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The Effect of Adding Hydroxyapatite Nanoparticles on Hardness of Heat Cured Acrylic Resin Material.

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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 hardness of heat-cured acrylic resin. **Materials and methods**: The total number of specimens was thirty divided into ten specimens (control, 0.5 % HA NPs, and 1 % HA NPs), the hardness was performed using shore D (Durometer), FTIR test was performed using (BRUKER LASER CLASS 1). 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 hardness for the PMMA-HA nanocomposite at HA nanoparticles (0.5%) and (1% when compared to the control. **Conclusion:** the use of hydroxyapatite nanoparticles as dental fillers at 0.5% and 1% by weight increased the hardness of PMMA denture base material.

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تاثير اضافة جسيمات الهيدروكسى ابيتايت النانوية على صلابة الراتنج الاكريلى المبلمر بالحرارة

الملخص

الأهداف: دراسة تأثير دمج جزيئات الهيدروكسي ابيتايت النانوية بحجم (20 نانومتر) بتركيزين (٠,٥٪ ،١٪) على الصلابة الراتنج الأكريليك المعالج بالحرارة. **المواد و طرائق العمل**: كان العدد الإجمالي للعينات ثلاثين مقسمة إلى عشر عينات (مجموعة السيطرة ، ٠,٥٪ ، ١٪) هيدروكسي ابيتايت النانوية ، تم إجراء فحص الصلابة باستخدام جهاز اختبار صلابة شور دي (مقياس التحمل) وتم احراء اختبار فورييه لتحويل طيف الاشعة تحت الحمراء باستخدام جهاز بروكر ليزر. تم إجراء التحليل الإحصائي باستخدام برنامج الاحصاء بما في ذلك الإحصاء الوصفي ، اختبارانوفا ، واختبار دنكن عند $p \leq 0.05$. **النتائج**: أظهرت النتائج أن هناك زيادة ملحوظة في الصلابة لمركب النانوي المتولد في جزيئات الهايدروكسي ابيتايت النانوية (٠,٥٪) و (١٪) ، عند مقارنتها بمجموعة السيطرة. **الاستنتاجات**: أن إضافة هيدروكسي ابيتايت إلى البولي ميثيل ميثاكريليت المعالج بالحرارة له تأثير ايجابي على المركب النانوي المتولد من حيث الصلابة لمادة قاعدة أطقم الاسنان.

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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 incompatibly 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 the hardness of heat-cured acrylic resin material and monitor the chemical structure of modified heat-cured denture base resin by using FTIR analysis.

MATERIALS AND METHODS

Sampling

Total number of specimens was thirty, those divided into three groups (control, 0.5 % HA NPs, and 1 % HA NPs). Ten specimens for each one. This study was done at the College of Dentistry / 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

A conventional flacking procedure was used during the mold preparation. 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.

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. Specimens of all groups were then stored in distilled water at 37ºC 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)].

Indentation Hardness Test

The specimens for the indentation hardness test were prepared with dimensions of 30mm length, 15mm width, and 3mm thickness [(14)], as shown in Figure (1).

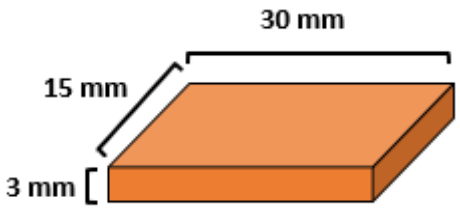

Figure (1): Dimensions of hardness testing specimen.

Shore D hardness tester (Durometer) was performed using an indenter (1.40 mm in diameter) which was attached to a digital scale starting from 0 to 100 units. The usual method was to press on the indenter and record the maximum reading (Figure 2). The surfaces of the specimen have been examined at three separate locations for hardness and the mean for each specimen has been determined. The sample had a set load of

44.5 N, after applying this load the hardness number had been recorded by 1 second following the instructions of the machine.

Figure (2): Shore D durometer hardness tester.

Fourier Transform Infrared Spectroscopy Test (FTIR)

FTIR analysis of the chemical interaction or change following the inclusion of the additives used in this study (Hydroxyapatite nanoparticles).

FTIR was performed using a CL Alpha-PFTIR spectrophotometer with a resolution of 2 $cm^{-1}$and a wavenumber region 400–4000 cm^{-1} Figure (3).

Figure (3): FTIR spectroscopy.

FTIR was performed on three specimens, one for each group (control, 0.5 %, and 1 % Hydroxyapatite nanoparticles) with dimensions of 10×4×4 mm [15].

The analysis was carried out by mixing the ground powder of the specimens with potassium bromide salt and compressing them under pressure to form a pellet that was analyzed by FTIR spectroscopy [16].

RESULTS

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

Indentation Hardness Test

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

Table (1) Mean and standard deviation for hardness of control and HA nanoparticles groups.

Groups	N.	Mean	Std. Deviation
Control	10	91.7500	1.62224
0.5% HA NPs	10	94.7600	1.10574
1% HA NPs	10	95.6600	1.04796

One-way analysis of variance (ANOVA) was used to assess adding of hydroxyapatite nanoparticles in two concentrations (0.5% and 1%) on the hardness of heat-cured acrylic resin denture base material, as shown in Table (2); A substantial difference ($P \leq 0.05$) between groups was discovered in this investigation.

Table (2) ANOVA indentation hardness test after adding Hydroxyapatite nanoparticles.

SOV	SS	Df	MS	F	P
Between Groups	83.861	2	41.930	25.399	.000
Within Groups	44.573	27	1.651		
Total	128.434	29			

Duncan's multiple range of hardness test demonstrated a significantly increased at (0.5%) and (1%), concentrations of HA nanoparticles respectively, when compared to control. The (0.5%) and (1%) HA nanoparticles groups did not differ significantly (Figure 4).

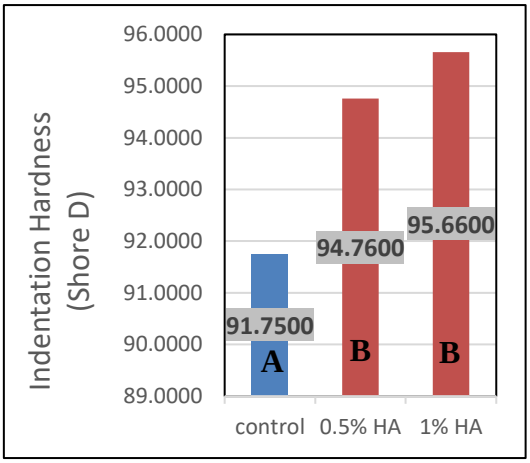

Figure (4) Duncan's multiple range test for indentation hardness after adding Hydroxyapatite nanoparticles.

Fourier Transform Infrared Spectroscopy Test (FTIR):

FTIR spectrum for control heat cured PMMA, Figure (5) showed absorption bands at 2992 cm^{-1} related to aliphatic (ν $-CH_3$) group and at 2950 cm^{-1} related to aliphatic (ν $-CH_2$) group; strong absorption bands at 1720 cm^{-1} for (ν C=O) group and at 1142 cm^{-1} for (ν C-O) in addition to absorption band at 1434 cm^{-1} related to (δ $-CH_2$) group.

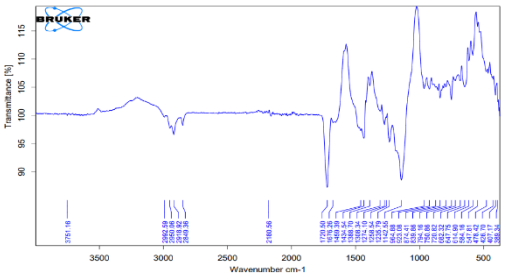

Figure (5): FTIR spectrum of control heat cured PMMA.

In comparison to the control group spectrum, the FTIR spectra for 0.5% and 1% hydroxyapatite nanoparticles (Figures, 6 and 7) reveal comparable FTIR spectral peaks.

The lack of new peaks suggests that adding hydroxyapatite nanoparticles at 0.5% and 1% did not stimulate the creation of new products in the PMMA matrix since there were no chemical reactions between the saturated heat-cured PMMA and the saturated HA nanoparticles. However, a composite was created using nano-sized additives as fillers in a polymer matrix, and this composite may result in a change in physical and mechanical qualities.

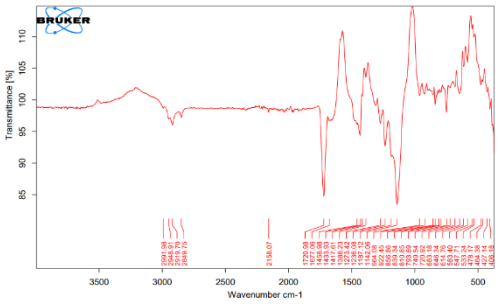

Figure (6): FTIR spectrum of the HA nanoparticles group at 0.5%.

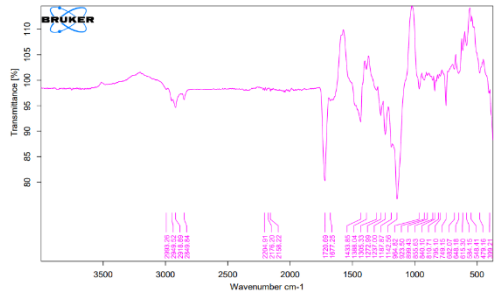

Figure (7) FTIR spectrum of the HA nanoparticles group at 1%.

DISCUSSION

Indentation Hardness Test

The hardness of denture base materials reflects the degree of polymeric matrix resistance to degradation and, as a result, determines the lifespan of the denture base inside the oral cavity [17]. Because higher hardness ratings are often associated with greater wear resistance [18].

The inclusion of hydroxyapatite nanoparticles resulted in relatively substantial differences in hardness values when compared to the control group, this result could be explained according to Zaynab *et al.* [19] who provided details regarding the process of the better hardness of the nanocomposite group as compared to control group to the higher stiffness of the resin matrix related to the reduction in its free volume and molecular mobility which might be a consequence of enhanced internal adhesion.

This substantial improvement in hardness may agree with the results of another study by Karadi and Hussein, [20] in which hydroxyapatite nanoparticles were added to "heat-cured PMMA" to enhance its hardness.

The findings of this test were consistent with Chladek *et al.* [21], who explained the change in nanocomposite hardness as a function of HA loading, demonstrating a progressive rise in the filler weight percentage.

The hardness of a composite is determined by the strength of the intermolecular bonds between the

nanoparticles and matrix; thus, its enhancement could be attributed to the uniform distribution of the HA nanoparticles within the PMMA matrix, which promoted a good interface between them, enhancing load transfer and consolidating resistance against shear stresses caused by volume compression [22].

The observed results further show that a low HA loading can improve the mechanical characteristics of PMMA, as opposed to excessive filler loadings, which promote agglomeration and degrade the material properties [23].

The decrease in hardness might be explained by the fact that the inserted fillers act as impurities, forcing themselves between resin chains, shifting them apart, and causing internal stresses and fracture development [24].

Fourier Transform Infrared Spectroscopy Test (FTIR):

FTIR analysis is used to identify organic, inorganic, and polymeric substances by scanning the samples with IR light. The transformation of material composition is defined by absorption band pattern alterations and detection of unknown substances, detection of impurities in the material, the discovery of additions, and oxidation [25].

The spectrum of a substance supplied information on the chemical content of the material as well as the identification of new copolymer synthesis [26].

FTIR chart of control, 0.5%, and 1% hydroxyapatite nanoparticles groups showed that no chemical interaction happened between them and that just a composite was established. Even though the experimental group's FTIR spectra showed the same FTIR absorption bands as the control group and revealed no change in the PMMA spectral range after the addition of 0.5 % and % hydroxyapatite nanoparticles, the data show that only physical and mechanical alterations occurred.

The absorbance band of nanoparticles in heat-cured acrylic resin did not emerge in the FTIR spectroscopy chart because its wavelength is between (200-400 cm-1) which is beyond the range of FTIR spectroscopy (400-4000 cm^{-1}) [27].

This finding was consistent with Flayeh [28] conclusion that FTIR analysis for heat-cured acrylic resin and zirconium oxide nanoparticles revealed no chemical interaction.

CONCLUSION

The incorporation of hydroxyapatite nanoparticles into the "heat-cured PMMA" enhanced the hardness 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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