

Nanotechnology applications to improve the mechanical properties of lime mortars in archaeological building

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تطبيقات تقنية النانو لتحسين الخواص الميكانيكية لمونة الجير في المباني الأثرية

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Abstract

The research discusses the different stages of consolidation, examination and analysis procedures that were carried out on the lime mortar that used as a basic building mortar. In Baibars Al-Khayat Mosque, in its dome and mihrab, lime mortar was used as a bonding mortar for cladding stones.

The damage aspects of structure mortar have been studied, with the aim of consolidating it, improving its resistance to compressive stress and making it more durable. The bonding mortar was also consolidated to have the highest resistance to tensile stress to which the mortar is exposed as a result of using cladding stones.

Samples were taken from the falling mortar and analyzed by different examination and analysis devices: X-ray diffraction (XRD) and scanning electron microscope equipped with an elemental analysis unit (EDX). Compression and tensile stress tests were carried out on the samples that had been consolidated at different stages.

The results of examination and analysis proved that the used bonding mortar is lime mortar, the stages of consolidation were done on the samples by adding some nanomaterial (Nano-silica, Nano-lime and WackerOH) to the mortar mixture to improve its properties. The samples were tested for compressive stress after salt aging, and then the results were determined, analyzed and discussed, which proved the the success of Nano-silica material with WackerOH then Nano-lime with WackerOH when determining the Compressive Strength, the success of Nano-lime with WackerOH then Nano-silica with Wacker when determining the tensile Strength and finally proved the success of Nano-lime with water, followed by Nano-silica with Wacker when determining the Compressive Strength after salt consolidation.

Keywords: Mortar, Baibars Al-Khayat mosque, compressive strength, tensile strength, salt weathering, Nanosilica, Nanolime, Wacker OH consolidation.

المخلص

يتناول البحث مراحل التقوية المختلفة وإجراءات الفحص والتحليل التي تمت علي مونة الجير المستخدمة كمونة بناء أساسية ، والمونة المستخدمة في الربط بين أحجار الزينة وحوائط البناء، تطبيقاً علي مسجد ومحراب القبة بجامع بيبرس الخياط بمنطقة درب سعادة بالقاهرة التاريخية، وتم معرفة وتعيين المونة التي تم استخدامها ومعرفة أهم الأضرار التي وقعت بها مع محاولة لتقويتها وتقويتها لما هو أفضل بحيث يجعلها أكثر قوة تحمل ومقاومة للضغط في مونة البناء ، وكذلك الذي يجعلها أعلى مقاومة للشد في مونة الربط لما تتعرض له المونة نتيجة استخدام ألواح التكبسية ذات الوزن الأكثر حملاً .

تم فحص العينات وتحليلها بأكثر من جهاز فحص وتحليل مثل جهاز حيود الأشعة السينية (XRD) والميكروسكوب الإلكتروني الماسح (SEM) المزود بوحدة التحليل، وتمت مراحل التقوية علي عدد من عينات من (مونة الجير) و تم إضافة بعض المواد النانوية لخلطة المونة لتحسين خواصها ، حيث استخدم كل من ، نانو السليكا ونانو الجير وكذلك مادة الفاكر OH المذاب في التريبتين المعدني، و تم تصنيع المونة علي شكل اسطوانات طبقاً للمواصفات القياسية، وتمت اختبارات اجهاد الضغط و الشد علي العينات التي تم تقويتها بالمراحل المختلفة قبل وبعد التقادم الملحي ثم مناقشة النتائج بكل مرحلة.

عند تعيين مقاومة اجهاد الضغط وكذلك مقاومة اجهاد الشد للعينات التجريبية، اثبتت مادة الفاكر OH جدارتها خاصة عند اضافة مادة نانو السليكا إليها وكذلك مادة نانو الجير ، وعند تعيين مقاومة اجهاد الضغط للعينات التي تم تقويتها بعد التقادم الملحي، اثبتت مادة الفاكر OH نجاحها عند اضافة نانو السليكا وكذلك مادة نانو الجير مع الماء.

الكلمات الدالة: مونة الجير ، مسجد بيبرس الخياط ، مقاومة إجاد الضغط ، مقاومة إجهاد الشد ، التجوية الملحية ، نانو السليكا ، نانو الجير ، مادة الفاكر OH ، التقوية

1- Introduction

Mortar is a mixture of natural and sometimes industrial materials used to adhesive main building materials such as stone and bricks horizontally and vertically. The thickness of the mortar usually does not exceed 4 cm, representing only less than 7% of the total volume of the walls of the building. When mixing the mortar, it is taken into account that its quantity is proportional to the work so that it can be used before it begins to dry. It is not allowed to add water to the mortar for its reusing again. The type of building, the type of building unit, the location of the wall in which the mortar is used, and the degree of its exposure to external and ambient conditions must be taken into consideration.1() (2) . The studied samples, table no (1), were taken from Baibars Al-Khayat Mosque. Al-Khayat Mosque is located on Al Joudriyah Street, Darb Saadah region, Al Darb Al Ahmar

1- A. H. Paul Maurenbrecher, Mortars for Repair of Traditional Masonry, Practice Periodical on Structural Design and Construction, Volume 9, Issue 2, 2004.

2- Jan Válek , John J. Hughes Caspar, J.W.P. Groot, Historic Mortars Characterisation, Assessment and Repair, Springer Dordrecht Heidelberg New York London, Library of Congress Control Number: 2012940739, , 2012,Pp 1-25.

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district, Cairo, monument number 191 fig. (1, 2). (3) (4) The mosque is exposed to many deterioration factors that need immediate intervention such as: a. the presence of street vendors in the mosque's urban, b. narrow streets surrounding the mosque causing poor preservation conditions, c. the deterioration of sewage networks, d. the excessive use of water, which moves the foundations of those buildings carrying salt and some natural components with mortar and stone fig. (5), e. water leaking into the soil, which immediately follows a partial settlement of the soil with the increase in the overloads of buildings, which quickly results in cracks and separations, whether in the walls and the building mortar, or due to the disintegration of components Bonding mortar, or falling in the decorative cladding stones as a result of the loss of the binding in the mortar due to the shortage of the tensile forces between its grains fig. (3), followed by falling the cladding layers fig. (4). Perhaps the aim of this research is to try to reach an optimal solution for the treatment and restoration of deteriorated mortar, whether main building or cladding mortar, using some modern nanomaterials, whether single or composite with some other polymers.



Fig. (1), The mosque and dome of the Baibars Al-Khayat mosque from outside, a, the main western façade of the mosque, b, the north façade of the mosque, c, the dome of the mosque

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3. Asim Muhammad Rizk, Doctor, Islamic Architecture in the Age of the Burji Mamluks, Part Three, Section Two, Madbouly Library, 2003, pp. 1951-68.
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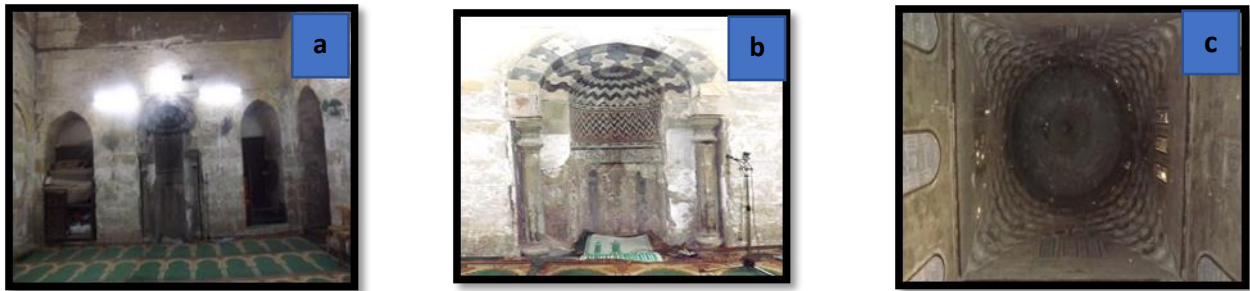


Fig. (2), The mosque and dome of Baibars Al-Khayat mosque from inside,a, the shrine from the inside,b, the shrine mihrab,c, the dome of the shrine from below



Fig. (3), Weakness of the bonds between the mortar and the stone,a, Describes the fall of mortar layers, b, showing the worn out parts of limestone,c, buckling layers of limestone



Fig. (4), falling off layers of stone cladding, a, Falling layers of cladding from the left side of the mihrab, b, Close up of the previous photo (a), c, Falling layers of cladding from the bottom of the mihrab

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Fig. (5), Salt crystallization of the outer layers of stones and mortar, a, Salt crystallization on cladding stone of mihrab, b, Close up of the previous photo (a), c, Separation of the cladding layers due to salts

2- Materials and Methods

Some modern techniques were used for the examination and analysis in order to diagnose the building material and determine its mineral components. Also, some Nano materials and some chemical polymers were selected with different concentrations; following some appropriate methods and steps in consolidating the stone samples to choose the most appropriate one, following some measurements and tests. The selected examination and analysis techniques were used to identify the physical and chemical composition of the stones and determine their mineral components. One of the studied samples was taken from the bonding mortars that used for the connection to the Baibars Al-Khayyat mosque. Other studied samples were taken from the building mortar. The mortar was prepared in the form of cylinders with a diameter of 2.5 cm and a height of 5 cm According to the ASTM C 170, C 880, C 5(99 test methods and ASTM C568/C568M-15 specifications) , then the Compression and tensile stress tests were carried out on the samples that had been strengthened at different stages. The results were discussed and then the samples were subjected to salt weathering by NaCl, as it is necessary to carry out salt aging to the different stages of consolidation.

5- Sayed Hemed, and Alghreeb Sonbol, Sustainability problems of the Giza pyramids, Hemed and Sonbol Herit Sci 8:8, <https://doi.org/10.1186/s40494-020-0356-9>, 2020, pp 1:28.

Table no (1), samples description .

Sample 1, fig(4)	sample of the cladding mortar	SEM with EDX
Sample 2, fig(3)	sample of the main building mortar	XRD, SEM with EDX

2-1- Scanning Electron Microscope with EDX

The scanning electron microscope is characterized by a very high magnification power, which gives researchers an opportunity to identify the problems that affect the internal structure of the materials, A device with the following specifications was used, SEM Model Quanta 250 FEG (Field Emission Gun) Attached with EDX Unit Energy Dispersive X-Ray Spectroscopy, with accelerating voltage 30 K. V., Magnification 14x up to 1000000 and resolution for Gun. In. K550X Sputter Coater, England. After examination and analysis, the current status of the samples that were examined and analyzed was determined by some figures. Where fig No. (8), a sample of the cladding mortar, and fig no. (9), shows a scanning electron microscope of a sample of the building mortar. Figure No. (6) Shows the result of the elemental analysis of a sample of the cladding mortar at Al-Khayat Mosque, and Fig No. (7) Shows a sample of the main building mortar.

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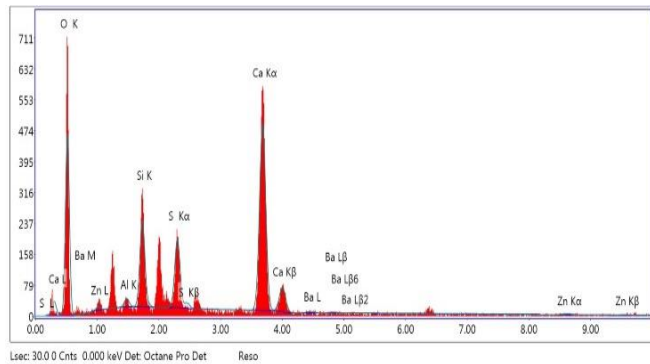


Fig No. (6)

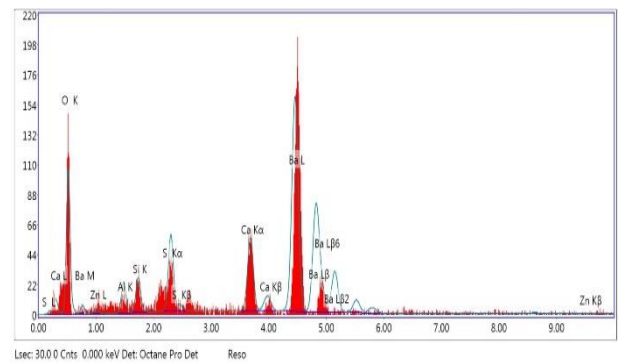


Fig No. (7)

Fig No. (6), elemental analysis result of the cladding mortar sample

Fig No. (7), elemental analysis result of the main building mortar sample

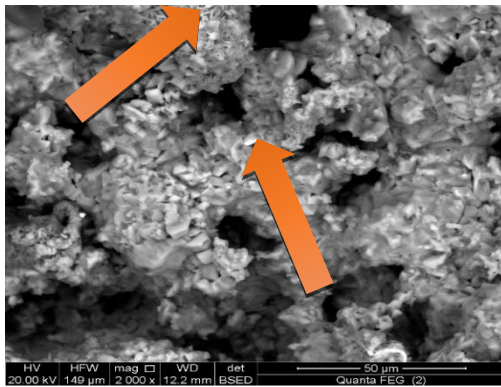


Fig No. (8) 2000x

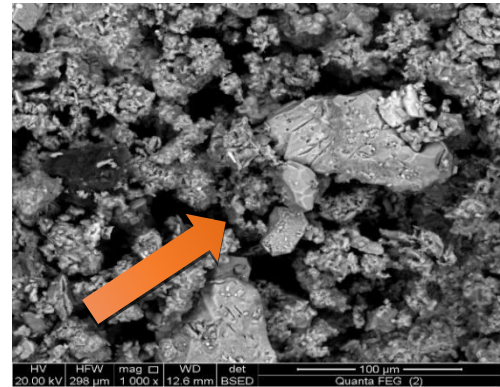


Fig No. (9) 1000x

Fig No. (8), scanning electron microscope examination of the cladding mortar sample, Fig No. (9), elemental analysis examination of the main building mortar sample

2-2- X-ray Diffraction results:

Results of the XRD analysis for the main building mortar materials at Baibars Al-Khayat Mosque. A device with the following specifications was used: Bruker model of Discover D8, TWIST-TUBE: Easy switch between point and line focus, Available anodes: Cr, Cu, Mo, Ag, Max. Power and filament: up to 3 kW depending on anode material (0,4 x 16 mm²), Patent: EP 1 923 900 B1, Temperature: Ranging from ~12 K up to ~2500 K, Pressure: 10⁻⁴ mbar up to 100 bar, Humidity: 5% to 95% RH, Turbo X-Ray Source (TXS), Line focus, 0.3x3 mm², Focal brightness of 6 kW/mm, max power depending on anode material: Cr 3.2 kW, Cu/Mo 5.4 kW, Co 2,8 kW, Pre-Aligned Tungsten filament (9).

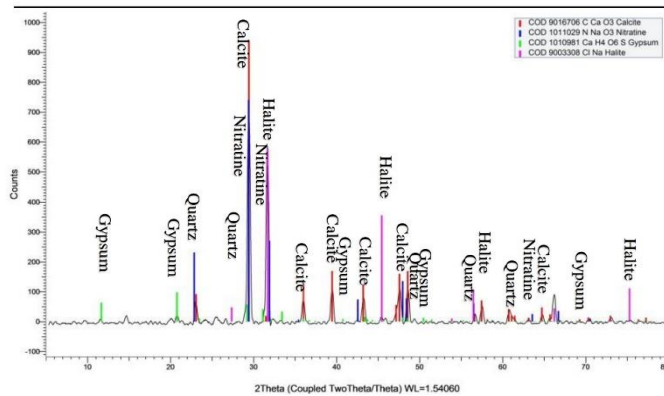


Fig No. (10)

Fig No (10). X-ray patterns of the identified components in main building mortar materials for the studied sample

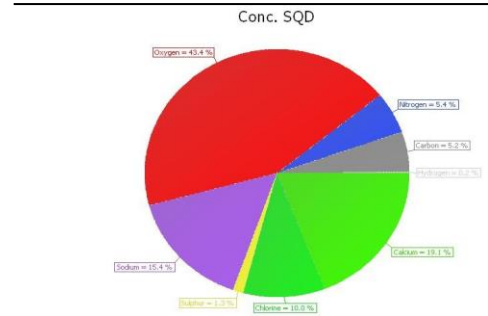


Fig No. (11)

Fig No (11).It shows the percentage of some elements in the sample

2-3- materials used in the consolidation

Nano is an English word meaning infinitesimally small ‘nanotechnology that science which cares of the study of nanomaterial in order to determine their chemical, physical, mechanical, thermal, electrical and magnetic properties. This is in addition to Study of its properties resulting from its reduction in size using the latest devices and various methods. Nanotechnology is an advanced science through which materials can be manufactured and their internal structure controlled by restructuring and arranging their constituent atoms.(9), Perhaps one of the most important characteristics of the materials used in the consolidation is to choose a material that corresponds to the nature of the original material that is being applied to it without leaving any harmful substances behind after the stage of dryness that follows the consolidation processes when applying different materials. Some different nanomaterial and some chemicals materials were selected for use in some samples of lime mortar, in order to choose the most suitable for restoration. The Nano materials that were used in the consolidation were divided into two main types as follows: Individual Nanomaterial. In this case, nanomaterial are used in a dispersed or suspended form in a carrier medium such as alcohol or water. Composite nanomaterial, in this case, Nano materials in different concentrations are added to the polymer with its solvent in order to increase the efficiency of the materials to which it is

9- Majid Hosseini , Ioannis Karapanagiotis, Advanced Materials for the Conservation of Stone, Springer International Publishing, Library of Congress Control Number: 2017963362, 2018, Pp. 25-42 .

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applied. Different nanomaterial have been used, Nano Lime and Nano Silica.)¹¹⁾ (10(. Wacker OH is considered one of the widely spread materials in the field of restoration and consolidation of monuments. It is one of the famous silane compounds. It is a ready-to-use product without dilution. The product is absorbed in layers of shale or stone and interacts with atmospheric moisture, where silicic acid is formed, which plays the role of a binder. One of the advantages of this product is that it penetrates deeply into the pores of the material, but it lacks the property of water repellency, and it is a product of the German WackerOH company. The color of the material is yellowish and contains tetraethyl ortho silicate with some ethyl polysilicate, also known as ethyl silicate or tetraethoxysilane, and it consists of organic and inorganic materials, and after treatment is completed, only inorganic materials remain within the pores of the treated material. The dissolved wacker substance was used in the mineral turpentine.As in table no (2), ,)¹³⁾ (12(The consolidating stages were carried out on a number of samples of (lime mortar) Cylindrical samples) ¹⁴⁾ at a ratio of 1 lime + 1 sand + 20% water, as well as 1 lime + 2 sand + 20% water, which is equivalent to 1: 5 of the weight of the mortar mixture (lime + sand). Of water, given that this ratio gives an ideal texture in the mortar mixture when mixed.)¹⁶⁾ (15(, Some nonmetric materials were added to the mortar mixture to improve its properties in integrating the main building granules, whether between building stones (limestone) or between cladding stones and building stones. The percentage of Wacker OH was dissolved in mineral turpentine at a concentration of 5%. The concentrations of different nanomaterial 5% and 7% were in grams per 100 grams of the weight of the mortar mixture (lime + sand).

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Preparing samples for mechanical tests.

After completing the various stages of consolidation, the samples are left to dry in the air in a period of not less than a week to complete the dryness and reach the fixed weight and to ensure the complete impregnation of the additives inside the mortar mix. After the drying stage is completed, the packaging stage begins. After that, the different mortars on which some tests will be carried out are distributed with writing the name of the tests, Compressive Strength Table(3), fig(12), and tensile Strength Table(4), fig(13), for Consolidated Mortars.)¹⁷(

Table no (2), consolidating stages with polymer and Nanomaterial

Consolidation Steps	Improved Lime Mortar	Mortar 1:1 Lime:Sand	Mortar 1:2 Lime:Sand
first stage	Lime & Sand Mortar.	200 gm. lime + 200 gm. sand + 80 ml water.	200 gm. lime + 400 gm. sand + 80 ml water.
Second stage	Nano Lime . 5 % + Wacker O H	200 gm. lime + 200 gm. sand + 20 gm. Nano-lime + 80 ml (mineral turpentine + water).	200 gm. lime + 400 gm. sand + 20 gm. Nano-lime + 80 ml (mineral turpentine + water).
Third stage	Nano Lime . 10 % + Wacker O H	200 gm. lime + 200 gm. sand + 20 gm. Nano-lime + 80 ml (mineral turpentine + water).	200 gm. lime + 400 gm. sand + 20 gm. Nano-lime + 80 ml (mineral turpentine + water)
Fourth stage	Nano Silica 2.5 % + Wacker O H	200 gm. lime + 200 gm. sand + 10 gm. Nano silica + 80 ml (mineral turpentine + water).	200 gm. lime + 400 gm. sand + 10 gm. Nano silica + 80 ml (mineral turpentine + water).
Fifth stage	Nano Silica 5 % + Wacker O H	200 gm. lime + 200 gm. sand + 10 gm. Nano silica + 80 ml (mineral turpentine + water).	200 gm. lime + 400 gm. sand + 10 gm. Nano silica + 80 ml (mineral turpentine + water).
sixth stage	Nano Lime 5 % + Water	200 gm. lime + 200 gm. sand + 10 gm. Nano-lime + 40 ml of water.	200 gm. lime + 400 gm. sand + 10 gm. Nano-lime + 40 ml of water.

17-17 - Sayed Hemeda, and Alghreeb Sonbol, Sustainability problems of the Giza pyramids, (2020), pp 1:28.

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Table (3), Compressive Strength test results of lime mortar compared to the reference sample

Consolidation Steps	Load (N)	Compressive Strength (N/mm ²)	Load % Compared with Control Sample	Compressive . St % Compared with Control Sample
Lime + Sand (Control Sample)	31.35	0.352	0	0
Nano lime 5%+ Wacker OH	36.5	0.371	16.42	5.39
Nano lime 7%+ Wacker OH	42.1	0.473	34.29	34.37
Nano Silica 5%+ Wacker OH	45.35	0.535	44.65	51.98
Nano Silica 7% + Wacker OH	56.98	0.695	81.75	97.44
Nano lime Only 5%	62.26	0.828	98.59	135.22

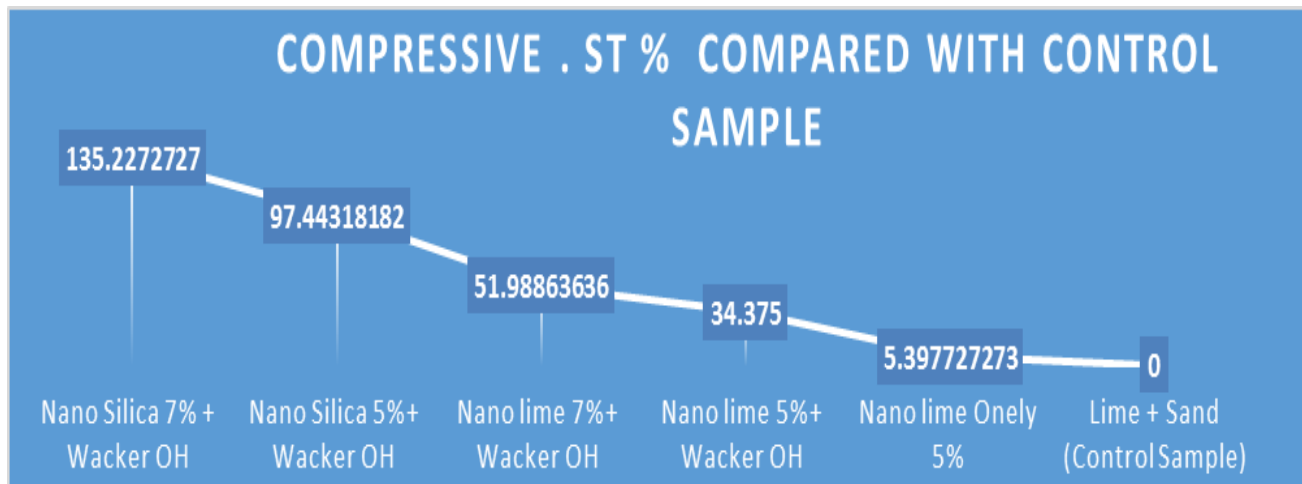


Fig (12), Compressive Strength test results of lime mortar compared to the reference sample

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Table (4), tensile Strength results with a comparison results of the reference samples.

No	Mortars Tensile (Splitting) strength Test Mortar After Consolidation	Load (N)	Splitting strength (N/mm2)	Load Compared with Control Sample	% Splitting. Compared with Control Sample
1	Nano lime + Wacker OH 7%	36.14	0.055	88.13	120
2	Nano lime + Wacker OH 5%	35.59	0.054	85.26	116
3	Nano Silica + Wacker OH 5%	35.13	0.049	82.873	96
4	Nano Silica + Wacker OH 7%	32.78	0.041	70.64	64
5	Nano lime Only 5%	22.54	0.028	17.33	12
6	Lime + Sand (Control Sample)	19.21	0.025	0	0

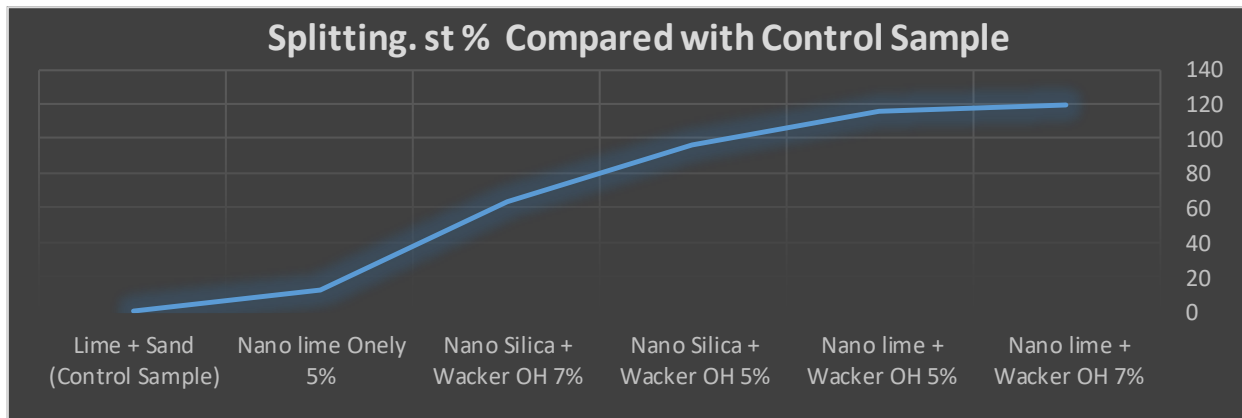


Fig (13), shows the results of the tensile Strength of the different consolidation stages with a comparison of reference mortar samples.

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Compressive Strength for Consolidated Mortars after Salt weathering

The results of the analysis that were conducted for the mortar samples showed that they contain sodium chloride salt NaCl, so it was necessary to carry out salt aging using this type of salt on the different lime mortars that were exposed to different stages of consolidation to know the extent of their resistance to this type of damage factors. A dilute solution of sodium chloride salt at a concentration of 10% of salt with water⁽¹⁸⁾,⁽¹⁹⁾ then immersing the samples in the saline solution for 24 hours. After the saline solution is completely impregnated, the samples are taken out and left to dry for a week until the salt crystallization appear on the surfaces of the mortar cylinders, and then some mechanical properties of lime mortar samples are measured after salt aging.⁽²⁰⁾Figure (14) shows .⁽²¹⁾ the results of the Compressive Strength test for the average results tested for lime mortar after salt aging, and Figure (15) shows the comparison of lime mortar samples before and after salt aging compared to the reference samples.

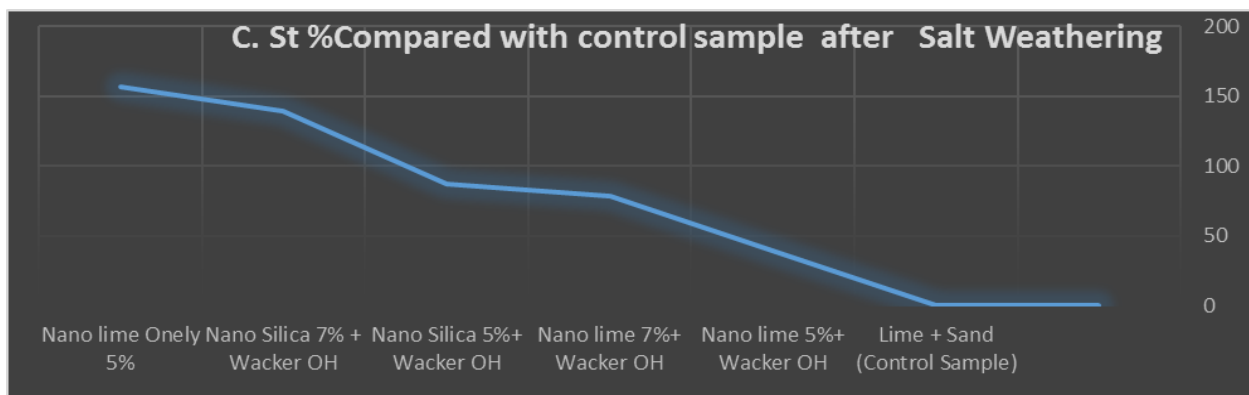


Fig (14) results of the Compressive Strength test after salt aging

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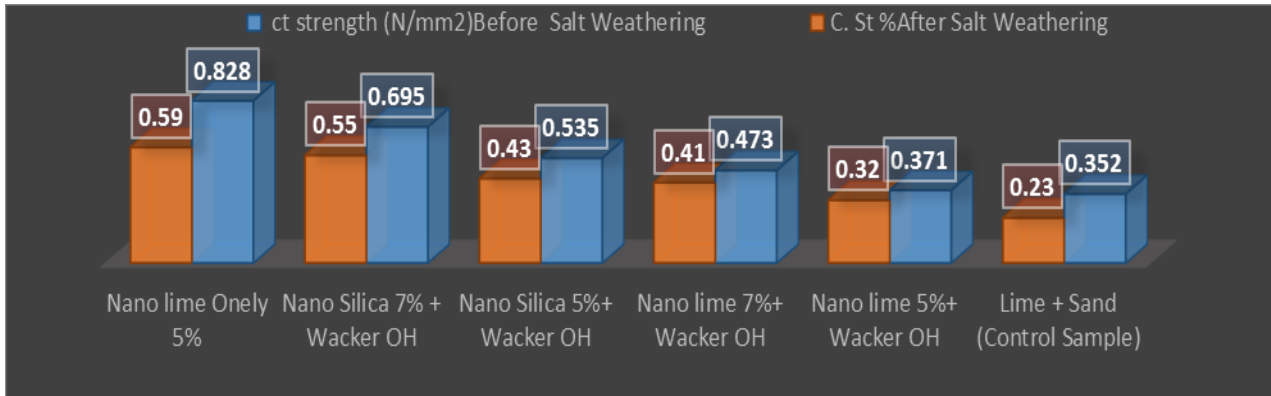


Fig (15) results of the Compressive Strength test after and before salt aging

3- Results and discussion

Various examination and analysis methods proved that the mortar that was used in the bonding was lime mortar, where the building mortar was in a ratio of 2:1 lime and sand, and the binding mortar was in a ratio of 1: 1 lime and sand. The results of the scanning electron microscope showed the extent of disintegration that reached the mortar, and also proved that the mortar that was applied as a basic building material or binding mortar is lime mortar. EDX analysis shows the lack of availability of calcium and silicon as a result of dissolution and the availability of chlorine, sodium, magnesium, aluminum, potassium and sulfur as impurities as a result of deterioration factors

SEM result show the extent of the deterioration that occurred to the physical composition of the mortar in addition to the disintegration of its grains and weakening of its internal structure as a result of salt deterioration. Represented by the sodium chloride salt, which is illustrated by some of the gaps and voids in the mortar as a result of groundwater and the deterioration of sewage networks in addition to dissolving calcium carbonate with various factors and forces of damage. The results of x-ray diffraction indicates that the compositions of the sample are: (major calcite CaCO_3 + Halite NaCl , minor gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, quartz SiO_2 + traces Dolomite $\text{CaMg}(\text{CO}_3)_2$, Nitratine NaNO_3 sodium oxide **Nitratine**). Fig.10, 11. Results also noted the presence of silica had decreased due to the weak bonding between lime and sand, and a large proportion of halite and Nitratine salts had been observed as a result of water leakage from the sewage surroundings the Mosque, The danger of these salts is on the internal structure of the mortar, creating mechanical pressures that break up the internal bonds of the mortar and act as a continuous source of moisture supply. It was also noted that there is a not small percentage of gypsum, which may have been used as a main component of mortar or resulted from some chemical changes that worked to transform the mineral calcite, whether through air pollution gases or the large presence of sulfur bacteria, where gypsum works to absorb moisture, which encourages biological damage to object.

Nanotechnology applications to improve the mechanical properties of lime mortars in archaeological building

When determining the Compressive Strength of mortar that was consolidated with different Nano materials, the success of Nano-silica material with WackerOH was proven in the first place, followed by Nano-lime with Wacker, then Nano-lime with water. When determining the tensile Strength of mortar that has been consolidated with various Nano materials, the success of Nano-lime with Wacker in the first place, followed by Nano-silica with Wacker, then Nano-lime with water. When determining the Compressive Strength of mortar that was reinforced with different Nano materials after salt aging procedures, the success of Nano-lime with water was in the first place, followed by Nano-silica with Wacker.

4- Conclusions

Nanotechnology has proven its ability to penetrate into the pores of the stone significantly, so it is necessary to focus on its study and application. The Nano-silica material has proven its success when applied in improving the properties of mortar that is used as a basic material in construction and the distribution of loads, especially those between stone courses that are used to withstand vertical effort. And resistance to loads, as shown by the results of Compressive Strength test.

The Nano-lime material has proven its success when applied in improving the properties of the mortar that is used in the bonding between the walls and the various decorative elements, especially when the tensile strength in the bonding is the basis and this is different from the mortar that is used to distribute the loads.

Wacker OH with Nano silica material has proven its worth in the field of consolidation, especially when subjected to compressive stress forces and continuous overloads, it is recommended to use it, especially in building mortar that is subjected to severe overhead loads. It also proved successful compressive stress, after salt aging

It also proved its effectiveness when used to strengthen mortars that are always subjected to different tensile forces like cladding mortars, especially when used with Nano lime.

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