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EFFECT OF ZIRCONIA NANOPARTICLES INCORPORATION ON SOME PROPERTIES OF ONE HIGH - IMPACT HEAT CURED PMMA RESIN

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ABSTRACT

PURPOSE: To evaluate the effect of ZrO_2 nanoparticles incorporation (in two different ratios 1% and 3%) on some properties of one high-impact heat cured PMMA resin material when it was processed by two different methods (Conventional and Microwave curing methods)**.**

Materials & Methods:

One high-impact heat cured PMMA resin (Trevalon Hi) was used in this study to be modified with nano-ZrO_2 in two different ratios (1% and 3%). Both unmodified and modified high-impact heat cured PMMA resin were subjected to three different tests (Flexural strength test, Microhardness test, and Color stability test). A total of ninety specimens were prepared, thirty specimens for each test (N=30). Within each test specimens, there were three groups (n=10), **(HI)** group: was prepared from high-impact heat cured PMMA resin without modification by nano-ZrO_2 (as a control group). **(1%n-ZrO_2 HI)** group: was prepared from high-impact heat cured PMMA resin modified by **1**% nano- ZrO_2. **(3%n-ZrO_2 HI)** group: was prepared from high-impact heat cured PMMA resin modified by **3**% nano- ZrO_2.Within each group, five specimens were prepared by conventional water-bath curing method, while the other five specimens were prepared by microwave curing. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

Results: Modification of high-impact heat cured PMMA resin with (1% and 3%) nano-ZrO_2 resulted in significant increasing in the mean values of flexural strength and VHN of all tested groups ($p<0.05$), while the effect on the color stability of the resin was slight.

Conclusion: Within the limitation of this study, modification of high-impact heat cured PMMA resin with zirconia nanoparticles up to 3% could improve the flexural strength and surface microhardness with slight effect on the color. Microwave curing of high-impact heat cured PMMA resin gives better results than conventional one.

Keywords: Zirconia Nanoparticles, High-impact heat cured PMMA resin**,** Microwave Curing, Flexural strength, Microhardness, and Color stability**.**

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INTRODUCTION

Polymethyl methacrylate acrylic (PMMA) resins have been widely used for construction of denture base since 1940s. As they have many advantages such as, simple laboratory procedures, easy to repair, good fit, sufficient stability in oral condition, accurate reproduction of tissue details, lightness in weight, low cost, lack of toxicity, and patient satisfaction in relation to color matching and good aesthetic.[1-3] Unfortunately, these resins have inadequate mechanical properties which may lead to premature fracture of the dentures during service. After few years of PMMA denture use, it may deteriorate under normal masticatory forces due to fatigue, continuous flexing, sudden fall, or uneven stresses associated with malocclusion or poor fit. [3,4]

Although, the reinforcement of PMMA resin with other materials, such as fibers or fillers (macro- and micro-fillers) is a common method to improve the physical and mechanical properties of the PMMA denture base resins and overcome their drawbacks.[5] But, recent studies have concentrated on evaluating the effect of inorganic nanoparticles incorporation instead of micro and macro ones. Properties of such polymer-nanocomposite mixture depend on shape, size, concentration as well as the interaction of these nanoparticles with the polymeric matrix.[6] Recently and with invasion of nanotechnology to prosthodontic and material fields, zirconium oxide nanoparticles (nano-ZrO_2) were widely used to strengthen the dental polymers.[7]

Nanoparticles of zirconium oxide have attracted attention due to several beneficial properties such as, biocompatibility, high strength properties, good wear and abrasion resistance, and having the highest hardness values among any other metal oxide nanoparticles. Beside this, the white color of ZrO_2 nanoparticles make them less likely to change the appearance of the reinforced dental material in comparison to other metallic oxide nanoparticles.[7-9]

High impact heat-cured PMMA resin material was developed mainly to get rid the low impact strength problem of conventional resin, that may lead to premature fracture of the denture on accidental fall.[10,11] Microwave curing method of PMMA resin is considered to be fast and clean method to process denture base material in few minutes and with better results.[12]

So, this study was conducted to evaluate the effect of ZrO_2 nanoparticles addition to one high impact heat-cured PMMA resin material in two different ratios (1% and 3%), on processing by two different methods (Conventional and Microwave curing methods). The null hypothesis was that, neither the addition of ZrO_2 nanoparticles to the resin, nor the processing method affect the tested properties.

MATERIALS AND METHODS

One high-impact heat cured PMMA resin (Trevalon Hi Dentsply G-7, Saket, New Delhi-110017) was used in this study to be modified with nano-ZrO_2 in two different ratios (1% and 3%). Both unmodified and modified high-impact heat cured PMMA resin were subjected to three different tests (Flexural strength test, Microhardness test, and Color stability test). A total of ninety specimens were prepared, thirty specimens for each test (N=30). Within each test specimens, there were three groups (n=10), **(HI)** group: was prepared from high-impact heat cured PMMA resin without modification by nano-ZrO_2 (as a control group), **(1%n-ZrO_2 HI)** group: was prepared from high-impact heat cured PMMA resin modified by **1**% nano- ZrO_2, while **(3%n-ZrO_2 HI)** group: was prepared from high-impact heat cured PMMA resin modified by **3**% nano- ZrO_2. Within each group, five specimens were prepared by conventional water-bath curing method, while the other five specimens were prepared by microwave curing.

Preparation of modified high-impact heat cured acrylic powder:

In this study, powder of high-impact heat cured PMMA resin was modified by nano-ZrO_2 powder of 90 nm particle size (99.9% purity, 1314-23-4; Sigma-Aldrich Co., St Louis, MO, USA). Silane coupling agent TMSPM (Sigma-Aldrich Co., St Louis, MO, USA) was added to nano-ZrO_2 particles to create reactive groups on their surfaces which allow for better adhesion between these nanoparticles and acrylic resin matrix.[13] For this purpose, 100 mL of acetone was used to dissolve 0.3 g of TMSPM, then 30 g of ZrO_2 nano-particles were added to the TMSPM/acetone solution and stirred for 60 min, using a magnetic stirrer (Cimarec Digital Stirring Hotplates, SP131320-33Q; Thermo Fischer Scientific, Waltham, MA, USA). Then, acetone solvent was completely removed using a rotary evaporator (Rotavapor R-200, Buchi Inc., Postfach, Switzerland) under vacuum at 60°C and 150 rpm for 30 min. When the sample was dried, it was heated for 2h at 120°C and allowed to natural cooling to obtain properly surface-treated nano-ZrO_2.[3,14-16]

Weighing of silanized nano-ZrO_2 powder was done using an electronic balance (S-234; Denver Instrument, Gottingen, Germany). Nano-ZrO_2 powder was added to pre-weighted high-impact heat cured PMMA resin powder in two different ratios (**1**% and **3**%) according to the studying group. Thorough mixing and meticulously stirring of nano-ZrO_2 oxide powder with the high-impact heat cured PMMA powder for about 30 min to achieve an even distribution of particles and to obtain a uniform, undisrupted color. Then, the monomer was added to the mixed powder in correct polymer/monomer ratio as recommended by manufacture.

Preparation of different test specimens:

In this study, ninety specimens were prepared (thirty specimens for each individual test). The same steps were followed to prepare all test specimens firstly, wax patterns (CavexSet UpWax, Cavex, Netherlands) were prepared using specially designed cupper mold with accurate dimensions according to the test type. Secondly, the wax patterns were invested within type III dental stone (GC Fujirock EP, Belgium) in suitable flask according to the curing method. In the case of water-bath curing, conventional brass metal flask (61B Two Flask Compress, Handler Manufacturing, USA) was used, while for microwave curing, a special type of fiber reinforced plastic flask (FRP Flask, G C America) was used. Mixing and pouring of dental stone inside the suitable flask were carried out according to manufacturer's instructions. After complete setting of stone, boiling water was used to remove wax and create clean mold spaces. The mold was allowed to dry in open air, and then a separating media (Isol Major; Major Prodotti Dentari Spa) was applied and allowed to dry. Finally, high-impact heat cured PMMA resin whether unmodified or modified by nano-ZrO_2 was mixed and packed in the flask according to the manufacturer's instructions. Trial closure was done to avoid under packing, after the final closure, the flask was kept under bench press for about 30 min before curing.

For conventional water-bath curing, flask was placed in water bath and processed for 8h at 74°C, and then the temperature was increased to 100°C for 1h in a thermal curing unit (Memmert, Germany).[7] After complete curing, the metal flask was allowed to cool at room temperature for 45 minutes before deflasking and removing of the specimens.[2] On the other hand, microwave curing was done by placing the FRP flask in the microwave (Samsung TDS, Korea) where the specimens were processed by heating for 3 minutes at (500W). The microwave-cured specimens were deflasked after overnight bench cooling.[2,14]

Excess resin material was carefully trimmed with a tungsten carbide bur (HM251FX-040-HP; Meisinger, Centennial, CO, USA). Specimens were finished and polished according to manufacturer's

instructions under running water to minimize heat generation and avoid distortion of specimens. All specimens were stored in distilled water at 37°C for 48h before subjecting to testing procedures.

1- Flexural Strength Test:

The flexural strength test specimens were rectangular in shape (65 length × 10 width × 2.5 thickness mm^3) in accordance with ANSI/ADA specification no 12[17]. Thirty specimens were prepared for flexural strength test, ten specimens were prepared from high-impact heat cured PMMA resin without modification by nano-ZrO_2 (as a control group), ten specimens were prepared from high-impact heat cured PMMA resin modified by **1**% nano- ZrO_2, while the last ten specimens were prepared from high-impact heat cured PMMA resin modified by **3**% nano-ZrO_2. In each group, five specimens were prepared by conventional water-bath curing method, while the other five specimens were prepared by microwave curing.

To determine the flexural strength, universal testing machine (Instron Model 3365; Tensile Tester 5 KN. USA) was used to fracture the specimens using a three-point bending test (3PB). The specimen was placed on two supports that were 50 mm apart and then, the load cell was adjusted at 50 kg to apply load at the midpoint of the specimen with a crosshead speed of 5 mm/min until the specimen fracture. The fracture load was determined and used in calculating the flexural strength from the following equation:

$$S = 3WL/2bd^2$$

where **S** is the flexural strength (MPa), **W** is the fracture load (N), **L** is the distance between the two supports (50 mm), **b** is the specimen width, and **d** is the specimen thickness.[3,18]

2- Surface microhardness test:

The specimens for microhardness test are rectangular in shap (30 length x 10 width x 2.5 thickness mm^3).[8] When these specimens were divested from the stone, they were trimmed by tungsten carbide bur to remove any excess resin material. Polishing of specimens were achieved by polishing carbide papers in a progressive increasing grit of 200, 500, 600, 800 and 1200 (Noroton, Indu´stria e Come´rcio Limitada, Sa˜o Paulo, SP, Brazil). The specimens were finally polished by a felt wheel and alumina paste to obtain a smooth, flat, and scratch-free surfaces.

There was a total of thirty specimens for microhardness test, they were divided in the same groups as mentioned before in flexural strength test. To measure the surface microhardness, Vickers diamond hardness tester was used (Microhardness Tester ZWICK/ROELL 2125 Barrett Park Drive, Suite107 30144 Kennesaw, GA USA). The applied load was adjusted at 50 g, and indentation time was set for 5 seconds.[8] A total of five indentations were made on the surface of each specimen at different points, with about 100 μm distance from each other and the mean values of individual specimens were obtained digitally and analyzed by one-way ANOVA and Tukey test ($p < 0.05$).

3- Color Stability Test:

Specimens for color stability test are rectangular in shape with dimensions of (35 length × 15 width × 0.5 thickness mm^3) according to ANSI/ADA no 12.[17] Portable Reflective spectrophotometer (X-Rite, model RM200QC, Neu-Isenburg, Germany) was used in this study to measure the color of specimens. Colorimetry was done to determine the degree of change in the color of one high-impact heat cured PMMA resin, processed by two different methods (Conventional and Microwave), in the case of modification by nano-ZrO_2 particles in two different ratios (**1**% & **3**%).

Before measurement, spectrophotometer was calibrated according to the manufacturer's instructions using the white calibration plate supplied by the manufacturer. The color difference (ΔE) was evaluated using the Commission Internationale de l'Eclairage (CIE L*a*b*) colorimetric system.[19,20]

This system is based on three parameters for color definition L*, a*, and b *. Where L* represents lightness-darkness; thence, the greater the L*, the lighter the specimen. The a* represents the chroma along the red-green axis, positive a* relates to the amount of redness, and negative a* relates to the amount of green of the specimen. While, the b* on the chromatic scale measure of the chroma along the yellow-blue axis, where positive b* relates to the amount of yellowness, and negative b* relates to the amount of blue of the specimen.

The color changes (ΔE) of the specimens were evaluated using the following formula:

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{½}$$

Delta L*, Δa*, and Δb* represent the differences measured in L*, a*, and b* values of high-impact heat cured PMMA resin (processed by two different curing methods) in the case of modification by nano-ZrO_2 particles in two different ratios (**1**% & **3**%). In order to quantify the levels of color change (ΔE), National Bureau of Standards (NBS) with the NBS units of color difference were calculated. NBS units are expressed by the following formula.[21]

$$\text{NBS unit} = \Delta E \times 0.92$$

The critical marks of such color difference according to the NBS were listed in (Table.4).

Statistical Analysis

The mean and standard deviation values were calculated for each group in each test. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests, data showed parametric (normal) distribution. Independent sample t-test was used to compare between two groups in non-related samples. One-way ANOVA followed by Tukey post hoc test was used to compare between more than two groups in non-related samples. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

RESULTS

As regard to flexure strength test (Table. 1), addition of 1% nano-ZrO_2 to the high-impact heat cure PMMA resin, was associated with significant increasing in flexural strength mean values (126.52 ±1.16 and 131.70 ±0.60) on processing by conventional and microwave methods respectively. While, addition of 3% ZrO_2 nanoparticles to the same resin, resulted in more significant increasing in flexural strength (141.76±1.24 for conventional method and 149.22±0.61 for microwave method) than adding only1% nano-ZrO_2. Within each individual group, the specimens which were processed by microwave method had a significantly higher flexural strength mean values than conventionally processed ones.

TABLE (1): The mean, standard deviation (SD) values of flexure strength in MPa of different groups.

Variables	Flexure strength MPa				
	Conventional water -bath		Microwave processing		p-value
	Mean	SD	Mean	SD	
HI	117.34 ^{cB}	0.54	121.50 ^{cA}	0.81	**<0.001***
1% n-ZrO_2 HI	126.5$^{2\ bB}$	1.16	131.70 ^{bA}	0.60	**<0.001***
3% n-ZrO_2 HI	141.76 ^{aB}	1.24	149.2$^{2\ aA}$	0.61	**<0.001***
	<0.001*		**<0.001***		

Means with different small letters in the same column indicate statistically significance difference, means with different capital letters in the same row indicate statistically significance difference. *; significant (p<0.05) ns; non-significant (p>0.05)

Results of surface microhardness (Table. 2) showed that, on processing of the specimens by conventional method, the mean values of Vickers hardness number (VHN) were significantly increased on modification by 1% and 3% nano-ZrO_2 (16.70 ±0.56 and 17.76± 0.24) respectively. The same was noticed in the specimens which were processed by microwave method, as the mean values of (VHN) were significantly increased on modification of the resin powder with 1% and 3% nano-ZrO_2 (17.50 ±0.39 and 18.38± 0.29) respectively. Within each group, processing of the specimens by microwave method resulted in higher (VHN) than processing by conventional method.

TABLE (2): The mean, standard deviation (SD) values of (VHN) surface microhardness of different groups.

Variables	Surface microhardness (VHN)				
	Conventional water -bath		Microwave processing		p-value
	Mean	SD	Mean	SD	
HI	13.08 ^{cA}	0.37	13.42 ^{cA}	0.41	**0.209ns**
1% n-ZrO_2 HI	16.70 ^{bB}	0.56	17.50 ^{bA}	0.39	**0.030***
3% n-ZrO_2 HI	17.7$^{6\ a}$B	0.24	18.38 ^{aA}	0.29	**0.006***
	<0.001*		**<0.001***		

Means with different small letters in the same column indicate statistically significance difference, means with different capital letters in the same row indicate statistically significance difference. *; significant (p<0.05) ns; non-significant (p>0.05)

Although the results of color stability test showed that, there were color differences between specimens which were prepared from unmodified high-impact heat cure PMMA resin powder and specimens which were prepared from modified one. But the mean values of ΔE (Table.3) on modifications of such resin powder with both 1% and 3% nano-ZrO_2 (whether it was processed by conventional or microwave method) were varied from 0.74 ±0.08 to 1.39 ±0.04, these values of ΔE according to the critical marks of color difference by NBS unites (Table.4) mean that, there are only a slight changes in color of the evaluated resin by such modifications.

TABLE (3): The mean, standard deviation (SD) values of (ΔE) color stability of different groups.

Variables	Color stability				
	Conventional water -bath		Microwave processing		p-value
	Mean	SD	Mean	SD	
1% n-ZrO_2 HI	0.7$^{4\ b}$A	0.08	0.76 ^{bA}	0.04	**0.615ns**
3% **n-ZrO_2 HI**	1.25 ^{aB}	0.12	1.39 ^{aA}	0.04	**0.034***
	<0.001*		**<0.001***		

Means with different small letters in the same column indicate statistically significance difference, means with different capital letters in the same row indicate statistically significance difference. *; significant (p<0.05) ns; non-significant (p>0.05)

TABLE (4): Critical marks of color difference according to the NBS:

Critical marks of color difference	Textile terms (NBS units)
Trace	0.0-0.05
Slight	0.5-1.5
Noticeable	1.5-3.0
Appreciable	3.0-6.0
Much	6.0-12.0
Very much	>12.0

DISCUSSION

High-impact heat cure acrylic resin is a graft copolymer which is produced by copolymerization of PMMA resin with rubber.[22] Although such modification was able to increase the impact strength of the resins, but unfortunately it decreases their transverse strength.[8,23] Many studies have found that, most of the fractured dentures within the first few years after insertion, were in the form of denture base breakage as a result of poor resistance to stresses caused by flexural and impact forces.[24]

Today and with great advancement in nanotechnology, nano-fillers became widely used for reinforcement of the denture base materials and improving their properties. In this study, ZrO_2 nanoparticles were selected as a filler to modify the properties of one high-impact heat cured PMMA resin, as they have several beneficial properties as mentioned before.[7-9] Silane coupling agent was used to treat the surface of ZrO_2 nanoparticles before their incorporation in resin matrix, to overcome their aggregation tendency problem and to improve the compatibility with the polymeric matrix.[7,8]

Many studies showed that, use of nanoparticles to modify the acrylic resins in a percentage up to 3%, were able to enhance some strength properties such as impact strength, flexural strength, and hardness.[25] While, surpassing this percentage could deteriorate or adversely affect the resin's properties.[26,27] So, in this study two different ratios of zirconia nanoparticles (%1 and 3%) were selected to modify one high-impact heat cure acrylic resin. Further comparison was made between conventional water-bath curing method that lasts about 9 hours and curing in microwave energy which requires only 3-4 minutes, because microwave curing of PMMA resins became an alternative technique to conventional one with fast, clean, more accurate, and better results.[12,28,29]

It is highly recommended to determine the effect of any additives on the flexural strength of acrylic materials to avoid any reduction in such strength below the standard level, the stander minimal value of flexural strength for any polymerized materials should not be less than 50 MPa.[30] In this study, flexural strength of tested high-impact heat cured PMMA resin varied from (117.34 to149.22 MPa), this average agrees with Kareem and Moudhaffer,[31] who found that the flexural strength of heat cured PMMA was varied from 111.58 MPa to 135.20 MPa before and after modification with nanoparticles respectively, and is close to Arora et al,[22] who found that the flexural strength of the same studied high-impact PMMA resin varied from (100.66 to 128.394 MPa). But it disagrees with Deepan et al,[24] as they found that the flexural strength of such high-impact PMMA resin varied from (82.8372 to 89.4968 MPa). In this study, microwave cured specimens in all tested groups showed higher flexural strength in comparison to conventionally cured ones. This agrees with Specification of American Dental Association (ADA) which indicated that, curing of acrylic resin with microwave energy produces dentures with higher strength properties and greater resistant to mechanical loading than conventionally cured material.[28] But, disagree with Aziz[2] who found that, microwave processed PMMA resin had lower strength properties than one which was cured in conventional water -bath.

It was noticed that, both added ratios of zirconia nanoparticles (1% & 3%) increased the flexural strength of the evaluated resin in a significant manner, whether the specimens were processed in conventional water-bath or microwave energy. This increasing in the transverse strength could be explained by transformation toughening mechanism of ZrO_2. When there is sufficient stress and cracks begin to propagate, a transformation of ZrO_2 from metastable tetragonal crystal phase to stable monoclinic phase takes place, which consumes the energy of crack propagation and so stop it. This transformation process is also accompanied by expansion of ZrO_2 crystals which places the crack under a state of compression instead of tension and so the crack propagation is arrested.[32] Beside this, zirconia nanoparticles which were used in this study were very small (about 90 nm) and this led to huge increase in the surface area, wide area was very successful in dissipation of energy and minimizing the chance of cracks propagation.[2]

Hardness of denture base material is an important property to be determined, as it indicates the degree of polymeric matrix resistance to degradation, and subsequently the longevity of the denture inside the oral cavity. High hardness of denture material minimizes the chance of denture scratch, which can compromise the strength of the denture and may lead to premature fractur during function. Surface scratches can also compromise the surface roughness and favoring plaque and pigments accumulation, eventually the aesthetic and appearance of the denture were deteriorated.[33]

This study showed that, the hardness of the high-impact acrylic resin was significantly increased on modification by 1% or 3% ZrO_2 nanoparticles. But, incorporation of 3% ZrO_2 nanoparticles had more significant effect than 1%, whether the specimens were processed by conventional or microwave method. This could be explained by the fact that ZrO_2 nanoparticles have the highest hardness values among any other metal oxide nanoparticles.[8]Another factor that can contribute to increase the hardness of such evaluated composite material is the good distribution of nanoparticles in the resin matrix and filling the free spaces between polymeric chains.[31] The mean values of VHN of the specimens which were modified by 1% and 3% nanoparticles were varied from 16.70 to 18.38 respectively, and this agrees with Ahmed and Ebrahim[34] who found that, VHN of heat cured PMMA was varied from 17.35 to 19.10 on modification with ZrO_2 nanoparticles in a ratio of 1.5% and 3% respectively.

Microwave cured specimens in all tested groups had higher mean values of Vickers Hardness Number than specimens which were processed by conventional water-bath method. This could be explained by homogeneous heating of the acrylic resin during microwave curing, instead of a centripetal heating during conventional curing.[35] Also, Ilbay et al[36] proved that, the acrylic resin material which was cured by microwave method could present lower incidence of porosity and so it had higher hardness values. According to Blagojevic and Murphy,[37] microwave energy irradiation of denture base materials, improves their mechanical properties as a result of decreasing in the amount of residual monomer and increasing in the degree of monomer conversion.

As regard to color stability, it was found that both added ratios of zirconia nanoparticles had a slight effect (according to NBS unites) on the specimens' color whether they were processed by conventional or microwave method. This can be explained by the white color of zirconia nanoparticles which are not expected to adversely affect or compromise the esthetic appearance of denture base material especially if they were used in such small percentages. This slight change in color is in agreement with Ihab et al,[38] who found that, there was an increase in the amount of light absorption with increasing in the concentration of nano-ZrO_2 within heat polymerized acrylic resin. According to Aziz,[2] the percentage of nanoparticles within acrylic resin

matrix shouldn't exceed 5%, otherwise this will lead to massive changes in the color.

Although the results of this study showed that, microwave cured specimens had more color changes in relation to conventionally cured ones, but these changes still slight according to (NBS unites). This disagrees with Aziz[2] who found that, there was no change in the color of acrylic resin by microwave curing, and also disagrees with Assunçao et al,[39] who found that the color of acrylic denture teeth was not affected by the polymerization methods, and the difference between conventional and microwave curing was non-significant.

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