




Flexural and Impact Strength of Poly Methyl Methacrylate Incorporated with Hydroxyapatite Nanoparticles.

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Article information

Received: 21 June 2022

Accepted: 1 August 2022

Available online: 1 September 2024

Keywords

Impact strength,
Flexural strength,
HA nanoparticles.

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Abstract

Aims: To investigate the effects of the incorporation of hydroxyapatite nanoparticles (HA NPs) with size (20 nm) at two concentrations (0.5% and 1%) on the flexural strength and impact strength of heat-cured acrylic resin. **Materials and methods:** The total number of specimens was sixty which were divided into two groups, thirty specimens for each test (flexural strength test and impact strength test), ten specimens for each subgroup (control, 0.5 % HA NPs and 1 % HA NPs), the flexural strength was performed using the universal testing machine, and impact strength using Charpy impact tester. The statistical analysis was done by using the SPSS program including descriptive statistics, ANOVA, and Duncan's test at $p \leq 0.05$. **Results:** The results demonstrated that there was a significant increase in the flexural and impact strength for the PMMA-HA nanocomposite at HA nanoparticles (0.5%) and (1%), when compared to control. **Conclusion:** the use of hydroxyapatite nanoparticles as dental fillers at 0.5% and 1% by weight enhanced the flexural strength and impact strength of PMMA denture base material.

القوة العرضية وقوة التأثير للبولى ميثيل ميثاكريليت لقاعدة اطقم الاسنان المدمج بجسيمات الهيدروكسي ابيتايت النانوية

الملخص

الأهداف: دراسة تأثير دمج جزيئات الهيدروكسي ابيتايت النانوية بحجم (20 نانومتر) بتركيزين (0.5% و 1%) على قوة الانحناء وقوة التأثير لراتنج الأكريليك المعالج بالحرارة. **المواد وطرائق العمل:** كان العدد الإجمالي للعينات ستين مقسمة إلى مجموعتين ثلاثين عينة لكل اختبار (اختبار قوة الانحناء واختبار قوة التأثير)، عشر عينات لكل مجموعة فرعية (مجموعة السيطرة، 0.5%، 1%) هيدروكسي ابيتايت النانوية، تم إجراء قوة الانحناء باستخدام آلة الاختبار الشاملة، وقوة التأثير باستخدام اختبار شاربي. تم إجراء التحليل الإحصائي من خلال برنامج spss وباستخدام الاحصاء الوصفي والمقارنات من خلال اختبار تحليل التباين مع اختبار دنكن عند $p \leq 0.05$. **النتائج:** أظهرت النتائج أن هناك زيادة ملحوظة في قوة الانحناء والتأثير لمركب النانوي المتولد في جزيئات الهيدروكسي ابيتايت النانوية (0.5%) و (1%)، على التوالي، عند مقارنتها بمجموعة السيطرة. **الاستنتاجات:** أن إضافة هيدروكسي ابيتايت إلى البولى ميثيل ميثاكريليت المعالج بالحرارة له تأثير ايجابي على المركب النانوي المتولد من حيث القوة العرضية وقوة التأثير.

DOI: 10.33899/RDENJ.2022.134388.1166 © Authors, 2024, College of Dentistry, University of Mosul

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INTRODUCTION

Teeth loss is one of the most prevalent oral health issues, particularly among the elderly ⁽¹⁾. Several therapeutic procedures are available to replace lost teeth, regardless of the underlying cause ⁽²⁾.

Dentures, either partial or complete, are the most common treatment options for replacing missing teeth, owing to the higher expense of dental implants ⁽³⁾. Resin, ceramic, and metal are the three types of dental materials utilized in denture manufacture. Acrylic resin, also known as polymethyl methacrylate (PMMA), is one of the most often used polymeric materials for denture bases because it has superior mechanical and physical qualities to other polymers ⁽⁴⁾. Though their properties are not perfect in every way, because they can be easily destroyed if dropped or if the patient applies a lot of mastication power to the denture base, there is a need to reinforce the denture base material to increase its mechanical characteristics ⁽⁵⁾.

Different processing strategies have been presented to produce a polymer with superior physical and mechanical qualities, in addition to the use of alternative materials or the modification of denture base acrylic resin by the incorporation of reinforcement materials ⁽⁶⁾. Unfortunately, improving some properties without jeopardizing others remains challenging ⁽⁷⁾.

It has been revealed that denture base material reinforcement includes the incorporation of foreign material into the prosthesis, which could be a risk factor for the development of prosthesis crack rather than crack prevention because it may act as an interfering feature in the integrity of the polymer matrix, resulting in stress concentration points due to inadequate concentration and/or incompatible or weak interfacial adhesion between the reinforcing component and denture base material ⁽⁸⁾.

As a result, care should be taken while selecting the appropriate kind and concentrations of reinforcement materials that have been utilized to improve the mechanical qualities of PMMA denture base resin without negatively influencing its other features ⁽⁹⁾.

Hydroxyapatite nanoparticles when employed as a reinforcing material, have a considerable impact on the mechanical characteristics of polymers ⁽¹⁰⁾.

This research aimed to study the influence of hydroxyapatite nanoparticle addition at two concentrations (0.5% and 1%) on flexural strength and impact strength of heat-cured acrylic resin material.

MATERIALS AND METHODS

Sampling

The total number of specimens was sixty which were divided into two groups thirty specimens for each test (flexural strength

test and impact strength test), then ten specimens for each subgroup (control, 0.5 % HA NPs and 1 % HA NPs), This study was done at the college of dentistry and technical institute at the University of Mosul. Approval of the study was from the Scientific Research Committee / Department of Prosthodontics / College of Dentistry (UoM. Dent / DM. L.34/22).

Preparation of the mold

Acrylic sheets were utilized to make the plastic model, which was produced using computer software (AutoCAD) and then carved with a computer-controlled laser-cutting machine. The length, width, and thickness of the plastic models used in mold fabrication were precisely established according to the specifications needed for each test.

During the mold preparation, a conventional flaking procedure was used for full dentures. A separating medium (cold mold seal) was used and allowed to dry for the layer of plastic before putting the lower part of metal flasks filled with dental stone and combining in vibration according to the directions of the manufacturer to release the trapped air, then left to set.

Specimens of all groups were then stored in distilled water at 37°C for 2 days using an incubator ⁽¹¹⁾.

Preparation of the Specimens

The mixing ratio of powder to liquid for heat-cured PMMA polymer material was 2:1 by weight, according to the manufacturer's instructions. The weight of

the hydroxyapatite nanoparticles was subtracted from the weight of the heat-cured PMMA polymer powder to produce the precise powder-to-liquid ratio stated by the manufacturer ⁽¹²⁾.

The specimens were first prepared by mixing the weight of hydroxyapatite nanopowder with "heat-cured PMMA" fluid monomer, which was sonicated and dispersed in the liquid monomer for 3 minutes using an ultrasonic probe of 20W and 60 kHz, and then the Heat-cured PMMA polymer powder was added and manually mixed to avoid particle agglomeration ⁽¹³⁾.

Flexural Strength Test

The specimen was made under International Standards Organization Specification No. 1567 (ISO) specifications (65 mm length × 10 mm width × 2.5 mm thickness), as presented in

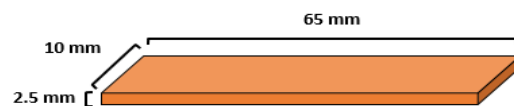


Figure (1).

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

The flexural strength test was performed using three-point bend tests (Universal testing machine), in which the specimen was placed on two parallel supports 50 mm apart for a three-point bending test, and it was then bent using a rod positioned in the center of the two supports with a weight of 50 kg and a speed

of 5mm/min until fracture occurred, as shown in Figure (2).



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

The transverse strength (σ) has been calculated by the following equation in (Mpa) for all specimens ⁽¹⁴⁾:

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

The following formula states that: F: Maximal force applied in the centre of the sample before fracture (N).

L: Distance between the two supports (mm).

b: Width of the specimen (mm).

h: Thickness of specimen (mm).

Impact Strength Test

The specimens for the impact strength test were designed according to iso standard No.179-1 in 2010 with measurements of 80 x 10 x 4 mm (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 (Figure 3).

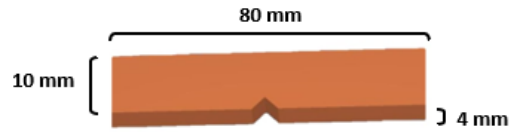


Figure (3): Dimensions of impact strength testing specimen.

The impact strength test was carried out utilizing an impact tester, as shown in Figure (4).



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

The specimen is held horizontally by two support arms 40 mm away from each other and struck by a free-swinging pendulum (25.81 Kg) released from a constant height at the middle on the opposite side of the notch, and the impact strength was calculated in KJ/m² for each specimen using the following formula ⁽¹⁴⁾:

Impact strength = $E/b \times d$; Where:

E: Absorbed energy.

b: Sample width.

d: Sample thickness.

RESULTS

The statistical analysis: Descriptive statistics and Inference statistics (ANOVA and Duncan's test) were done by using the SPSS program version (19).

Flexural Strength Test:

Descriptive statistics (mean and standard deviation) of three groups of flexural strength tests which included (control, 0.5 % HA NPs, and 1 % HA NPs) were presented in Table (1).

Table (1): Mean and standard deviation for flexural strength of control and HA nanoparticles groups.

Groups	N.	Mean	Std. Deviation
Control	10	79.5000	5.61690
0.5% HA NPs	10	92.7810	2.31709
1% HA NPs	10	89.6380	1.67795

One-way analysis of variance (ANOVA) was used to assess the flexural strength data of the control and (0.5% and 1%) hydroxyapatite nanoparticles; Table (2): A substantial difference ($P \leq 0.05$) between groups was discovered in this investigation.

Table (2): ANOVA for flexural strength of control and HA nanoparticles groups.

SOV	SS	Df	MS	F	P
Between Groups	963.475	2	481.737	36.372	.000
Within Groups	357.606	27	13.245		
Total	1321.081	29			

Duncan's multiple range test of flexural strength demonstrated a significant increase at HA nanoparticles (0.5%) and (1%), respectively, when compared to the control. The (0.5%) and (1%) HA nanoparticle groups did not differ significantly (Figure 5).

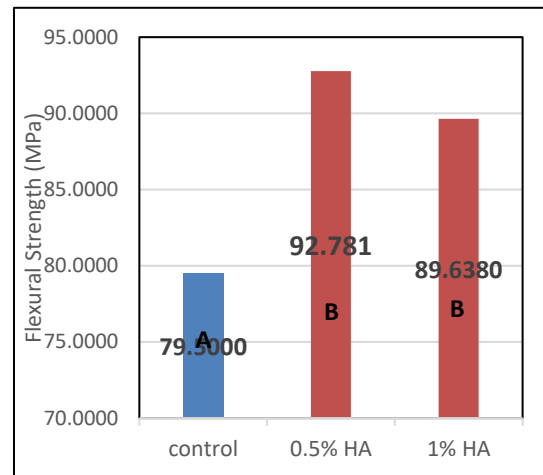


Figure (5): Duncan's multiple range test for flexural strength of control and HA nanoparticles groups.

Impact Strength Test

Descriptive statistics (mean and standard deviation) of three groups of impact strength tests which included (control, 0.5 % HA NPs, and 1 % HA NPs) were presented in Table (3).

Table (3): Mean and standard deviation for impact strength of control and HA nanoparticles groups.

Groups	N.	Mean	Std. Deviation
Control	10	36.7280	0.32737
0.5% HA NPs	10	48.5570	0.41862
1% HA NPs	10	58.4530	4.89077

One-way analysis of variance (ANOVA) was used to assess the impact strength data of the control and (0.5% and

1%) hydroxyapatite nanoparticles; Table (4): A substantial difference ($P \leq 0.05$) between groups was discovered in this investigation.

Table (4): ANOVA for impact strength of control and HA nanoparticles groups.

SOV	SS	Df	MS	F	P
Between Groups	2366.106	2	1183.053	146.647	.000
Within Groups	217.819	27	8.067		
Total	2583.924	29			

Duncan's multiple range test of impact strength demonstrated a significant increase in HA nanoparticles (0.5%) and (1%), respectively, when compared to control. There was a significant difference between the (0.5% and 1%) HA nanoparticles groups, with the 1% HA nanoparticles group significantly higher than the control group and (0.5%) HA nanoparticles group (Figure 6).

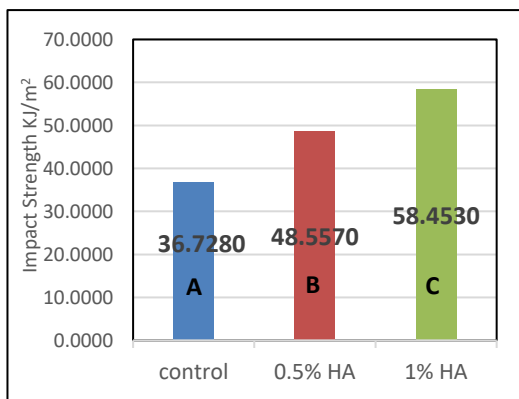


Figure (6): Duncan's multiple range test for impact strength of control and HA nanoparticles groups.

DISCUSSION

Flexural Strength Test

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⁾.

The inclusion of hydroxyapatite nanoparticles resulted in relatively substantial differences in flexural strength values when compared to the control group, as shown in Table (2).

The findings reveal that the 0.5% and 1% HA groups' flexural strength values were greater than that of the control group, Figure (5). This might be due to better interatomic forces as a result of higher crosslinking. The strength of an improved polymeric network with a higher degree of crosslinking is increased as a result of better load transmission inside the resin matrix⁽¹⁶⁾.

The reinforcing impact of the nanoparticles in the polymer matrix was attributed to the heat-cured PMMA- HA nanocomposite's greater transverse strength. A strong connection between the nanoparticles and the polymer chains results in a three-dimensional network, which was anticipated to restrict the polymer chain's movement against the nanoparticle's surface as a consequence of the strengthening process⁽¹⁷⁾.

According to Ranganathan *et al.*⁽¹⁸⁾, massive surface energy created by crosslink among the polymeric networks prevents stress concentration at the filler/matrix interphase, resulting in higher flexural strength of the corresponding resin composites, or according to Fatihallah⁽¹⁹⁾, as the crystallinity of polymeric composite

increases, the interfacial strength among polymer increases, resulting in higher flexural strength of the corresponding resin composites.

The strength of polymeric composites improved as the homogeneity of the polymeric matrix increased ⁽²⁰⁾. The inhomogeneity of the polymeric matrix acts as a structural defect, causing stress concentrations and lowering the material's strength.

according to Karci *et al.* ⁽²¹⁾ the agglomeration of incorporated fillers at higher addition percentages that reached supersaturation works as defects and stress concentration centers that disrupt the crosslinking of polymer networks, resulting in a reduction in flexural strength of PMMA composite ⁽²²⁾.

Another factor that might explain the decrease in PMMA's flexural strength is porosity. These flaws cause stress to build up and cracks to spread, making the denture base more prone to fatigue failure ⁽²³⁾.

Impact Strength Test

Impact strength is still one of the most essential requirements of denture base resin material that has to be enhanced to overcome its proclivity to breakage since a high strain rate of denture fracture occurs as a result of being bent while cleaning or dropped unexpectedly on the floor ⁽²⁴⁾.

The inclusion of hydroxyapatite nanoparticles resulted in relatively substantial differences in impact strength values when compared to the control group, as shown in Table (4).

The findings reveal that the 0.5% and 1% HA groups' impact strength values were greater than that of the control group, Figure (6). This could be due to the nano filler's high interfacial strength, which is caused by cross-links or supramolecular connections shielding the nanofillers and restricting crack propagation, and by attaching functional groups on nanoparticle surfaces to polymer chains; fracture propagation can be prevented.

Furthermore, nanoparticles may fill the spaces between polymer chains, resulting in a heterogeneous mixture and limiting polymer chain segment displacement. Nanoparticles can minimize fracture propagation and so boost impact resistance because of their large surface area ⁽²⁵⁾. It's important to note that including HA nanoparticles at a concentration of more than 1% reduces the impact strength of the PMMA nanocomposite to levels lower than those seen in pure PMMA ⁽¹⁷⁾.

This was in agreement with Gad and Abualsaud ⁽²⁶⁾, who said that the interactions between the polymer matrix and the included fillers had a significant impact on the characteristics of composites.

The incorporation of nano-hydroxyapatite to PMMA increased impact strength significantly, which might be attributed to interfacial interaction between the two materials. The tiny size and low concentration of HA nanoparticles, on the other hand, aided in their dispersion and embedding in the polymer matrix, which improved the impact strength ⁽²⁷⁾.

These findings were consistent with those of Schulze *et al.* ⁽²⁸⁾, who stated that increasing the concentration of incorporated fillers does not necessarily increase the strength because fillers tend to cluster, especially when incorporated at higher fraction content and act as defects, causing stress concentration and material weakness. Somani *et al.* ⁽²⁹⁾ found a comparable drop in PMMA strength along with an increase in the concentrations of integrated fillers.

Also, agree with Anjali *et al.* ⁽³⁰⁾, who found that the distribution of filler particles inside the matrix has a significant influence on the impact strength of composites.

This observation may be compatible with Ghahremani *et al.* ⁽³¹⁾ studies in which nanofillers (hydroxyapatite) were added to PMMA and the impact strength was increased.

CONCLUSIONS

The incorporation of hydroxyapatite nanoparticles into the "heat-cured PMMA" enhanced the flexural strength and impact strength of the heat-cured PMMA denture base material.

Conflicts of Interest

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

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