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NANOSTRUCTURED MATERIALS FOR STRENTHENING AND PRESERVATION OF HISTORIC STRUCTURAL MARBLE COLUMNS

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Abstract

One of the most durable building materials, stone has been used throughout history to build buildings. In particular, marble is the most common material in historical structures. For thousands of years, these materials have been valued for their beauty and versatility. Despite the permanent aura, marble can suffer from wear and tear. As a result of increased urbanization today in Rashid city, a high level of groundwater, salt accumulation and pollutions, marble degradation is occurring much faster than in the past. Accumulation of dust, cracked construction, spelling, and loss of structural integrity are among the problems affecting marble structures. There are many sources of marble deterioration, especially in the city of Rashid. Damage occurs through physical, chemical, and environmental weathering factors, air pollution, and soiling build-up. More damage can occur through corrosion, as a result of movement in marble (thermal expansion / shrinkage), damaged or broken impact points, and even a human error in design, engineering, or construction. The experimental group study was conducted on samples of historical marble using some traditional reinforcing materials and some nano materials to reach the best one in the treatment and maintenance of historical structural marble. The evaluation methodology was performed using a set of stress analysis of the treated sample and compared to the untreated sample or the clear sample that was not exposed to the treatments. Several parameters such as marble resistance point were measured using ultrasonic velocity testing, and mechanical properties, as well as physical properties of the sample including fine porosity, were determined The results proved the superiority of the samples that have been strengthened by Nano Restore with EucoColle materials in 50% concentration and diluted in water by 1:1 ratio. Finally, the present study involved strengthening and preserving some of the historical structural marble columns inside the Al-Mahaly Mosque in the city of Rashid.

***Keywords**: Nanostructured materials; Consolidation; Marble columns; Preservation; Architectural heritage; Marble restoration.*

Introduction

In recent times, nanomaterials have been applied in the construction and maintenance of the world´s cultural heritage with the aim of improving the consolidation and protection treatments of damaged stone. These nanomaterials include important advantages that could solve many problems found in the traditional interventions. The present paper aims to study the state of art on the application of nanotechnology to the conservation and restoration of the structural marble columns. We highlight the different types of nanoparticles currently used to produce conservation treatments with enhanced material properties and novel functionalities.

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The use of nanotechnology applied to the marble heritage conservation field creates possibilities to produce conservation treatments with enhancing material properties and novel functionalities. These nanomaterials include important advantages that could solve many problems found in the traditional interventions through the development of new nanomaterials or the improvement of the traditional treatments with the incorporation of nanoparticles. In this way, as has been described in the work, innovative applications have reached by using nanoparticles in building materials such as marble deterioration and preservation. While the development in material science has generated important nanostructured materials long time ago, conservation of cultural heritage was, until recently, mainly based on the traditional conservation and restoration treatments. Recently, nanomaterials are being applied in the construction and maintenance of the architectural heritage with the aim of improving the consolidation and protection treatments of damaged marble columns. Firstly, Baglioni et al were pioneering in the application of nanoparticles for the cleaning and consolidation of mural paintings [1]. Later, the application of nanoparticles to stone heritage started through works of *Dei and Salvadori* [2] and *Manoudis et al.* [3]. Subsequently, the nanoparticles specifically designed for cleaning, consolidation, water repellence, as well as for antimicrobial and anti-graffiti treatments have been developed.

Consolidation is one of the most important conservation treatments carried out on stone heritage. This treatment is used to improve the cohesion of weathered stone when serious decay patterns and in depth cohesion loss are present. Consolidation treatments are the most risky conservation actions due to their irreversibility and the likeliness to cause undesired effects, such as the possible loss of the surface that was supposed to be preserved. This risk justifies the numerous studies that have been developed on stone consolidants. The application of nanotechnology in the cultural heritage conservation is marked by the possibility to design consolidant products highly compatible with the original stone substrate. Moreover, when particles have dimensions of about 1÷100 nanometers, the materials properties change significantly from those at larger scales. In this sense, nanomaterials have larger surface areas than similar masses of larger-scale materials, which increase their chemical reactivity. In addition, these nanomaterials present the possibility to penetrate deep into the damaged stone materials due to the particle size.

Since ancient times, these properties are known in different elements: Ag, Ti, Cd, Fe, Pd, Zn, Pt, Co, etc. The nanoparticles must have the following attributes: stability and sustained photoactivity, biologically and chemically inert, nontoxic, low cost, suitability towards visible or near UV light, high conversion efficiency and high quantum yield, could be react with wide range of substrate and high adaptability to various environment and good adsorption in solar spectrum [4]. In addition, these treatments can also have water repellent properties which favor this self-cleaning action [5, 6], and prevent the generation of damage caused by water. The nanostructured particles can provide not only hydrophobic and repellence properties to the stone substrate but also super-hydrophobic properties [7], which generate water contact angle greater than 150°. The nanoparticles can also be used as additives in construction materials or to modify the synthetic polymers in order to enhance its outdoor performance, and its mechanical and thermal properties [8]. Moreover, capsules with high initial flexibility are even used for self-healing concretes. On the other hand, the presence of soluble salts is recognized as an important decay agent of stone heritage. Thus, in the last few years, the study of the application of nanoparticles as a de-sulphating agent for stone, mortars and wall paintings are being carried out.

Finally, it can be pointed out that the study of the effectiveness, compatibility and durability of these new nanomaterials are necessary in order to avoid the use of inadequate treatments [9, 10], which modify the aesthetic, physical and chemical properties of marble materials, causing new pathologies. Also, the knowledge of the industrial production, the capacity utilization, and the price of raw materials are important aspects need to be considered.

The human health risks and environmental implications resulting from the use of the new nanomaterials should be taken into account when designing treatments based on nanoparticles, [11].

Figure 1 show the present state of preservation of the main entrances of Al Mahalli Mosque. Figure 2 illustrate the effect of ground water on the marble columns inside Al Mahalli Mosque, and the observed in and out of plane deformations of the structural marble columns. Figure 3 illustrates the decomposition of marble building materials and the permanent deformation of the structural mortar between the column parts.

Fig. 1. The present state of preservation of the main entrances of Al Mahalli Mosque. rick disintegration due to the ground water rise and salt accumulation is obvious

Fig. 2. Degradations and deteriorations of columns: *left* - ground water rising above the surface of the earth and the growth of algae within the mosque; *center* - staining and of soot layers cover some of the marble columns; *right* - intensive erosion of the surface layer

Methodology

X-Ray Diffraction Analysis

X-ray diffraction (XRD) test method was carried out on marble samples from the mosque. XRD technique using a PW1050/70 diffractometer (Philips, Holland) was recorded with a diffractometer with Cu-Kα radiation generated at 40 KV and 40mA. It covers 2Θ from 5^o to 50^o. Figure 4 shows XRD for the marble sample. The diffractometer shows that this sample composed of calcite and halite. Figure 4 and Table 1 show the percentage chemical compounds of mortar.

Fig 3. The pillars of the mosque and its abyss show signs of deterioration and imbalanced

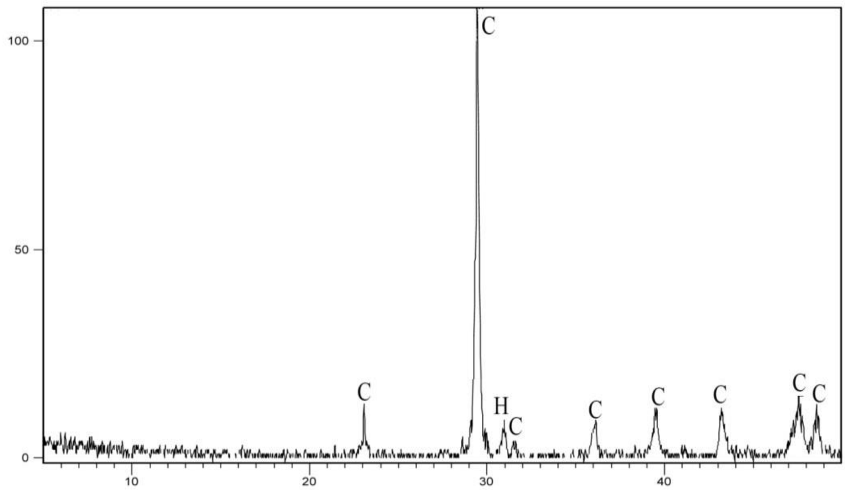


Fig. 4. XRD pattern for the marble sample

Table 1. The percentage chemical compounds of marble sample

Name of minerals	Chemical formula	%
Calcite	$Ca\ CO_3$	91,71
Halite	Na Cl	7,44

Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) model Inspect S (FEI Company) was used to investigate and analysis the marble sample. The SEM machine was equipped with an energy dispersive X-ray analyzer (EDAX). Figure 5 shows micro photograph of marble sample (control sample; U) with a magnifying force of 1000× for the marble sample, which was taken from one deteriorated structural marble columns in the mosque. It shows cracks and gaps as well as calcite crystals salts crystallized between them and on them. Also Figure 5 shows the elements analysis by EDAX for the control sample, it confirms that the general component of the sample is calcium, CaO (27.69%) and carbon, C (23.90%).

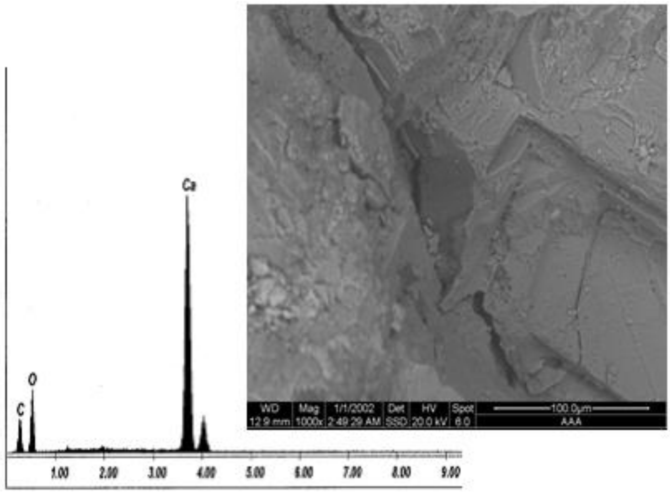


Fig. 5. EDX spectrum and microstructure of marble control sample.

Thin section analysis by Polarizing Light Microscope

Polarizing Microscope model N. Kon Eclipse LV 100 PoL was used for investigation. Figure 6 shows a photographic for the marble (control sample; U) Marble samples are consisting of rough crystallization calcite granules. The size of these granules varies between small and large, which are bound and overlapped with each other during the transformation and give mosaic texture.

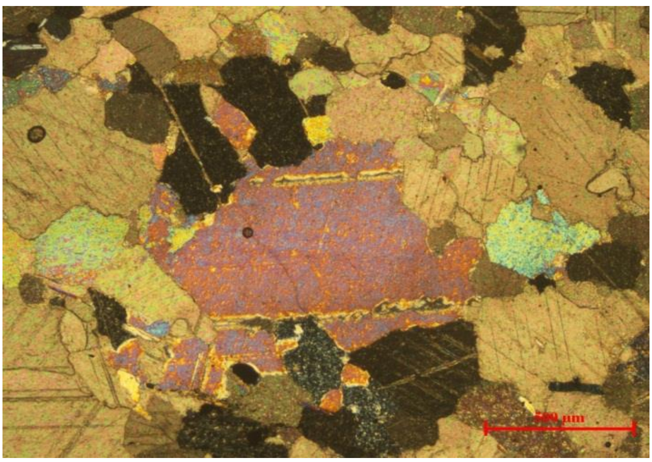


Fig. 6. Thin section under polarizing microscopy of marble sample collected from the mosque.

Experimental

Samples Preparation

El-Mahalli mosque in Rashed city was chosen for this experimental study. Number of marble fragment throw on the ground due to the distortion of some parts of the column which exposed to serve factors in this site was collected. The specimens were cut to cubes of 4×4×4cm, and then dried in electric oven for 24 hours at 100°C (+/- 5°C). Sixty specimens were prepared and divided into 11 groups, each group were composed of three specimens. Figure 7 and 8 shows the specimens with their code number before and after the consolidation processes.

Consolidation material used

Consolidation process required one of the important proceed to valid coherence for the internal structure of the marble in historic buildings in Rashed city. Consolidation material takes into consideration its ability to improve the physical and mechanical properties and it's compatible to the nature of building material fabric. Two types of consolidation materials were used as summarized in Table 2.

Fig. 7. Marble specimens with their code number before consolidation treatment.

Fig. 8. The specimens with their code number after the consolidation treatment.

Table 2. The traditional and nano materials used for consolidation

	Traditional materials	Nano-materials
1.	Waker BS 15	Nano silica
2.	Waker SKM 550	Nano-titanum
3.	Estel 1100	Nano calcite
4.	Eucocolle	Nano restore

Asessment of consolidation materials. Mechanical properties of consolidation or treated samples.

Uniaxial Compressive Strength.

Three cubes samples of 4×4×4cm were used to carry out the uniaxial compression test of the control and the treated samples. Compressive strength carried out using 50 ton Italy Matest pressing machine with a loading rate of 6.5kN/min as the specimens. The pressure resistance (compressive strength) of each sample was calculated as follows in Equation (1).

$$\sigma_c = P/A = kg/cm^2 \qquad (1)$$

where: σ_c is the compressive strength in kg/cm^2; P = load in kg; A = area, in cm^2.

Table 3. The consolidation materials used in each treatment

No.	Sample Symbol	The consolidation materials used
1	U	Control sample
2	A	EucoColle materials with 25% concentration in water
3	B	(Waker Bs15) Bs15 materials with 20% concentration in water
4	C	(Waker SKM 550) SKM 550 with 10% concentration in water
5	D	Estel 1100
6	E	Nano Silica with 3% concentration in alcohol
7	F	Nano Silica with 2% concentration in (Waker Bs15) Bs15 materials with 20% concentration in water
8	G	Nano restore with 50% with EucoColle materials with 50% concentration and diluted in water by 1;1 ratio in water
9	H	Nano Titaniun with 2% in Estel 1100
10	J	Nano Silica with 1.5% concentration and Nano Titaniun with 1.5% with (Waker BS15) BS15 materials with 20% concentration in water
11	K	Nano Silica with 2% concentration with (Waker SKM 550) SKM 550 with 10% concentration in water

Figure 9 shows the average Compressive Strength for three samples for each treatment. The figure shows that sample with code number G treated with Nano restore with 50% and EucoColle materials with 50% concentration and diluted in water, gives the highest σ_c, where its value is $629.23 kg/cm^2$ which increase the control one by about 23.59% which give $509.18 kg/cm^2$.

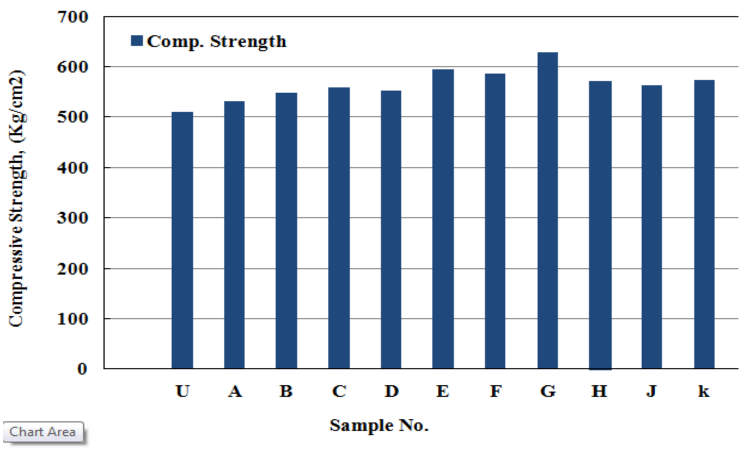


Fig. 9. The average compressive strength of samples after consolidation

Ultrasonic Pulse Velocity (UPV)

Wave velocity through materials gives indication about the quality of building materials by the way of waves flow. The velocity of waves flow of ultrasonic through any materials depend on two factors, one of them is internal factor depend on the type of materials, grain size, the fabric, the density and porosity of the material. The other factor is external factor depend on the temperature, porosity pressure of liquid and the stress on materials. For this test an ultrasonic tester model E4b was used, in which it consists of two microphones for transmit and receive waves connected with the main device. The tow microphone put on both side of the specimen to determine the time of the wave travel [12-16]. With the recent advancement in transducer technology, the test has been widely accepted in testing concrete materials. Ultrasonic testing of concrete is an effective way for quality assessment and uniformity, and crack depth estimation. The test procedure has been standardized as "Standard Test Method for Pulse Velocity through Concrete" (ASTM C 597, 2016).

Figure 10 shows the average wave velocity for three samples for each group. The figure shows that sample with code sample with code number G treated with Nano restore with 50% with EucoColle materials with 50% concentration and diluted in water gives the highest velocity of 6252m/s which increase the control one by about 39.99% which is 4266m/s.

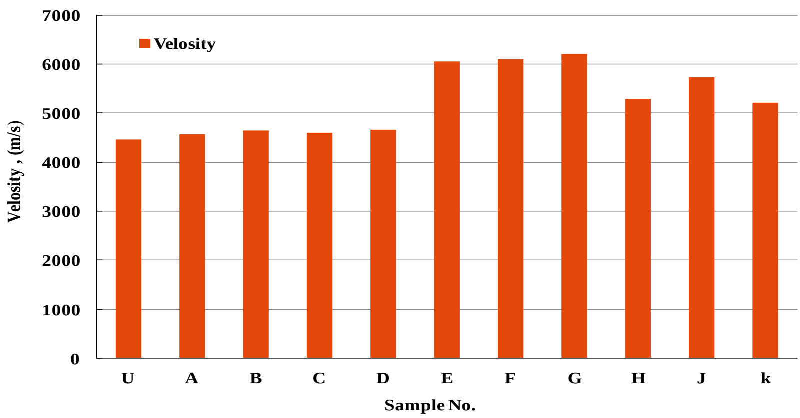


Fig. 10. The average wave velocity of samples after consolidation

Physical properties

The physical properties (bulk density, water absorption and apparent porosity) of these samples were determined by calculating the volume of each sample, measuring the dry weight and the wet weight of each sample. The physical properties were calculated as follows:

a) *Bulk density* (ρ) in gm/cm^3 was determined as follows in equation (ASTM C97/C97M − 15):

$$\rho = M/V = \ldots \text{ g/cm}^3, \qquad (2)$$

where: ρ is the density in g/cm^3, M is the Mass in g and V is volume in cm^3;

b) *Water absorption* (Wa) in % was determined as follows in equation (ASTM C97/C97M − 15):

$$Wa = 100 \cdot (W_2 - W_1)/W_1, \qquad (3)$$

where: Wa is the water absorption in %, W_1 and W_2 is dry and wet weight in grams;

c) *Apparent porosity* (η) in % was determined as follows in equation (ASTM C97-83):

$$\eta = 100 \cdot (M_2 - M_1)/V, \qquad (4)$$

where: M_1 is the Mass of the dried specimen, M_2 the Mass of the soaked specimen, and V the total volume of the sample.

Figures 11 shows the physical properties of samples. Figure 12 shows the bulk density in which sample with code number G treated with Nano restore with 50% with EucoColle materials gives the highest bulk density of 2.7g/cm^3 which increase the control one by about 5.46% which is 2.56g/cm^3.

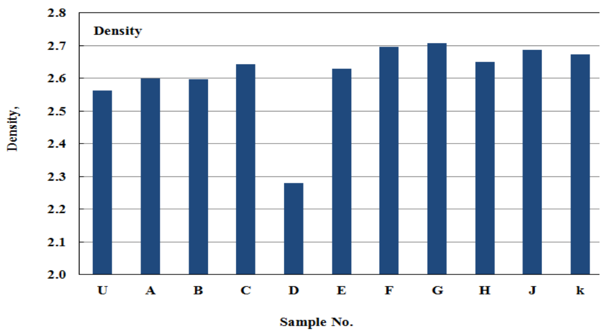


Fig. 11. The average bulk density of samples after consolidation

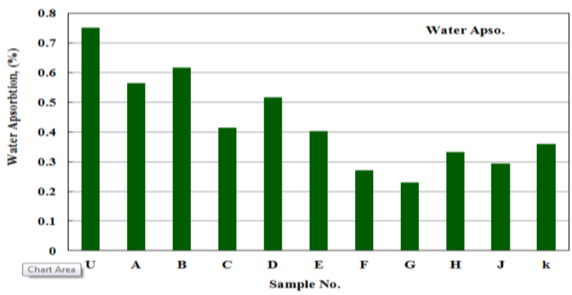


Fig. 12. The average water absorption of samples after consolidation

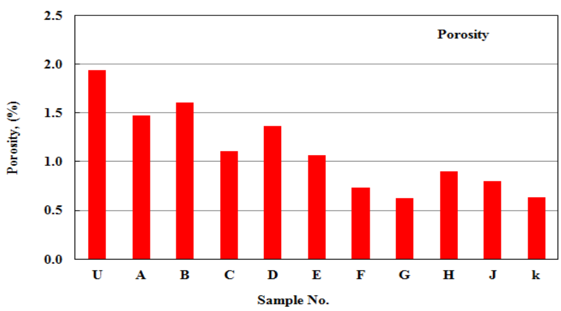


Fig. 13. The average apparent porosity of samples after consolidation

Figure 12 shows the water absorption in which sample with code number G treated with Nano restore with Nano restore with 50% with EucoColle materials with 50% concentration and diluted in water by 1:1 ratio in water gives absorption of 0.23% which decrease the control one by about 69.33% which is 0.75%.

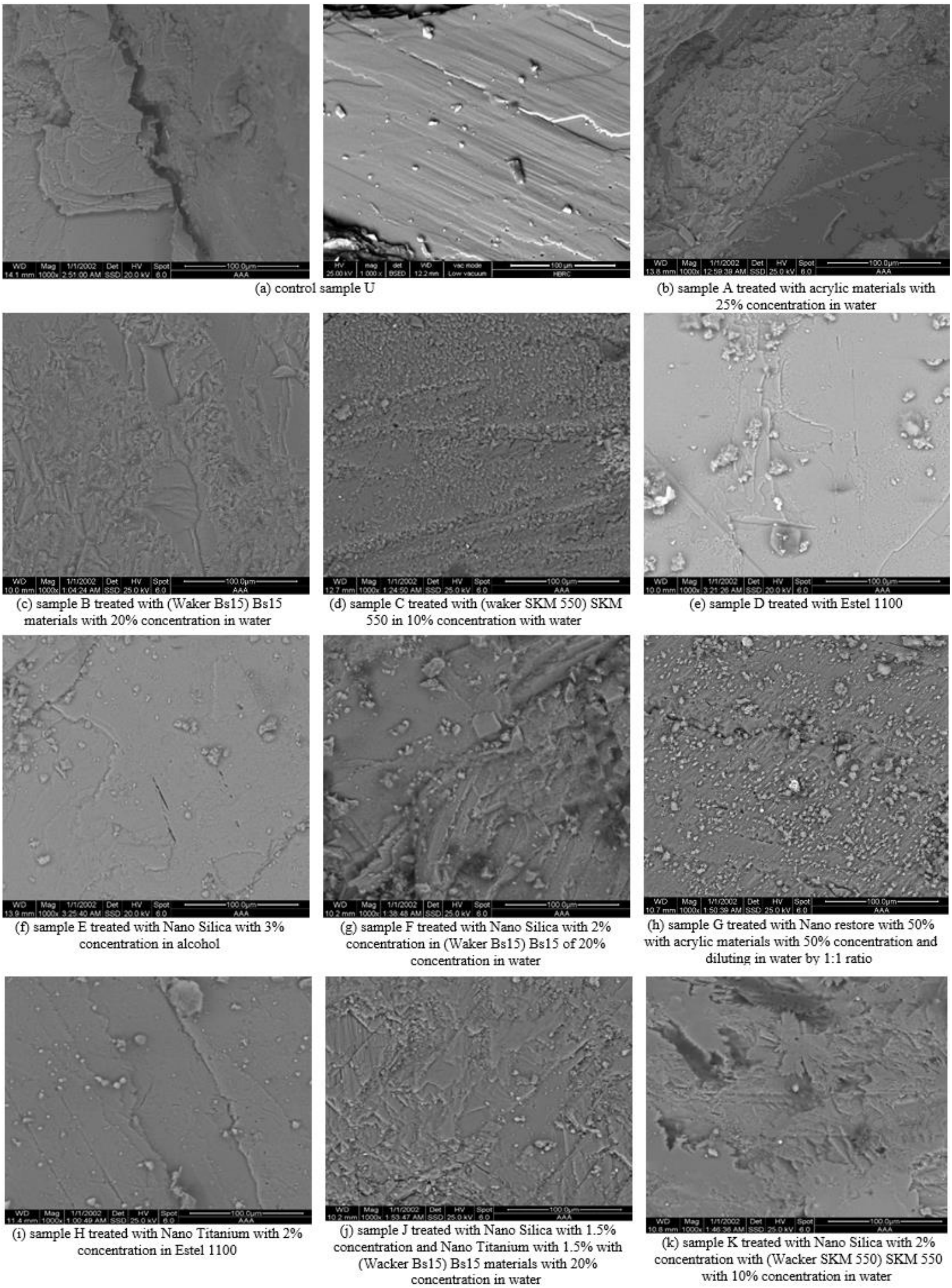
Figure 13 shows the apparent porosity in which sample with code number G treated with Nano restore with 50% with EucoColle materials with 50% gives the lowest apparent porosity of 0.63% which decrease the control one by about 67.52% which is 1.94%.

SEM analysis for marble after treatment

Scanning Electron Microscopy (SEM) for marble specimens after treatment were carried out using SEM model Inspect S (FEI Company) supplied with EDAX to investigate the effect of reinforced materials and its penetration through the texture of marble by comparing them with the untreated sample.

Microphotographs of the control sample U

Figure 14 shows microphotographs of SEM with magnification 1000× for control and treated samples by different method.



(a) control sample U

(b) sample A treated with acrylic materials with 25% concentration in water

(c) sample B treated with (Waker Bs15) Bs15 materials with 20% concentration in water

(d) sample C treated with (waker SKM 550) SKM 550 in 10% concentration with water

(e) sample D treated with Estel 1100

(f) sample E treated with Nano Silica with 3% concentration in alcohol

(g) sample F treated with Nano Silica with 2% concentration in (Waker Bs15) Bs15 of 20% concentration in water

(h) sample G treated with Nano restore with 50% with acrylic materials with 50% concentration and diluting in water by 1:1 ratio

(i) sample H treated with Nano Titanium with 2% concentration in Estel 1100

(j) sample J treated with Nano Silica with 1.5% concentration and Nano Titanium with 1.5% with (Wacker Bs15) Bs15 materials with 20% concentration in water

(k) sample K treated with Nano Silica with 2% concentration with (Wacker SKM 550) SKM 550 with 10% concentration in water

Fig. 14. Micro photographs of SEM with magnification 1000× for control and treated samples by different method

Figure 14a present the investigation of the untreated marble sample of code (U), which has a severe fragility, cracks and inconsistency and consistency with the presence of salty impurities. Figure 14b illustrates the good propagation of the polymer but blocks the pores. Figure 14c shows the filling of the blanks and pores between the calcite granules and the emergence of a surface protection layer. Also, this figure shows the granule packaging which is characterized by a dense and homogeneous layer with small gaps. Figure 14d shows the spread of the polymer well and regularly without closing the pores. Figure 14e shows the appearance of nano-silica granules almost regularly with some gaps between silica granules. Figure 14f shows good polymerization and calcite granules, but the silica granules are distributed almost homogenously. Figure 14g presents marble sample treated with material, the excellent polymer diffusion and penetration on the surface of the sample and the work of a layer of good surface protection without closing the pores. Also, this image shows the coherent granules with the proliferation and penetration of the excellent polymer, with nano granules which restore regularly and homogenously on the surface of the sample. Figure 14h shows the polymer penetration and its fusion are well demonstrated with nanotubes of titanium with the appearance of some small gaps. Figure 14i shows the good penetration and granule packaging are shown in a homogeneous polymer layer, and the nano-silica and nano-titanium granules are uniformly distributed over calcite granules. Figure 14j presents the spread of the polymer which is shown homogeneously between the calcite granules, with the appearance of nano-silica particles on a semi-regular basis and the presence of some fine cracks and gaps. Figure 14k presents the treated samples with Nano Silica with 2% concentration with (Wacker SKM 550) SKM 550 with 10% concentration in water.

Figure 15 shows electron microscopy SEM scanner with a magnification of 1000× for marble samples treated with material Nano restore (50%), with code (G).The complete endurance of the sample is demonstrated by the effect of saline weathering, where the surface appeared in a coherent manner with the excellent polymer diffusion and penetration, as well as Nano restore particles distributed evenly and uniformly and did not clog the pores.

Strengthening and preservation of historic structural marble columns

The restoration and strengthening of Al-Mahalli mosque involved the structural marble columns restoration. The challenge in cleaning the interior masonry decorative components in particular the marble columns was to avoid more damaging and instability. This was achieved by using carefully placed protection in these areas of interface.

Marble columns and natural stone were cleaned and repointed to enhance the overall look and feel of the sacred mosque, which is an amazing combination of marble, cast stone, stucco, and other decorative materials. Marble columns were carefully resurfaced and filled to produce a smooth and uniform marble surface that is now capable of withstanding the abrasion of constant use. Other marble decorative elements also received the standard clean and re-point treatment.

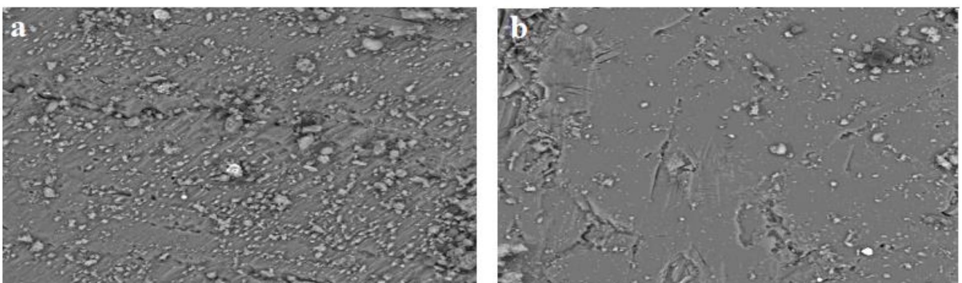


Fig. 15. Micro photographs of SEM with magnification 1000× for sample G treated with Nano restore with 50% with acrylic materials with 50% concentration and dilute in water by 1:1 ratio agent time: **a** – before; **b** – after weathering

Marble columns were cleaned using the dry tools and mild detergents with a low-pressure (900psi) water rinse (Fig. 16).

The challenge in cleaning these interior decorative components was to avoid damaging the adjacent highly stenciled surfaces. This was achieved by using carefully placed protection in these areas of interface. Cleaning and repointing of the pits and gaps s was carefully executed to avoid contact with the leaded components.

Interior marble columns were carefully resurfaced and filled to produce smooth and uniform marble columns that are now capable of withstanding the abrasion of constant use.

Other marble columns were anchored and restored to preserve the exterior details of the facade. The final step of the restoration process included the consolidation and protection of the marble surfaces with Nano restore with 50% with acrylic materials.

Fig. 16. Cleaning and consolidating process process of the marble columns: **a** - dry cleaning; **b** - salts removal process using poultices saturated with mild detergents with a low-pressure (900 psi) water rinse; **c** - final step, staining removing with detergents; **d** - repointing of the pits and gaps inside the marble columns; e – consolidating with Nano restore with 50% with acrylic materials with 50% concentration and dilute in water by 1:1 ratio agent time

Conclusions

The deterioration actions of historical building in Rashid mainly are the ground water (GWT), rains, and salt weathering, variation of temperature, air pollution and biological factors.

The damage factors of historical building in Rashid are the ground water, rains, and salts, variation of temperature, air pollution and biological factors.

Also, XRD, photographic analysis and SEM indicate that the marble of Rashid consists mainly of calcite and halite.

The present study indicates that the Nano-materials improve the physical and mechanical properties of marble in historical building in Rashid city and overcome the ordinary materials.

Compressive strength for sample (G) after treatment with Nano restore with 50% with EucoColle materials with 50% gave the highest load of 629.23kg/cm^2 by an increase of 23.59% with respect to the control one which give a compressive strength of 509.12kg/cm^2.

Ultrasonic pulse velocity for sample (G) after treatment with Nano restore with 50% with EucoColle materials gave the highest velocity of 6252m/s with an increase of 39.99% of the control sample which give 4266m/s.

Physical properties for sample (G) after treatment with Nano restore with 50% with EucoColle materials gave the highest bulk density of 1.87g/cm^3 with an increase of 19.10% of the control sample which give 1.57g/cm^3.

The assessment of consolidation effectiveness is not straightforward, particularly because there is interdependence between the stone and the applied treatment.

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