



## Transverse and Impact Strength of Poly Methyl Methacrylate-Zirconium Oxide Nanocomposite Denture Base Material

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### Abstract

**Aims:** this study aims to evaluate the effect of adding two concentrations of zirconium oxide nanoparticles (1.0% and 2.0%) to "heat-cured PMMA" in order to improve its mechanical properties such as transverse strength and impact strength. **Materials and Methods:** The particle size of Zirconium Oxide ZrO<sub>2</sub> was 20nm. The ZrO<sub>2</sub> nanoparticles were added to the "heat-cured PMMA" resin base at 1.0% and 2.0% by weight, respectively, to create a PMMA-ZrO<sub>2</sub> nanocomposite with two various percentages compared to PMMA without additives. For the transverse strength and impact strength tests, the traditional heat-curing technique was cured with a water bath to polymerize the specimens. The results of the study were statistically analyzed by using one-way ANOVA and Duncan's multiple range test with a significant P-value of 0.05. **Results:** The results showed that there was an increase in the transversal and impact strength for the PMMA-ZrO<sub>2</sub> nanocomposite at concentrations of 1.0% and 2.0% respectively, after comparison of the findings at 0.05. **Conclusions:** The use of Zirconium Oxide nanoparticles as dental fillers at 1% and 2% by weight improved the transverse strength and impact strength of PMMA denture base material.

### الخلاصة

**الاهداف:** تهدف هذه الدراسة الى تقييم تأثير إضافة تركيزين من جزيئات أكسيد الزركونيوم النانوية (1.0% و 2.0%) إلى البولي ميثيل ميثاكريليت المعالج بالحرارة لتحسين بعض الخصائص الميكانيكية مثل القوة العرضية و قوة التأثير. **المواد و طرائق العمل:** بلغ العدد الإجمالي للعينات في هذه الدراسة 305 عينة، بينما كان عدد العينات في الدراسة التجريبية 80 عينة و عدد عينات الدراسة الرئيسية 225 عينة، تم فصلهم إلى ثلاث مجموعات بناءً على تركيز المادة المضافة من النانو أكسيد الزركونيوم. تم تصنيع العينات من البولي ميثيل ميثاكريليت المعالج بالحرارة مع جزيئات أكسيد الزركونيوم النانوية (1.0 و 2.0%)، حيث كانت طريقة تحضير العينة المستخدمة في هذه الدراسة على النحو التالي: تم تحضير العينات أولاً بخلط كل تركيز بمفرده (بنسبة 1.0% و 2.0% مسحوق نانو أكسيد الزركونيوم) بالوزن مع مونومر سائل البولي ميثيل ميثاكريليت المعالج بالحرارة بعد ان تم طرح من وزن مسحوق البولي ميثيل ميثاكريليت بقدر ما تمت اضافته من مادة النانو أكسيد الزركونيوم ، ثم تم مزجها وتشتيتها في المونومر السائل بواسطة مسبار فوق صوتي بقوة 20 وات و 60 كيلو هرتز لمدة ثلاث دقائق ثم تمت إضافة مسحوق البوليمر ومزجها بنفس الطريقة لتجنب تكثف الجسيمات ، ثم تم اختبار العينات و تحليل النتائج إحصائياً عن طريق (المعدل ± الانحراف المعياري ، الأنوفا واختبارات المدى المتعددة لدانكن. **النتائج:** أظهرت النتائج وجود فروق ذات دلالة إحصائية عند 0.05 ≤ في القوة العرضية ، وقوة التأثير عند مقارنة المجموعات بإضافة أكسيد الزركونيوم بتركيز (1.0% و 2.0%). **الاستنتاجات:** أن إضافة أكسيد الزركونيوم إلى البولي ميثيل ميثاكريليت المعالج بالحرارة له تأثير ايجابي على المركب النانوي المتولد من حيث القوة العرضية و قوة التأثير ، علاوة على ذلك، لم يكن هناك فروق ذات دلالة إحصائية عند 0.05 ≤ بين 1.0 و 2.0% أكسيد الزركونيوم.

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## INTRODUCTION

PMMA "poly methyl methacrylate" is an excellent biocompatible organic polymeric substance that is utilized to make denture bases <sup>(1)</sup>. It was initially used in denture construction in 1937. Since then, its strong physical and mechanical qualities make it a suitable material <sup>(2)</sup>. In all denture base materials, acrylic resins have been most often used and approved and it has been estimated that (95%) of dental polymers are composed of "Poly methyl methacrylate" thermoplastic polymers <sup>(3)</sup>.

However, when used alone, PMMA has inadequate mechanical properties and inadequate surface hardness. It was readily damaged in an event involving a strong impact or when a patient applied severe chewing pressure to the tooth base <sup>(4)</sup>.

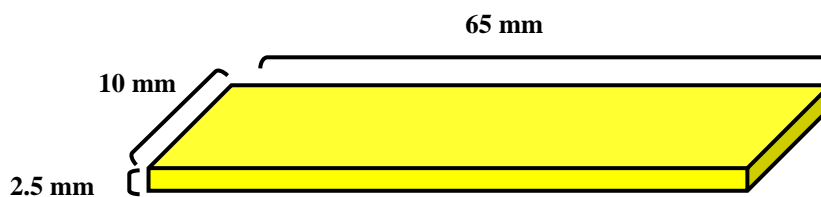
On the other hand, low mechanical characteristics against impact, bending and fatigue are major concerns that must be addressed enhancing acrylic polymers for removable dental and acrylic appliances <sup>(5)</sup>. Recently, a great deal of attention has been paid to the integration of inorganic nanoparticles into PMMA to enhance their characteristics. The characteristics of nanoparticles depend on the type of nanoparticles that are included, their size

and shape, their concentration and their interaction with the polymer matrix <sup>(6)</sup>. Metal oxide "zirconium oxide" has several benefits, including mechanical strength, toughness, rigidity, wear strength, chemical tolerance, and high thermal stability <sup>(7)</sup>.

## MATERIALS AND METHODS

In this present study sixty specimens were used, which were split into two major groups. Impact and transverse mechanical tests were performed on a subdivided group of 30 specimens (n=30).

The proposed PMMA-ZrO<sub>2</sub> nanocomposite study was prepared according to the following measurements: "ZrO<sub>2</sub> of 1.0% wt. was added to the heat-cured PMMA resin base at 99% wt., ZrO<sub>2</sub> of 2.0% wt. was added to a heat-cured PMMA resin base of 98% wt. using sensitive balance to achieve an even ZrO<sub>2</sub> distribution within the PMMA matrix", then; the sample preparation was done by mixing 1.0% and 2.0% by weight ZrO<sub>2</sub> nano powder with "heat-cured PMMA" fluid monomer, then; sonicated and dispersed in the liquid monomer by an ultrasonic probe of 20W and 60 kHz for three minutes, after that; the "heat-cured PMMA" polymer powder was added and sonicated in the same way to avoid particles agglomeration <sup>(8)</sup>.



**Figure (1):** Transverse (Flextural) testing specimen dimensions.

During mold preparation, a conventional flaking process was performed for full dentures. Separating media (cold mold stitch) was employed and allowed to dry for the layer of plastic before placing the lower part of metal bottles filled with dental stone and combined in vibration according to the directions of the manufacturer to remove the trapped air, and then left to set. Acrylic sheets were used to create the plastic model which was designed using computer software (AutoCAD), and then they were engraved using a computer-controlled laser cutting machine. The length, width, and thickness of the plastic models used in the fabrication of the molds were determined according to the specifications required for each test. In both groups, the specimens were washed

and stored in distilled water at 37°C for 2 days before being analyzed <sup>(4)</sup>.

### Testing Procedures

#### Transverse (Flextural) strength test:

The specimen was made in accordance with International Standards Organization Specification No. 1567 (ISO) specifications "65 mm length × 10 mm width × 2.5 mm thickness", as illustrated in Figure (1).

Test was performed with the specimen mounted on two parallel supports 50 millimeters apart and bent using a rod at the center of the two supports at 50 kg and 1mm/min until fracture occurred. For all specimens, the transverse strength (Q) has been determined as follows <sup>(4)</sup>:

$$Q = \frac{3FI}{2BH^2} \text{ N/mm}^2$$

**F = the maximal load or force (N)**

**I = the distance between the two surface rests (mm);**

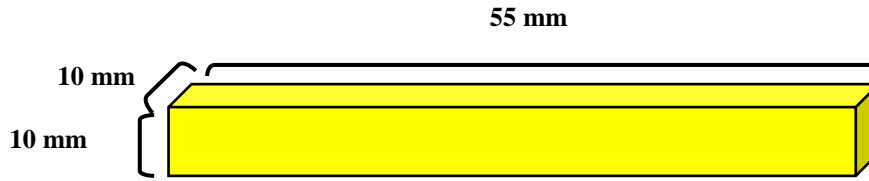
**B = the specimen's width (mm)**

**H = the specimen's height or thickness (mm)**

#### Impact strength test:

In room temperature (22°C), the impact strength test was conducted & in accordance to ISO 148, the standard

specimen sizes were (55 mm length × 10 mm width × 10 mm thickness) as illustrated in Figure (2).



**Figure (2):** Impact testing specimen dimensions.

Charpy impact test equipment was used to strike the unnotched specimen with a 2 joule pendulum (gunt HAMBURG Impact Tester). a given mass and length pendulum that is dropped from a known height to strike the specimen's material is

$$I = E / (bd)$$

**E = the energy absorbed by joules**

**B = the breadth of the specimen in millimeters**

**D = the thickness of the specimen in millimeters.**

used as part of the equipment. A hammer's height can be used to determine how much energy is transferred to the material (energy absorbed by the fracture event).

The following equation was used to determine impact strength (I) in KJ/m<sup>2</sup> (9):

## RESULTS

### Transverse (Flextural) strength test:

Table (1) was shown the results of a descriptive statistical analysis of

transverse strength for all groups were tested.

**Table (1):** Descriptive statistical analysis of roughness test

Groups	N	Descriptive Statistics			
		Minimum	Maximum	Mean	Std. Deviation
Control	30	62.4	130.8	93.24	16.05
ZrO <sub>2</sub> 1.0%	30	1	3	2.00	0.830
ZrO <sub>2</sub> 2.0%	0				

Significant differences were found in one way analysis of variance (ANOVA) of (1.0% ZrO<sub>2</sub> and 2.0% ZrO<sub>2</sub>) groups and control group table (2).

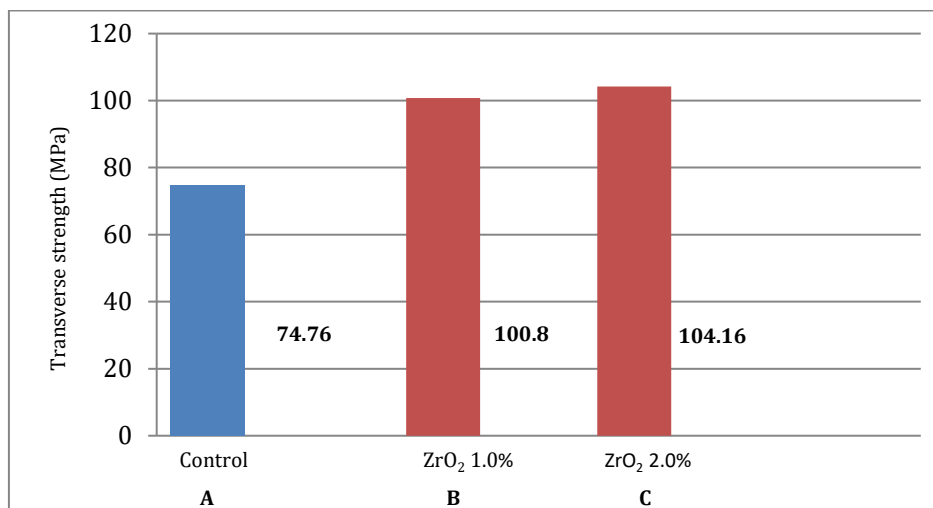
**Table (2):** ANOVA was used to compare the transverse strength of different ZrO<sub>2</sub> groups

SOV	SS	df	MS	F	Sig.
Between Groups	5179.104	2	2589.552	30.466273	.000
Within Groups	2294.928	27	84.997333		
Total	7474.032	29			

SOV: Source of variance; SS: Sum of squares; df: degree of freedom; MS: Mean square

Duncan's multiple range test Figure (3) revealed a significant difference between the (1.0% and 2.0%) ZrO<sub>2</sub> & the control groups. There was no significant

difference between the (1.0% and 2.0%) ZrO<sub>2</sub> groups, with the (2.0%) ZrO<sub>2</sub> group was substantially higher than the control groups and (1.0%) ZrO<sub>2</sub>.



**Figure (3):** ZrO<sub>2</sub> groups were compared using mean, standard deviation, and Duncan's multiple range test of transverse strength.

**Impact strength test:**

Table (3) was shown the results of a descriptive statistical analysis of impact strength for all groups were tested.

**Table (3):** Descriptive statistical analysis of roughness test

Groups	N	Descriptive Statistics			
		Minimum	Maximum	Mean	Std. Deviation
Control	30	0.006	0.024	0.143	0.004
ZrO <sub>2</sub> 1.0%	30	1	3	2.00	0.830
ZrO <sub>2</sub> 2.0%	0				

Significant differences were found in one way analysis of variance (ANOVA) of (1.0% ZrO<sub>2</sub> and 2.0% ZrO<sub>2</sub>) groups and control group table (4).

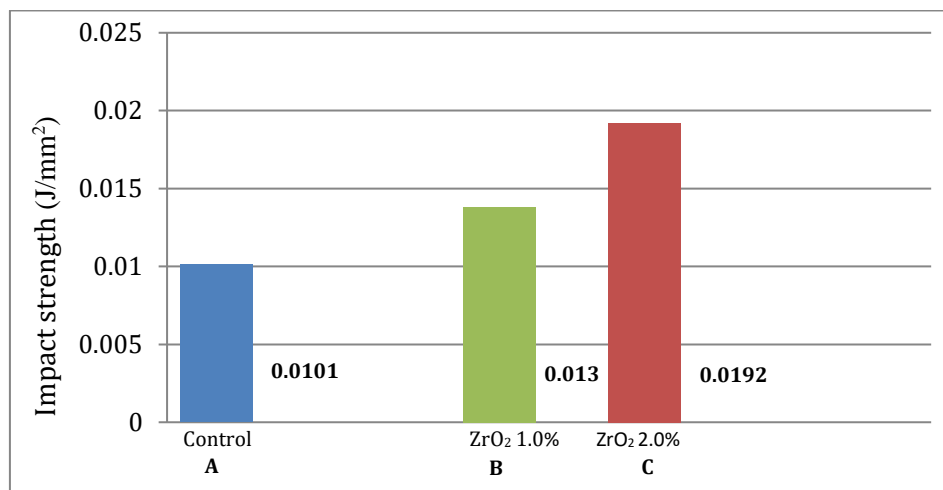
**Table (4):** ANOVA was used to compare the impact strength of different ZrO<sub>2</sub> groups

SOV	SS	df	MS	F	Sig.
Between Groups	0.000	2	112497.233	76.404	.000
Within Groups	0.000	27	1472.407		
Total	0.001	29			

SOV: Source of variance; SS: Sum of squares; df: degree of freedom; MS: Mean square

Duncan's multiple range test Figure (4) revealed a significant difference between the (1.0% and 2.0%) ZrO<sub>2</sub> & the control groups. There was a significant

difference between the (1.0% and 2.0%) ZrO<sub>2</sub> groups, with the (2.0%) ZrO<sub>2</sub> group was substantially higher than the control groups and (1.0%) ZrO<sub>2</sub>.



**Figure (4)** ZrO<sub>2</sub> groups were compared using mean, standard deviation, and Duncan's multiple range test of impact strength.

## DISCUSSION

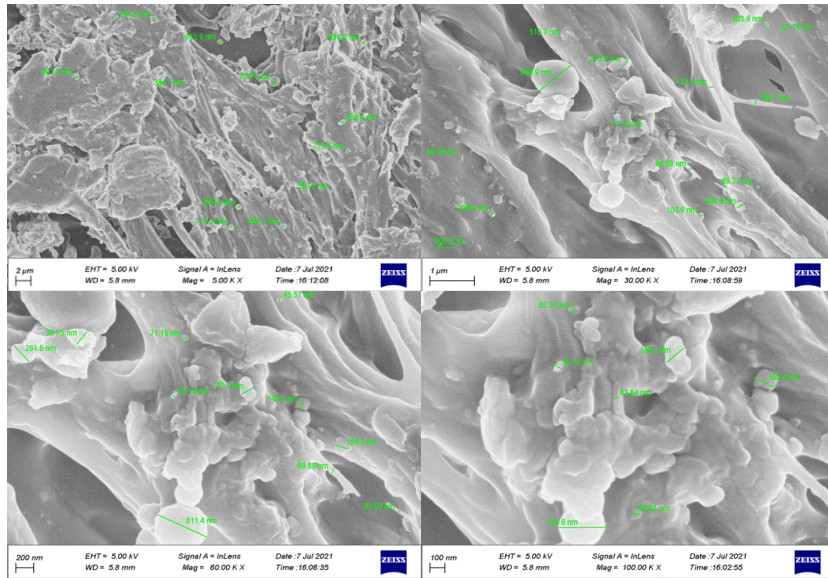
### Transverse (Flexural) strength

#### test:

Statistics were shown that ZrO<sub>2</sub> nanoparticle additions of 1.0%, and 2.0% increased transverse (flexural) strength significantly as compared to the control group. According to the SEM images Figure (5), the higher transverse strength of heat-cured PMMA-ZrO<sub>2</sub> nanocomposite

was due to the reinforcing impact of the nanoparticles in the polymer matrix. It is possible to trap nanoparticles in a "heat-cured PMMA" matrix. Interactions between the nanoparticles and the polymer chains result in a three-dimensional network, which is supposed to obstruct the movement of the polymer chain against the nanoparticle surface <sup>(10)</sup>.

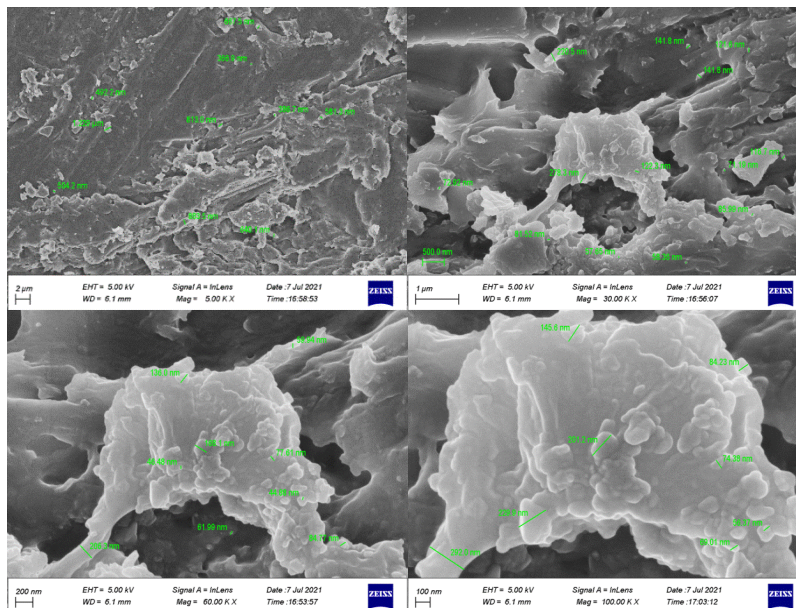




**Figure (5):** FESEM of ZrO<sub>2</sub> groups

It could also inhibit the activity of other polymer chains on one another, which could lead to a change in the matrix's overall network density subsequently; the polymer stiffness was increased significantly as a result of the process <sup>(11)</sup>. The source of a relatively substantial rise in transverse strength following ZrO<sub>2</sub> nanoparticles integration has been

discussed yet, but when the mean transverse strength of the 1.0% ZrO<sub>2</sub> group was compared to that of the 2.0% ZrO<sub>2</sub> group was no discernible change in transverse strength, which might be attributed to nanoparticles' proclivity to agglomerate when the concentration was raised. These findings were verified using SEM imaging<sup>(12)</sup> Figure (6).



**Figure (6):** FESEM imaging

### Impact strength test:

The mean values of the ZrO<sub>2</sub> nanoparticle addition of 1.0% and 2.0% enhanced the impact strength considerably when compared to the control group.

The impact strength values of the 1.0% and 2.0% ZrO<sub>2</sub> were greater than those of the control group. This might be attributed to the nanofiller's high interfacial strength, which is caused by cross-links or supra-molecular connections shielding the nanofillers and preventing crack propagation and by attaching functional groups on the nanoparticle surfaces to polymer chains thereby inhibiting fracture propagation. Furthermore, nanoparticles may fill the spaces between polymer chains, resulting in a heterogeneous mixture and limiting the polymer chain segment displacement. Nanoparticles have the ability to decrease fracture propagation and hence enhance impact resistance because of their large surface area<sup>(13)</sup>.

It should be mentioned that the inclusion of ZrO<sub>2</sub> nanoparticles to a ratio greater than 2.0% decreases the impact strength of the PMMA nanocomposite to values lower than those found in pure PMMA<sup>(14)</sup>.

### Declaration of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript

### CONCLUSION

The Addition of Zirconium Oxide nanoparticles as a dental filler (1.0% and 2.0% by wt.) to PMMA denture base material enhanced the transverse strength and impact strength of the material.

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