The effect of addition nano particle ZrO₂ on some properties of autoclave processed heat cure acrylic denture base material

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ABSTRACT

Background: Polymethyl methacrylate (PMMA) is used in denture fabrication and considered as the most reliable material for the construction of removable prosthodontic appliances. The material is far from ideal in fulfilling the mechanical requirements and the effect of autoclave processing has not been fully determined. The purpose of this study was to evaluate the effect of addition of salinized (ZrO₂) Nano fillers in percentages 3%, 5% and 7% by weight on some properties of heat cured acrylic processed the by autoclave and compare it with 0% (control) group.

Materials and methods: The silanized(ZrO₂) Nano-particles was added to PMMA powder by weight in three different percentages 3%, 5% and 7%, mixed by probe ultra-sonication machine.Two hundred specimens were constructed and processed by autoclave and divided into 5 groups according to the test (each group consist of 40 specimens) and each group was subdivided into 4 sub-groups according to the percentage of added (ZrO₂) nano-particles(with 10 specimens for each subgroups). The tests conducted were transverse strength, hardness (shore D), Impact strength test, surface morphology and apparent porosity. AFM can provide 3D image of the specimen the homogeneity of nanostructure film, roughness of surface and crystallite size. Scanning electron microscope SEM of control and salinized Nano ZrO₂ reveal the Nano fillers distribution and it is shaped.

Results: Highly significant increase in impact strength recorded when acrylic (vertex) mixed with 3%, 5% ZrO2 nano filler, while a non-significant reduction was observed with 7% ZrO2 addition in comparison to control. Non-significant improvement in transverse strength when 3% ZrO2 was added, 5% nano addition ZrO2 improved transverse strength significantly while 7% nano addition showed anon significant reduce when these groups compared to the control group .Anon significant reduction in the deformity was seen within 3% nano addition ZrO2 also 5% nano ZrO2 addition reduce the deformity significantly and significant increase was recorded when 7% nano addition ZrO2 was add and when these groups are compared to the control group. A significant increase in surface hardness was observed with the addition of (ZrO2) nano-particles to (PMMA) at the percentage of 3%, highly significant increase at 5% and 7% with addition of modified nano-ZrO2. A non-significant decrease in apparent porosity at 3% and highly significant decrease in apparent porosity at 5% and 7% with addition of modified nano-ZrO2 were observed. SEM results showed a good distribution of the modified nano-ZrO2 fillers at 3%, 5% and showed aggregation at 7% in the polymer matrix. Conclusion: The addition of modified nano-ZrO₂ particles to acrylic resin cured by autoclave improved impact and transverse strength of denture base nano composite containing 5% of nano-ZrO2. And this strength decreases with further increase of nano-ZrO₂ filler content. Also addition of modified nano-ZrO₂ slightly increaseshardness, thesurface roughness and the apparent porosity also decrease by addition of nano ZrO_2 percentage increase. Key words: Salinized (ZrO₂) nano fillers, PMMA. (J Bagh Coll Dentistry 2015; 27(1):32-39).

INTRODUCTION

Polymethylmethacrylate (PMMA) is the most commonly used material in construction of denture base since 1930 ⁽¹⁾. This material is not ideal in every respect and it is the combination of various rather than one single desirable of properties that accounts for its popularity and usage.

Despite its popularity which satisfy aesthetic, simple processing and easy repair, the main problems associated with PMMA as denture base material are poor strength particularly under fatigue failure inside the mouth, impact failure outside the mouth ^{(2).} In order to overcome these problems, several attempts were made to modify and improve the strength, thermal properties, and hardness of the PMMA. These attempts included the addition of filler particles such as zirconia, glass fiber, alumina, tin, and Copper or addition of whisker to resin ^{(3-8).} Recently, much attention has been directed toward the incorporation of in organic nanoparticles into PMMA to improve its properties. The properties of polymer nanocomposites depend on the type of incorporated nanoparticles, their size and shape, as well as the concentration and interaction with the polymer matrix ^{(9).} Nanoparticles were undergone surface treatment with saline coupling agent and embedded into PMMA ⁽¹⁰⁾.

MATERIALS AND METHODS

1. Surface modification of Nano fillers (ZrO2)

The introduction of reactive groups onto fillers surface was achieved by reaction of N-propyltrimethoxysilane with zirconium oxide.

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Typical process was as follows

30g of Nano filler ZrO2 and 250ml pure toluene were placed into a flask then sonicated at ambient temperature for 20min.After that, the Nano filler and toluene were placed into a flask equipped with a magnetic stirrer at room temperature for 10 min. Then 1.5g of silane (5% wt. to Nano filler) was added drop wisely by sterile syringe under rapid stirrer (750 rpm) for 20 min. The flask was covered by parafilmand the slurry was left standing in flask for 2 days. The solvent (toluene) was removed by rotary evaporator under vacuum at 60°C at rotary 150 rpm for 30 min. After that the modified Nano filler was dried in vaccum oven at 60°C for 20 hours. Then Nano filler stored at room temperature before use ^(10, 11). The infrared (IR) spectra were performed on Shimadzu FTIR-

8400S infra-red spectrophotometer to determine whether or not functional groups of the TMSPM have been attached to the Nano filler by analyzing the characteristic vibrations of functional groups (10).

2- Specimens grouping

(200) specimens were prepared to be used in the present study. The specimens were divided into (5) groups according to the tests used, for each test (4) subgroups (three for zirconium oxide addition and one control) where each sub group contain ten specimens to be tested.

3-General preparation of test specimens

1. Preparation of the acrylic specimens (Test specimens):

Include Plastic model preparation

2. Proportioning and mixing of the acrylic

Cable 1: Percentages and amounts of polymer, monomer and zirconium oxide Nano fil	ller
powder used in the study	

ZrO ₂ percentage	Amount of ZrO ₂	Amount of PMMA	Amount of monomer
0%	0	22g	10ml
3%	0.66g	21.34g	10ml
5%	1.1g	20.9g	10ml
7%	1.54g	20.46g	10ml

3. Addition of filler

4. Packing

5. Curing (Fast curing cycle).

6. Finishing and polishing.

4-Mechanical and physical tests utilized to examine properties

Evaluations of the mechanical and physical properties of the prepared nano-composite denture base were compared with conventional denture base (heat cure acrylic resin).

Including:

1. Impact strength test

i. Specimen design

The specimens used were prepared as described with dimensions (80mmX 10mm X 4mm \pm 0.2mm), international standard ISO. 179-1. 2000 for unnotched specimens. Ten specimens of each concentration were prepared make a total of (40) specimens for impact strength measurements.

Specimens tested after being conditioned in distilled water at 37°C for 48 hours.

ii. Testing procedure

Impact strength test was conducted following the procedure given by the ISO 179 with charpy type impact testing instrument. The specimen was supported horizontally at its ends ad struck by a free swinging pendulum which released from a fixed height in the middle. A pendulum of 2 joules testing capacity was used. The scale reading gave the impact energy absorbed to fracture the specimen in joules when struck by a sudden blow. The charpy impact strength of unnotched specimen was calculated in KJ/mm2 as given by the following equation:

Impact strength = $\frac{E}{b,d} X10^3$ (Anusavice, 2008)

Where:

E: is the impact absorbed energy in joules.b: is the width in millimeters of the test specimens.d: is the thickness in millimeters of the test specimens.

2. Transverse strength

i. Specimen design

The specimens used were prepared as described with dimensions (65mm X 10mmX 2.5mm \pm 0.2mm). Ten specimens of each concentration make a total of 40 specimens for measurements of transverse strength. All the specimens were immersed in distilled water at 37°C for 48 hours before being tested (ADA No. 12, 1999).

ii. Testing procedure

The test was achieved using instron testing machine, each specimen was positioned on bending fixture, consisting of 2parallel supports (50)mm apart, the full scale load was 50kg, and the load was applied with cross head speed of 1mm/min by rod placed centrally between the supports making deflection until fracture occurred. The transverse bend strength was calculated using the followingformula:

Transverse strength = $\frac{3FL}{2hd^3}$

Where:

P: is the peak load.
L: is the span length.
b: is the sample width.
d: is the sample thickness. ⁽¹²⁾

3. Surface hardness test

i. Specimen design: like Specimen design in Transverse strength

ii. Testing procedure

Surface hardness was determined using durometer hardness tester from type (shore D) that was fabricated by TIME group INC company according to American National standard/American Dental Association (ANS/ADA) No. 12, 1999 which is suitable for acrylic resin material? The instrument consists of blunt-pointed indenter 0.8mm in diameter that tapers to a cylinder 1.6mm. The indenter is attached to a digital scale that is graduated from 0 to 100 units. The usual method is to press down firmly and quickly on the indenter and record the maximum reading as the shore "D" hardness, measurements were taken directly from the digital scale reading. Five measurements were done on different areas of each specimen (the same selected area of each specimen), and an average of five reading was calculated.

4.Apparent Porosity test

This test method covers procedures for determining apparent porosity by:

1. Balance, of adequate capacity, suitable to weigh accurately to 0.001 g.

2. Oven, capable of maintaining a temperature of 150 65°C (302 6 9°F).

3. Wire Loop, Halter, or Basket, capable of supporting specimens under water for making suspended mass measurements.

4. Container—A glass beaker or similar container of such size and shape that the sample, when suspended from the balance by the wire loop is completely immersed in water with the sample and the wire loop being completely free of contact with any part of the container.

5. Distilled Water.

Test Specimens

At least five representative test specimens selected. Sharp edges or corners shall be removed. The specimens shall contain no cracks.

(A.P)% = $\frac{\frac{\text{Calculation}}{W_s - W_d}}{W_s - W_n} \times 100$ Where: Ws: saturated weight Wd: dry weight Wn: hooked weight

5. Surface morphology (AFM)

Most AFMs use a laser bean deflection system, where a laser reflected from the back of the reflective AFM lever and onto a position sensitive detector. AFM tips and cantilevers are micro fabricated. Typical tips radius is from a few to 10s of nm because the AFM relies on the forces between the tip and samples. AFM can provide 3D image and we can know the homogeneity of nanostructure film, also roughness of surface and crystallite size. The surface morphology of the prepared sample with different percentage of salinized ZrO2 was observed by AFM type (AA 2000 SPM) as shown.

6. Scanning electron microscope SEM

Scanning electron microscope of control and salinized Nano ZrO2 fillers with magnification up to (20, 50 and 100) Mm to show the Nano fillers distribution and its shaped.

RESULTS

1. Characterization of modified nano fillers (ZrO2)

The absorption bands of TMSPM can be assigned to the presence of the functional groups, which are two prominent peaks at 2945cm-1 and 2841cm-1 can be attributed to the (C-H) stretching, and the characteristic (C=O) stretching occurs at 1720cm-1,and the characteristic for (C=C) stretching occurs at 1637cm-1, for (CH2- and CH3) occurs at 1413cm-1, and group of four peaks between 1296cm-1 and 1166cm-1 can be attributed to (C-O-C) stretching, the characteristic for (Si-O-CH3) Stretching occurs between 400-470 cm1.

2. Descriptive data of analysis by dependent variable groups:

From table (2) which plots the different means of transverse strengths across different concentrations of the incorporated Nano-particles dependent on variable groups.



Figure 1:Zr-O band FTIR spectrum of modified ZrO₂

Demendent			Groups							
Dependent		Control 3%			5%	7%				
variable	No.	Mean <u>+</u> S.E	No.	Mean <u>+</u> S.E	No.	Mean <u>+</u> S.E	No.	Mean <u>+</u> S.E		
Impact strength	10	6.22 <u>+</u> 0.330♦	10	8.50 <u>+</u> 0.317	10	8.95 <u>+</u> 0.139 ♣	10	6.55 <u>+</u> 0.341		
Transverse strength	10	93.03 <u>+</u> 2.09	10	97.13 <u>+</u> 2.73	10	103.14 <u>+</u> 3.01♣	10	92.17 <u>+</u> 1.72♦		
Deformation	10	6.97 <u>+</u> 0.4955	10	5.93 <u>+</u> 0.2814♦	10	4.38 <u>+</u> 1.0397	10	7.73 <u>+</u> 0.5766 ♣		
Hardness	10	80.96 <u>+</u> 0.4818♦	10	84.08 <u>+</u> 0.7402	10	84.53 <u>+</u> 0.7641	10	84.97 <u>+</u> 0.8294 ♣		
Apparent porosity	10	2.785 <u>+</u> 0.1465♣	10	2.291 <u>+</u> 0.1266	10	1.719 <u>+</u> 0.1562	10	1.242 <u>+</u> 0.1339♦		

Table 2: Descriptive data of analysis by dependent variable groups parameters

♣ Highest mean in each groups

♦lowest mean in each group

3. Multiple comparisons of nanofillers composite with control for dependent variable groups:

Table (3) showed significant difference between groups was investigated by further complement analysis of data Tuky's test.

Table 3: Multiple comparisons of nano filler composite with control for dependent variable groups

	Dependent variable by P value for Tuky's with significance									
Experimental groups	Impa streng	ict gth	Transverse strength		Deformation		Hardness		Apparent porosity	
Control & 3%	0.000	HS	0.639	NS	0.686	NS	0.019	S	0.082	NS
Control & 5%	0.000	HS	0.029	S	0.041	S	0.006	HS	0.000	HS
Control & 7%	0.835	NS	0.994	NS	0.843	NS	0.002	HS	0.000	HS
3% & 5%	0.804	NS	0.319	NS	0.355	NS	0.972	NS	0.034	S
3% & 7%	0.000	HS	0.486	NS	0.233	NS	0.816	NS	0.000	HS
5% & 7%	0.000	HS	0.015	NS	0.005	HS	0.971	NS	0.098	NS

4. Surface Morphology (roughness)

Surface morphological examination was done for all tested groups with AFM as shown from Figures (2-5) it was appeared that there is a



homogenous distribution of the filler with nano surface morphology as the percentage of nano addition increased with an even nano surface roughness table (4)



Figure 2: AFM control (without Nano)

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The effect of



 Table 4: AFM data for sample prepared from acrylic and nano ZrO2

Sample mane	Roughness average (nm)	Average grain size (nm)							
Control	62.1	83.41							
3%	61.0	53.57							
5%	58.9	39.23							
7%	50.4	11.71							

5. Scanning Electron Microscope test

SEM examination was done under (20X, 50X, and 100X) Mm with deferent magnification. This test was done for all tested groups (control 0%, 3%, 5% and 7%) as shown in Fig (6).

As appear in the figure (6) the individual particle was in nano scale size and it is spherical shaped with diameter approximately 20-30 nanometers.





50 Mm 20 Sample with 5% nano ZrO₂

Figure 6: Control, 3%, 5%, and 7%): Scanning electron microscope shows particles of nano

DISCUSSION

In the present study some mechanical properties of acrylic denture base material (Vertex) cured by autoclave are evaluated after the addition of modified silanized nano- ZrO_2Nano Zirconia (ZrO₂) was used because it is excellent biocompatible material also esthetic because of white color and is less likely to alter esthetic.. The nano-sized zirconia has been successfully used to fabricate nano-composite with highhardness, high refractive index and improved scratch resistance (13).

1. Silane coupling agent

A properly applied coupling agent can improved physical and mechanical properties and inhibit leaching by prevent water from penetrating along the filler resin interface, also excellent filler to matrix adhesion is needed for minimizing wear. Silanization of the nano-filler particles yields a better dispersion, eliminate aggregation and improve its compatibility with organic polymer ⁽¹⁴⁾ Although zirconates can be used as coupling (12) In the present study agents Npropyltrimethoxysilanewas used as a coupling agent. Toluene was used as the solvent for Npropyltrimethoxysilane to aid in dispersion of Npropyltrimethoxysilane to cover all the nano-ZrO₂ Particles.

The atomic number of zirconium (Zr) is 40, so it has unsaturated empties orbitals (5S, 4d), the alone pair electron of each (O) present in the coupling agent can attack the unsaturated empties orbitals of Zr (form coordination bonds with an inorganic nano-ZrO₂ fillers ⁽¹⁵⁾.

Moreover, due to the presence of more than one active side so this will lead to physical interaction (molecular interaction) by Vander Waal forces which enhanced the bond strength. Both Vander Waal force and covalent bonding will increase the shear strength and adhesion force (16).

2. Impact strength

Impact strength is defined as the energy required fracturing a material by an impact force ^{(17).}

The results of impact strength test showed that the addition of modified nano- ZrO_2 powder increased the value of the impact strength compared to control group, 5wt% group has the highest impact strength, but increasing the percentage of modified nano- ZrO_2 to 7wt% lowered the impact strength.

The increase in impact strength due to the interfacial shear strength betweennano-filler and matrix is high due to formation of cross-links or supra molecular bonding which cover or shield the nano fillers that in turn prevent propagation of crack. Also the crack propagation can be changed by good bonding between nano filler and resin matrix ^{(16, 18).}

The aggregation of modified nano-ZrO₂ at 7wt%, that lead to the reduction in impact strength because of higher surface area of the fillers, which may reached to μ m in size, so stress concentration around this aggregation which lead to crack proportion. Also the increase in percentage of modified-nano-ZrO₂ powder effects the interface region lead to lowering of energy dissipation per unit volume and consequently lowers the impact strength ⁽¹⁶⁾.

Similar finding was reported by Nabil ⁽¹⁵⁾. when he added zirconia nano-particles and found that the Impact strength increase at 3% and 5% percentage of addition Nano-ZrO₂, Than slight decrease at thepercentage 7% addition of nano-ZrO₂ fillers.

3. Transverse strength

The transverse strength test is one of the mechanical strength tests especially useful in comparing denture base materials in which a stress of this type is applied to the denture during mastication ⁽¹⁹⁾.

The addition of Nano ZrO₂ and curing by autoclave could be attributed to increase in cross linking. Cross-linkage provides a sufficient number of bridges between linear macromolecules to form a three-dimensional network that decreases water sorption, decreases solubility, and increases the strength and rigidity of the resin⁽¹⁷⁾.

The Addition of modified nano- ZrO_2 fillers increased the value of transverse strength significantly compared to control; 5% wt group has the highest transverse strength (result). And increasing the percentage of modified nano- ZrO_2 at 7% wt lowered the transverse strength.

The increase in transverse strength that occur with addition of 3 % and 5% wt ZrO_2nano particles is due to well dispersion of the very fine size of nano-particles enable them to enter between linear macro molecular chains of the polymer and fill spaces between chains, segmental motions of the macromolecular chains are restricted and lead to increase strength and rigidity of the resin, so this improve fracture resistance and lead to improve transverse strength (20).

At the 7%, reduction in transverse strength may be because all spaces between the PMMA chains have been filled at 5%, above this percentage the excess fillers caused separation of PMMA chains and weak force between them lead to decrease fracture resistance and to decrease in mechanical properties of the polymer. Also reduction in transverse strength at 7% wt is due to aggregation of modified nano-ZrO₂ fillers because of higher surface area (reached to μ m in size), this aggregation actually caused a micro fracture that weakened the nano-composite at this percentage.

"Single individual Nano filler exhibited no distinct fracture, or single distinct fracture. In contrast, the nano cluster (aggregate) fillers exhibited multiples fractures, the failure occurredalong the lines of internal porosity within the aggregate (nano cluster)"^{(21).}

Similar finding were reported by Nabil ⁽¹⁵⁾ when he added zerconianano-particles and found that the transverse strength increase with the concentration of nano-fillers at 3% and 5%, and decrease at 7% nano addition of ZrO₂.

4. The deformation test

Deformation testing evaluates the effect that load has on the shape of a sample.Deformation is

measured as the percent change in height of a sample, under a specified load, for a specified period of time. The decrease in deformation that occur with addition of 3% and 5% ZrO_2 nanoparticles due to well dispersion of the very fine size of nano-particles enable them to enter between linear macro molecular chains of the polymer and fill spaces between chains, segmental motions of the macromolecular chains are restrict lead to increase rigidity of the resin and decrease the ability to deformation ^{(21).}

The addition of nano ZrO_2 3%,5 % lowered the deformation, At 7 % the deformation increase due to excess fillers caused separation of PMMA chains and weak force between them lead to decrease fracture resistance and to decrease in mechanical properties of the polymer ⁽¹⁵⁾.

5. Surface Hardness

Hardness defined as the resistance of material to plastic deformation typically measured under an indentation load ^{(17).} Shore (D) hardness tester was used which is suitable for ensuring the hardness of acrylic resin ^{(22).}

The significant increase in hardness of the nano-composite at low nano-particle concentration level 3% nano addition ZrO_2 would be dominated by the cross linking density, while highly significant increase in hardness of the nano-composite at 5% and 7% nano addition ZrO_2 may be attributed to the randomly distributed particles of a hard material nano- ZrO_2 into acrylic matrix.

Similar finding were reported by Nabil⁽¹⁵⁾ when he added zerconia nano-particles and found that the surface hardness increase with the increase in the concentration of nano-fillers.

6. Apparent Porosity

Apparent Porosity in acrylic resin is a complex phenomenon of multifactorial origin. The addition of nano filler lead to decrease the porosity because the nano that fill the space inside the material lead to decrease the number of pores that open on the external surface, Also the using the autoclave demonstrated a reduction in porosity in a denture base resin polymerized ^{(23).}

The well dispersion of the very fine size of nano-particles ZrO_2 enable them to enter between linear macro molecular chains of the polymer and fill spaces between chains ⁽²⁰⁾. That would lead to decrease the apparent porosity at 3%, 5% and 7% respectively.

In conclusion; the addition of modified nano-ZrO2 particles to acrylic resin cured by autoclave improves impact and transverse strength of denture base nano composite containing 5% of

nano-ZrO2. And this strength decreases with further increase of nano-ZrO2 filler content. Also addition of modified nano-ZrO2 slightly decreases the indentation hardness (surface morphology) the apparent porosity also decreases by increase addition of nano ZrO2 percentage increase.

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الخلاصة

بيان المشكلة. ميتاكريلات (PMMA) المستخدمة في تصنيع اطقم الأسنان وتعتبر من المواد الأكثر موثوقية لبناء الأجهزة التعويضية القابلة للإزالة.وتعتبر مواد بعيدة عن المثالية في الوفاء بالمتطلبات الميكانيكية، وتأثيرات الطبخ بالأوتُوكيليف لم تحدد بشكل كامل كان الغرض من هذه الدراسة لتقبيم تأثير إضافة حبيبات ثنائي اوكسيد الزركونيوم النانويةالمعالجة سطحيا بنسب 3٪، 5٪، 7٪ الى مادة الاكريليك.

مينية من الإصادة على المريسة عمرية. ودر استه تأثير هذه الإضافة على القوة عرضية، قوة الصدمة، والصلادة السطحية والمسامية وشكل السطح بعد معالجه المادة بالأوتوكلاف. المواد والطرق: بالإضافة إلى المجموعة السيطر قالمعالجة بالاوتوكليف تم اضافه حبيبات اوكسيد الزركونيوم النانوية لباودر البولي ميثيل ميثا أكريلات وزنا في ثلاث نسب مختلفة 3٪، 5٪ و7٪، ويخلط بواسطة مسبار آلة صوتيه فائقة. صنعت 200 نموذج بواسطة الأوتوكيليف وقسمت إلى 5 مجموعات وفقا للاختبارات (تتكون كل مجموعة من 40 عينه)، وتنقسم كل مجموعة إلى 4 مجموعات فرعية وفقا للنسب المضافة من أوكسيد الزركونيوم النانوية (وتحتوي كل مجموعة فرعية على 10 عينات). وكانت التجارب التي أجريت القوة عرضية، قوةالصدمة، والصلادة السطحية, المسامية وشكل السطح, وقد تم تحليل النتائج إحصانيا باستخدام اختبار ANOVA.

النتائج.سجلت زيادة معنويه في تأثير القوة العرضية للأختبار التي تحدث في الاكريليك عند اضافه 3% و5% بينما هناك انخفاض معنوي قد سجل عند أضافه 7% عند مقارنته مع مجموَّعه السيطرة سجلت زياده غير معنوية في اختبار العرضية عند أضافه حبيبات أوكسيد الزركونيوم النانوية بنسبه 3%و7% , بينما تظهر زيادات معنويه في اختبار العرضية عند أضافه 5% عند مقارنه المجاميع بمجموعه السيطرة بتظهر زيادات غير معنويه في نسبة التشوهات ضمن النسب المضافة 3% و7% بينما تظهر زيادة معنوية في قله نسبه التشوهات عند أضافه 5% من حبيبات أوكسيد الزركونيوم النانوية عند مقارنه المجاميع بمجموعه السيطرة . هناك تحسن معنوي في قوة الصلادة عند أضافه حبيبات أوكسيد الزركونيوم بنسب 3% وهناك زيادة معنويه ملحوظه في قوة الصلادة عند أضافه الحبيبات بنسبة 5% و7% مقارنة بمجموعه السيطرة . عدم ظهور نقصان معنوي في المسامية عند أضافه 3% وحدوث نقصان معنوي ملحوظ في المسامية عند أضافة 5% و7% . وأظهرت نتائج فحص المجهر الالكتروني توزيع جيد للحبيبات النانوية بنسب 3% و5%، وأظهرت التجميع في7٪ في البوليمر .

الاستنتاج: اضافه حبيبات اوكسيد الزركونيوم النانويةالمعالجة سطحيا عن طريق الأوتوكيليف بنسب 3% و 5 % أظهرت تحسن في القوهالعرضية وقوه الصدمة وبنسب 3% و 5% و 7 % لوحظ زياده في صلادة السطح ونقصان في المساميةالظاهرية.