



Evaluation of Flexural Strength and Impact Strength of CAD/CAM and Heat Cured Acrylic Resin Modified by Zinc Oxide Nanoparticles

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Abstract

Aims: To analyse the effects of the incorporation of Zinc Oxide nanoparticles (ZnO NPs) with actual size (10-30 nm) at one concentration (1%) and compare it with milling CAD/CAM and non-modified heat cure denture base materials on the flexural strength and impact strength. **Materials and methods:** samples' number were 60, which was divided into thirty specimens for each test and sub-grouped into 10 for each group (control, 1 % ZnO NPs, CAD/CAM). Using the SPSS program, the statistical analysis was carried out using ANOVA, descriptive statistics, and Duncan's test at a significant $p \leq 0.05$. **Results:** The findings showed a substantial difference between the groups. Moreover, milled CAD/CAM PMMA material had the highest flexural strength and impact strength than groups. Moreover, Flexural strength and impact strength for the PMMA-ZnO nanocomposite at ZnO nanoparticles (1%) increased significantly, when compared to control. **Conclusions:** For edentulous patients, milled CAD/CAM dentures are more suitable because of their improved mechanical properties. In addition, that use of ZnO nanoparticles as dental fillers at 1% by weight significantly increased the PMMA denture base material's flexural and impact strength.

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الخلاصة

الأهداف: تهدف الدراسة الى دراسة تأثير دمج جزيئات الزنك وأكسيد النانوية بحجم (10-30 نانومتر) بتركيز (1%) على مقاومة الانحناء ومقاومة الصدمة لراتنج الأكريليك المعالج بالحرارة ومقارنته بمادة أطقم الاسنان المصنع عن طريق أنظمة الكمبيوتر (CAD/CAM). **المواد و طرق العمل:** كان العدد الإجمالي للعينات ستون مقسمة إلى ثلاثون عينة لكل فحص وعشرة عينات لكل مجموعة (مجموعة السيطرة ، 1% الزنك وأكسيد النانوي، CAD/CAM) تم إجراء التحليل الإحصائي باستخدام برنامج الإحصاء بما في ذلك الإحصاء الوصفي ، اختبار انوفا ، واختبار دنكن عند $p \geq 0.05$. **النتائج:** أظهرت النتائج أن مجموعة (CAD/CAM) تمتلك معدل قيمة الأعلى عند مقارنته ببقية المجموعات و أيضاً هناك زيادة ملحوظة في مقاومة الانحناء و أيضاً زيادة ملحوظة في مقاومة الصدمة لمركب النانوي المتولد في جزيئات الزنك وأكسيد النانوي (1%) ، عند مقارنتها بمجموعة السيطرة. **الاستنتاجات:** أن الراتنج لمادة قاعدة أطقم الاسنان المصنع عن طريق أنظمة الكمبيوتر معدل القيمة الأعلى بين المجموعات من حيث مقاومة الانحناء ومقاومة الصدمة و أيضاً إضافة الزنك وأكسيد بتركيز (1%) إلى البولي ميثيل ميثاكريليت المعالج بالحرارة له تأثير إيجابي على المركب النانوي المتولد من حيث مقاومة الانحناء و مقاومة الصدمة لمادة قاعدة أطقم الاسنان .

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INTRODUCTION

The loss of teeth is a significant consideration for most individuals. Their replacement by artificial replacements, such as dentures processed from acrylic PMMA resin, are essential to the continuation of everyday life. Regardless of the underlying reason for missing teeth, multiple treatment options are obtainable to replace missing teeth ⁽¹⁾.

The material of choice for the design of removable partial dentures and complete denture bases is poly methyl methacrylate (PMMA), which was initially introduced in 1937. It remains in use because of its reasonable fit, dimensional stability, and ease of manipulation ⁽²⁾. These prostheses, especially implant-retained over-dentures, help prevent bone loss and supply more stability and retention during functioning ⁽²⁾.

However, a few crucial drawbacks are in this resin, such as insufficient strength, specifically under fatigue failure inside the mouth, residual monomer allergy, low abrasion resistance and brittleness on impact, These drawbacks regularly direct to dentures fractures ⁽³⁾.

Reinforcing denture bases with filling materials, modifying the chemistry of the denture base polymer by co-polymerization or cross-linking of resin materials, and making new materials with increased fracture resistance are continuing efforts to improve material strength to diminish the risk of denture fractures ⁽⁴⁾.

One of the most encouraging techniques newly used to overcome these drawbacks is integrating different nanoparticles into the PMMA denture base to act as the reinforcing material, Different nanocomposite fillers, metal oxide, and carbon graphite fiber are incorporated into the composition to overcome these problems and improve the resin's mechanical and physical properties ⁽⁵⁾.

The properties of the polymer nanocomposite depend on the type of included nanoparticle, their size, shape, concentration, and how particles interact with the polymer matrix ⁽⁶⁾.

Nano-scaled filler zinc oxide may have excellent antibacterial and antifungal capabilities. Denture base resins' characteristics, particularly the biological properties of acrylic resins, can be considerably improved by adding ZnO ⁽⁷⁾.

Since the 1980s, computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies have been used in prosthodontics to overcome some of these drawbacks. CAD/CAM technology was presented to provide a denture base acrylic resin manufactured by machining. As a result, polymerization shrinkage of the resin is minimized, and the fit of the denture base is improved over that produced conventionally ⁽⁸⁾. Compared to the material produced by conventional technique, it appears to have less residual monomer and to be more hydrophobic. This leads to a more bio-hygienic denture as a

result. Due to the lack of porosity, bacteria like *Candida Albicans* cling less to dentures' bases, lowering the risk of infections⁽⁹⁾.

This study aimed to analyze the influence of zinc oxide nanoparticles addition at concentration (1%) on flexural strength and impact strength of heat cured denture base material and compare them with CAD/CAM PMMA .

MATERIALS AND METHODS

Sampling: There were 60 total specimens, which were divided into 30 specimens and grouped into three groups for each test (control ,1 % ZnO NPs,CAD/CAM). This research was done at the college of dentistry and technical institute at the University of Mosul. Approval of research was from the Scientific Research Committee / Department of Prosthodontics/College of Dentistry (UoM.Dent/DM.L.39/22).

Preparation of the samples

The molds were prepared using the conventional flasking procedure. A separating medium (Alginic isolator, Zhermack®) was used and left to dry for the layer of plastic before putting the lower part of metal flasks filled with die stone (Elite® stone, Zhermack®) and combined in vibration to remove trapped air ; then left to set according to manufacturer's instructions. A computer-controlled laser cutting machine was used to cut the plastic model from acrylic sheets which designed

by using computer software (AutoCAD). Mold fabrication was involved precisely determining the plastic models' dimensions, widths, and thickness according to the test specifications. An incubator was used to store samples of all groups at 37°C for two days⁽¹⁰⁾.

The mixing ratio of powder to liquid for heat polymerized acrylic resin (SR Triplex® Hot, Ivoclar Vivadent) was 2:1 by weight, according to the manufacturer's instructions, A curing cycle (heat up to 100 °C for 90 min and let boil for 45 min), using a water bath that was thermostatically controlled. Then the flasks were removed and left to cool down slowly on the bench before opening the flask⁽¹¹⁾.

The weight of the Zinc oxide nanoparticles was subtracted from the weight of the heat-cured PMMA polymer powder to create the accurate powder-to-liquid ratio framed by the manufacturer⁽⁵⁾.

The specimens were first prepared by mixing the weight of zinc oxide Nano powder with "heat-cured PMMA" fluid monomer, which was sonicated and dispersed in the liquid monomer for 3 minutes using an ultrasonic of 20W and 60 kHz, and then the Heat cured PMMA polymer powder was added and manually mixed to avoid particle agglomeration⁽¹²⁾.

The CAD/CAM specimens; a 3D specimens design with dimensions were virtually designed using software program SketchUp Pro (2020) were saved to as standard tessellation language (STL) file form ; Then, STL files were imported to

CAD software (Exocad Dental DB) which eventually connected to the milling machine to be used for fabricating the milled samples from (Ivotion CAD Base) pre-polymerized PMMA blocks (SPEC 98.5x30 shade Pink Monolayer) with CAD/CAM milling machine (MAXX DS 200-5Z, Korea) with milling PMMA burs (2.5, 1, 0.5 mm) respectively; the number of the axis was five-axis with dry milling subtractive technique ⁽⁸⁾.

Flexural (Transverse) strength test

Thirty specimens, ten specimens for each group, were designed in accordance with ADA specification No.12 (2002) for

denture base polymers. (65mm × 10 mm x 2.5 mm) as shown in figure (1) . Before the testing, all samples were kept in distilled water at (37) °C for (48) hours ⁽¹⁰⁾ .

This test was achieved by operating a three-point loading (Universal testing machine), as shown in figure (2). This apparatus is supplied with a central loading plunger and two supports and a 50 mm distance between supports. The support should be parallel to each other and perpendicular to the central line. The loading plunger is mid-the way between the two supports with a weight of 50 kg and a speed of 5mm/min until fracture happened ⁽¹¹⁾.

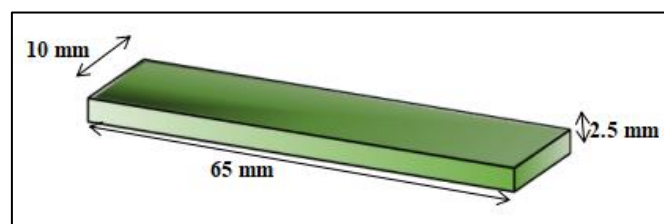


Figure (1): Dimensions of flexural strength testing specimen.

The flexural strength (σ) has been computed by the subsequent equation in (Mpa) for all specimens:

$$\sigma = 3FL/2bh^2$$

F: Max load before fracture.

L: Distance between the support.

b: Width of the specimens.

h: Height of the specimens.



Figure (2): Universal testing machine for measuring flexural strength.

Impact strength test

Thirty specimens, ten specimens for each group, were prepared according to ISO standard No.179-1 in 2010⁽¹³⁾ with dimensions of 80 x 10 x 4 mm ±0.02 (length, width, and height, respectively)

with a V-shaped notch. The specimens were notched in the center to a depth of 2.0 mm using a 45° notch angle and a notch radius of 1.0 ± 0.05 mm as shown in Figure (3).

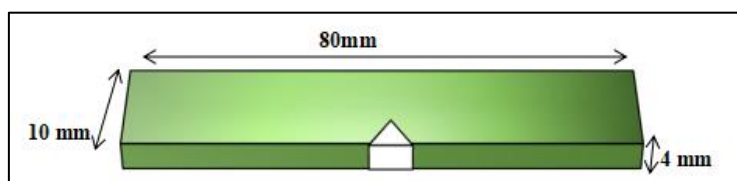


Figure (3): Dimensions of impact strength testing specimen.

The impact strength test was accomplished by utilizing the Charpy impact tester, as shown in figure (4). The specimen was secured in position horizontally utilizing two support arms 40mm away from each other and struck by

a free-swinging pendulum of (25.81 Kg) which was released from a fixed height in the middle on the opposite side of the notch and the result was documented for each specimen in kJ/m² using the following formula⁽¹⁴⁾.

$$I.S = E/b \times d$$

Where:

E: Absorbed energy (Joules).

b: specimen's width (mm).

d: specimens thickness (mm).



Figure (4) : Charpy impact tester for measuring impact strength.

Using the SPSS software version 26, the statistical analysis was performed using inference statistics analysis of variance (ANOVA) and Duncan multiple range tests .

RESULTS

Flexural (Transverse) strength test

(ANOVA) was used to assess the flexural strength data of the control , (1%) zinc oxide nanoparticles, and CAD/CAM table (1), there is significant difference ($P \leq 0.05$) between groups was discovered in this investigation.

Table (1) ANOVA for flexural strength of control , zinc oxide nanoparticles and CAD/CAM groups.

SOV	SS	Df	MS	F	P
Between Groups	10429.6	2	5214.8	409.03	0.000**
Within Groups	344.2	27	12.7		
Total	10773.9	29			

Figure (5), which showed the Duncan's multiple range test results, showed that adding ZnO nanoparticles at a concentration of 1% significantly increased the (1%) ZnO group flexural strength. Additionally, the flexural strength of the

CAD/CAM group was substantially higher than the control group and (1%) ZnO group . This difference was also shown to be significant difference between (1%) ZnO group and CAD/CAM group.

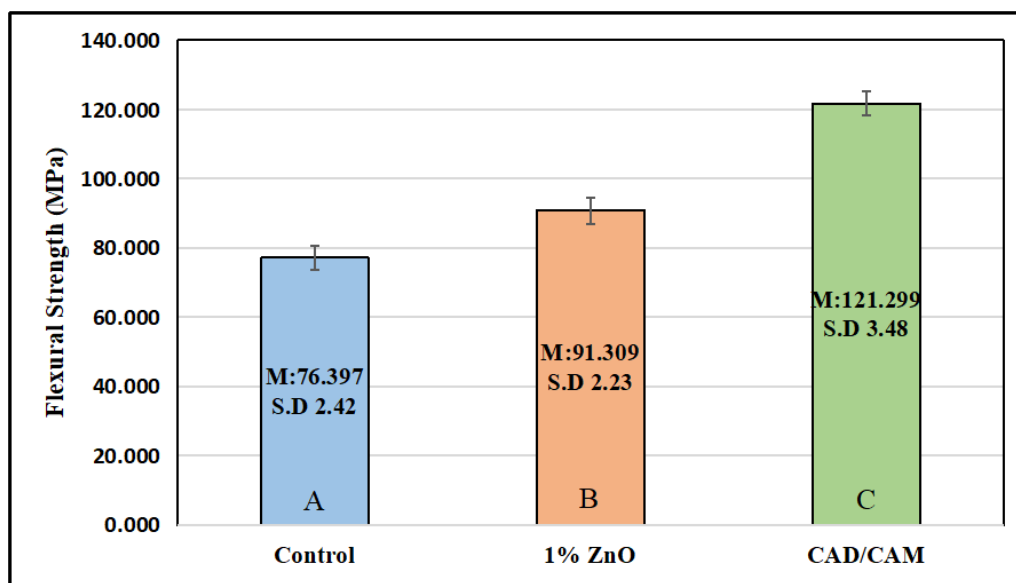


Figure (5): Mean, standard deviation and Duncan’s multiple comparison test for flexural strength of control , (1%) ZnO nanoparticles and CAD/CAM groups.

Impact strength test

One-way analysis of variance (ANOVA), table (2) showed there is

significant difference ($P \leq 0.05$) between groups was discovered in this test.

Table (2) ANOVA for impact strength of control, zinc oxide nanoparticles and CAD/CAM groups.

SOV	SS	Df	MS	F	P
Between Groups	4861.85	2	2430.9	484.4	0.000**
Within Groups	135.48	27	5.01		
Total	4997.33	29			

The Duncan's multiple range test Figure (6), showed that adding ZnO nanoparticles at a concentration of 1% significantly increased in the impact strength. Additionally, the impact strength of the

CAD/CAM group was substantially higher than the control group and the (1%) ZnO group. This difference was also shown to be significant between (1%) ZnO group and CAD/CAM group.

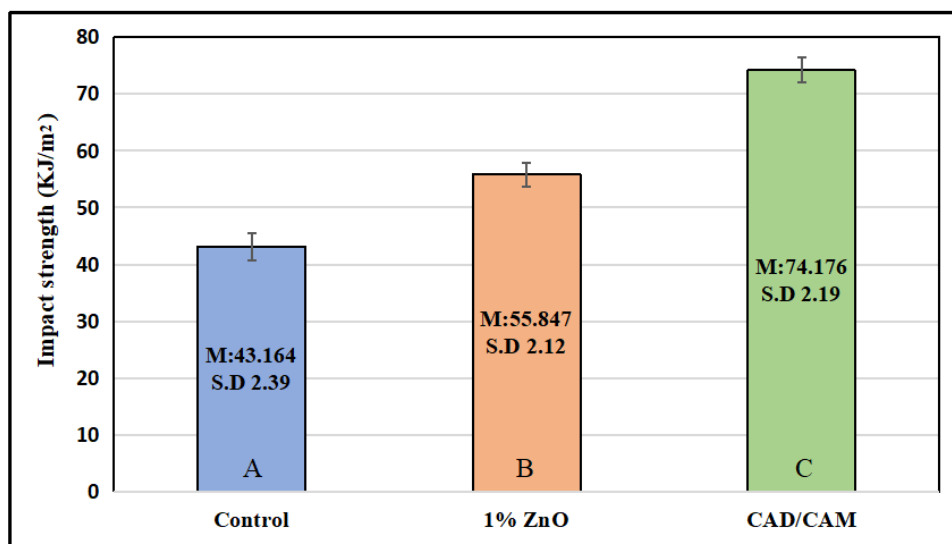


Figure (6): Mean, standard deviation and Duncan's multiple comparison test for impact strength of control , (1%) ZnO nanoparticles and CAD/CAM groups.

DISCUSSION

Flexural Strength Test

The denture base may fracture for different causes; it is critical that its material has increased flexural strength. Optimizing the flexural strength of oral appliances, especially denture bases, is critical because they are subjected to deforming pressures that can lead to fracture, particularly during use ⁽¹⁵⁾.

In this study, the CAD/CAM milled specimens showed a statistically significant higher flexural strength mean value followed by (1%) Zinc oxide nanoparticles group , and (control) group showed a lower mean values, as shown in figure (5). The superiority values of the CAD/CAM group due to the difference in the processing technique of the CAD/CAM group and the incorporation of nanoparticles (1%) zinc oxide group, which lead to a significant

increasing of flexural strength as compared to control .

The polymerization process significantly influences the mechanical properties of denture base resin. The presence and generation of free radicals and temperature control during polymerization are essential factors that affect the material's properties, such as flexural strength .⁽⁸⁾

As a result of the milled CAD/CAM denture base's high polymerization pressure, its average molecular weight has been elevated, and residual monomers and porosity have been reduced ⁽⁹⁾.

The incorporation of (1%) to heat cure PMMA leads to a significant increase in flexural strength; this might be due to better interatomic forces as a result of more increased crosslinking ⁽²³⁾. The strength of an improved polymeric network with a higher degree of crosslinking is increased.

Since there were more contact points or a larger surface area between the matrix and filler, which improved mechanical interlocking and improved stiffness properties, the smaller particle size provided higher mechanical properties. As a result of better load transmission inside the resin matrix ⁽¹⁶⁾.

The finding of the flexural strength of the CAD/CAM group in the study are in agreement with Al-Dwairi *et al.* (2020)⁽⁸⁾; Hada *et al.* (2021)⁽¹⁷⁾ ; Iwaki *et al.* (2020)⁽¹⁸⁾ . On the other hand, our study's findings disagree with those Pacquet *et al.* (2019)⁽¹⁹⁾ reported that CAD/CAM PMMA had lower Flexural strength than heat-polymerized acrylic resin PMMA.

The findings of the flexural strength of the (1%) Zinc oxide NPs group in the study are in agreement with those Vikram and Chander,(2020)⁽⁶⁾; Salahuddin *et al.* (2017)⁽⁵⁾ . Additionally, study Kamonkhantikul *et al.* (2017)⁽²⁰⁾ showed that adding ZnOnps as filler did not improve the mechanical properties of PMMA in different concentrations.

Impact Strength Test

Impact strength remains one of the essential requirements of denture base resin material that still needs to be improved in order to overcome its liability to breakage, since a high strain rate of denture fracture occurs because of being bent during cleaning or dropped suddenly on the floor ⁽²¹⁾.

In this study, the results of the CAD/CAM milled samples showed the statistically significant high-impact strength mean value followed by Modified heat-polymerized with Zinc oxide nanoparticles samples, and conventional heat-polymerized denture base samples showed the lower mean value, as shown in figure (6). The superiority by a high value of impact strength , with the significant difference due to the difference in the processing technique to CAD/CAM group as compared to the control group. The reinforced heat-cured PMMA with (1%) zinc oxide NPs group showed a significant increase in impact strength as compared to a control groups.

Findings of the impact strength test of the milled CAD/CAM group in the study are in agreement with Al-Dwairi *et al.* (2020)⁽⁸⁾ ; Dugal *et al.* (2020)⁽²²⁾ .

The greater degree of polymerization, which is one of the important factors affecting resin strength, may be connected to the superiority of milled CAD/CAM. A highly condensed resin mass with low porosities is accomplished because the CAD/CAM resin blocks are pre-polymerized to a very high degree using equipment more sophisticated than conventional procedures. ⁽²²⁾

The result of zinc oxide group was in line with study of Salahuddin *et al.* (2017)⁽⁵⁾ which concluded that the impact strength improved as a result of the addition of ZnO NPs.

The improvement in impact strength of zinc oxide group may be attributed to the superior dispersion of the nanoparticles that improve the interfacial shear strength between the PMMA matrix and nanoparticles strength due to their nano size, which help fill the matrix interstitially. Moreover, the incorporation of the metal nanoparticles into the polymer matrix enhances the stiffness of the Nanocomposites by restricting the mobility of the polymer matrix chains ⁽²³⁾.

On the other hand, the findings of our study are in disagreement with Hummudi *et al.* (2022)⁽²⁴⁾ who showed that the addition of ZnO as a filler might impair the impact strength of the denture material and reported that the reason might be result in less energy absorption due to stress concentration around the zinc oxide particles and therefore create crack propagation.

CONCLUSION

CAD/CAM dentures exhibit more favorable mechanical properties, trying to make them more acceptable as acrylic resin denture base material for edentulous patients. In addition, the use of ZnO nanoparticles as dental fillers at 1% by weight significantly improved the flexural strength and also improves the impact strength of denture base material (PMMA).

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