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ARCHAEOMETRIC INVESTIGATION TO EVALUATE ACRYLIC, SILICON MATERIALS AND NANO-ADDITIVES AS CONSOLIDATION MATERIAL TO SANDSTONE MONUMENTS OF THE SPHINXES AVENUE (LUXOR, EGYPT)

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ABSTRACT

This research aims to shed light on an experimental study to evaluate acrylic, silicone materials and nano additives used to consolidate the sandstone with an application to a statue in Sphinxes Avenue at Luxor, Egypt. Samples of sandstone were taken from one of the statues of Sphinxes Avenue to determine their components and current status by X-Ray Diffraction analysis (XRD), scanning electron microscope's examination (SEM) and EDAX elemental analysis to choose the standard sample which should be very close to the sandstone of the statue. Six materials were selected to the experimental study; two of them belong to acrylic group (Addicon and Paraloid B72), Kemtekt and Wacker (OH 100) which are silicone materials, Nano silica which is an additive material to Paraloid B72 and Wacker (OH 100). The selection of the best material by the evaluation of visual examination, measuring physical properties such as density, porosity and water absorption as well as mechanical properties (compressive strength) of standard and treated samples in addition to examination by scanning electron microscope (SEM).

The treated samples were subjected to artificial aging (thermal and salt weathering) and the results were also evaluated by previous methods before weathering, the best consolidation material is Wacker (OH 100)+ Nano silica which achieved the best results comparing to the other materials in physical and mechanical properties and scanning electron microscope 's examination. It's found that high temperatures have good effect on some hydrophobic materials like Siloxanes which make them Superhydrophobic, also nanoparticles improve the performance of some consolidation materials like Wacker (OH 100) and Paraloid B72.

KEYWORDS: Experimental Study, Acrylic, silicone, consolidation materials, nano additives, physical and mechanical properties, Sphinxes Avenue, Sandstone.

1. INTRODUCTION

Luxor is located in the southern Upper Egypt region between latitudes 25-36 north and 32-33 east and is located 670 south of Cairo. It also contains a third of Egypt's antiquities, Luxor city consists of two parts: Eastern bank: It is called the city of neighborhoods in the Pharaonic eras, where the religious temples and palaces of kings and the common people. Western bank: It was called the city of the dead, where the tombs and funerary temples (Weeks, K.R., 2005).

In the late eras, the city of neighborhoods on the eastern bank of Luxor has almost disappeared, leaving only some simple archaeological features, the most important of which are the Karnak temples and the Luxor Temple, where the Luxor Temple is located about three kilometers north of the Karnak Temple. It was connecting the two temples by a sacred road known as the Sphinxes avenue. Which was established by King Nectanebo I, founder of the Thirtieth Dynasty. This road was later known as the Rams Road, The Sphinx Road (The Rams) is one of the largest ancient sacred roads built by the ancient civilization to link the two largest holy areas on the eastern bank of Luxor (Boraik. 2013) (Fig 1).

The statues of the Sphinx in the era of the eighteenth dynasty are characterized in their construction by the larger size of the statues than their counterparts in the era of the thirtieth dynasty, and the head of the statue takes the form of a ram (lamb's head) in the era of the eighteenth dynasty, which symbolizes the god Amun. While in the era of the thirtieth dynasty it takes the form of a head the statue is shaped like a Sphinx (the god of Sekhmet), as well as the size of the statue and the base is smaller than the era of the eighteenth dynasty. The base also contained cartouches in the name of the king (Boraik, 2013)(Fig 2).

Figure 1. The Sphinxes Avenue in Luxor

Figure 2. Sphinxes statues in Luxor

A. Study of the type of sandstone used in the construction of Sphinx statues

The road was built entirely of Nubian sandstone, which was brought from the quarries of Jabal al-Silsila and Masa'id, the same types of stones from which Luxor Temple and Karnak Temples were built.

The Nubian sandstone contains various hard iron nodules, shells and fossils, the stone's mineral composition includes "micaceous minerals", the stone is named in this case "micaceous sandstone" besides the micaceous minerals, there are gypsum crystals and pyrite crystals (FeS_2) which are considered the cement material for quartz crystals "the main mineral of the sandstone SiO_2 "silicone di- oxide".

The Jabal al-Silsila quarry is a series of mountains consisting mainly of Nubian sandstone, which extends on both banks of the Nile for an estimated distance of about 800 km, and is located 30 km north of Aswan between Edfu and Kom Ombo, the thickness of the mountain is 140 m. Its stones are pink, brown and gray, and are characterized by their high porosity. There are writings of the names of kings indicating that this quarry has been used since the eighteenth dynasty, including Hatshepsut, Seti I, Ramses II, and Merneptah (Radi et al., 2019).

B. Acrylic, silicone consolidation materials and nano-additives used in the experimental study

Six of the consolidation materials were identified to be used in the experimental study, and it was taken into account in the selection process that they include different consolidants, where the consolidation materials included two materials from the silicone materials group, namely (Wacker OH100, Kemtekt), It also included two materials of the acrylic group consolidants, namely (Paraloid B72, Addicon). It also included two synthetic nanomaterials, to which nano silica was added to Wacker (OH100), and Paraloid

B72 to which nano-material was added. The following is a presentation of these materials.

1. Wacker (OH 100)

Wacker is one of the Tetra Ethoxy Silane (TEOS) compounds (Doehne, E., & Price, AC, 2010), and it is one of the chemical compounds that are used to strengthen ancient stones with a high penetrating power (Malaga, K. et al,) (Abdel Kader, RR & El-Sayed, SSM, 2019)

It was efficiently used to strengthen and isolate archaeological stones with low porosity in the form of consolidation cycles with the need to wait until each consolidation cycle dries (El-Sayed, SSM., 2012), so it was chosen in the processes of consolidation and isolation of the stone objects.

2. Kemtekt

Kemtekt is a transparent silicone-based consolidation material that has a low viscosity, as it quickly absorbs into the pores without forming a clear-coloured film or film, the material works to prevent water droplets from accumulating and expelling them from surfaces, which helps prevent the absorption of rainwater or ground water. It is economical, recyclable and easy to apply (El-Sayed, SSM., 2016).

3. Paraloid B72

Paraloid B72 is a class of acrylic resins. It is a solid transparent crystals that can dissolve in many solvents including toluene, xylene, acetone and trichloroethylene. It is characterized by its tolerance of high and low temperatures and combines all the advantages of acrylates, one of its disadvantages is that when the humidity rises by a large percentage, fungi can grow on it (Horie, C.V., 1998).

4. Addicon

Addicon is also an Egyptian commercial product, it is an acrylic resin. It consists of Dimethyl Metacrylate; it is transparent and is stable in moisture and weather conditions. Drying time is from 2-4 hours and density is 1.01 kg / liter. It is applied to a clean surface either by brush or by spraying.

5. Wacker (OH 100) reinforced with silica nanoparticles

Composite materials are prepared where nanoparticles (reinforcement materials) are added to the polymeric medium (base material), and these materials are mixed well with each other to obtain a homogeneous nanocomposite material, characterized by good properties, which are not available in these materials when used in a single way.

Wacker (OH 100) nanoparticles supported material is a mixture of silica nanoparticles suspended or dispersed in Wacker (OH 100) compound. This material was prepared by adding silica nanoparticles to Wacker (OH 100), at a concentration of 2% w/v, and then mixing these components well, until the silica nanoparticles are homogeneously dispersed in the polymer (Manoudis, P., et al., 2009).

6. Paraloid b72 reinforced with silica nanoparticles

It is a mixture of nano-silica particles suspended or dispersed in Paraloid B72 dissolved in trichloroethylene at a concentration of 5%, this material was prepared by adding silica nanoparticles to Paraloid B72, at a concentration of 2% w/v, and then mixing these components well, until the silica nanoparticles were homogeneously dispersed (Manoudis, P. et al., 2009) (Ntelia, E., and Karapanagiotis, I., 2020).

This Research aims to shed the light on evaluating acrylic and silicone materials which are used in the consolidation of sandstone statues in sphinxes avenue at Luxor, Egypt and comparing them before and after adding nanoparticles to highlight the role of them in improving performance of the selected consolidation materials.

2. MATERIALS AND METHODS

A sample was taken from a statue at sphinxes avenue to identify the components of sandstone by analysis by X-ray diffraction and examination by scanning electron microscope in addition to the elemental analysis by EDAX (Fig 3,6) (Table 1).

A. Preparation of sandstone samples for experimental study

Through the study that took place on sphinxes avenue in Luxor, it was found that most of the stones used in its construction were sandstones brought from Jabal Al-Silsila quarry, so a group of sandstone blocks were brought from the same quarry, then they were refined and cut into regular cubes with dimensions 3 cm x 3 cm x 3 cm, to be used in the experimental study (Fig 7).

B. Determination of the physical and mechanical properties of the experimental samples

The physical properties of the experimental samples of sandstone are studied and determined before and after the consolidation processes, in order to identify the impact of the consolidants on the properties of the treated sandstones, as well as a comparison between the different consolidation materials to choose the most appropriate material, among the most important physical properties that must be studied and tested, Density, Porosity, and Water absorption. Compressive strength is one of the mechanical properties that must be tested for sandstone samples before and after consolidation processes.

C. Consolidation processes of the experimental samples

The previous six consolidation materials were applied to the sandstone cubes used in the laboratory:

1- The sandstone cubes are equipped with sizes (3 cm x 3 cm x 3 cm).

2- Preparing the consolidation materials that will be used in the consolidation at a concentration from 2-5%.

3- Drying the cubes in an oven at a temperature of 105°C for 24 hours to ensure dryness and weight stability.

4- Measuring the physical and mechanical properties before consolidation.

5- The consolidation process was carried out by two methods: immersion of samples in consolidants and brushing, then leaving the samples for 24 hours and repeating the process for three consecutive days, taking into account starting with the lowest and then higher concentrations in the diluents, and then leaving the samples in the ordinary room's temperature covered with polyethylene for three weeks, which is the time sufficient to complete the polymerization process (Table 2).

6- Measuring the physical and mechanical properties after consolidation.

Table 2. The consolidation materials and samples' symbols

Symbol of samples	Consolidation Material
A	Wacker (OH 100)
B	Kemtekt
C	Paraloid B72
D	Addicon
E	Wacker (OH 100) reinforced with Silica Nanoparticles
F	Paraloid B72 reinforced with Silica Nanoparticles

Figure 3. A statue where the samples were taken (as arrow refers)

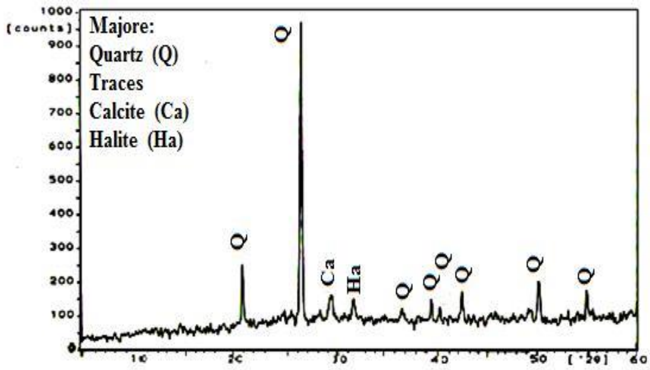

Figure 4. X-Ray Diffraction pattern of the sand stone's sample taken from the statue

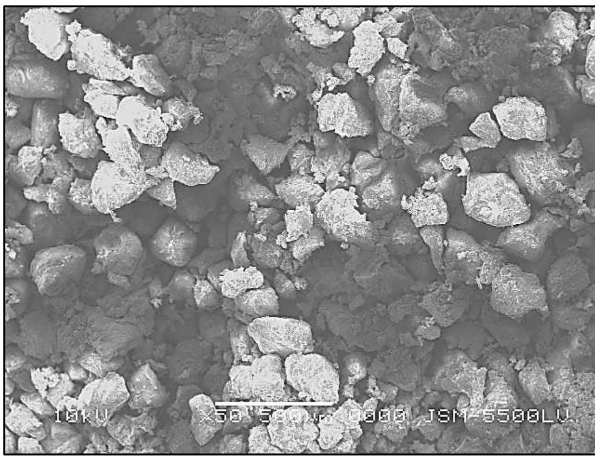

Figure 5. Scanning Electron Microscope (SEM) Examination of the sandstone's sample (500X)

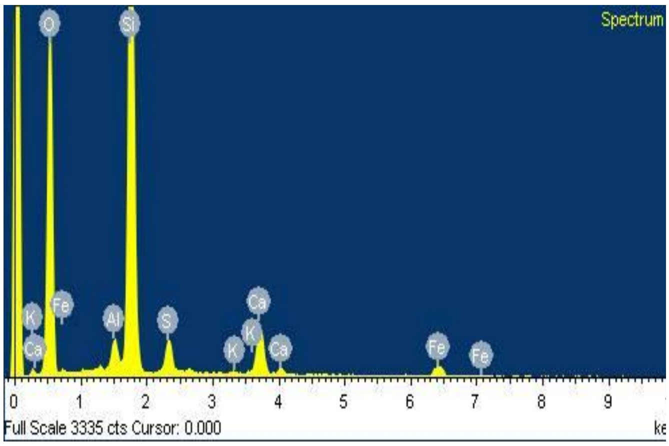

Figure 6. EDAX elemental analysis of the sandstone's sample

Table 1. Elements of the sandstone's sample (EDAX analysis).

Element	Weight%	Atomic%
O K	37.66	53.16
Al K	1.35	1.13
Si K	46.15	37.11
S K	3.31	2.33
K K	0.55	0.32
Ca K	9.53	5.37
Fe L	1.46	0.59
Totals	100.00	

Figure 7. Preparation of the experimental samples

D. Evaluating of the suitability and efficiency of materials used to consolidate sandstone samples (before artificial weathering)

The validity and efficiency of the materials used to strengthen the sandstone samples were evaluated by measuring the effect of the various consolidation materials on the physical and mechanical properties of the samples, as well as through visual examination and scanning electron microscope (SEM) to determine the extent of the ability of these materials to bond between sandstone grains and their efficiency in penetration and spread between these grains.

1. Visual examination

The results varied in terms of the general appearance of the sandstone samples after the consolidation process, as there were materials that had no effect, while other consolidation materials had a slight effect on the general appearance as shown in the following table (Table 3) (Fig 8).

Table 3. Visual examination (effect on appearance) of the consolidated samples

Effect on appearance	**Consolidation Material**	**Symbol of samples**
No surface changes	Wacker (OH100)	A
Slight color change	Kemtekt	B
The appearance of a weak luster	Paraloid B72	C
Intense darkening and glossy	Addicon	D
No surface changes	Wacker (OH 100)+Nano silica	E
Darkening and pallor	Paraloid B72 + Nano silica	F

A B C D E F

Figure 8. The consolidated samples after drying

2. Measuring of physical and mechanical properties after consolidation processes

Physical properties like density, water absorption and porosity are measured for the untreated and treated samples to evaluate the used consolidation materials (Table 4, Fig 9).

Table 4. Values of physical properties of the consolidated samples

Porosity %	Water Absorption %	Density g/cm³	Consolidation Materials	Symbol of samples
25.80	18.15	1.65	Untreated sample	-
2.85	1.25	1.74	Wacker (OH100)	A
15.70	1.23	1.54	Kemtekt	B
17.20	9.15	1.52	Paraloid B72	C
13.80	10.47	1.85	Addicon	D
2.80	1.20	1.64	Wacker (OH 100)+Nano silica	E
14.30	9.10	1.55	Paraloid B72 + Nano silica	F

A B C D E F

Figure 9. The effect of consolidation materials on water repellent properties of sandstone samples.

3. Measuring of mechanical properties

The mechanical properties of the treated samples were determined and compared with an untreated sample in order to identify the ability of consolidation materials to improve the mechanical properties of the treated samples (Table 5).

4. Scanning electron microscope (SEM) examination

Examination by scanning electron microscope (SEM) of the samples treated with the consolidation materials to identify the extent of the spread and penetration of the hardener between the grains and components of the stones and to evaluate its efficiency in the consolidation processes (Fig 10, 15).

Table 5. The compressive strength values of the untreated and treated samples

Compressive strength Kg/cm²	Consolidation materials	symbol
107	Untreated sample	———
204	Wacker (OH 100)	A
187	Kemtekt	B
190	Paraloid B72	C
199	Addicon	D
230	Wacker (OH 100)+Nano silica	E
200	Paraloid B72+Nano silica	F

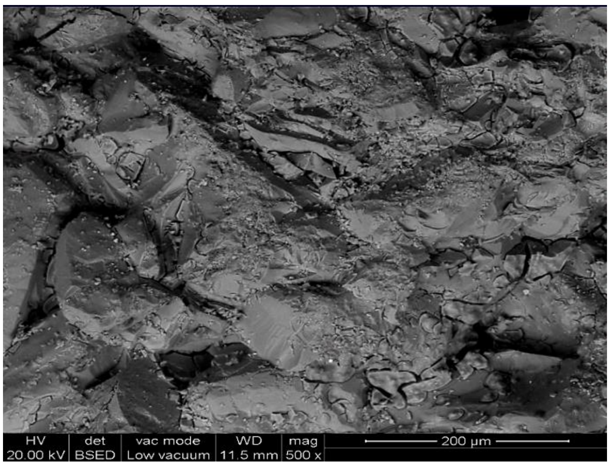

Figure 10. The SEM examination of the sample treated with Wacker (OH 100) (500X).

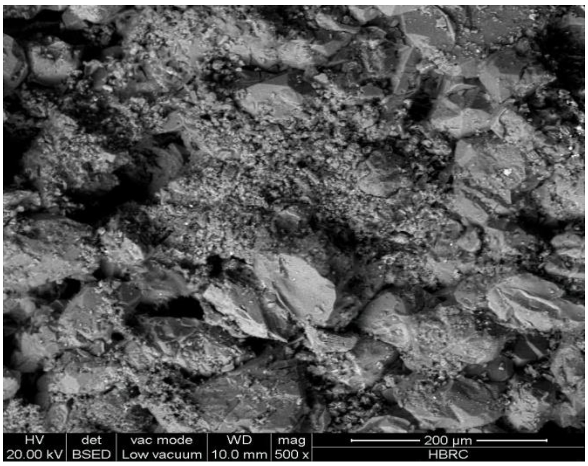

Figure 11. The SEM examination of the sample treated with Kemtekt (500X).

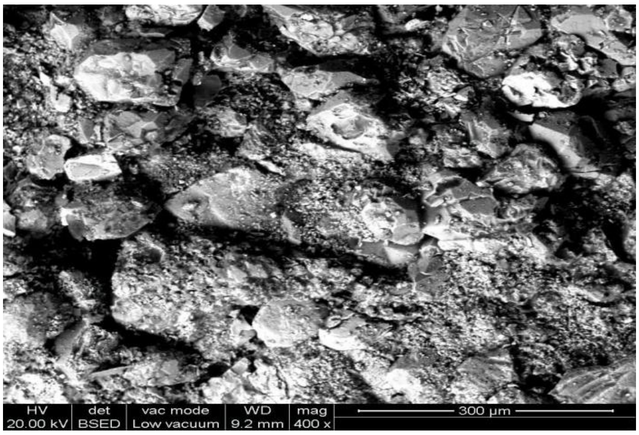

Figure 12. The SEM examination of the sample treated with Paraloid B 72 (500X).

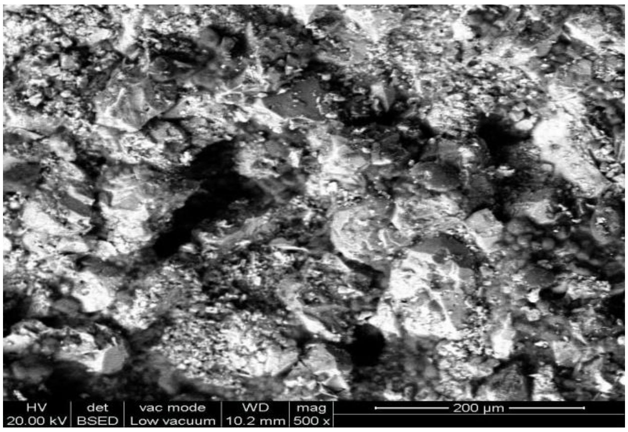

Figure 13. The SEM examination of the sample treated with Addicon (500X).

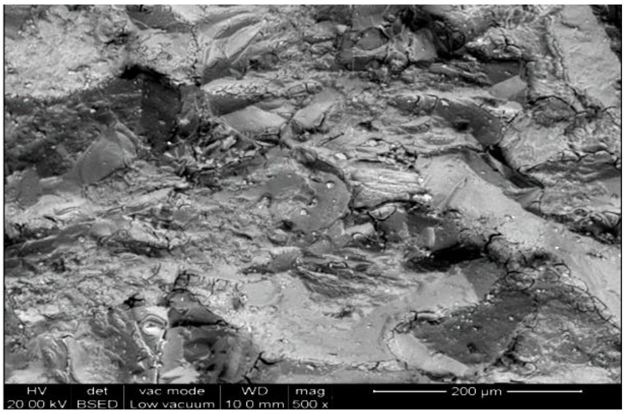

Figure 14. The SEM examination of the sample treated with Nano silica+ Wacker (OH 100) (500X).

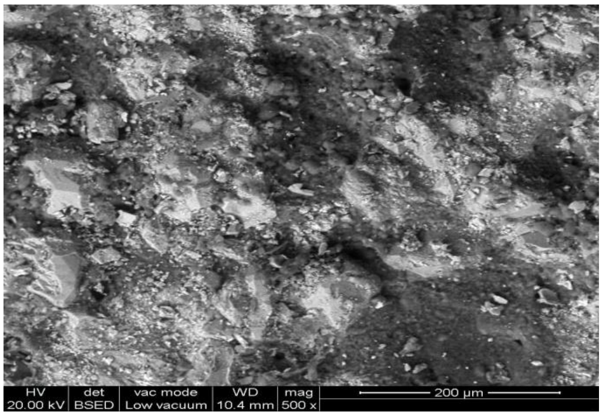

Figure 15. The SEM examination of the sample treated with Nano silica+ Paraloid B72 (500X).

E. Artificial aging of the consolidated samples (weathering)

The importance of artificial weathering tests lies in the fact that they represent the process of keeping pace with the environmental conditions surrounding the antiquity, especially that open environment that is characterized by variable weather factors, very complex and sharp, as is the case in the Sphinxes Avenue, in addition to the importance of these tests in identifying the success of the consolidation materials used in the experimental study. And the performance of its role under those different environmental conditions, and therefore the possibility of comparison among them to choose the best and most stable one in consolidating and protecting the treated samples. These tests were carried out as follows:

1. Expose the samples to wet and dry heating cycles:

This test was carried out by following the following steps:

- Dry the samples in a drying oven for 24 hours at a temperature of 105°C, then record their weights.
- Submerge the samples in water and then place them in a drying oven at a temperature of 60°C for 5 hours.
- Put the samples while they are dry in the drying oven at a temperature of 60° C for 10 hours.
- Cool the samples at room temperature for 15 hours. This process was repeated for ten consecutive cycles.

B. Thermal weathering

The samples were immersed in a solution of sodium chloride at a concentration of 25% for 4 hours, then the samples were dried in an oven at a temperature of 45° C for 8 hours, then the samples were left at room temperature for 12 hours. This process was repeated for ten successive cycles. The effect of salt weathering on treated stones was also evaluated.

F. Evaluating of the suitability and efficiency of materials used to consolidate sandstone samples (after artificial weathering)

The evaluation process was carried out in the same previous methods after artificial weathering by visual examination and measurement of physical and mechanical properties in addition to examination by SEM microscope (Table 6,9- Fig 16).

Table 6. The density values of the treated samples after aging

Density g/cm³	Consolidation materials	Symbol
1.50	Untreated sample	——
1.55	Wacker (OH 100)	A
1.53	Kemtekt	B
1.50	Paraloid B72	C
1.52	Addicon	D
1.58	(Wacker OH100) +Nano	E
1.51	Paraloid B72+Nano	F

Table 7. The rate of change in porosity after weathering

%Porosity	Consolidation materials	Symbol
14.80	Untreated sample	——
10.70	Wacker (OH 100)	A
3.44	Kemtekt	B
12.80	Paraloid B72	C
3.50	Addicon	D
4.70	Wacker (OH100) +Nano	E
13.20	Paraloid B72+Nano	F

Table 8. Water absorption rate and rate of decrease in absorption

Water % absorption	Consolidation material	Symbol
10.80	Untreated sample	——
7.10	Wacker (OH 100)	A
2.84	Kemtekt	B
8.95	Paraloid B72	C
1.80	Addicon	D
7.0	Wacker (OH100) +Nano	E
8.90	Paraloid B72+Nano	F

Table 9. Compressive strength values of the treated samples after aging

Compressive strength (Kg/cm²)	Consolidation materials	Symbol
95	Untreated sample	——
195	Wacker OH 100	A
171	Kemtekt	B
132	Paraloid B72	C
167	Addicon	D
222	Wacker+ Nano (OH100)	E
185	Paraloid B72+Nano	F

- **Scanning electron microscope examination after artificial aging**

The examination is conducted using the scanning electron microscope for treated samples after exposing artificial weathering courses to see the extent to which they are affected or reflected in different deterioration factors, and therefore we can identify the materials that are given the best results in the process of consolidating and preserving of stones (Fig 17,22).

A B C D E F

Figure 16. The appearance of the treated samples and their water repellence after weathering operations

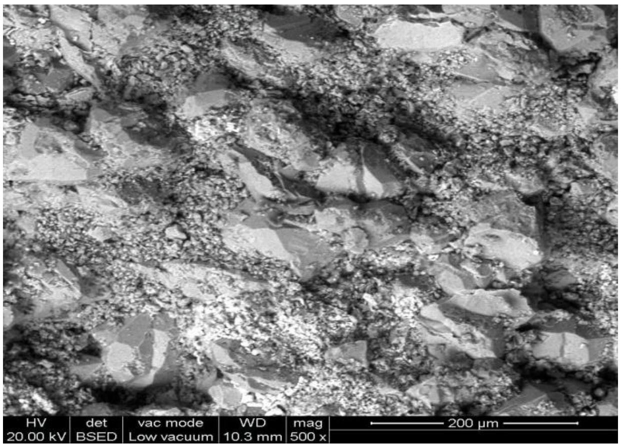

Figure 17. The SEM examination of Wacker (OH 100) after weathering (500X).

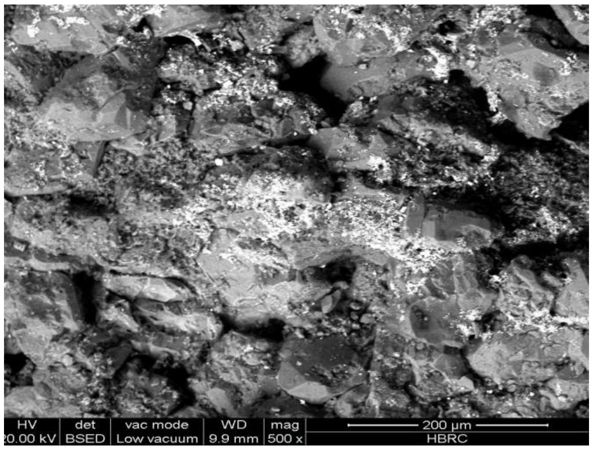

Figure 18. The SEM examination of Kemtekt after weathering (500X).

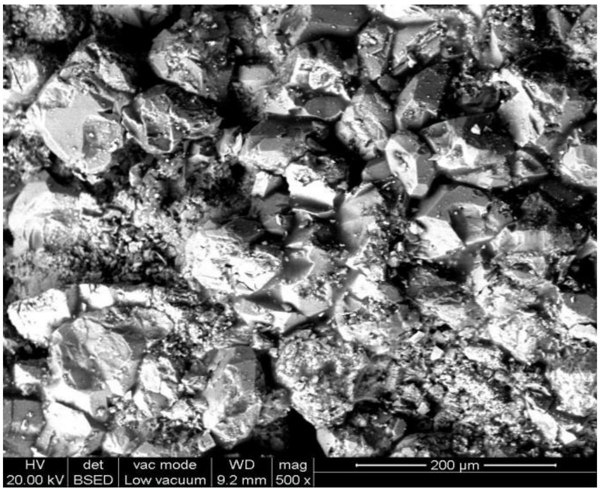

Figure 19. The SEM examination of Paraloid B72 after weathering (500X).

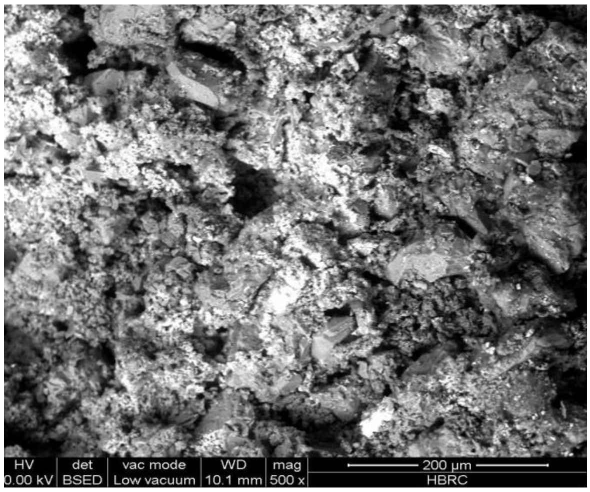

Figure 20. The SEM examination of Addicon after weathering (500X).

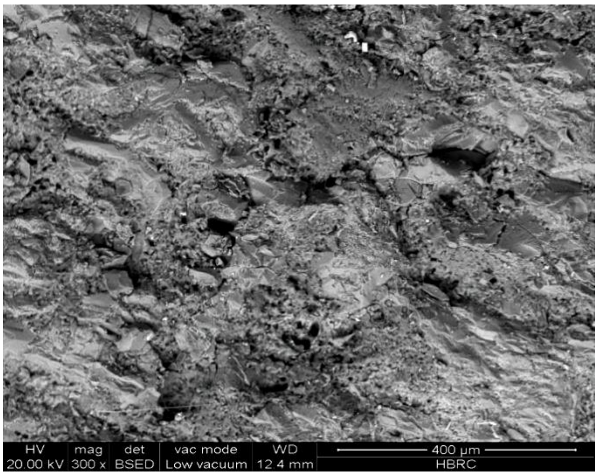

Figure 21. The SEM examination of Nano silica+ Wacker (OH 100) after weathering (500X).

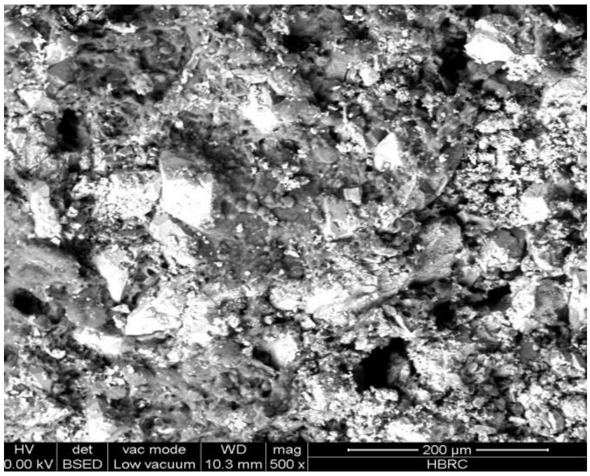

Figure 22. The SEM examination of Nano silica+ Paraloid B72 after weathering (500X).

3. RESULTS AND DISCUSSION

The results of the examination and analysis of the sandstone sample taken from one of the statues in Sphinxes avenue showed that the sandstone consists of quartz by 85% in addition to the presence of calcite by 8% and halite by 7%.

The scanning electron microscope's examination shows the presence of gaps and weakness in the inner tissue of the sandstone's sample surface.

The result of the elemental analysis (EDAX) of the sandstone sample taken from the selected statue was that it consists of silicon (Si) by 46.15%, Oxygen (O) by 37.66%, Calcium (Ca) by 9.53%, sulfur element (S) by 3.31%, Iron element (Fe) by 1.46% and Potassium element (K) by 0.55%. As shown in (Table 1) and (Fig.6).

By calculating the average values of the physical and mechanical properties of the untreated experimental samples (standard samples), it was found that the average value of density was 1.5 g/cm^3, porosity 14.70%, and water absorption 10.6%, while the average value of compressive strength was 105 kg/cm^2.

After the consolidation processes of the sandstone samples (before weathering), it was noticed through the visual examination that the materials Wacker (OH 100) and Wacker (OH 100) + Nano silica did not show any changes in color on the surface of the sample and there was no glossiness while the other treated samples had changes in color and glossiness appearance so they were excluded from the following comparisons.

By measuring the physical properties as a density, porosity and water absorption show that Wacker (OH 100), Wacker (OH 100) + Nano silica is one of the best materials where the density was recorded in the sample of Wacker (OH 100) 1.74 g/cm^3 and sample of Wacker (OH 100) + Nano silica 1.64 g/cm3, the porosity in Wacker (OH 100) sample 2.85% and Wacker (OH 100) + Nano silica 2.80%, while the proportion of water absorption of Wacker (OH 100) 1.25% and Wacker (OH 100) + Nano silica 1.20% which explains the benefit of these materials in isolation of the sphinxes statues beside their consolidation.

Through measurement of compressive strength, Wacker (OH 100) sample was recorded 204 kg/cm^2 while the sample of Wacker (OH 100) + Nano silica was recorded 230 kg/cm^2.

The examination using scanning electron microscope for Wacker (OH 100) shows its success in good and homogeneous proliferation between stone granules and fully packed, and some accurate crackdowns were found in parts of the polymer filled with relatively large paramedics, and the polymer was highly observed density in some places while the examination for Wacker (OH 100) + Nano silica showed the success of the material in reducing the proportion of water absorption and porosity for treated stone samples and thus achieving isolation function and is illustrated by its good packaging for granules and linking them to all spaces and gaps in stone's texture.

Through the procedure of artificial aging (thermal and salt weathering), it was found that there weren't any changes in color and appearance of the treated samples of Wacker (OH) 100 and Wacker (OH 100) + Nano silica, the values of the physical properties of the samples differed after weathering from before weathering, as well as the mechanical properties (compressive strength), Where the values of density 1.55 g/cm^3, porosity 10.70%, and water absorption 7.10% of Wacker (OH 100) sample after weathering, while Wacker (OH 100) + Nano silica recorded density 1.58 g/cm^3, porosity 4.70%, and water absorption 7.0%.

The compressive strength value was 195 kg/cm^2 for Wacker (OH 100) while it was 222 kg/cm^2 for Wacker (OH 100) + Nano silica sample.

In order to obtain an integrated evaluation of the treated samples, the samples were examined after weathering using a scanning electron microscope (SEM), the examination with SEM showed for sandstone samples treated with Wacker (OH 100), a slight fading of the polymer material in some parts and the appearance of some salts on the surface sporadically.

Sandstone's sample treated with Wacker (OH 100) + Nano silica gave the best result, as well as showed a large degree of stability for weathering cycles, and also showed a high degree of resistance to artificial aging.

Through the previous results, it was found that the Wacker (OH 100) material added to it Nano-silica is the best experimental material ever, which can be used to consolidate the deteriorated sandstone statues by the Sphinxes avenue, as it has the property of repelling water and thus doing the isolation next to the consolidation, followed by the Wacker (OH 100) material in the second place, as for the other consolidation materials Paraloid B-72, Kemtekt, Addicon, and Paraloid B 72 with nano-silica additives were excluded from the beginning, as color changes appeared in the treated samples with the occurrence of glossiness and were less in measuring the physical and mechanical properties compared to the selected samples in addition to assessing their efficiency using a scanning electron microscope (SEM).

It was also noted that the addition of nano-materials to acrylic and silicate compounds improved their properties in consolidation processes. It was noted that the results of adding nano-silica to Wacker (OH 100) were better than Wacker (OH 100) alone, and adding nano-silica to Paraloid B72 gave better results than Paraloid B72 alone.

Nanoparticles are used to improve the mechanical and the wetting properties of OH 100 and Paraloid B72. The addition of the nanoparticles is a standard method to enhance the hydrophobic character of the aforementioned polymers (Karapanagiotis, 2015) (Chatzigrigoriou et al., 2013) (Chatzigrigoriou et al., 2020).

It is demonstrated that the Superhydrophobic and water-repellent siloxane surfaces exhibit self-cleaning properties, good durability, and furthermore do not practically affect the optical transparency of glass substrates after thermal weathering, the siloxane materials, also transform from hydrophobic to superhydrophobic in high temperatures (Karapanagiotis et al, 2014).

4. CONCLUSION

Consolidation is a very important process to save the deteriorated statues in Sphinxes Avenue at Luxor, six materials used to consolidate stones were chosen, tested and evaluated to reach the best selected material to conserve the chosen statue (Wacker (OH 100) + Nano silica where the best results were recorded in visual examination, physical and mechanical properties measurement and scanning electron microscope examination (SEM).

This material consists mainly of silica, which is the same as the sandstone component and it has achieved an important condition of consolidation (convergence in the components and physical and mechanical properties).

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