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LABORATORY EVALUATION OF NANOLIME CONSOLIDATION OF LIMESTONE STRUCTURES IN THE ROMAN SITE OF JERASH, JORDAN

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ABSTRACT

This study investigates the consolidation effect of nanosized particles of calcium hydroxide dispersed in alcoholic medium on limestone. The treatment materials were applied on limestone samples from Jersah archaeological site as well as other fresh samples. Different parameters were taken into consideration to evaluate the efficacy of the consolidation material and include: porosity, water uptake, compressive strength, drilling resistance and salt crystallization damage resistance.

Comparison between these properties before and after consolidation showed that the application of nanoparticles prepared in propanol-1, significantly improved the mechanical properties of the treated stone. Compressive strength increased by about 37% for archaeological stone and by about 25% for fresh ones, the drilling resistance increased by about 75% for archaeological stone and by about 52% for the fresh ones. Nano-sized lime has no significant effect on porosity; decreased by 4.6% and 3.2% for archaeological and fresh stones respectively, while water uptake value (w-value) decreased by 8.4% for archaeological stones and by 17.2% for fresh stones.

Nanolime consolidant improved salt crystallization damage resistance by about 29% for fresh stone and by about 32 % for archaeological samples. The main disadvantage of the nanolime consolidants is the relatively low penetration depth; the average consolidant uptake value ranged between 6.14 kg/m²hr^{0.5} for archaeological stones and 1.52 kg/m²hr^{0.5} for fresh stone.

KEYWORDS: consolidation, calcium hydroxide, calcium carbonate, nanotechnology, porosity, capillary water uptake, compressive strength, drilling resistance, salt crystallization resistance.

1. INTRODUCTION

The history of mankind has been accompanied by the use of natural stones in constructing and monuments. Due to their availability, durability and aesthetic appearance natural stone is used in building facades, concrete, foundations, floors and other architectural elements that form buildings (Siegesmund & Sneath, 2011; Al-Share *et al.* 2012; El-Gohari, 2010; Samanian *et al.*, 2012; Salama *et al.*, 2017a, b).

Among the natural stones, limestone is the most common natural building stone in Jordan, it was used for constructing not only Jerash archaeological site but also many other important archaeological sites in Jordan and the region, e. g. the Decapolis (Nizar Abu-Jaber, al Saad & Smadi, 2009; Yaseen *et al.*, 2013) and in paintings (Salama *et al.*, 2018). Furthermore, it is the main material in modern constructions in Jordan.

Although natural stones are some of the most weathering-resistant building materials, they are not immune to physical and/or chemical deterioration factors; the deterioration of stone is known to anyone closely observes historic building or monument. While there are a few stones that seem to be minimally affected by centuries of exposure to weather, most of stones are undergoing gradual and continuous deterioration (Amoroso & Fassina, 1983; Torraca, 2009).

Degradation of building materials is essentially attributed to weathering phenomena, a phenomenon that arises from physical. Chemical and biological mechanisms which can cause many deterioration forms on two scales:

a. Macro scale weathering phenomena such as structural damage, cracking, loss of plumb and bulging of walls.

b. Micro scale weathering phenomena and includes: bursting, flaking, coloration, scaling, skinning, exfoliation, soiling and chemical alteration of the original mineralogical composition (El-Gohary, 2010; Samanian *et al.* 2012; El-Derby *et al.* 2016).

There are different methods that can be implemented to retard or even prevent the deterioration of building materials, of which the consolidation treatment is one of the most important. A wide spectrum of consolidants has been used to consolidate natural building materials; this includes organic and inorganic materials (Horie, 2010).

The most frequently products used to consolidate degraded building stones are mainly based on tetraalkoxy- or alkylalkoxy-silanes, resulting in the formation of relatively stable silica inside the stone pores. Unfortunately, silica is not chemically compatible with carbonate stones; in this respect,

nanocalcite may be a suitable alternative (Coltelli *et al.* 2018).

The aim of this research is to examine the efficiency of $\text{Ca}(\text{OH})_2$ nanoparticles (nanolime) for limestone consolidation, by evaluating porosity, capillary water uptake value, compressive strength, drilling resistance and salt damage resistance for the study samples before and after treatment. To assess the penetration depth of the nanolime dispersed in propanol, consolidant uptake value and drilling resistance were measured.

Nanolime is a relatively recently developed material used in consolidation treatment. Derived from an older limewater method, nanolime is synthesized at nanoscale as calcium hydroxide particles in alcohol for suspension stability (Giorgi, Dei, & Baglioni, 2000; D'Armada & Hirst, 2012). As a particle size ranges between 100 nm and 500 nm is smaller than the pore size range of porous and carbonate stones, research has shown that nanolime can be an effective, deep penetrating consolidant for various types of limestone. The nanoparticles are formed very rapidly during the first immersion in the solution to form a thin surface on the pores of the treated materials.

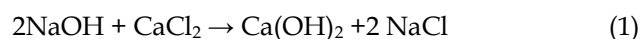
In addition to the successful application of the nanolime as a consolidant; the material has been successfully used to clean fresco paintings (Salama *et al.*, 2017a, b; Salama *et al.*, 2018).

2. MATERIALS AND METHODS

For the purpose of this study, fresh and archaeological limestones from Jerash archaeological site were sampled. Limestone in the area can be quarried from the Upper Cretaceous Limestone Formations exposing in northern Jordan. Cubic samples ($5 \times 5 \times 5 \text{cm}^3$) were prepared for this study.

Alcoholic - based calcium hydroxide ($\text{Ca}(\text{OH})_2$) nanoparticles were synthesized as suggested by Ambrosi *et al.* (2001). The procedures were applied as follows:

Five hundred mL of NaOH solution (0.4 M) and 500 ml of CaCl_2 solution (0.2 M) were separately heated to a temperature in the range 60-90 °C. When the selected temperature was reached, the two solutions were then rapidly stirred keeping the temperature of the mixture constant within ± 1 °C., during this stage the following reaction took place:



The suspension was allowed to gradually reach room temperature under a nitrogen atmosphere to avoid the carbonation of $\text{Ca}(\text{OH})_2$. The supernatant solution was discarded, and the remaining suspension was washed 3 times with water to reduce NaCl concentration. Each time, the dilution ratio between

the concentrated suspension and washing solution was maintained 1:10. The complete removal of NaCl from the suspension was controlled by AgNO₃ tests. Nanolime used in this study was synthesized at Department of Chemistry at Yarmouk University. The morphology and size of prepared Ca(OH)₂ nanoparticles were investigated by using scanning electronic microscopy SEM at the NanoCenter, Jordan University of Science and Technology. As the nano particles are more stable in short-chain aliphatic alcohols such as ethanol, propanol-1, or isopropanol (Ambrosi et al, 2001; Chelazzi et al., 2013; Giorgiet al, 2002; Baglioni & Chelazzi, 2013), the prepared nanolime was dispersed in propanol-1, the concentration was nearly 6 g/l.

The procedure followed to treat the samples was applied as suggested by Borsoi et al (2016) as follows:

The test samples were first dried in the oven at 60 °C for 24 h and then conditioned at 20 °C and 50 % RH. The prepared nanolimes were applied on the limestone specimens by capillary absorption until full saturation. The bottom surface of the specimens was partially immersed in a Petri dish filled with nanolime suspension and with a grid on the bottom. The specimens were stored at 50 % RH and $T = 20$ °C for at least 4 weeks to allow the carbonation process to take place before further testing (Daniele, Taglieri, & Quaresima, 2008).

Physical properties of the samples, such as porosity, capillary water uptake, compressive strength, drilling resistance and salt crystallization resistance, were measured before and after consolidation treatment. Porosity of the studied samples was measured according to the procedures of RILEM (1980); tests No. I.1 and I.2, The procedures were applied as follows: After drying to a constant mass in the oven at 70±5 °C for overnight, the samples were placed in an evacuation vessel; the pressure was gradually lowered to 2.0 ± 0.7 kPa (15 ± 5 mm Hg). This low pressure was maintained constant for 24 hours in order to remove the air contained in pores of the samples. Then distilled water was slowly introduced into the vessel until the samples were completely immersed. The samples were left for another 24 hours under water at atmospheric pressure. Then they were weighed separately in water (hydrostatic weight). The samples were quickly wiped with a dampened cloth and the mass of each sample saturated with water was measured. The following formula was used to calculate the porosity:

$$\rho = \frac{M3-M1}{M3-M2} \times 100 \text{ in Vol. \%} \quad (2)$$

ρ : Porosity, $M1$: dry mass, $M2$: mass taken under water (hydrostatic weight), $M3$: wet mass in air.

The capillary water absorption coefficient (*w-value*) of a stone is the amount of water absorbed through the surface area of the stone per square root of time. It provides a measure for assessing the extent of stone damage and the success of conservation treatments.

The water uptake coefficient was measured in the laboratory on dry specimens following the standard test DIN EN 1925. The samples were dried in a drying oven at 60 °C until a consistent weight was achieved. Dried samples were then placed in a desiccator and cooled at room temperature. The specimens were placed in a water tank, so that the water level is maintained around 2 mm above the bottom of the specimen. The specimens were weighted on specific time intervals, which were selected depending on the type of the stone and its water absorption capacity. Highly absorbing stones (weathered stones) are weighted after 0.5, 1, 2, 5, 10, 10, 20, 30, 60, 240 and 1440 minutes. The water absorption coefficient is then calculated as the slope of the linear part of the curve depicting the amount of water absorbed per area against the square root of time.

$$w - value = \frac{\Delta m}{A\sqrt{t}} \quad (3)$$

Where: *w-value*: water absorption coefficient [kg/(m².h^{0.5})];

Δm : mass of absorbed water in time interval;

A : the area of the stone surface in contact with water [m²];

t : time interval [h].

Consolidants uptake value was calculated by applying the same procedures used to measure the *w-value*; the only difference was the replacement of water by the consolidant.

The compressive strength was determined by applying an axial compressive force along the length of the sample at a constant rate. Testing Machine ELE Autotest available at the Laboratory for Research on the Structure of Matter at Jordan University for Science and Technology was used in this study. Constant rate of load was applied until failure was observed. The failure was determined when the maximum load was reached.

Drilling resistance is a micro-destructive technique that can be used, both in situ and in the laboratory, to evaluate the weathering extent of stone in depth profile and to assess the effectiveness and penetration depth of consolidation treatments (Johnson et al., 1996). The resulting profiles of drilling resistance provides information about the quality of the stone and the consolidation effect of the applied products. This test was conducted on fresh and archaeological samples before and after treatment at Yarmouk University–Faculty of Archaeology and

Anthropology, using SINT Drilling Resistance Measurement System (DRMS), which measures the drilling resistance of stone materials and mortars. It measures the force needed for drilling and the position of the bit during drilling and is equipped with a software program that allows continuous recording and monitoring of that force in relation to advancement of the bit. The speed of rotation and penetration speed are kept constant during testing. The drilling resistance was measured at a depth between 0 and 10 mm.

Untreated and treated limestone samples were artificially weathered by salt crystallization damage test, which was carried out as recommended by DIN EN 12370. The procedures were applied as follows:

Untreated and treated limestone samples of known mass were totally immersed in a saturated solution of sodium sulfate decahydrate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) for 24 hours, the samples were then dried in the oven at 40 °C in presence of silica gel. 25 salt weathering cycles were performed on the sam-

ples. Finally, the samples were submerged for one week in distilled water that was changed daily before eventually rinsed thoroughly in water to remove all the salt from their pores. The samples were finally dried in oven and the loss of stone material after the test was calculated from the change in the mass of specimen as a percentage of initial mass by applying the following formula:

$$\text{Mass loss ratio} = \frac{\text{initial mass} - \text{final mass}}{\text{initial mass}} \times 100\% \quad (4)$$

Samples with the highest loss ratio are the least resistant to salt damage.

3. RESULTS AND DISCUSSION

SEM micrographs of the prepared nanolime showed a typical $\text{Ca}(\text{OH})_2$ nanoparticles agglomerate, where the particles were crystalline and regularly shaped, ranging in size from 200 to 450 nm (Figure 1).

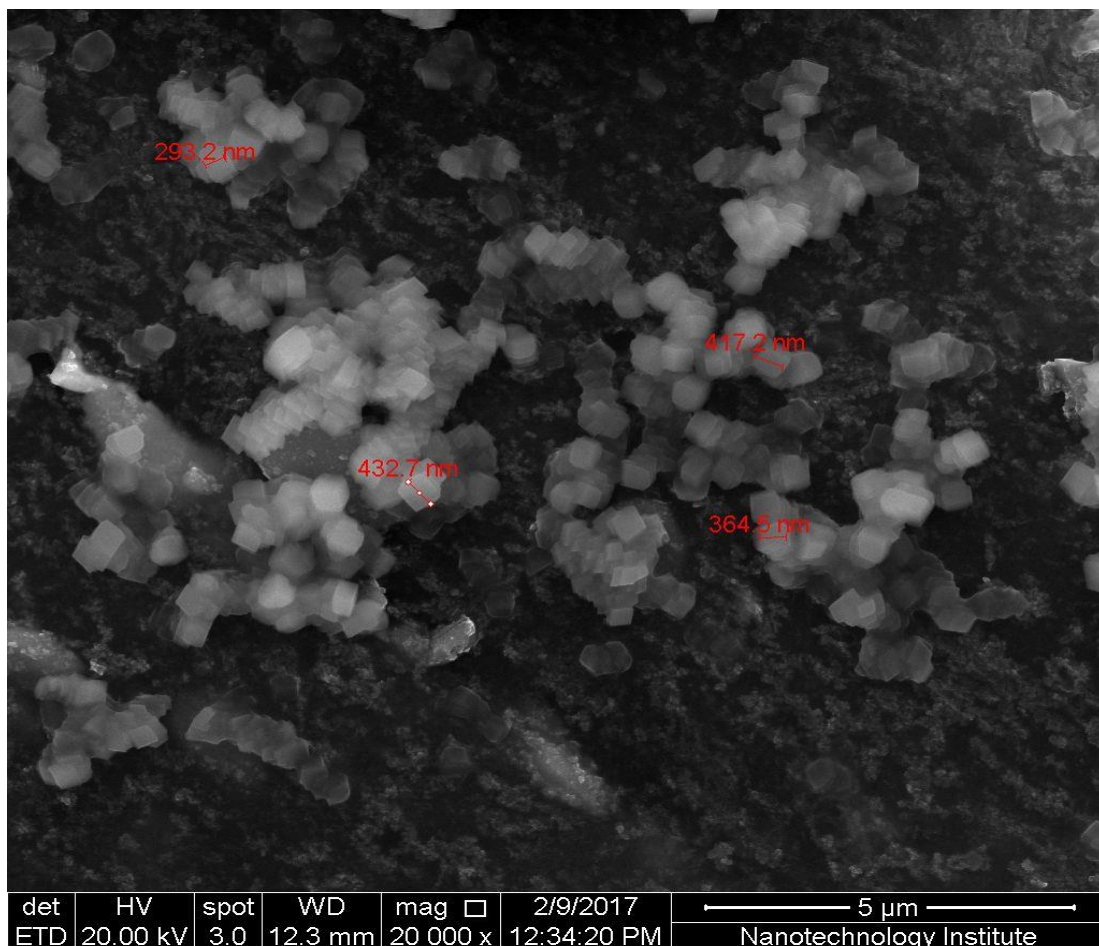


Figure 1. SEM micrograph of the alcoholic nanolime suspension.

Nanolime consolidation appears to have had little impact on the pore structure of the limestone samples. As can be seen in table 1, the porosity of the studied samples before treatment ranges between

15.8 and 23.8 with an average of 16.4% and standard deviation (Std) of 0.66 for the fresh samples and with an average of 22.7 % and standard deviation of 1.03 for the archaeological samples, while after treatment

it ranges between 15.3 and 22.7 with an average of 15.9% and standard deviation of 0.60 for the fresh samples and with an average 21.6 and standard deviation of 0.97 for the archaeological samples. Porosity decrease ratio due to consolidation treatment is

only 3.2% for the fresh samples and 4.6% for the archaeological samples; the change in the pore system is negligible, the relatively low standard deviation indicates the homogeneity of the studied samples.

Table 1: physical and mechanical properties of the samples before and after treatment

Sample Number		Fresh samples					Archaeological samples				
		F1	F2	F3	Average	Std	A1	A2	A3	Average	Std
Porosity (%)	untreated	16.3	17.1	15.8	16.4	0.66	23.8	21.8	22.4	22.7	1.03
	treated	15.8	16.5	15.3	15.9	0.60	22.7	20.8	21.4	21.6	0.97
	decrease%	3.1	3.5	3.2	3.2	0.21	4.6	4.6	4.5	4.6	0.06
W- value (Kg/m ² hr ^{0.5})	untreated	2.4	2.5	2.6	2.5	0.10	9.6	9.7	9.5	9.6	0.10
	treated	2.0	2.0	2.1	2.0	0.06	8.8	8.9	8.7	8.8	0.10
	decrease%	16.7	17.1	17.6	17.2	0.45	8.3	8.2	8.5	8.4	0.15
Consolidants uptake value (Kg/m ² hr ^{0.5})		1.40	1.61	1.55	1.52	0.11	6.12	6.09	6.21	6.14	0.06
Compressive strength (N/mm ²)	untreated	36.1	36.6	35.3	36.0	0.66	12.8	12.0	12.5	12.0	0.40
	treated	44.1	47.0	44.2	45.1	1.65	17.3	16.4	17.0	16.9	0.46
	increase%	22.3	28.5	25.3	25.4	3.10	34.6	36.6	35.9	35.7	1.01
Drilling resistance (N)	untreated	20.1	22.5	19.8	20.8	1.48	10.3	13.7	13.2	12.4	1.84
	treated	29.2	35.3	30.5	31.7	3.21	18.7	23.9	22.6	21.7	2.71
	increase%	45.3	56.9	54.0	52.0	6.04	81.6	74.5	71.2	75.7	5.31
Mass loss ratio due to salt crystallization (%)	untreated	12.3	12.8	11.6	12.2	0.60	21.5	19.2	20.7	20.5	1.17
	treated	9.5	8.8	8.5	8.9	0.51	13.6	13.1	15.4	14.0	1.21
	decrease%	22.8	31.3	26.7	26.9	4.25	36.7	31.8	25.6	31.4	5.56

The average w-value of the fresh untreated samples is 2.5 kg/m²hr^{0.5} (Std=0.10), decreased to 2.0kg/m²hr^{0.5} (Std=0.06) after treatment with an average decrease of about 17.2%, while it was higher for the archaeological samples, with an average of 9.60 kg/m²hr^{0.5} (Std= 0.10) dropped to 8.8 kg/m²hr^{0.5} (Std=0.10) after consolidation, with an average decrease percentage of 8.4% (Figure 2). The effect of the nanolime on the w-value is very minimal compared to the effect of polysiloxane or silica nanoparticles; Lettieri and Masieri (2016) reported that alky siloxane could decrease the w-value of a highly porous calcarenite to a less than 2% of the original w-value

(Lettieri and Masieri, 2016). Aslanidou et al. (2018) reported that silica nanoparticles dispersed in an aqueous emulsion of alkoxy silanes and organic fluoropolymer decreased the capillary water absorption of marble and sandstone by 45 - 75%. (see also Manoudis et al 2017).

The limited impact of the consolidation treatment on the w-value, as the low decrease percentage indicates, may be attributed to the little impact of the nanolime on the pore structure of treated stone, taking into consideration the fact that the pore structure of building materials is the main factor affecting its capillarity (Taylor, 2013; Coltelli et al. 2018).

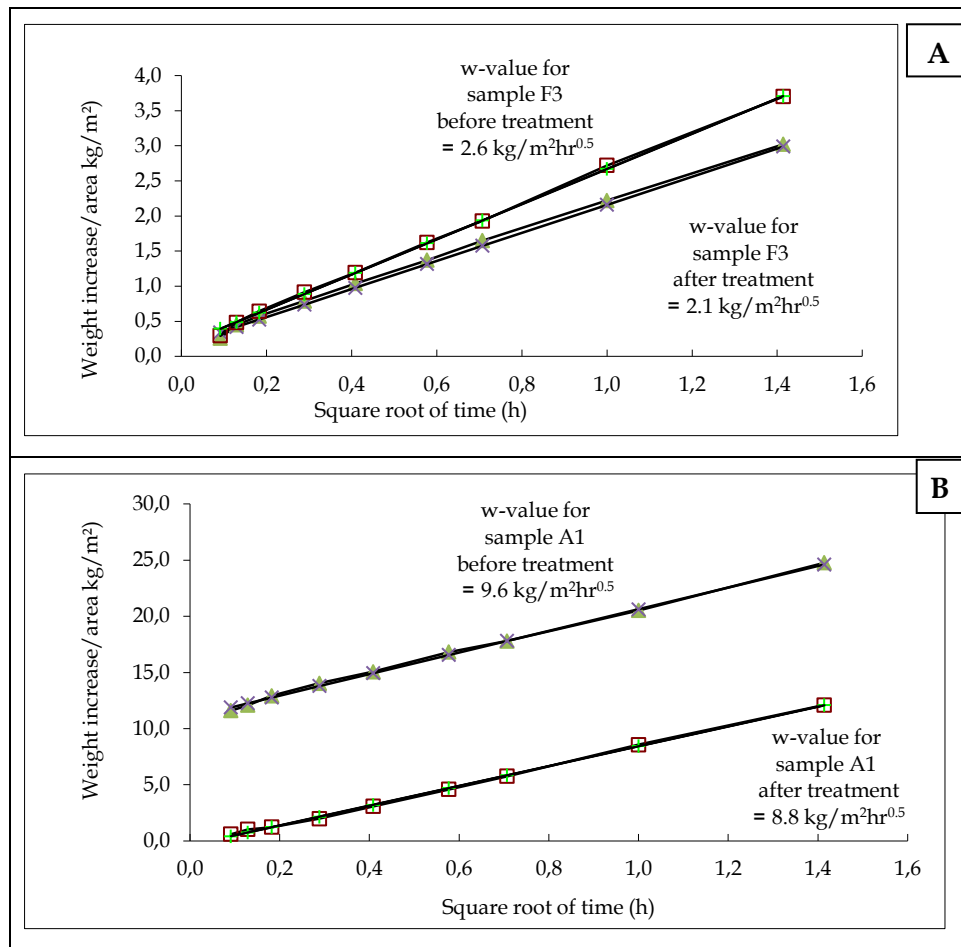


Figure 2. W-value measurements before and after treatment; a. For fresh samples b. For archaeological samples.

One of the most important properties of a good consolidation treatment is the capability of the consolidant to reach a substantial depth, which can be assessed by different methods such as measuring the consolidant uptake value (c-value); as the c-value of a consolidant increases, its penetration capability increases. Table 1 shows that the average c-value of the nanolime suspended in propanol is $1.52 \text{ kg/m}^2\text{hr}^{0.5}$ with a standard deviation of 0.11 for the fresh samples and $6.14 \text{ kg/m}^2\text{hr}^{0.5}$ with a standard deviation of 0.06 for the archaeological samples.

The average compressive strength of the untreated fresh samples is 36 N/mm^2 and increased to about 45 N/mm^2 after treatment with an average increase percentage of about 25%, while the average for the untreated archaeological sample is 12 N/mm^2 and increased to about 17 N/mm^2 after consolidation with an average increase percentage of about 36%. A result which can be attributed to the good adhesion of the nanolime and its capacity to form bridges of nanocalcite on the grains of the treated stone (Coltelli, 2018)

The untreated fresh samples have an average drilling resistance of 20.8 N that increased to 31.7 N after consolidation with an increase of 52.0%. While the untreated archaeological samples have an average drilling resistance of 12.4 N that increased to 21.7 N after consolidation with an average increase of 75.7%. The drilling resistance profiles (Fig. 3) show that the penetration depth of the used consolidant ranges between 3 and 5 mm only.

One of the most important deterioration factors for building materials are salts. Consequently, an efficient consolidant should improve salt crystallization resistance (Amoroso & Fassina, 1983). The average mass loss ratio due to the 25 cycles of salt crystallization for the untreated fresh samples is 12.2% dropped to 8.9% after treatment, with an average decrease percentage of about 26.9%. On the other hand, the untreated archaeological samples lost 20.5% of their masses due to the 25 cycles of salt crystallization, after consolidation the mass loss ratio dropped to 14.0%, the average decrease percentage is 31.4%.

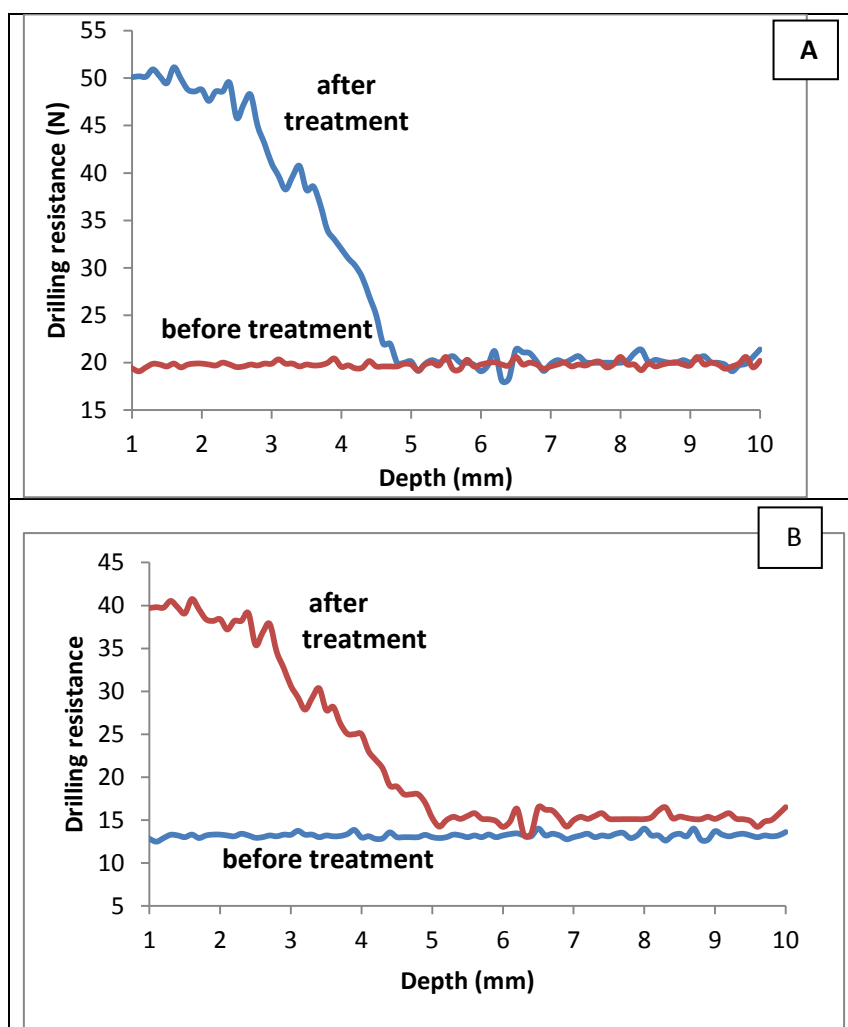


Figure 3. drilling resistance measurements before and after treatment; a. For fresh samples, b. For archaeological samples.

4. CONCLUSIONS

The low impact of the nanolime consolidant on the porosity and capillarity of the treated limestone makes such a consolidant suitable for the conservation of building stones; as the outer part of the stone treated with this material will have almost the same porosity as that for its original internal part, the water movement from internal to external parts will not be considerably retarded due to the decreased porosity.

Comparing the mechanical properties, compressive strength and drilling resistance, and the mass loss ratio for the samples before and after consolidation with nanolime, it can be clearly seen that nanolime can considerably improve the mechanical durability of the treated samples and increase its resistance to salt crystallization damage.

In all of the studied samples, the c-value is lower than the w-value. Propanol has a surface tension that is lower than that of water; 23.7 and 72.8 mN/m at 20 °C for propanol and water respectively (www.surface-tension.de). Consequently, it should

have a higher capability to penetrate, as the attraction force among its molecules is too much lower than the attraction force between its molecules and the pores surface. However, the presence of the suspended nanoparticles decreases its penetration capability. The main disadvantage of the nanolime consolidant is the relatively low penetration depth; however, the penetration depth of nanolime can be enhanced by reducing the concentration of the nanolime and increasing the number of applications. Another minor disadvantage of the nanolime is its whitening effect on the treated material, especially on darker stones, however, this effect is reduced after weathering (Otero et al. 2018).

The conclusions of this study are based only on laboratory evaluation; and since environmental conditions greatly affect the cure of nanolime, field tests are highly recommended to evaluate the efficiency of the nanolime and to optimize the application conditions to consolidate archaeological limestone in Jerash.

In general, this study demonstrates that archaeological limestone can be strengthened by consolidation with propanol-based nanolime at low concentrations without displaying significant physico-chemical changes. Like many other lime-based building materials, performance of the nanolime

consolidant is associated with the completion of the carbonation process, which may persist for many years. Therefore, to understand a long-term performance of nano lime consolidant on limestone, further testing is required.

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