).

Fig.9. Cleaning of the gravestone (mechanical and chemical cleaning)

4.2 Consolidation processes:

The Consolidation process of the deteriorated archaeological marble is carried out in order to increase its mechanical properties as well as its resistance to environmental effects.

Consolidator, protective and hydrophobic polymeric materials have been used in conservation science like: acrylic (Paraloid B-72 for example)(A.M.Bakr.,2011) , silicate and alkoxy silane materials (C. Giancristofaro., *etal*., 2014), the most common products used to consolidate deteriorated building stones are mainly based on tetralkoxy- or alkylalkoxy-silanes, resulting in the formation of relatively stable silica inside the stone pores. Unfortunately, silica is not chemically compatible with carbonate stones like: marble and limestone, nanolime is a relatively recently developed material used in the consolidation process, derived from an older limewater method, nanolime is synthesized at nanoscale as calcium hydroxide particles in alcohol for suspension stability, as a particle size ranges between 100 nm and 500 nm is smaller than the pore size range of porous and carbonate stones (R.A. Al-Omary., *etal*.,2018), (E.Caner., and E.N.C.Saltık., 2018) , nano lime is called by some commercial names(Calosil®, Nanorestore ®,and Merck®)(A.S.Fernandez., *etal*., 2017)(Y.Y. Abdel–Aty., *etal*., 2020).
Hydroxyapatite (HAP) also was used in the consolidation processes of carbonate stones like: marble and was converted recently into nanoparticles (E. Sassoni., 2018). Another nanomaterial which is used in the consolidation process is nanosilica (silicon dioxide - SiO2 in nano particles) (M.E. David., *etal*., 2020) and nano titanium (TiO2) which can be used for archaeological marble consolidation(M. Abd El Hady., *etal*., 2019).The marble gravestone was consolidated with Paraloid B-72 dissolved in acetone at concentration of 2% (Considering the low porosity of marble), This is by 7 cycles of consolidation using the brush for a period of 7 days, where the object was left for 24 hours between each consolidation session and the other until it was completely dry, taking into account the observation of the result of each consolidation session through a visual examination (the absence of a film or the presence of color change and surface gloss), Paraloid is considered one of the acrylic resins most commonly used in the consolidation of monuments due to its characteristic mechanical properties and ease of use. Therefore, using a polymer requires dissolving it in a solvent, such as ethyl alcohol, acetone or toluene. There are many kinds of Paraloid like: Paraloid B-66, Paraloid B-67, Paraloid B- 44 and Paraloid B-82 but Paraloid B72 is the most used acrylic polymer. When using Paraloid B72 in consolidation, it doesn't cause discoloration for the surface, and thus it is considered acceptable material for marble stone preservation. Moreover, Paraloid B72 also increases the water-resistance of the stone (M.M. Ibrahim., *etal*., 2021), Paraloid B-72 is characterized with filling most of the pores and obscured many of particles, good stability, resistance to friction, flexibility and resistance to yellowing (A. Shoaib., and H. Kamal., 2020), (Fig.10).

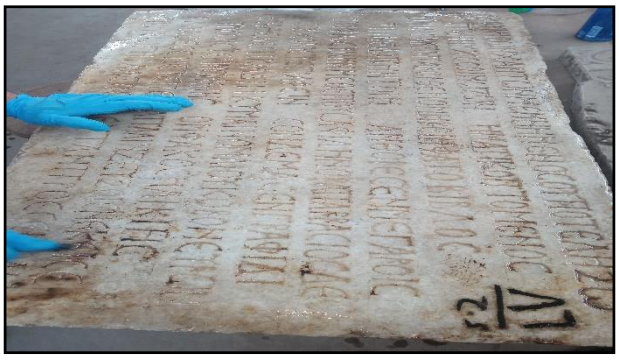

Fig.10. Consolidation processes of the gravestone with Paraloid B-72 at a concentration of 2%

4.3 Isolation process:

The process of isolation or coating the archaeological marble surfaces is carried out after the completion of the treatment or restoration processes, in order to preserve them from the factors of damage and deterioration, the same consolidator that was previously used in the consolidation processes can be used in the isolation process as a water repellent, but with a concentration ranging from 5-10%.

Acrylic polymers, tetraethoxysilane, nano lime ($CaOH_2$), nano Titanium (TiO_2) and nano silica (SiO_2) are the most common coatings of stone objects (M. Zuena., *etal*., 2021). Hydroxyapatite (HAP) can be used also as an isolation material, it has a much lower dissolution rate and solubility than calcite, especially in an acidic environment, so it has been proposed for the protection of marble against acidic rain erosion (G. Graziani., *etal*., 2016). Methyl cellulose is experimented for marble monuments coating (indoor marble statues) to prevent them from staining and damage factors, the methyl cellulose performed very well. Aesthetically, it was not visible on the samples and had the best reversibility when using non-toxic materials (L. Kubick., and J. Giaccai., 2012).

The process of isolation of the gravestone after its restoration was carried out by using the same consolidation material, which is Paraloid B-72 dissolved in acetone (Water repellent material), but with a concentration of 5%, with only two cycles, and by brushing, taking into consideration the dryness of each isolation cycle before applying the next cycle .The purpose of the isolation process is to conserve or preserve the object from the effects of the surrounding environment and future damage, (**Fig 11,13**).

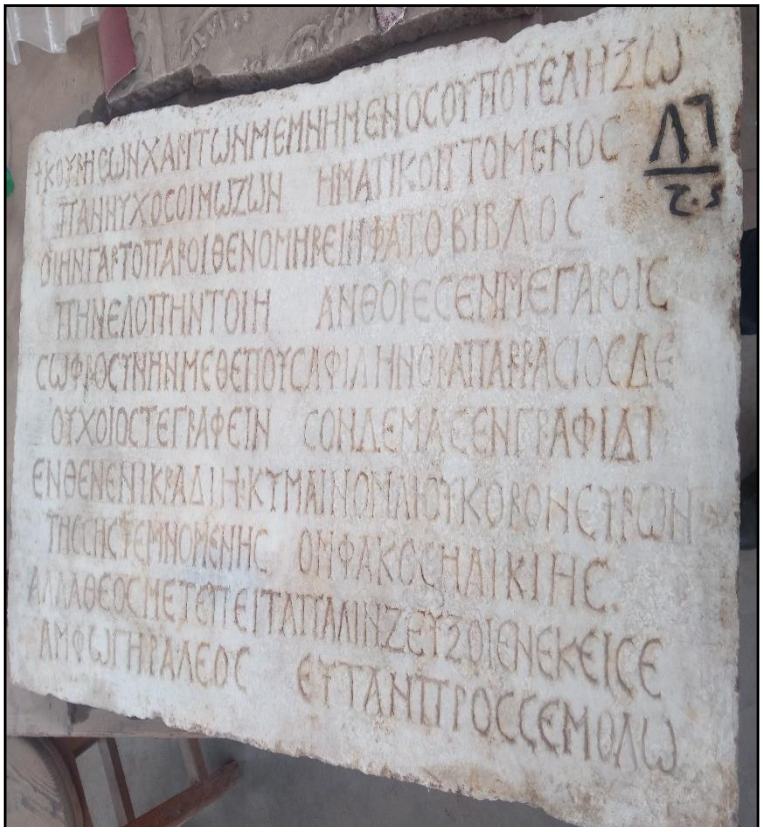

Fig.11. The selected gravestone after isolation process

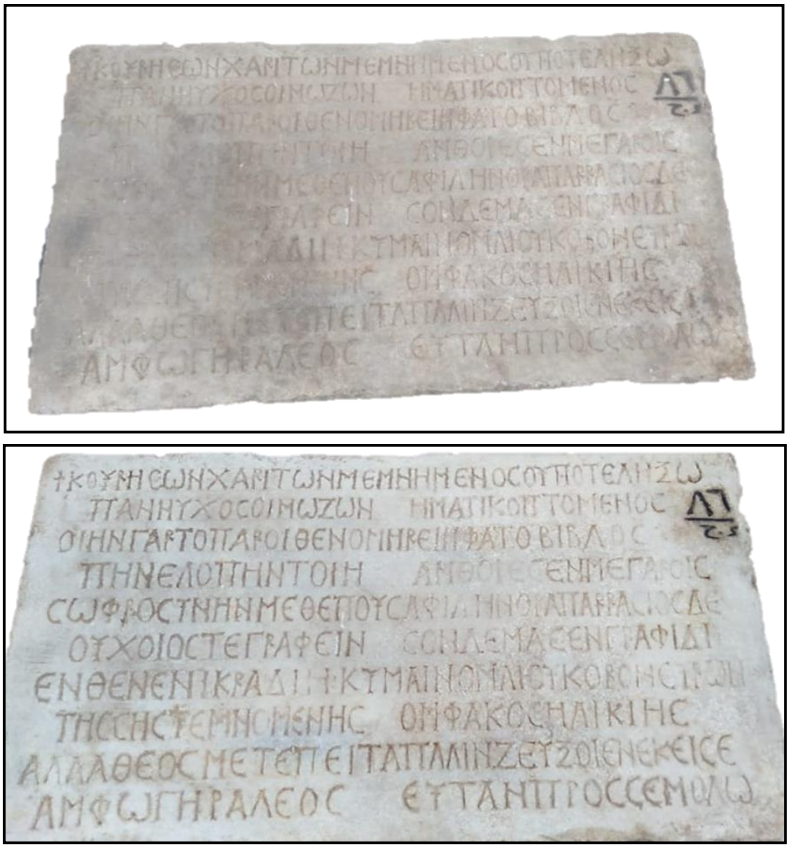

Fig .12-13. Marble gravestone no.142 before and after the restoration and conservation processes

5. Conclusion

The gravestones are one of the important dating methods, the stones from which these gravestones were carved were numerous, the most important of which is marble, as it is a rock that is easy to carve and ornament, the museum stores of the Ministry of Tourism and Antiquities - Egypt are rich in many archaeological gravestones from the ancient Egyptian civilization until the Ottoman era, the forms of these gravestones differed. In the Ottoman era, the gravestones of women were distinguished from that of men. The importance of this research is due to the preservation of stone gravestones and the important decorations and writings they contain, especially of marble, through the selection of a marble gravestone no.142 from the Greek era which is preserved in seven rooms museum store - Al Fustat museum stores- Ministry of Tourism and Antiquities – Egypt , the mineral components of the marble and the aspects of the damage were identified through various methods of examination and analysis like : stereomicroscope , scanning electron microscope, x-ray diffraction and elemental analysis by EDAX , the restoration and conservation plan included mechanical and chemical cleaning , consolidation and isolation processes , for the purpose of preserving this important cultural heritage and its important writings.

The research recommends the necessity of preserving this gravestone after its restoration and conservation by packing it with acid-free materials such as polyethylene in a relative humidity ranging between 40-45 %, a temperature of 18-20 ° C, 100 lux lighting (cold lighting-fluorescent), and in the environment Free pollutants.

6. Acknowledgment

The Author extends its sincere thanks and appreciation to the conservators in Al-Fustat museum stores – Ministry of Tourism and Antiquities - Egypt for their great help to accomplish this research.

7. Abbreviation List :

-XRD: X- Ray Diffraction.

- SEM : Scanning Electron Microscope.

- EDAX: Energy-dispersive X-ray spectroscopy.

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