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Evaluation of Effectiveness between Wacker Polymeric Material and its Silver Nanocomposite in Glazed Pottery Consolidation

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Abstract

The research dealt with one of the modern applications of restoring and conserving antiquities. One of the most important applications is the use of nanoparticles and their addition to traditional consolidation materials to improve their properties in consolidating glazed pottery samples. This paper aims to evaluate the use of wacker OH100 in its traditional form and wacker OH100 + nano silver to improve its properties and apply it to the experimental glazed pottery samples. This study used some investigation and analysis methods for the evaluation process, TEM was used to measure the size of prepared nanocomposites. Microscopes such as SEM and USB digital were used to investigate the surface morphology. Static water contact angles were measured to assess the hydrophobic characteristics of the consolidants utilized in this investigation. Color change, as well as physical and mechanical testing, were all examined. According to the findings of this investigation, the treated sample with Wacker OH100 + nano Silver at 3% achieved the best results where the contact angle reached 116o, colour change stops at ΔE value 5.43, and compressive strength maximized 518 Kg/cm2 with an improvement rate of 64.96%.

Keywords: Wacker polymeric, Nano silver, contact angle, consolidation, glazed pottery, TEM, SEM

1. Introduction:

One of the deterioration processes of ceramic material is the exfoliation of the surface, which may cause a partial removal of the glaze [1]. Improper grinding of different glaze layers, since there is a grade of grinding smoothness that must achieve, as the improper grinding process causes defects to appear, so that increased grinding improves the surface texture of the glaze layer but, in the case of its increase in some coatings, it cracks during drying and collects during the firing process [2,3]. Many of The SiO_2- PbO glaze layers heavily deteriorated [4]. Consolidation is a process in which the cohesion and strength of the internal object structure are restored to enhance the mechanical and physical properties [5]. The success of the consolidating material depends on its suitability to the composition and properties of glazed pottery [6]. The strengthening material must be able to consolidate and protect the fragile surface layers [7]. The consolidating material must have the ability to penetrate inside the body to improve its mechanical properties [8]. TECHNICAL-Characteristics of WACKER® BS OH 100 consolidant is a solvent-less, ready-to-use product for consolidating construction materials. It produces a mineral binder that is convenient with the construction material. The binder is acid-resistant and therefore resists rainwater. It does not close pores, and the construction material remains penetrable to water vapour. WACKER® BS OH 100 is based on ethyl silicate. When applied, it penetrates through the capillaries deep within the construction material. It is Color from colourless to yellowish - Ethyl silicate content, approx. [wt %] 100 - Density at 25 °C,

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approx. [g/cm3] 1.0 - Catalyst neutral - Flashpoint 40 °C – Ignition temperature 230°C. Nanotechnology plays an important and effective role in the processes of consolidating and improving the characteristics of inorganic archaeological materials. In addition, overcoming many obstacles faced by archaeological materials [9,10]. Especially in the topic of the research about using nano silver with traditional material in Consolidate glazed pottery objects and comparing the results after using this technique to improve the properties of traditional materials used to strengthen glazed pottery. Since traditional methods failed to produce positive results compared to nano methods, using a brush in the application of consolidation helps to form a thin layer on the outer surface [11]. However, the application by immersion can harm the red-firing pottery, and there are other ways to apply reinforcement materials, such as spraying and injections. We compare both methods in consolidating glazed pottery with (Wacker OH100 + nano Silver) at concentrations of 1%, 3%, and Wacker OH100 without nano Silver additive. Then discussed the results. Adding nanoparticles to various traditional boosters increases their resistance to light and heat, and the more homogeneous the nanoparticles, the more they react chemically, and the use of nanomaterials contributes to high heat resistance and is highly capable of resisting biological damage [12]. Nanomaterials are highly capable of penetrating the deepest depth within the pottery artifact to strengthen, which helps to bind the inner granules and the strength of their cohesion and improves visual properties and the absence of colour change after the application of Reinforcement materials. Nanomaterials have been widely used in recent times because of their ability to improve the properties of traditional boosters [13]. When nanoparticles are in different polymers, this leads to resistance to pressure strains, and the addition of nanoparticles in treatment and maintenance increases the effectiveness of these effects on water ejection that the water's contact angle with the surface is greater than 150° and is considered a waterproofing material [14]. Whenever the water's contact angle with the surface exceeds 120°, the material is considered to be highly water-ejecting [9].

Nano silver particles are tiny silver grains, meaning they are silver particles of a size of between (nm 1-100 nm) and usually described as "silver", some of which consist of a large percentage of silver oxide due to the large surface ratio they possess compared to bulk silver atoms. Nanoparticles are one of the most important of these materials due to their potential applications in many areas, usually high-purity silver. There are various methods for manufacturing silver nanoparticles, and they can be divided into three broad categories: physical steam deposition (PVD), ion infusion, or wet chemistry [15]. The ability of silver nanoparticles to resist bacteria efficiently has no effect on the substance of monuments [16]. As anti-bacterial agents, AgNPs were applied in a wide range of applications, from disinfecting medical devices and home appliances to water treatment [17]. The Effect of silver nanoparticles appears on MMA/HEMA behaviour in enhancing and restoring the lost chemical bonds of paper [18]. Moreover, this encouraged the textile industry to use AgNPs in different textile fabrics. In this direction, silver nanocomposite fibres were prepared to contain silver nanoparticles incorporated inside the fabric [17].

2. Materials and Experiments

2.1. Materials

WACKER® BS OH 100 purchased from BRESCIANI company for Materials and Equipment for the Restoration and Conservation. Silver nanoparticle with an average particle size of 80 nm and density of 0.02mg/ml was supplied from Thermo Fisher Scientific – China.

2.2. Preparation of consolidation materials

Wacker / Silver nanocomposite solution was prepared by adding 0.03 g of silver nanoparticles into 100 ml Ethanol. The solution was mixed for about 10 min; then, 1.0 g of wacker crystal was added and mixed vigorously. The mixture was sonicated for 15 min to obtain 3% concentrations of Wacker / Silver nanocomposite. Wacker / Silver nanocomposite prepared in the Polymer Department, National Research Centre, Dokki, Giza, Egypt.

Table 1. The samples and materials used in consolidating the experimental standard samples.

Sample code	Consolidation material
U	Untreated sample
A	Wacker OH100
B	Wacker OH100 + Nano Silver 1.0%
C	Wacker OH100 + Nano Silver 3.0%

2.3. Preparation of glazed pottery samples

The appropriate clay was chosen to form the required experimental glazed pottery cubes on certain bases, including having suitable properties for forming such as plasticity - accepting glass coatings - mixing it with clay or other materials to obtain a good paste, from binders, melting materials, glazed materials [19]. One of the valid clays available in Egypt that can give good results is the ball clay. The glaze was prepared with different components, including:

A - Powder glaze (the vitrified material - silica, which is the basis for the glaze).

B - Different types of clays are added in specific proportions to the glaze mixture, and they are often of the same material as the body.

C - A melting substance (whether basic or alkaline, such as lead oxide or borax) that lowers the melting temperature of the glaze.

D - Materials to give different properties (coloured, opaque, or other materials).

E - Other binding materials.

1- The components of the body samples were weighed, and the clay was mixed to avoid structural defects during the preparation of the samples (Table 2), to obtain the requisite plasticity, the proper amount of water has been applied. The time required for the clay to form can be days. Then the clay is shaped into cubes using a wooden mold with a dimension of 3 cm edge of the cubes [20].

2- The samples were placed in the kiln to fire at 855 degrees, gradually, where the oven temperature was adjusted to 50 degrees and then raised 10 degrees every quarter of an hour until the temperature reached 855 degrees [21].

Table 2. Pottery body samples were prepared in the lab with the following ingredients.

Ingredients				**Firing temperature**
170 gm. water	20 gm. kaolin with high silica content	30 gm. quartz	75 gm. ball clay	855 degrees

2.4. Applying the glazing layer to experimental standard samples

The ingredients for the glaze stir well to become homogeneous (Table 3). Then the glaze layer was applied by brush to the cubes of the body, then left to dry in preparation for firing in the kiln, and then fired at temperatures of 1000 degrees (Fig. 1).

Table 3. The glaze layer is prepared with the following ingredients
.

Ingredients				**Firing temperature**
100 gm. water	1 gm. Egyptian iron	2 gm. copper	100 gm. Feldspars White 60%	1000 degrees

Fig.1. standard glazed pottery samples.

2.5. Thermal and light aging

Thermal aging on treated samples carried out for 100 hours at 100 °C, and relative humidity of 60%.

The samples were placed in an oven with temperature control (FN500 - Germany) on a custom frame. A UV lamp was used for a 100-hour long light aging test (268 improved/small-format UVA sensor heads). Lamp in the following case: (power: 600 watts, irradiation wave range: 400 nm, the distance between sample, lamp: 15 cm).

2.6. Characterizations

2.6.1. Transmission Electron Microscope (TEM)

(JEM-1230 electron microscope operated at 60 KV (JEOL Ltd., Tokyo, Japan). Prior to examination, the sample diluted at least 10 times by water. A drop of well-dispersed diluted sample was then placed onto a copper grid (200-mesh and covered with a carbon membrane) and dried at room temperature). The examination carried out at the National Research Center in The Dokki district of Cairo – Egypt. To make sure that the size of the granules arrival to the nano scale, as the smaller the dimensions of the substance, the greater the number of its atoms on the surface.

2.6.2. USB Digital Microscope

USB 2.0 interface, Linux, Mac OS & above 10.5.5, from (10X-500X), Model: PZ01, made by Shenzhen Supereyes Co., Ltd., China. This procedure was achieved at the Conservation Dept., Faculty of Archaeology, Cairo University, Egypt.

2.6.3. Scanning electron microscope (SEM)

SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Ana-lyzes), with accelerating voltage 30 K.V., magnification14x up to 1000000 and resolutions for Gun.1n). FEI Company, Netherlands was used. This procedure was executed at the General Authority for Mineral Resources in Dokki – Egypt.

2.6.4. Static water contact angle

This test was performed on samples before and after the paint. Canon's high-resolution camera with an 18-55 lens is used to take pictures, and a dedicated program is then used to calculate the water droplet contact angle values on the sample's surface. The test is being conducted at the National Research Center in Dokki district of Cairo.

2.6.5. Color alteration

This test is based on measuring color change using Optimatch 3100® from the SDL Company at the National Institute of Measurement and Calibration.

2.6.6.. Physical properties

The physical characterization of these samples was determined by measuring their dry and wet weights. The dry weight is set by placing the samples in a dryer at a temperature of 105°C for 24 hours, and then the wet weight is set after the harsh experimental samples are immersed for 24 hours. The following physical characteristics were calculated [21]:

Water Absorption: Through dry and wet weight, the water absorption is set according to the following equation:

$$\text{Water absorption ratio} = \frac{\text{Wet Weight - Dry Weight}}{\text{Dry Weight}} \times 100$$

Porosity: Porosity is the ratio of the size of empty pores to the total size of the standard sample and depends [1]. The porousness ness of the archaeological pottery is mainly on the mechanical fusion of the metal constituents of the clay during the process of firing. Moreover, the size of the molecules and the amount of organic matter to be found within the components of pottery and did not lose during the firing phase and left internal cavities in place, and the

$$\text{Visible porosity ratio} = \frac{\text{Wet Weight - Dry Weight}}{\text{Total volume}} \times 100$$

ratio of virtual porosity is set according to the following equation [23]:

Bulk Density: Density is the mass-to-size ratio, density influenced by transcendence, the amount of spaces, and pores affected by temperature and fire rate, and the density is estimated g/cm [24]

$$\text{Bulk Density} = \frac{\text{Mass (dry weight)}}{\text{volume}} \quad g/cm^3$$

2.6.7. Compressive Strength

Tinius Olsen QMat5.37/Q3214. The operating circumstances (Load Range: 10000N, Expansion range: 10mm, Speed: 305 mm/min, Endpoint: 5.0 mm, Preload: 1.0N). The testing was carried out at the National Institute of Standards, Egypt.

2.6.8. Determination of the percentage of residual resin solid

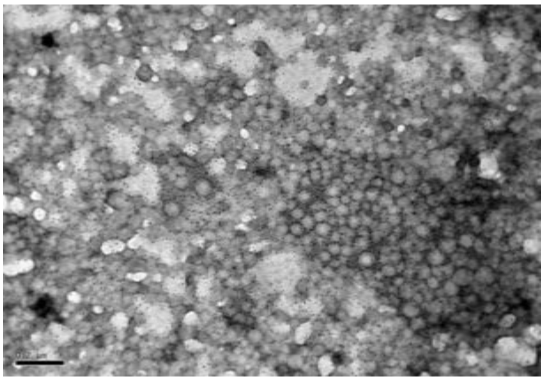
This method depends on the saturation degree of the solution and its concentration, as well as the number of saturation cycles. This ratio is determined by the weight of samples before the application of different Consolidating materials and after the completion of its application, at least one month until the polymerization process of those materials used in Consolidation has been completed, and the remaining solid resin material calculated through the following equation:

$$\text{Percentage of solid material remaining} = \frac{\text{Weight after Consolidation - weight before consolidation}}{\text{Weight before Consolidation}} \times 100$$

3. Results and discussion

3.1. Transmission Electron Microscope (TEM)

The use of Transmission Electron Microscope (TEM) proof the success of the interaction and mixing between polymer and nanometer, and make sure the size of the granules and their arrival to the nano scale, it explained in the following panel **(Fig.1, 2)**:



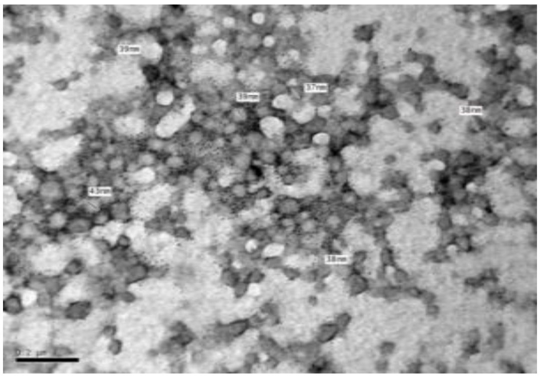


Fig.1, 2. Showing the compound of the material (Wacker H100 + Nano Silver) used to strengthen under the powerful electron microscope**.**

3.2. USB Digital Microscope

Using a USB digital microscope both before and after treatment has the advantage of illustrating how the application of consolidated materials changed the brightness of the ceramic sample surfaces [1,**25**]. The coverage of the outer surface illustrates good coverage without any surface brightness, and therefore it is considered Wacker OH100 + (Nano Silver 3.0%) the best material from its positive result (Fig. 3).

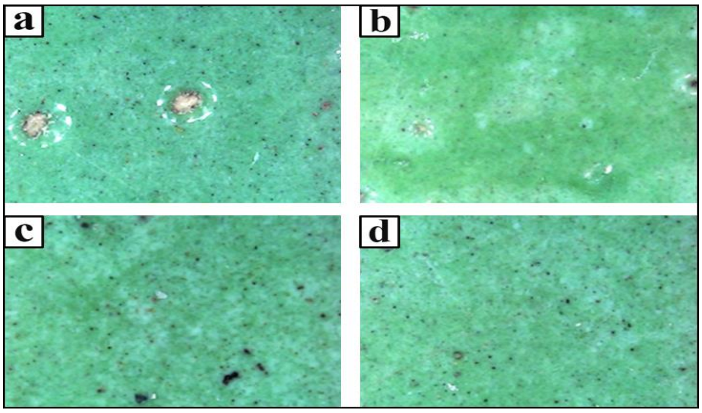


Fig. 3. USB digital microscope images of the untreated and treated/consolidated samples in magnification 200-500, as in the same order in fig. 2.

3.3. Scanning Electron Microscope (SEM)

Scanning electron microscope-(SEM) is an investigation technique used to characterize the preservation state and determine the possible causes of variation and deterioration phenomena of glazed pottery and other archaeological materials [1].

Using a scanning electron microscope to examine the surface morphology of the untreated and treated samples. (a) Untreated sample, (b) sample treated/consolidated with Wacker OH100, (c) sample treated/consolidated with Wacker OH100 + Nano Silver 1% nanocomposite, (d) sample treated with Wacker OH100 + Nano Silver 3% nanocomposite. To show the most effective polymer selected depending on the results of previous analytical methods to investigate the surface morphology of each selected sample (Fig. 4).

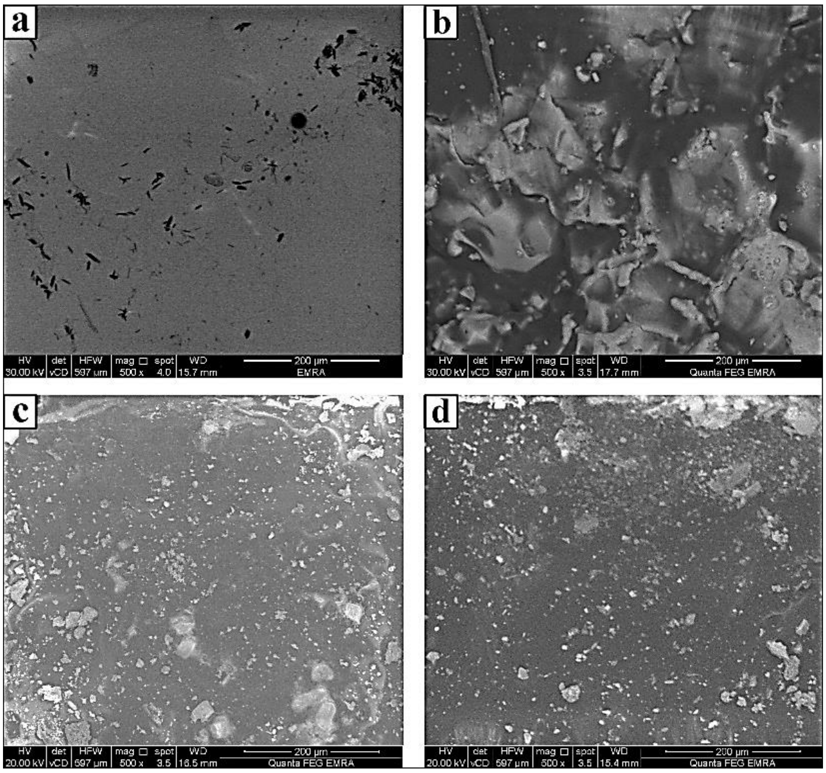


Fig. 4. SEM microscope images of the untreated and treated/consolidated samples. (a) Untreated sample, (b) the sample treated/consolidated with Wacker OH100, (c) sample treated/consolidated with Wacker OH100 + Nano Silver 1% nanocomposite, (d) the sample treated with Wacker OH100 + Nano Silver 3% nanocomposite.

3.4. Static water contact angle

The assessment of the extent to which the surfaces on which the reinforcement materials have been applied is able to be affected by water by determining the angle at which water droplets contact a surface, where the angle value of less than 90° indicates that the surface is water-friendly and attractive. vice versa indicates the value of the angle greater than 90° that the surface is considered water-repellent [26].

Measuring the angle of the water connection is also very necessary. it aims to evaluate the consolidating materials that are characterized by the characteristic of water ejection, which is also used to determine the susceptibility of surfaces to wetness, where interest has increased in recent years in the study of surfaces above water-repellent using nanomaterials that gain these characteristics traditional materials (Fig. 5).

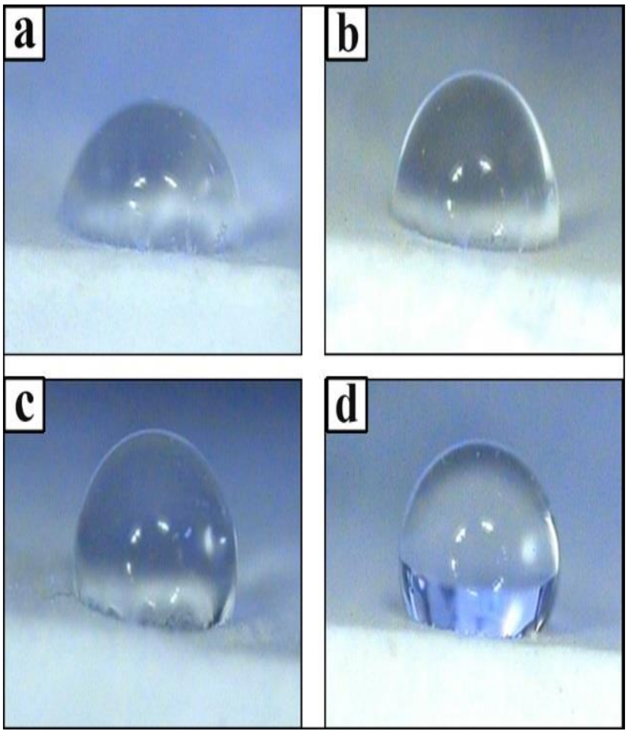


Fig. 5. The results of measuring the water contact angle of untreated glazed pottery samples and treated/consolidated samples.

(Table 4) results of measuring the angle of water contact on experimental standard samples after being subjected to industrial obsolescence and applying the consolidating material to them.

Table 4. Water contact angle values.

Sample code	Water contact angle
U	50
A	80
B	107
C	116

It is clear from the table and the results of measuring the angle of water contact that sample C, consisting of synthetic wacker OH100 + nano silver at a concentration of 3%, gave the highest angle, 116%, compared to other samples and is therefore considered one of the best materials that can be used in consolidation
.

3.5. Color change

The glazed pottery's visual appearance was assessed utilizing colorimetric methods [27]. The termination of the industrial obsolescence process is essential to measure the color change to reach the samples that gave color stability. In addition, the absence of an apparent color change affects the external form of samples applied to damaged reinforcement materials, and therefore the possibility of selecting the best materials that gave good results and applying them in the consolidation process for fragile pottery objects. Testing the color change of standard samples after obsolescence and applying boosters is one of the most important tests that show whether different reinforcement materials can make a clear difference in the external appearance of these samples. The color change of the samples is measured using the (CIE Lab) system; it is an international system based on the measurement and determination of (I^* - a^* - b^* - ΔE).

Color change measured according to the following equations, (Table 5);

$$(\Delta l^* = l^*t - l^*0)\ (\Delta a^* = a^*t - a^*0)\ (\Delta b^* = b^*t - b^*0)$$

$$\Delta E = \sqrt{(\Delta l^*)2 + (\Delta a^*)2 + (\Delta b^*)2}$$

Table 5. Explain equation symbols.

symbol	The meaning of the symbol
0	Standard sample before Consolidation materials are applied to them
t	Symbolizes samples after the application of Consolidation materials
I*	Bright sample lighting
a*	Refers to red to a green where it is slanted to red when the symbol +a
b*	Represents the color from yellow to blue, where the color is yellow when the symbol +b and is slanted to blue when the symbol -b
ΔE	Expresses the value of color change of samples

The obtained **ΔE** Values below 3 reflect a simple color change that is not noticeable to the eye of the abstract. While values of 3-6 reflect a slight and noticeable color change to the naked eye or values greater than 6 indicate a perceptible and significant change in colour to the bare eye. The results of color change are as evident in (Table 6):

Table 6. The results of the color change of the experimental standard samples

Sample	l	Δl	a	Δa	b	Δb	ΔE
U	56.65	----	-8.43	----	8.86	----	----
A	69.49	3.93	-9.09	-0.66	8.21	-0.65	4.08
B	62.70	-2.86	8.88	-0.45	8.48	-0. 38	2.92
C	70.83	5.27	-8.17	0.26	10.13	1.27	5.43

The results mentioned in the previous table show that, samples A, B and C gave a slight color change that was not noticeable to the naked eye because they gave grades ranging from 3-6.

3.6. Physical properties:

The study of the physical properties of experimental standard samples is one of the most important steps used in the evaluation of traditional and nano-reinforcement materials to strengthen pottery to determine the extent to which these materials are used on experimental samples for comparison and achieve results to choose the best-strengthened material (Table 7) [28].

Water absorption test results

(Table 7) shows that samples B and C consisting of nano silver 1.0% - 3% nano silver + (Wacker OH100), recorded a water absorption rate of between 20.154%, and then in the last stage comes to sample A Consisting of pure + (wacker OH100), and recorded the highest water absorption rate of 27.377%.

The porosity test results

Sample C Consisting of Nano Silver 3.0% + Wacker OH100, recorded a virtual porous ratio of 28,344 percent, followed by samples B and a virtual porous ratio of 32 % to 33 %. Then in last place comes sample A, which consists of pure OH100 (Wacker) and recorded the highest apparent porous rate of 36,033%.

Density test results

The following table shows that sample C, consisting of Nano Silver 3.0% + (Wacker OH100), recorded the highest total density value of 1.406g/cm^3.

Table 7. The results of the appointment of physical properties of experimental samples after the application of consolidating materials

Sample code	Dry weight (gm.)	Wet weight (gm.)	volume (cm3)	Water absorption%	porosity %	density (g/cm^3)
U	36.184	45.389	27	25.439	34.092	1.340
A	35.536	45. 265	27	27.377	36.033	1.316
B	36.716	45. 525	27	23.992	32.652	1.359
C	37.972	45. 625	27	20.154	28.344	1.406

3.7. Compressive Strength

The pressure resistance test is essential for determining the mechanical characteristics of pottery samples. Where it shows how well the reinforcement materials used are able to strengthen experimental samples and withstand pressures and loads or not, in addition, to giving a general indication of the extent to which these clubs used in consolidation have good physical and mechanical properties (Table 8) [29].

Table 8. Determination of the compressive strength and its improvement rate %.

Sample code	Measurement of compressive strength (kg/cm2)	Rate of improvement Measurement of compressive strength(%)
U	314	———
A	369	17.51
B	448	31.40
C	518	64.96

According to the previous table, sample C consisting of 3.0% Nano Silver + Wacker OH100, recorded a pressure resistance score of 518 kg/cm^2, an increase of 64.96%. The following sample B consisting of nano (Wacker OH100+ nano silver 1.0%), recorded a pressure resistance score of 448 kg/cm^2, an increase of 31.40%.

3.8. Determination of the percentage of residual resin solid

It is clear from the table that sample **C** consisting of (Wacker OH100+ nano silver 3.0%), is one of the best samples that gave good results in consolidation, with the remaining solid material accounting for 8.56%. Then comes sample **B** with 4.97%, followed by sample **A** with the remaining solid material ratio of 1.60% (Table 9).

Table 9. The remaining solid ratios after the application of different consolidate materials.

Sample code	Sample weight (g)	Percentage of solid material remaining %
U	34.977	—
A	35.536	1.60
B	36.716	4.97
C	37.972	8.56

4. Conclusion

This study compared the fundamental characteristics of glazed pottery samples before and after consolidation with (Wacker OH100) in traditional/conventional and nano forms. Moreover, this was accomplished through some examination/investigation and the achievement of mechanical and physical properties. TEM Microscopes used to examine and ensure the success of the interaction and mixing between polymer and nanomater. Microscopes such as SEM and USB digital to investigate the surface morphology. Static water contact angles were measured to assess the hydrophobic characteristics of the consolidants, indicating that nano (Wacker OH100+ Nano Silver) at 3% was the ideal material for glazed pottery consolidation. Consequently, it created a consistent and homogeneous coating and improved the capacity of the polymer's ability to penetrate by filling the pores. Traditional polymer application resulted in significant colour changes from untreated samples, but the (Wacker OH100+ Nano Silver) had essentially no visual impacts/effects on the pottery colour objects. Additionally, this coating gives significant resistance/isolation versus water permeation. The samples' hydrophobic properties after being treated with nanoparticles compared to those which received pure polymer treatment better than those treated with pure polymers. Basically, it revealed that using nano (Wacker OH100 + Nano Silver) at 3% enhanced the mechanical and physical properties of the pottery samples and its high resistance against microorganisms. It can be advised to use this material to consolidate ancient glazed pottery based on the results of this study.

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تقييم الفعالية بين مادة الفاكر البوليميرية التقليدية ومادة الفاكر بمركب الفضة النانوية لها في تقوية الفخار المزجج

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الملخص

تناول البحث أحد التطبيقات الحديثة لترميم الآثار والحفاظ عليها . ومن أهم التطبيقات استخدام الجسيمات النانوية وإضافتها إلى مواد التقوية التقليدية لتحسين خصائصها في تقوية عينات الفخار المزجج . يهدف هذا البحث إلى تقييم استخدام مادة الفاكر wacker OH100 في شكله التقليدي ، والفاكر wacker OH100 مع النانو فضة wacker nano silver لتحسين خصائصه ، وتطبيقه على عينات الفخار المزجج التجريبية . وفى هذه الدراسة استخدمت بعض طرق الفحص والتحليل لعملية تقييم هذه المادة من خلال: الفحص بالميكروسكوب الإلكتروني النافذ TEM لقياس حجم جزيئات النانو المركبة المحضرة ، استخدام الميكروسكوبات مثل الميكروسكوب الإلكتروني الماسح SEM والميكروسكوب الرقمى USB لفحص طبيعة ومظاهر تلف السطح . وتم قياس زوايا اتصال الماء لتقييم الخصائص الطاردة للماء للمواد المقوية المستخدمة في هذا الاختبار ، كما تم فحص التغير اللوني ، وكذلك إجراء الاختبارات الفيزيائية والميكانيكية . ووفقًا لنتائج هذه الفحوص والاختبارات فقد حققت العينة المعالجة بمادة الفاكر wacker OH100 مع النانو فضة wacker nano silver بنسبة 3٪ أفضل النتائج ، حيث وصلت زاوية اتصال الماء إلى 116 درجة ، وتوقف التغير اللونى عند قيمة ΔE 5,43 ، وقوة الضغط وصلت إلى 518 كجم / سم 2 مع التحسن بمعدل 64.96٪ .