Effect of addition ZrO_2-Al_2O_3 nanoparticles mixture on some properties and denture base adaptation of heat cured acrylic resin denture base material

Ali MA Aljafery, B.D.S. [1]
Basima MAH, B.D.S., M.Sc., Ph.D. [2]

ABSTRACT

Background: The PMMA polymer denture base materials are low mechanical properties, adaptation of the denture base to underlying tissue is important for retention and stability of denture. The aim of the study was toevaluate the effect of mixtureZrO_2-Al_2O_3 nanoparticles on impact strength, transverse strength, hardness, roughness, denture base adaptation of heat cured acrylic resin denture base material.

Materials and methods: One hundred (100) specimens were prepared, the specimens were divided into five groups (20 specimens to each) according to the test type, each group was subdivided in to two subgroups (control and experimental) each subgroup consist of 10 specimens, the experimental group included mixture of 2% (ZrO_2-Al_2O_3ratio2:1) by weight. Theimpact strength was measured by Charpy's impact testing machine, the transverse strength was measured by Instron testing machine while the hardness was measured byshore D durometer and roughness was measured by Profilometer. Denture base adaptation was measured by digital microscope and evaluated by computerized tomography (CT).

Results: Highly significant increaseofimpact and transverse strength, non-significant increase ofhardness, significant increase ofroughness andreduction of denture base adaptation (measured at 3 point A, B and C) occurred in experimental groupwhen compared to control group. CT evaluation, gap between the denture base and master cast (control and experimental groups) increased from the anterior to posterior side of palate and from the alveolar ridge to the mid palatal line.

Conclusion: The polymer nanocomposites had mechanical properties higher than neat PMMA at same time less denture base adaptation.

Keywords: Acrylic denture base, nano fillers, mechanical properties, denture base adaptation. (J Bagh Coll Dentistry 2015; 27(3):15-21).

INTRODUCTION

Acrylic resin polymethyl methacrylate (PMMA) is the most extensively used material in fabrication of dentures. Although it is very popular, this material is still insufficient in fulfilling the ideal mechanical requirements of such appliances [1]. Clinicians still encounter fracture of this material due to low resistance to impact, flexural, or fatigue stresses [2]. In order to prevent fracture of the dentures, the thickness of acrylic resin in susceptible regions, such as the palatal midline, and the mandibular lingual and labial frenal attachments has been increased[3].

In addition, improvement on mechanical properties of denture base materials were tried to be achieved either by adding a polyfunctional cross-linking agent such as polyethylene glycol dimethacrylate [4] or by incorporating a rubber phase [5], metal oxides, metal wire [6,7] or fiber [8]. The reinforcement of polymers used in dentistry with metal-composite systems has been a prime interest. Addition various amounts of powdered Cu, Ag and Al into the PMMA resin and reported increased compressive strength but decreased tensile strength [9].

(1)M.Sc. Student. Department of Prosthodontics, College of Dentistry, University of Baghdad.
(2)Assistant Professor. Department of Prosthodontics, College of Dentistry, University of Baghdad.

Evaluation the changes in the mechanical properties of PMMA, polyethyl methacrylate (PEMA) and poly isobutyl methacrylate (PIMA) resin matrices by reinforcingwith oxides of Al, Mg, Zr and pulverized E-glass particles [10]. They suggested that 2% admixtures by volume in PMMA resin matrix resulted in better mechanical properties. Much attention has been directed toward the incorporation inorganic nanoparticles in to PMMA to improve its properties. The properties of polymer nanocomposites depend on the type of incorporating nanoparticles, their size and shape, as well as the concentration and interaction with the polymer matrix [11].

Nanoparticles were undergone surface treatment with silane coupling agent and embedded in to PMMA [12]. Alumina nanoparticles were treated with trimethoxysilylpropylmethacrylate (TMSPM) to get PMMA/alumina Nano composite with improved properties over pure PMMA [13]. Also, using modified ZrO_2 with trimethoxysily-lpropylmethacrylate to get PMMA/ ZrO_2 Nano composite to improve properties of PMMA [14]. Furthermore, studying an experimental investigation of mixture HA/AL_2O_3 nanoparticles on mechanical properties of restoration materials [15]. Also, evaluation of influence of mixture of ZrO_2-TiO_2 on mechanical and physical properties

of heat-cured polymethyl methacrylate denture base resins [16].Though the incorporation of fillers like rubber and fibers to heat-cured poly methyl methacrylate resin improves the impact strength and fatigue resistance, it may affect some of the properties of heat-cured poly methyl methacrylate resin such as fitness accuracy (denture adaptation), dimensional stability and the effect of water sorption [17]. Various investigators have compared the dimensional changes between different denture base materials [17,18], palatal vault configurations [19], methods of packing [18], modes of polymerization [20] and curing cycles [21]. This study was conducted to use inorganic mixture of ZrO_2-Al_2O_3 Nano fillers that were added to heat cure PMMA and test the effect of this addition on the some mechanical properties and denture base adaptation of heat cured acrylic denture base material.

MATERIALS AND METHODS

Some of the materials used in this study are summarized in table (1).

Table 1: List of the materials that were used

Material	ZrO_2 nanofiller	Al_2O_3 nanofiller	Trimethoxysilylpropyl methacrylate(TMSPM)	Heat-curing acrylic resin
Manufacturer	HWNANO China	NS6130 01-123 Germany	2530-85-8 Germany	Vertex Netherlands

Test specimens preparation

Two different plastic patterns were constructed according to the required test. The pattern that was constructed for impact strength a bar shaped specimen (80mm X 10mm X 4mm) length, width, thickness respectively [22]. For transverse strength test: a bar shaped specimen was constructed (65mm X 10mm X 2.5mm) length, width, thickness respectively [23] (Figure 1). Same specimen measurement was used to prepare hardness test and roughness test. For denture base adaptation test: prepare acrylic resin denture bases with their corresponding master casts byconventional denture flasking technique using a Biostar sheet as record base (2mm thickness) without teeth.

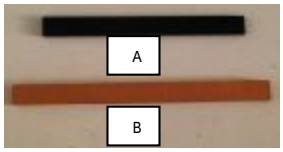


Figure 1: Plastic patterns; A for transverse strength, B for impact strength

Surface modification of nanofillers (ZrO_2, Al_2O_3)

The introduction of reactive groups to the fillers surface was achieved by reaction of 3-trimethoxy silylpropyl methacrylate (TMSPM) with zirconium oxide and aluminum oxide Nano fillersthrough salinization procedure [24]. For ZrO_2, TMSPM was used in 5% wt. of nanofiller, toluene was used as a solvent to ZrO_2 [12,14,24], while Al_2O_3,TMSPM was used in 75% wt. of Al_2O_3, ethanol was used as a solvent toAl_2O_3[13].

Mould preparation

Addition of fillers

This only for experimental groupincluded mixture of 2% (ZrO2-Al2O3 ratio 2:1) by weight, electronic balance (Sartorius, Germany)with sensitivity of (0.0001g)was used to weigh then nanofillers powder weight in 2%wt. of the PMMA powder weight. The filler was added to the monomer of PMMA mixed by the probe sonifier apparatus (DANBURY, U.S.A.) for 3 minutes [13,14] to disperse the nanoparticles in the monomerand reduce the possibility of particle aggregation.

Mixing of the acrylic

Acrylic materialwas mixed and manipulated according to manufacturer's instructions using a conventional water bath denture flasking technique for both groups (control and experimental). For experimental group the suspension of the monomer with nanofiller was immediately mixed with acrylic powder.

Mechanical and denture base adaptation tests

1-Impact strength test.

a- All theprepared specimens (20 specimens, 10 for each control and experimental groups) stored in distilled water inside incubator at 37°C for 48 hours before testing [23].

b- Testing procedure: The impact strength test was carried out following the procedure recommended by the ISO 179 usingimpact testing device (Tmi, testing machine Inc. Amity Ville, New York, USA) [22] (Figure 2).The specimen was supported horizontally at each end and strucked by free swinging pendulum of 2 Joules.

The scale readings give the impact energy in Joules. The charpy impact strength of un-notched specimen was calculated in Kilo-joules per square meter using the following formula:

Impact strength (kj/m^2) $= \frac{E}{b.d} \times 10^3$ where E: The impact energy in Joules, b: Is the width of the specimens in millimeters, d: Is the depth of the specimens in millimeters.

2-Transverse strength test.

a. All the prepared specimens (20 specimens, 10 for each control and experimental groups) stored in distilled water inside incubator at 37°C for 48 hours before being tested [23].

Figure 2: Impact strength testing device

b. Testing procedure: The test was performed using Instron universal testing machine (WDW-200 E, UK) (Figure 3), each specimen was positioned on the bending fixture which consist of two parallel supports (50 mm apart). The load was applied by a rod placed centrally between the supports with across head speed of 1mm/min applied making deflection until fracture occurs. The transverse strength was calculated using the following formula:

Transverse strength (N/mm2) $= -\frac{3PL}{2bd^2}$ Where P: is the peak load, L: is the span length, b: is the sample width, d: is the sample thickness [25].

Figure 3: Instron testing device

3- Measuring hardness property

a- All the prepared specimens (20 specimens, 10 for each control and experimental groups) stored in distilled water inside incubator at 37°C for 48 hours before being tested [23].

b- Testing procedure: Test was performed using durometer hardness tester (shore D hardness, TH210, Italy)which is suitable for acrylic material [23]. The instrument consists of a blunt pointed indenter (0.8 mm in diameter) that present in a cylinder (1.6mm in diameter) .The indenter was attached to a digital scale that is graduated from 0 to 100 unit. The usual method was to press down firmlyand quickly on the indenter, a measurements were taken directly from the digital scale reading. Five measurementswere recorded on different areas of each specimen and an average of these five readings was recorded.

4-Measuring surface roughness.

a- All the prepared specimens (20 specimens, 10 for each control and experimental groups) stored in distilled water inside incubator at 37°C for 48 hours before being tested [23].

b- Testing procedure: The Profilometer device surface roughness tester (TH 210, China) was used to test the micro geometryof the surface for experimental and control group.The device has surface analyzer (sharp stylus made from diamond) to trace the profile of the surface irregularities. It moves for maximum distance of 11 mm. The Profilometer records by its scale all the peaks and recesses which characterized the surface of the specimen under testing. The analyzer pass along the specimen surface for 11 mm distance. Three locations were selected in every specimen making 3 readings then the mean of these readings were recorded as a surface roughness value for each specimen.

5- Denture base adaptation testing

a- Microscopic measurement:The cast- denture base sets (20 specimens, 10 for each control and experimental groups)was sectionedto a horizontal line 5 mm away from theposterior end of the cast using a cutting saw device under water cooling [26,27]. Three points were marked on the cast on transverse line at the posterior border of the cast specimens (deepest point of the left vestibule, left ridge crest andmidline point which is marked according to the line bisecting the incisive papilla and extending posterior on the cast) as (A, B and C) respectively (figure 4) [26].

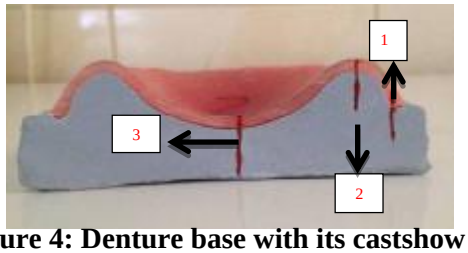


Figure 4: Denture base with its castshow position of 3 points: (1) point A, (2) point B and (3) point C

The gap between the cast and the denture base margin at these 3 points was measured with the use of digital microscope (Dino- Lite, Taiwan) of magnification 200x capability and accuracy of 0.001 mm. Two measurements were made, first one made immediately after deflasking and sectioning for all the samples. Second measurement was done after incubation in distilled water at 37°C for 14 daysfor all the samples [27], then each denture base was seated on its corresponding cast and measurement of the gab was done while a weight of 1kg was placed over the denture base to ensure a proper seating of the denture base over the cast [26].

b- To observe the overall gap formation of the denture base, All denture bases placed on theirrespective master cast for each group was scanned by computerized tomography(Light Speed, Philips, Netherland), (figure 5). The frontally-sectioned images of the denture-cast set and sagittal images obtained at the palatal midline were taken from the CT data [27].

A **B**

Figure 5: A- computerized tomography device, B- The casts with their corresponding denture bases under scanning

RESULTS

Mean values, standard deviation, t-test, p-value and Significances of mechanical properties presented in table (2).

Table 2: Descriptive and statistics of mechanical properties

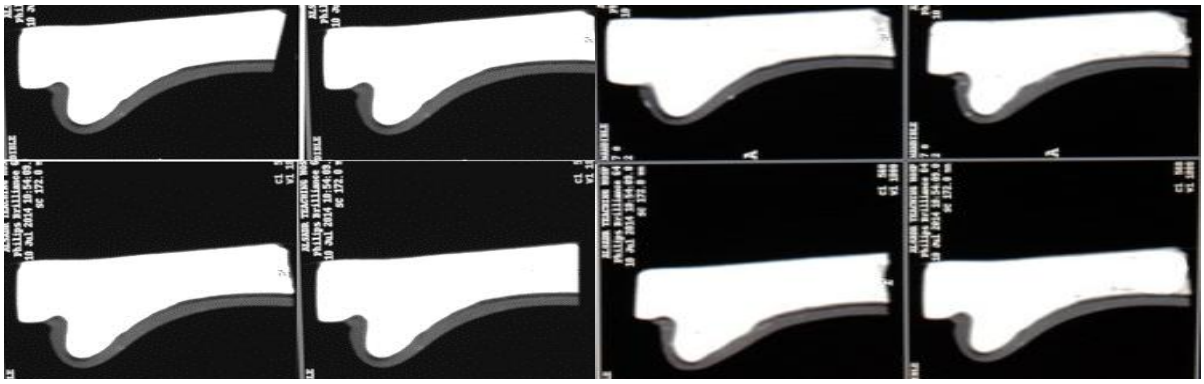
Property	Tested groups	N	Mean	S.D.	T-test	P. value	Sig.
Impact strength (Kj/m^2)	Control group	10	7.94	0.25	-16.02	0.000	HS
	Experimental group	10	9.63	0.22			
Transverse strength (N/mm^2)	Control group	10	88.50	0.77	-11.85	0.000	HS
	Experimental group	10	93.48	1.08			
Surface hardness	Control group	10	84.64	1.12	-1.316	0.205	NS
	Experimental group	10	85.35	1.27			
Surface roughness (µm)	Control group	10	1.29	0.08	-2.309	0.033	S
	Experimental group	10	1.37	0.07			

Mean values, standard deviation, t-test, p-value and Significances of the gap at three selected points (A, B and C) to measure denture base adaptation presented in table (3).

Table 2: Descriptive and statistics of the gap(mm)at three point

point	Time	Tested groups	N	Mean	S.D.	T-test	P-value	Sig.
Point A	Immediately After deflsking	Control group	10	0.122	0.018	-2.160	0.045	S
		Experimental group	10	0.160	0.054			
	After incubation 14 day	Control group	10	0.143	0.038	-2.266	0.036	S
		Experimental group	10	0.195	0.063			
Point B	Immediately After deflsking	Control group	10	0.065	0.016	-2.61	0.018	S
		Experimental group	10	0.081	0.013			
	After incubation 14 day	Control group	10	0.088	0.035	-1.64	0.111	NS
		Experimental group	10	0.113	0.036			
Point C	Immediately After deflsking	Control group	10	0.244	0.040	-0.711	0.486	NS
		Experimental group	10	0.257	0.033			
	After incubation 14 day	Control group	10	0.284	0.053	-0.524	0.607	NS
		Experimental group	10	0.301	0.087			

Evaluation of denture base adaptation made by CT images (Figure 6) was at the mid sagittal line of denture bases on the respective master cast for all tested specimen (control and experimental groups), gap formation between the tissue surface of the denture base and master cast increased from the anterior to posterior side of palate and also from the alveolar ridge to the mid palatal line. However, the gap distance or volume could not be measured from the CT images due to the low resolution.



A **B**

Figure 6: Computerized tomography images for denture-cast sets: A. control group, B. experimental group

DISCUSSION

The present study was conducted to evaluate and compare the effect of addition (ZrO_2:Al_2O_3nano-fillers mixture) to PMMA on some mechanical properties and denture base adaptation of heat cured acrylic denture base.The introduction of nanofillers into PMMA produced highly significant increase in the value of impact strengthwhen compared with control group. The increase in the impact strength could be due to thehigh interfacial shear strength between nanofiller and matrix resulted from the formation of cross-links or supra molecular bonding which cover or shield the Nano fillers which in turn prevent propagation of cracks. Also the crack propagation may be changed by good bonding between nanofiller and resin matrixresulted from interaction between the functional groups introduced by salinization process [28]. The small size and high surface area and relatively low concentration may helped in a good distribution of these fillers that may cause a restricted motion of macromolecule chains and enhance mechanical properties [29], that means the PMMA nanocomposite has mechanical stability more than neat PMMA.

Also, the transverse strength test result showed highly significant increase with nanocomposite when compared with control group.This increase in transverse strength may be explained on the basis of transformation toughening, when sufficient stress develops and crack begins to propagate, a transformation of ZrO_2 and Al_2O_3 which depletes the energy of crack propagation, also, in this process expansion of ZrO_2 and Al_2O_3 crystals occurs and places the crack under a state of compressive stress and crack propagation is arrested [25].

Increase in transverse strength also could be due to transfer of stress from more flexible polymer to the higher modulus, more rigid and stiffer filler particles [25]. The addition of nanofillers at 2wt.% to PMMA led to increase of surface hardness beyond that of pure PMMA, statistically was not-significant,this could be due to the relatively low concentration of the nanofillers used in the study, although, this improvement may be attributed to the inherent characteristics of the nanoparticles. Nanoparticles possess strong ionic interatomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength. On these bases it may be expected when nanoparticles disperse in a matrix, they increase its hardness and strength [30].

The surface roughness of modified PMMA with nanofiller was significantly increased when compared with control group.This is may be due to the differencein roughness of Nano particles and acrylic denture base matrix and also probably attributed to the difference in micro structural characteristics of the materials and the form of the particles [31].

With regard to this study, the significantly increase insurface roughness can be considered uninfluential since microorganism colonization occurs when the roughness more than 0.2µm [32].The gap between denture base and cast was measured at 3 point (A, B, C) in two time to

evaluate denture base adaptation, where it mostly depend on polymerization shrinkage and water sorption of PMMA [33,34]. So, in first measurement made immediately after deflasking showed a significant increase of gap in experimental groupwhen compared to control groupat point A and B, and non- significant increase of gap in experimental group at point C. This increase explained may be due to addition of nanoparticle lead to increase in thermal conductivity of acrylic resin [13,30], and degree of polymerization effected considerably by heat dissipation and thermal conductivity [35], lead to contraction of denture base due to further polymerization shrinkage that occur due to exposure to high temperature with reduction in the spaces between the chain of the polymer this result in agreement with Ogawaand and Hasegawa [36].

In second time after incubation 14 day showed in a significant increase of gap in experimental group when compared to control groupat point A, and non-significant increase of gap in experimental group at point B and C. This result may be due to that the addition of nanoparticles to PMMA may decreased in water sorption when compared with unmodified PMMA [13,16], So decrease expansion of acrylic denture base which considered antagonist effect to polymerization shrinkage that occur in experimental group more than control group as discussed previously [37].

The CT images of denture base-cast sets did show this tendency of gap formation in medial-lateral and anterior-posterior areas (Figure 6). These findings are also predictable with the results reported by Consani et al. [38], who compared the posterior border gap of the denture base-cast sets sectioned transversally at each area of the canine, molar and posterior ends. Moreover, the magnitude of the posterior border gap generally increased medially along the palatal vault reaching a maximum at the midline of the palate [39,40].

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الخلاصة

خلفية:مادة قاعدة الطقم البوليمثيلميثاكريليت(الراتنجالأكريلك)البوليمر ذات خواص ميكانيكية منخفضة، وتكيف قاعدة الطقم إلى الأنسجة الكامنة مهم لاستبقاء واستقرار قاعدة الطقم. وكان الهدف من هذه الدراسة هو تقييم تأثير خليط الحبيبات النانوية لأوكسيد الالمنيوم وأوكسيد الزركونيوم على قوة الصدمة والقوة العرضية، صلابة و خشونة السطح، وتكيف قاعدة الطقم لمادة قاعده طقم الراتنجالأكريلك الحراري.

المواد والطرق: أعدت مائةعينة، تم تقسيم هذه العينات إلى خمسمجموعات (عشرونعينة لكل مجموعة) وفقا لنوع الاختبار، ومن ثم تم تقسيم كل مجموعة إلى مجموعتينفرعيتينهي(السيطرة والتجريبية)كل مجموعة فرعية تتكون من عشرة عينات، وتضمنت المجموعة التجريبية خليط من 2٪ وزنا ل(أوكسيدالزركونيوم وأوكسيدالالمنيوم)من مسحوق البوليمثيلميثاكريليت، وبنسبة(ZrO_2:Al_2O_3 2:1). تم قياس قوة الصدمة بواسطة آلة اختبار الصدمة(Charpy) ، تم قياس القوة العرضية بواسطة آلة اختبار انسترون(INSTRON) بينما تم قياس صلابة السطح بواسطة مقياس (ShoreDdurometer) , وتم قياس خشونة السطح من قبل Profilometer. تم قياس تكيف قاعدة الطقم بواسطة المجهر الرقمي وتقييمها من قبل جهاز التصوير المقطعي المحوسب (CT).

النتائج: زيادة كبيرة للغاية في قوة الصدمة والقوة العرضية، وزيادة غير كبيرة في الصلابة، وزيادة كبيرة في الخشونة ونقصانفيتكيف قاعدة الطقم الذي تم قياسه عند ثلاث(3)نقاط هي(A، B و C) حصلت في المجموعة التجريبية مقارنة بالمجموعة السيطرة. أما تقييم جهاز التصوير المقطعي المحوسب ، الفجوة بين قاعدة الطقموالقالب الرئيسي لكلا المجموعتين(السيطرة والتجريبية) يظهر فيه زيادة من الجانب الأمامي إلى الخلفي من سقف الحلق ومن قمة عظم الفك إلى خط منتصف الحنك.

الاستنتاج:مادة النانو المركبة على أساس البوليمرلديها خواص ميكانيكية أعلى من الراتنجالأكريلك البوليمر الحراري النقي وفي نفس الوقت نقصان في تكيف قاعدة الطقم.

الكلمات الرئيسية: قاعدة طقم الاكريليك ، حشوة النانو، الخواص الميكانيكية، وتكيف قاعدة الطقم.