

# Experimental study for the consolidation of load-bearing limestone walls applied to the building of Wekalat (Caravan Sarai) Uda-Basha in the city of Cairo

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## Experimental study for the consolidation of load-bearing limestone walls applied to the building of Wekalat (Caravan Sarai) Uda-Basha in the city of Cairo

دراسة تجريبية لتقوية الحوائط الجيرية الحاملة تطبيقاً على مبنى وكالة أودة باشا بمدينة القاهرة

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### Abstract

The research discusses the various steps of examination, analysis and consolidation of the limestone that was used in the construction of Islamic buildings in the city of Cairo, in the application of the Wekalat Uda-Pasha in Historic Cairo. Standing on the current status it has reached, to protect it from collapse and fall as a result of the severe neglect that it reached due to the lack of archaeological awareness. Fallen limestone Samples were taken from different places to know the chemical composition and to identify the most important damages that occurred to it, then evaluate and consolidate it for the best, which makes it stronger and bear the pressures and loads that fall on it and it's resistant to various damage factors.

X-ray diffraction was used, as well as the X-ray fluorescence device. A scanning electron microscope equipped with the EDX unit was also used to identify the chemical and mineral composition, as well as some impurities and salts in the limestone. The results of the analysis confirmed the presence of sodium chloride salt. The samples were examined and analyzed, which proved that the building material is limestone, and the consolidation stages were carried out on a number of limestone samples with special tools and materials for that. The stages of the experimental side were carried out on cubes of limestone in order to carry out some physical and mechanical properties. The limestone was cut into cubes with dimensions of 3 centimeters, then they were numbered in preparation for consolidating them with different consolidation materials according to four cubes of limestone with each of the different consolidation materials giving each stage a special code to facilitate dealing with it.

The process of preparing samples for consolidation was carried out in three stages. Nano-calcite and Nano-lime were used with Wacker OH and it's solvent (mineral turpentine), in concentrations of 5%, 7% with the polymer and the solvent. Some physical properties were studied after consolidation in order to know the effectiveness and resistance of the various consolidation materials that were applied, and to know their behavior and the extent of their impact, whether on the internal structure of the stone or on its external texture. Where the following properties were determined: bulk density, natural water content, water absorption, and apparent porosity. Where the Nano-calcite material 7% with Wacker OH proved its effectiveness and recorded the highest bulk density, as well as the lowest natural water content and the lowest apparent porosity, as well as the lowest water absorption rate.

Some mechanical properties of limestone samples were determined after the various stages of consolidation, the most important of which is the compressive strength, as the Nano-calcite 7% with Wacker OH also proved its effectiveness and recorded the highest percentage. The stages of salt weathering were carried out using sodium chloride salt, which found in the analysis at a concentration of 10% with water, then the compressive strength test was determined after salt weathering with the effectiveness of nanocalcite 7% with wacker OH.

**Key words:** Uda-Pasha(Caravan Sarai), Limestone, X-ray diffraction, X-ray fluorescence, scanning electron microscope, Nanocalcite, Nanolime, Salt weathering

## المخلص

يتناول البحث مراحل التقوية المختلفة وإجراءات الفحص والتحليل للحجر الجيري الذي استخدم في تشييد بعض العمارات الإسلامية المملوكية بمدينة القاهرة تطبيقاً علي وكالة أودة باشا بالقاهرة التاريخية، حيث تم تناول تاريخ تأسيسها وموقعها و الوصف الأثري ونبذه تاريخية مبسطة عنها، مع الوقوف علي الحالة الراهنة التي وصلت إليها مع إلقاء الضوء علي سرعة التدخل لحمايتها من الإنهيار والسقوط نتيجة للإهمال الشديد التي وصلت إليه من انعدام الوعي الأثري لدي المستفيدين منها من أصحاب الورش والمحلات . تم أخذ بعض العينات من مادة البناء من أماكن مختلفة في محاولة لمعرفة التركيب الكيميائي والوقوف علي أهم الأضرار التي وقعت بها مع محاولة لتقويتها وتثبيتها لما هو أفضل، والذي يجعلها أكثر قوة تحمل للضغوط والأحمال التي تقع عليها ومقاومة لعوامل التلف المختلفة والتي تم فحصها وتحليلها بأكثر من جهاز فحص وتحليل، والتي أثبتت أن مادة البناء التي استخدمت هي من الحجر الجيري وتمت مراحل التقوية علي عدد من عينات الحجر الجيري وتمت مراحل التقوية بالأدوات والمواد الخاصة والمعدة لذلك حيث تم عمل بعض المكعبات وتهذيبهم بأبعاد 3 سنتيمتر لكل من الطول والعرض والإرتفاع ، ثم ترقيمهم تمهيداً لتقويتهم بمواد التقوية المختلفة بموجب أربع مكعبات من الحجر الجيري بكل مادة من مواد التقوية المختلفة مع إعطاء كل مرحلة كود خاص لها تسهياً للتعامل معها.

تم استخدام حيود الأشعة السينية ، وكذلك جهاز تفلور الأشعة السينية. كما تم استخدام الميكروسكوب الإلكتروني الماسح المزود بوحدة التحليل العنصري EDX للتعرف على التركيب الكيميائي والمعدني وكذلك بعض الشوائب والأملاح في الحجر الجيري وتم فحص وتحليل العينات التي أثبتت أن مادة البناء من الحجر الجيري وقد أكدت نتائج التحليل وجود ملح كلوريد الصوديوم ، وتمت مراحل التقوية علي عدد من عينات الحجر الجيري بأدوات و مواد خاصة لذلك وتم تنفيذ مراحل الجانب التجريبي علي مكعبات من الحجر الجيري للقيام ببعض الخواص الفيزيائية والميكانيكية، حيث تم تقطيع الحجر الجيري إلى مكعبات بأبعاد 3 سم ، ثم تم ترقيمها تمهيداً لتقويتها ب مواد مختلفة وفقاً لأربعة مكعبات من الحجر الجيري مع كل مادة من مواد التقوية المختلفة مع إعطاء كل مرحلة رمزاً خاصاً لتسهيل التعامل معها. تمت عملية تحضير العينات للتقوية علي ثلاث مراحل، تم استخدام النانو كالسيت ونانو الجير مع بتركيزات 5% و 7% مع البوليمر Wacker OH والمذيب (زيت التريبتين المعدني)، وتمت دراسة بعض الخواص الفيزيائية بعد التقوية لمعرفة مدى فاعلية ومقاومة مواد التقوية المختلفة التي تم تطبيقها ومعرفة سلوكها ومدى تأثيرها سواء على التركيب الداخلي للحجر أو على النسيج الخارجي للحجر، حيث تم تحديد الخواص التالية: الكثافة الظاهرية ، محتوى الماء الطبيعي ، امتصاص الماء ، المسامية الظاهرة. حيث أثبتت مادة النانو كالسيت بنسبة 7% مع Wacker OH فعاليتها وسجلت أعلى كثافة حجمية وأقل محتوى مائي طبيعي وأقل مسامية ظاهرة وأقل معدل امتصاص للماء. تم تحديد بعض الخواص الميكانيكية لعينات الحجر الجيري بعد مراحل التقوية المختلفة ، وأهمها مقاومة إجهاد الضغط ، حيث أثبتت نانو كالسيت 7% مع Wacker OH أيضاً فعاليتها وسجلت أعلى نسبة، تم تنفيذ مراحل التجوية الملحية باستخدام ملح كلوريد الصوديوم بتركيز 10% مع الماء ، ثم تم تحديد اختبار مقاومة إجهاد الضغط بعد التجوية الملحية حيث أثبتت النتائج مدى نجاح مادة النانو كالسيت مع Wacker OH .

**الكلمات الدالة:** وكالة أودة باشا، الحجر الجيري، حيود الأشعة السينية، تفلور الأشعة السينية ، الميكروسكوب الإلكتروني الماسح ، نانو الكالسيت، نانو الجير، التجوية الملحية

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## 1- Introduction

Building technology developed during the various Islamic eras in Egypt. Brick was widely used as a main building material in early Islamic buildings such as the Amr Ibn Al-Aas Mosque and the Ahmed Ibn Tulun Mosque, and it remained in use for this purpose until the Fatimid era. Then the Fatimids used limestone along with bricks in their facilities in the city of Cairo, and in the Ayyubid, Mamluk and Ottoman eras, limestone became the main building material in their facilities, Islamic architecture is shaped and matured by a number of religious, civilizational and climatic tributaries.<sup>1,2,3</sup> . The research deals with the study of the most important aspects of damage to Wekalat Uda-Pasha, especially limestone, the main component of the building material, with the identification of the most important factors and causes that led to some damages. The study also carried out some different consolidation stages on some samples of fallen limestone that were taken from different places of the building using some modern nanomaterials to consolidate them.<sup>4</sup>

Wekalat Uda-Basha (Zulfiqar Caravan Sarai) on Jamaliah Street, Object No.: 19, the it goes back to its owner, Prince Muhammad Katkhuda, brother of Prince Zulfiqar (Ottoman Era), it is considered an extension of the Mamluk style, where the founding text on the northwestern façade mentions that. located in neighborhood on Al-Timbeksha Street. Al-Gamaliya, at the end of Habs Al-Rahba Street, branching from Al-Muizz Lidin Allah Al-Fatimi Street in Historic Cairo, and meters away from Bab Al-Nasr and the Wekalat Qaitbay. Its history dates back to 349 years ago, where it was established in 1084 AH 1673 AD. Archaeological Description: It is a huge commercial Wekalat with a large number of internal and external shops and an ancient house attached to it. There are some agencies in Historic Cairo that are threatened with loss. At least they have received some restoration and attention, but Wekalat Uda-Basha did not get. It is in poor condition and needs to be restored.<sup>5</sup>. Therefore, it was necessary to intervene too late, as

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the violations on the street are many and require immediate intervention, especially a number of them have extended to the main building itself, Where some street vendors sit in front of it, in addition to the narrowness of the street and its poor condition, which is not commensurate with this historic place, but this is nothing for what is inside, it is filled with shops and workshops for the manufacture of copper, aluminum and iron, and it was exposed to a major fire because of those workshops where it has been sitting for a long time as well as the goods, raw materials and scrap that threaten to burn again. As a result of the workshop owners' lack of awareness of how to deal with this place, its condition has deteriorated so much that it has become from the inside in a miserable form, as shown in the attached photos. Dilapidated building and walls on the verge of collapse, the electrical wires, and rubbish dumped on the sides of the building, In addition to the deterioration of the sewage, a number of people do not know the value of the place or the building. As a result of the status reached by Wekalat Uda-Pasha, wooden and iron shores were made for shoring the building and its walls from the outside, as shown by the attached Fig. (1) which shows the building , as well as the iron beams and steels for the building and its walls from the outside , due to its poor condition and its protection as a temporary solution from collapse, fig. (2) shows the damage to limestone, the main building material, as a result of natural and man-made deterioration, Steel and wooden shores were also made from the inside in order to protect the entrance vault from the inside and outside to avoid passers-by during entry and exit, Some wooden pillows were also made to prevent the iron steel from directly scratching the building material, as illustrated by the group of Fig. (3), so this work was done as an initial contribution in an attempt to save the Wekalat Uda-Basha from its expected collapse and fall.

Undoubtedly, there are many physicochemical, chemical, mechanical, biological and man-made damage, to which the stone monuments in general <sup>6</sup>,and the Wekalat Uda-Basha in particular, caused the disintegration of their mineral components as a result of the processes of dissolution that occur for the bonding materials between the grains, or as a result of the damage of these Bonds due to the pressures resulting from the crystallization of salts or the swelling of the clay minerals present within the natural composition of the stone material, which are most harmful to the stone monuments or those that result in increasing the loads on them. This was clearly observed without any care.<sup>7,8</sup>And with the problems of damage that were previously talked about, the damage

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also appeared in separation in the form of crusts that separate layer by layer resulting from the crystals created by the salts, as in a group of Fig. (4), which requires re-installing these grains subject to prevent continuous loss, as well as those The crusts layers due to the factors of damage, which makes the stone lose its resistance to external load pressures, which resulted in some cracks and separations, from of the owners of workshops and stores, or those that occur as a result of weak soil layers, as in a group of Fig. (5). In addition to human damage from wrong restorations using newly manufactured bricks without specifications that preserve the effect of its components, in addition to the use of Portland cement laden with salt, Man-made factors such as electric wires and scattered garbage, which spreads animals and activates biological damage. with all its repercussions and problems, which is explained by Fig. (6).

Perhaps this is what prompted the researcher to find alternatives to the natural bonding materials that are affected by various damage processes, and to apply them to stone monuments, which gives them new qualities and properties that make them more tolerant to the processes of damage, which requires:, The safety and security of the stone effect, the effectiveness of the materials used in the treatment and not causing a change in the color of the treated stone, This is in addition to the penetration of the consolidation material deep enough into the stone installation.<sup>9,10</sup> The ability of the consolidation materials to penetrate is related to the period of their stay in a good condition in the liquid moving image during application, due to the low viscosity of the liquid used and the ability of the consolidation liquid to spread on the surfaces of the stone, which improves the mechanical and physical properties of these materials by adding some improved materials that merge With the inner tissue of the object and becomes part of it and works to bond its grains.

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10 - Omar Mohamed Adam, , A study of the manifestations of damage and methods of treatment and maintenance of movable artifacts carved from limestone, applied to some selected models, Faculty of Archeology, Cairo University, Master Thesis, 2009, pg. 97.

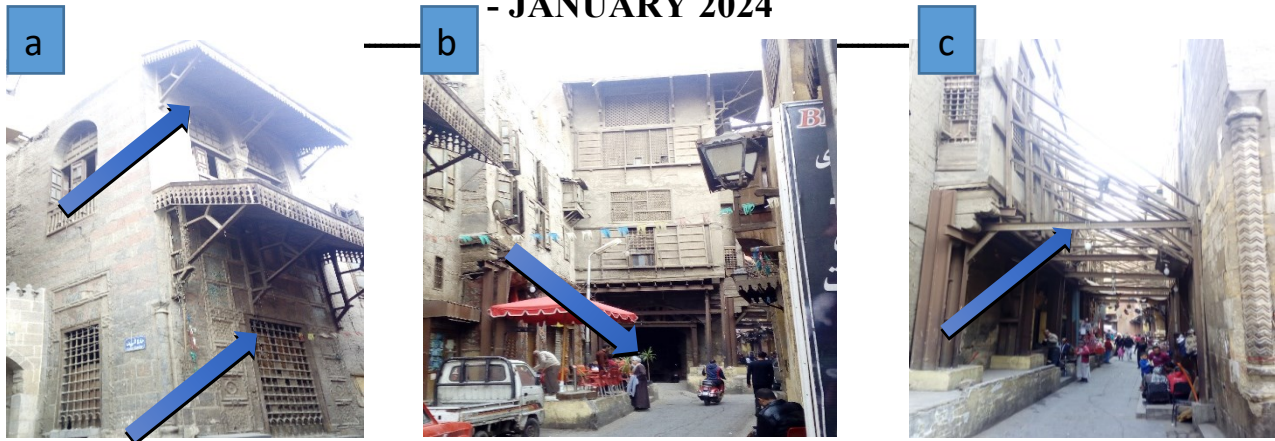


Fig. (1). The main facade of Wekalat Uda-Basha after being supported with different types of shoring. (a)Sabil & kottab Ouda basha (b)The main entrance (c)Different types of shoring resting on the wall of Madrasat Jamal Al-Din

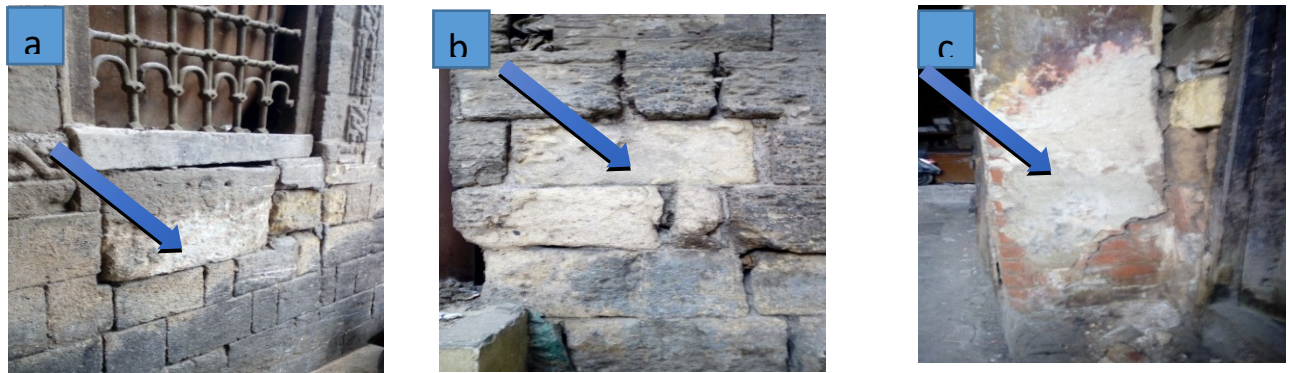


Fig. (2). shows the limestone damage, the main building material, as a result of natural and man-made deterioration. A & b. shows the limestone deterioration from outside. (c) The use of Portland cement as wrong restoration

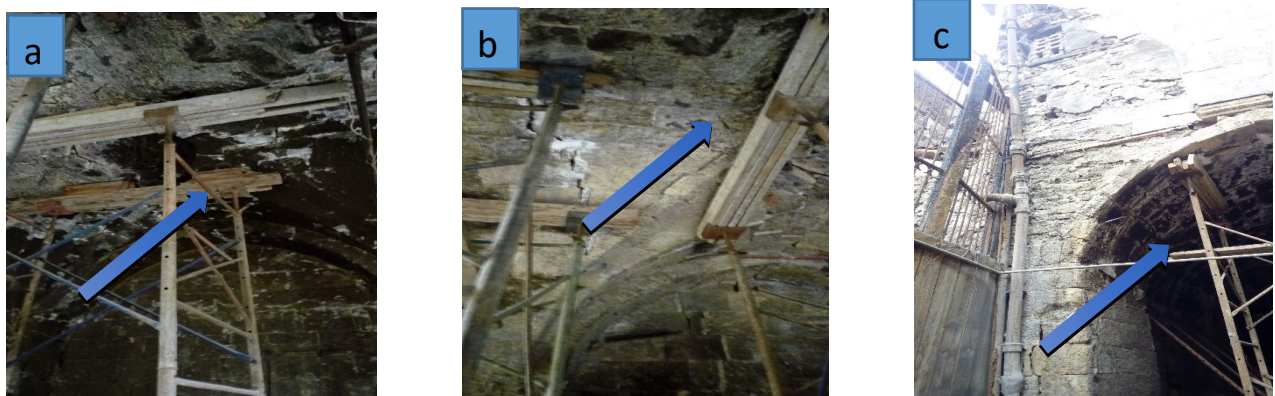


Fig. (3). Steel and wooden shores from inside to protect the entrance from the inside and outside to avoid passers-by during entry and exit with wooden pillows to prevent shores from directly scratching the building material. (a) Inside shoring (b)Wooden pillows (c) entrance shoring

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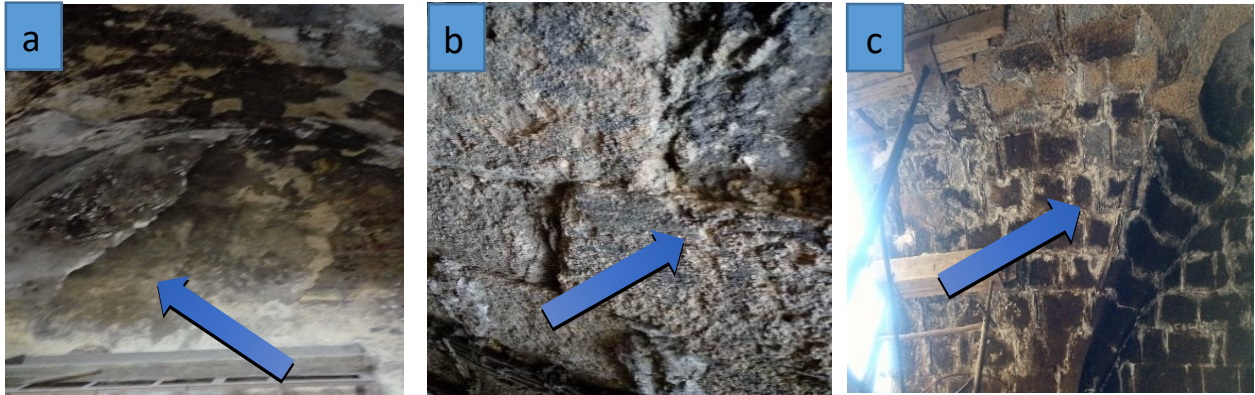


Fig. (4). Separation in the form of crusts by salt crystallizations. (a) Crusts Separation forms. (b)Effect of salt damage on limestone. (c) Salt crystals between walls units

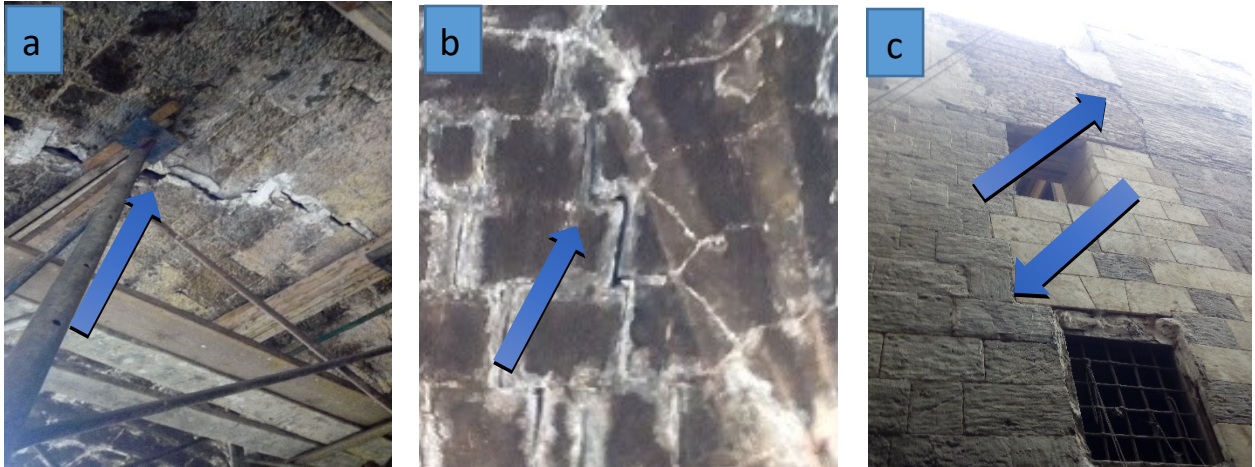


Fig. (5). Cracks that occur as a result of soil weakness and mechanical vibrations. (a) Cracks in the ceiling of the building from inside. (b)Cracks in the walls of the building from inside. (c) Cracks in the walls of the building from outside.

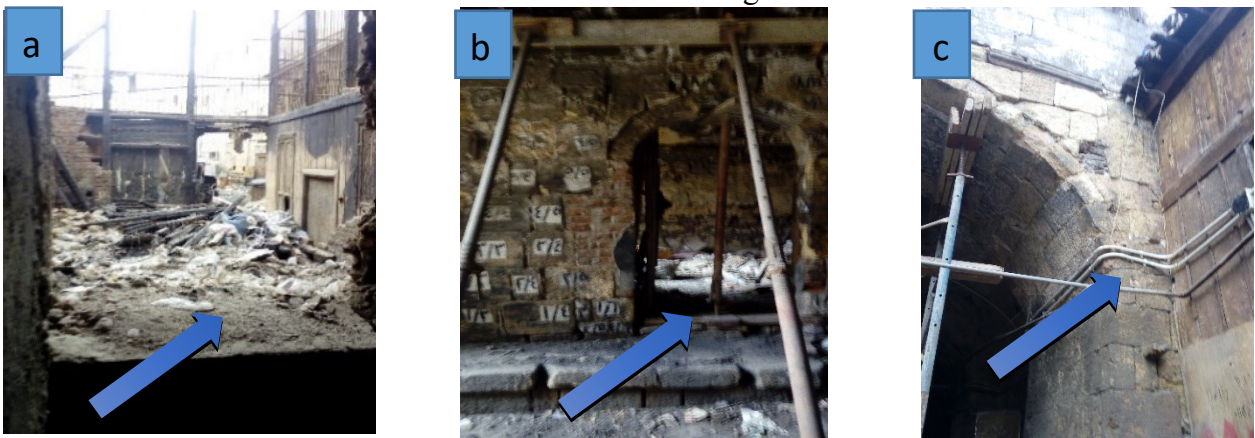


Fig. (6). Man-made factors such as electric wires and scattered garbage, which spreads animals and activates biological damage. a. & b. scattered garbage inside the building. (c) Electric wires outside and inside the building

## **2- Materials and Methods**

The limestone consolidation and protection program has been applied using some modern technologies from specialized devices in examination and analysis, in order to diagnose the building material and determine its mineral components. A group of different consolidated and protected materials were also used, varied between nanomaterial and polymers, and this was done at different concentrations in order to compare them and try to find what is best and most suitable for application in the field of consolidation, restoration and preservation of archaeological heritage.

The stones was prepared which carried out on cubes of limestone in order to carry out some physical and mechanical properties. The limestone was cut into cubes with dimensions of 3 centimeters, then they were numbered in preparation for consolidating them with different consolidation materials according to four cubes of limestone with each of the different consolidation materials giving each stage a special code to facilitate dealing with it. ( 11 According to the ASTM C 170, C 880, C 99 test methods and ASTM C568/C568M-15 specifications) .

The process consolidation was carried out in three stages. Nano-calcite and Nano-lime were used with Wacker OH 10% in it's solvent (mineral turpentine), in concentrations of 5%, 7% nanomaterial with the polymer and the solvent. then the physical and mechanical properties were carried out on the samples that had been consolidated 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.

### **2-1- Scanning Electron Microscope with E D X**

Scanning Electron Microscope with E D X equipped with an elemental analysis unit and is used in examination by its high magnification power. 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<sup>(12)</sup>.

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

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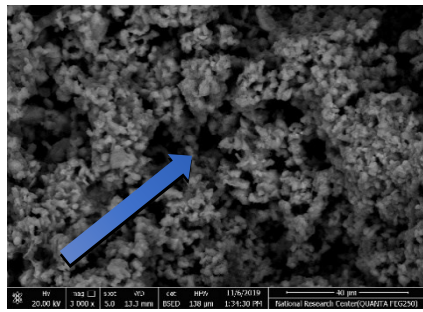


Fig. (7)

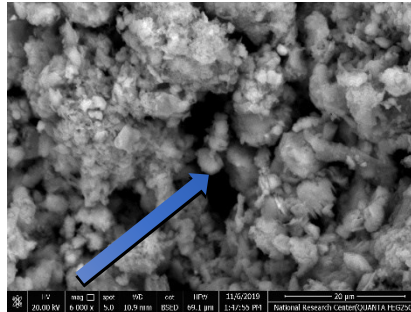


Fig. (8)

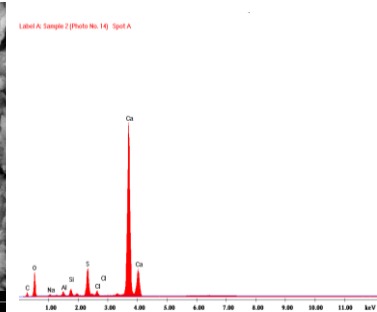


Fig. (9)

Fig No. (7), scanning electron microscope examination of limestone sample with 3000x  
 Fig No. (8), scanning electron microscope examination of limestone sample with 6000x  
 Fig No. (9), elemental analysis result of limestone sample

## 2-2- X-ray Diffraction results:

Results of the XRD analysis for the main building limestone at Wekalat Uda-Basha . 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 mm<sup>2</sup> ), Patent: EP 1 923 900 B1, Temperature: Ranging from ~12 K up to ~2500 K, Pressure: 10<sup>-4</sup> mbar up to 100 bar, Humidity: 5% to 95% RH, Turbo X-Ray Source (TXS), Line focus, 0.3x3 mm<sup>2</sup>, 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)<sup>13</sup>(.

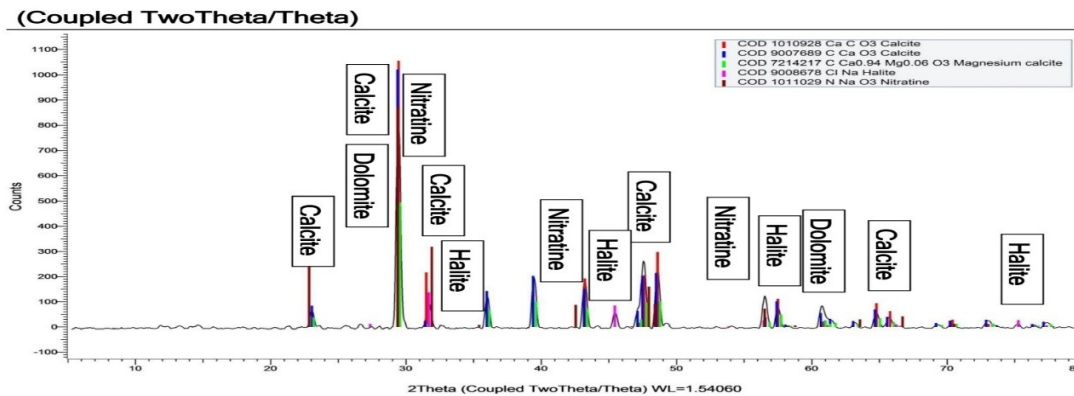


Fig No. (10), X-ray patterns of the identified compounds in main building stone materials for the studied sample

### 2-3- X-Ray Fluorescence (X R F)

It is an elemental analysis device, a device that is used only to identify the elements, as it plays the role of the (EDX) unit in the scanning electron microscope and the spectroscopy device by directed laser beams. Calculate the percentage of the presence of each element in the sample.<sup>14,15,16,17</sup>. A device with the following specifications was used, used : A Philips X-Ray Fluorescence equipment, model Philips PW/1404 , with RH target and six analyzing crystal for determining major and trace elements, The concentration of the analyzed elements were determined by using software Kernl X-44, The maximum power for the equipment 3 Kwt. The samples were prepared by fusion bead machine perlx 2 presser for pressed pellets(18). The results were as shown in Table (1)

Table No. (1) Shows the results of the element analysis using the fluorescence device

	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	CaO
<b>Limestone Sample</b>	8.3	0.03	0.72	0.11	1.02	<b>48.22</b>
	6.3	0.06	0.61	0.22	1.25	<b>48.22</b>
	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	Cl	SO <sub>3</sub>	<b>C.O.I</b>
	<b>2.31</b>	<b>1.04</b>	<b>1.55</b>	<b>2.8</b>	<b>1.4</b>	<b>31.12</b>
	<b>1.25</b>	<b>1.02</b>	<b>0.95</b>	<b>1.91</b>	<b>2.24</b>	<b>30.25</b>

### 2-4- The materials used in the consolidation

Some different nanomaterial and some chemical polymers were selected for use in consolidating samples of limestone, in order to choose the most suitable for the treatment and maintenance of limestone used as a basic building material as follows:• Individual Nanomaterial, In this case, Nanomaterial are used in the form of dispersed or suspended in a carrier medium such as alcohol or water in different concentrations, where they are

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17- Tan Chin Ling, X-Ray Fluorescence (XRF) Analysis in Masonry Heritage Building Conservation in Malaysia , Vol 11, Issue 12, Dec 2020, Sys Rev Pharm 2020;11(12):484-488.

<sup>18</sup> \* The Ministry of Petroleum, The Egyptian Mineral Resources Authority, Central Laboratories Sector, 1 Ahmed El-Zaiat St, Dokki, Gezah, Egypt.

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applied directly afterwards, at concentrations of 5% with water.<sup>19,20</sup> • Composite nanomaterial, In this case, nanomaterial at different concentrations are added to the polymer with its solvent in order to increase the efficiency of the materials to which nanotechnology is applied from physical properties or mechanical properties such as the material's resistance to stress fracture, and in this case Nano-calcite and Nano-lime were used with Wacker OH and its solvent (metal turpentine), nanomaterial in this case applied in concentrations of 5%, 7% with the polymer and the solvent. The process of preparing the samples for consolidation was carried out in three stages using nanomaterial and Wacker OH with different concentrations, where the nonmetric materials were concentrated at a concentration of 5, 7%, singly or combined with Wacker OH, which was at a concentration of 5% with the solvent (mineral turpentine),<sup>21</sup> and they are as follows:, As Table No. (2) Shows, the stages of consolidation that took place with their different concentrations. A group of building material stones (limestone) available at the study site were selected and prepared, which were cut into small cubes (3 cm each for length, width and height). For building material. Various nanomaterial have been added, whether in water or in the appropriate polymer for each stage so that they are dispersed or suspended, then the different samples of stones are immersed and covered tightly so that it works to preserve the solvent and not volatilize it in a period not exceeding three hours, which is an appropriate time that allows the penetration of the dispersed nanomaterial in the solvent inside the limestone more, due to its high porosity and acceptability to absorb liquid materials. The stage of consolidation with different concentrations of Nano-materials that were made on some different samples of limestone. The stages were carried out with special tools prepared for that in six stages, with each stage given a special code to facilitate dealing with it. Limestone samples after hardening in preparation for testing the physical and mechanical properties.

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19- Manjusha Hariharan, Neethumol Varghese, Dr. A. Benny Cherian, Dr. P.V. Sreenivasan, Jenish Paul, Asmy Antony. K.A, Synthesis, Characterisation of CaCO<sub>3</sub> (Calcite) Nano Particles from Cockle Shells Using Chitosan as Precursor, International Journal of Scientific and Research Publications, Volume 4, Issue 10, October 1 ISSN 2014,2250-3153.

20- Aboelkasim Diab & Zhanping You Moisture susceptibility of Nano-sized Hydrated Lime-modified foamed Warm Mix Asphalt mixes, Asphalt Pavements – Kim (Ed), Taylor & Francis Group, London, ISBN,(2014), 978-1-138-02693-3.

21- Zeynep Adali-Kaya, Bernadette Tse Sum Bui, Molecularly Imprinted Polymer Nanomaterials and Nanocomposites, Atom-Transfer Radical Polymerization with Acidic Monomers, Angew. Chem, 2015, 127, 5281 –5284.

**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 Wacker OH 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. <sup>( 22)</sup>

Table No. (2) Shows, the stages of consolidation that took place with their different concentrations

Consolidation Steps.	Limestone
The first stage	samples of limestone did not undergo the stages of consolidation
The second stage	Nano-lime at a concentration of 5%, which is dispersed in water
Third stage	Nano-lime 5% + Wacker OH 10% + solvent (mineral turpentine)
Fourth stage	Nano-lime at a concentration of 7% + Wacker OH at a concentration of 10% with the solvent (metallic turpentine)
Fifth stage	Nano-calcite 5% + Wacker OH 10% with solvent (mineral turpentine)
Sixth stage	Nano-calcite 7% + Wacker OH 10% with solvent (mineral turpentine)

#### **2-5- Limestone Physical Properties after Consolidation.**

There is no doubt that the mechanics of damage resulting from the various damage factors and the manifestations they cause affect the stones in general and limestone in particular, leading to weakness and destruction of the internal structure as well as the

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22- Omar Mohamed Adam Abdel Hameed, Nanotechnology applications to improve the mechanical properties of lime mortars in archaeological building, JOURNAL OF THE FACULTY OF ARCHAEOLOGY –VOLUME 26 - JANURAY 2023, p 423-483.

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external texture of the various building materials, which requires treatment so that the stone continues to resist the continuous factors of damage.

Among the most important physical properties that have been studied to know the status reached by the limestone, the effectiveness and resistance of the various consolidation materials that have been applied, whether on the internal or external texture, The following properties were determined: Bulk Density, Natural Water Content in the stone, Water Absorption and Apparent Porosity, Where the physical properties were determined on the limestone samples after consolidation.

### **2-6- Compressive Strength Test for Limestone Sample**

The compressive strength of a material is the value of the axial compressive stress that the material reaches upon complete collapse. The compressive strength resistance of limestone samples after consolidation which was done with Nano-materials dispersed in water or in organic solvents was determined with Waker OH with comparing the results with the reference samples that have not undergone any stages yet.

### **2-7- Compressive Strength for Consolidated Limestone after Salt Weathering.**

The results of the analysis that were conducted for the limestone samples showed that they contain sodium chloride salt NaCl. A salt aging was carried out using this type of salt on the limestone samples that were subjected to different consolidation stages to determine the extent of their resistance to this type of damage factors fig (11).<sup>23,24,25,26</sup>, Therefore, it was necessary to supply limestone samples with sodium chloride salt after consolidation and then subject them to Compressive Strength in order to find out which of the consolidation materials resist sodium chloride salt.

## **3- Results and discussion**

**3-1-** Result of SEM with EDX, Where fig (7)., with 3000 x, and the following Fig. (8), with 6000 x. They show the extent of the deterioration that occurred to the physical structure of the stone in addition to the disintegration of its grains and weakening of its

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23 - Silvestro A. Ruffolo, Mauro F. La Russa, Piergiorgio Aloise, Cristina M. Belfiore, Andrea Macchia, Antonino Pezzino , Gino M. Crisci, 2014, Efficacy of nanolime in restoration procedures of salt weathered limestone rock, *Appl Phys A* (2014) 114:753–758.

24 - Simona Scrivano · Laura Gaggero, An experimental investigation into the salt-weathering susceptibility of building limestones, *Rock Mechanics and Rock Engineering* (2020) 53:5329–5343.

25 - Silvestro A. Ruffolo, Mauro F. La Russa, Piergiorgio Aloise, Cristina M. Belfiore, Andrea Macchia, Antonino Pezzino , Gino M. Crisci, 2014, Efficacy of nanolime in restoration procedures of salt weathered limestone rock, *Appl Phys A* (2014) 114:753–758.

26 - Silvestro A. Ruffolo, Mauro F. La Russa, Michela Ricca, Cristina M. Belfiore, Andrea Macchia, Valeria Comite, Antonino Pezzino, Gino M. Crisci, new insights on the consolidation of salt weathered limestone: the case study of Modica stone, *Bull Eng Geol Environ*, 2017, 76:11–20.

internal structure, as the picture shows some crystallized sodium chloride on the surfaces of calcite crystals. <sup>27,28</sup> Fig. (7, 8, 9). - As shown in Fig. (9), the result of the elemental analysis of the limestone sample consists mainly of calcium and the presence of silicon, sodium, magnesium, aluminum and sulfur as impurities as a result of deterioration factors. Fig. (7,8). the deterioration that occurred to the physical structure of the stone in addition to the disintegration of its grains and weakening of its internal structure, it also explains the result of the elemental analysis of the limestone sample.

**3-2-** The results of x-ray diffraction indicates that the compositions of the sample are: (major calcite  $\text{CaCO}_3$  + Halite  $\text{NaCl}$ , minor + traces Dolomite  $\text{CaMg}(\text{CO}_3)_2$ , Nitratine  $\text{NNaO}_3$  sodium;oxido Nitratine). Fig.9. It was noted a large proportion of halite and Nitratine salts had been observed as a result of water leakage from the sewage surroundings the object, the danger of these salts is on the internal structure of the stone, creating pressures that weak the internal bonds of the stone grains and make as continuous source of moisture. . <sup>(29)(30)</sup>

**3-3-** The results of XRF proved the presence of many elements that are present in varying proportions in the limestone sample, as it was proven that the presence of each of the elements: calcium and carbon in a large proportion, and then the presence of each of the elements: silicon, sodium, sulfur and chlorine varies, and Table No. (1) Shows the results of the element analysis using the fluorescence device, as it shows the proportions of the presence of each element separately.

**3-4-** The results of physical properties, Perhaps the most important treatment methods by using different materials and then identifying the physical properties of the stone after that and comparing it with the physical properties of the treated stone and comparing those different materials and choosing the best one and then applying the best possible way , Table No 3 and Fig No. (11, 12) shows the results of some physical properties that occurred on the limestone after the different stages of consolidation.<sup>31, 32</sup>., when

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27- MOHAMMED A. AMIN. Weight loss, polarization, electrochemical impedance spectroscopy, SEM and EDX studies of the corrosion inhibition of copper in aerated NaCl solutions, Springer, Journal of Applied Electrochemistry(2006), , 36:215–226.

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29 - Simona Scrivano & Laura Gaggero, An experimental investigation into the salt-weathering susceptibility of building limestones, Rock Mechanics and Rock Engineering volume 53, pages5329–5343 (2020).

30 - Mustafa Yavuz Çelik & Murat Sert, The role of different salt solutions and their concentration ratios in salt crystallization test on the durability of the Döğer tuff (Afyonkarahisar, Turkey) used as building stones of cultural heritages, Bulletin of Engineering Geology and the Environment volume 79, pages5553–5568 (2020).

31- H.YavuzS.DemirdagaS.Caran, 2010, XThermal effect on the physical properties of carbonate rocks, International Journal of Rock Mechanics and Mining Sciences, Volume 47, Issue 1, January 2010, Pages 94-103.

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determining the bulk density showed that the samples that were consolidated with Nano-calcite with Wacker OH at a concentration of 7%, where the highest density, followed directly by the samples that had been consolidated with Nano-calcite with a concentration of 5 % with Wacker OH.

Table No 3, physical properties results after the different stages of consolidation

Consolidation stages	volume cm <sup>3</sup>	normal weight gm.	saturated weight gm.	dry weight gm.	bulk density g/cm <sup>3</sup>	normal water content	water absorption %	apparent porosity %
Reference sample	26.4	42.3	50.3	36.2	1.37	16.85	38.95	53.41
Nano-lime 5%	27.1	43.2	51.2	38.4	1.41	12.52	33.33	47.23
Nano-lime with wacker OH 5%	26.7	47.5	55.5	42.7	1.59	11.24	29.97	47.94
Nano-lime with wacker OH 7%	27.3	48.7	56.7	43.9	1.61	10.93	29.15	46.88
Nano-calcite with wacker OH 5%	25.9	52.6	56.6	47.1	1.81	11.67	20.16	36.67
Nano-calcite with wacker OH 7%	26.5	50.2	53.2	49	1.84	2.44	8.57	15.84

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32 - Hong Tian , Thomas Kempka , Neng-Xiong Xu , Martin Ziegler, 2012 , Physical Properties of Sandstones After High Temperature Treatment, Rock Mech Rock Eng, Springer-Verlag , 45:1113–1117.

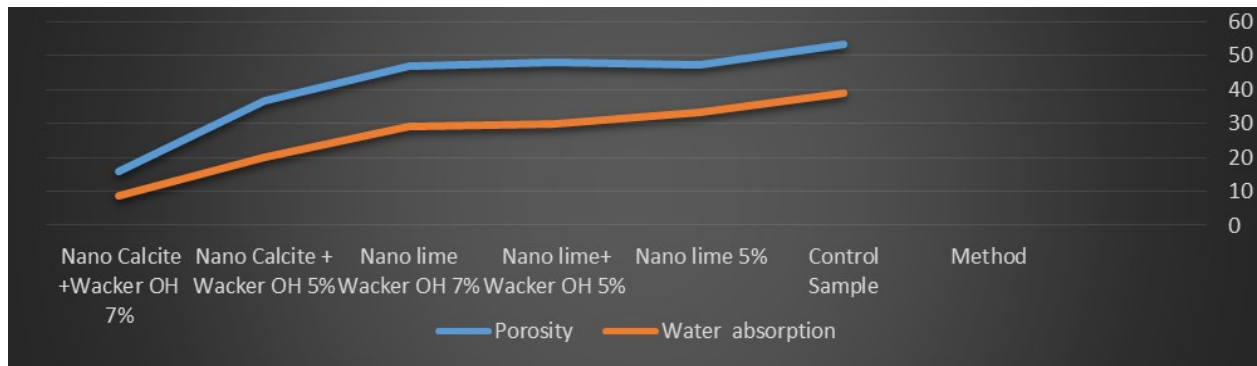


Fig. 11, Bulk Density determination after consolidation

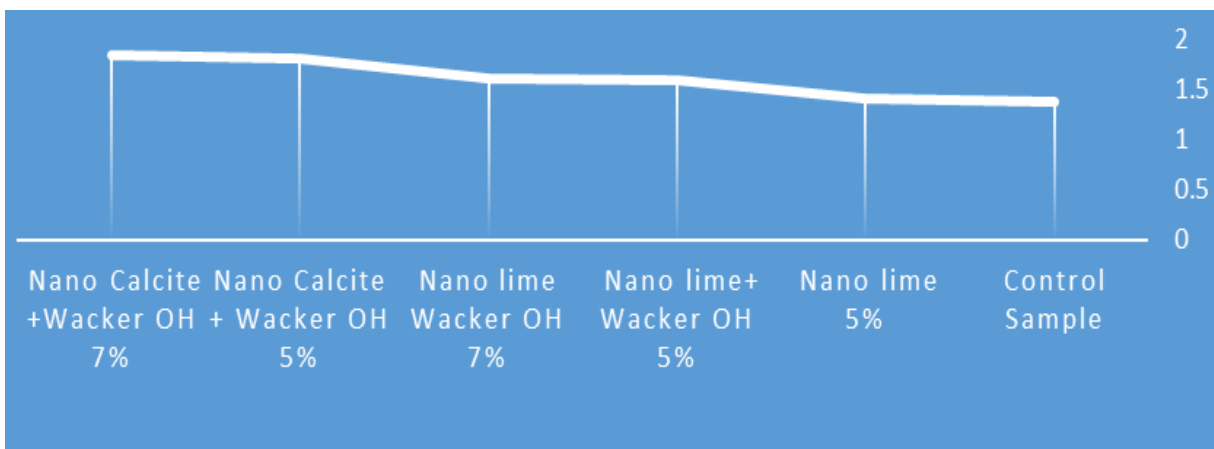


Fig. 12, Apparent porosity and water absorption before and after consolidation ...%

**3-5-** The results of compressive strength, were arranged from the highest resistance to the lowest compared to the reference sample, as shown in Table No. (4). Figure No. (13) Show the results of the compressive strength values for the average results tested for limestone, after the various consolidation processes that were conducted on it, with a comparison of the results with the average of the reference samples that were not subjected to the consolidation stages. When measuring the Compressive Strength, samples that were consolidated with Nano-calcite with a concentration of 7% took the first place with the highest resistance, with an increase of 64% compared to the reference sample, immediately followed by the samples that were consolidated with Nano-calcite in a concentration of 5% with Wacker OH and then the samples that consolidated with Nano-lime at a concentration of 7% and 5%, respectively, then in the Nano-lime with water samples.



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Table No. (4) Arrangement of Limestone Compressive Strength compared to the reference sample.

Arrangement of Compressive Strength	Limestone after consolidation	Limestone compared to the reference sample ...%
The first resistance	Nano-calcite 7% + Wacker OH	64.71
The second resistance	Nano-calcite 5% + Wacker OH	59.81
The third resistance	Nano-lime at a concentration of 7% + Wacker OH	26.98
The fourth resistance	Nano-lime at a concentration of 5% + Wacker OH	11.13
The fifth resistance	Nano-lime at a concentration of 5%, dispersed in water	15.73
reference sample	did not undergo the stages of consolidation	0

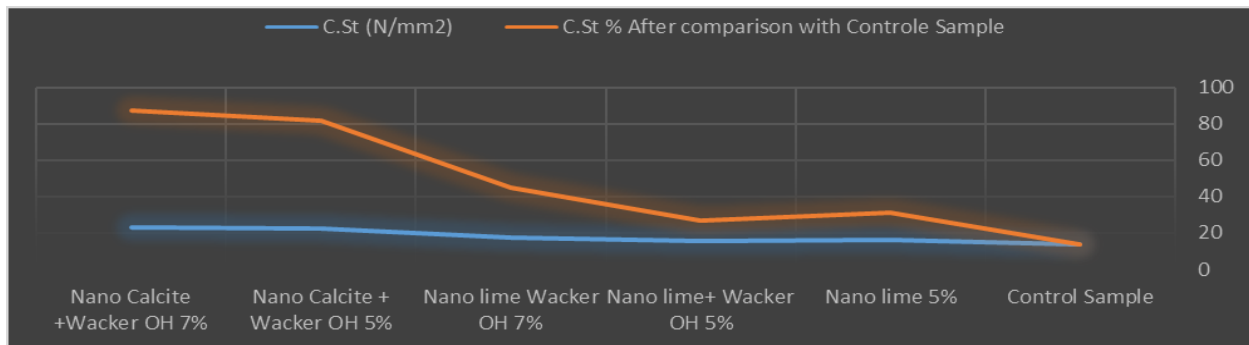


Fig. 13, consolidated Limestone samples after Compressive Strength compared to the reference sample

**3-6-** Result of Compressive Strength for Consolidated Limestone after Salt Weathering, were arranged from the highest resistance to the lowest compared to the reference sample, as shown in fig no 14, When Compressive Strength was determined after salt aging, the samples that were consolidated with Nano-calcite with Wacker OH proved a better resistance result when compared to Nano-lime with Wacker OH.

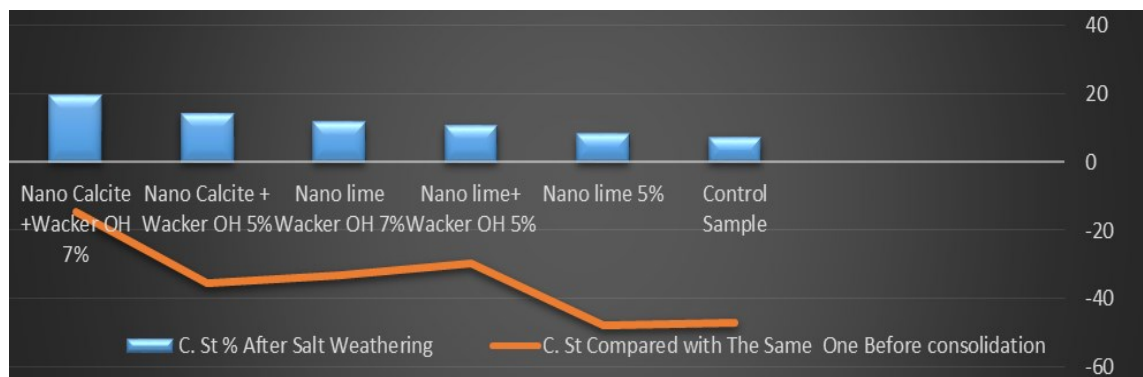


Fig. 14, Compressive Strength after Salt Weathering (NaCl)

#### **4- Conclusions**

The results of the physical properties measurements that were carried on limestone samples after the different consolidation stages when determining the bulk density showed that the samples that were consolidated with Nano-calcite with Wacker OH at a concentration of 7%, where the highest density, followed directly by the samples that had been consolidated with Nano-calcite with a concentration of 5 % with Wacker OH. When measuring the Compressive Strength, samples that were consolidated with Nano-calcite with a concentration of 7% took the first place with the highest resistance, with an increase of 64% compared to the reference sample, immediately followed by the samples that were consolidated with Nano-calcite in a concentration of 5% with Wacker OH and then the samples that consolidated with Nano-lime at a concentration of 7% and 5%, respectively, then in the Nano-lime with water samples. When Compressive Strength stress was determined after salt aging, the samples that were consolidated with Nano-calcite with Wacker OH proved a better resistance result when compared to Nano-lime with Wacker OH.

Nanotechnology has recently proven its worth and leadership in use and application due to the ability of these materials to penetrate into the pores of the stone, so it is necessary to focus on its study and application. Wacker OH material has proven its effectiveness when adding with various Nano materials, especially with Nano-calcite when consolidated limestone. When applying both Nano-calcite and Nano-lime at different concentrations 5%, 7% with the polymer (Wacker OH dissolved in mineral turpentine at a concentration of 10%), as well as Nano-lime applied individually with water at a concentration of 5%, the success of Nano-calcite is significantly different from Nano-lime with polymer or with water, and this has been shown when measuring physical properties or Compressive Strength testing, so it is recommended to use and apply it in the field of restoration and conservation of limestone monuments.

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