ORIGINAL RESEARCH

Enhancement of Mechanical Properties of Polymethylmethacrylate Denture Base Material by Zirconium Oxide Nanofiller

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Abstract

Aim: The aim of this work was to study the effect of adding zirconium oxide (ZrO_2) nanofiller at different concentrations (0.5 wt%, 1 wt%, and 3 wt%) on the impact strength (IS) and fracture toughness (FT) of heat-polymerized polymethylmethacrylate (PMMA).

Materials and methods: Zirconium oxide nanofiller (ZrO_2) was added at different concentrations (0.5 wt%, 1 wt%, and 3 wt%) to PMMA. Total of eighty specimens was prepared for both tests, 40 for an individual test. For the individual test, four groups were formed (10 specimens each). There were three experimental groups with ZrO_2 powders at different concentrations (0.5%, 1%, and 3%) by weight and one control group without any addition. For measuring IS, Charpy's impact test was used, and the universal testing machine was used for FT. The values obtained for IS and FT were tabulated and statistically analyzed. Analysis of variance (ANOVA) and Tukey's test were utilized to analyze the level of significance in between the obtained means of the various groups, where the level of significance was set at a p value of ≤0.05.

Results: Reinforcement of PMMA with ZrO_2 nanofiller significantly increased the IS and FT values between study groups ($p = 0.000$). Highest values obtained for IS and FT by adding 3 wt% nanofiller were 3.89 kJ/m^2 and 2.76 MPa.m$^{1/2}$, respectively in comparison to control values of 1.53 kJ/m^2 and 1.23 MPa.m$^{1/2}$.

Conclusion: It was found that addition of ZrO_2 at different concentrations improved the values for IS and FT of PMMA in comparison to the control group. The maximum values were obtained with 3 wt% of ZrO_2 nanofiller.

Clinical significance: PMMA resin with improved properties obtained by addition of ZrO_2 nanofillers can serve in prosthodontic applications.

Keywords: Denture base, Fracture toughness, Impact strength, Nanofiller, Polymethylmethacrylate, Resin, Zirconium oxide.

Introduction

The main goal of using removable dentures is to replace missing teeth and surrounding structures in oro-dental applications. PMMA is suffering from low flexural strength, impact strength and fatigue resistance,[1,2] still it is the material of choice for fabrication of complete denture bases. This is due to the several advantages such as biocompatibility, color matching ability, ease of use, low cost and ease of finishing and polishing.[3]

The major problem of acrylic resin is that it is low in fracture toughness. Denture bases are exposed to two kinds of stresses. Intraorally repeated force by mastication that causes fatigue failure. Extraorally, dropping the prosthesis leads to impact failure.[4]

Seventy percent of dentures fracture within 3 years after fabrication. This is commonly caused by heavy biting and masticatory forces that lead to deformation, or it can be accidental damage; any factor that changes the distribution of stresses can cause a fracture. Most of the upper dentures fracture due to combined fatigue and impact failure. For lower dentures, fractures are mostly (80%) due to an impact. Twenty-nine percent of the fractures are in mid-line, and 33% fractures occur due to debonded/detached teeth.[2] Fractures are more common in upper dentures compared to the lower dentures.[5]

The PMMA is weak in terms of mechanical properties, therefore, researchers are always in search of newer modified material with improved properties.[6]

Change of the composition of acrylic base materials for improvement in strength has been achieved by addition of a cross-linking agent or rubber group,[4] frame of metal,[7] oxides of metal,[8] or various kinds of fibers.[9] Despite the various researches conducted to enhance fracture resistance of acrylic resin, only some approaches gave positive results.[10,11] The strengthening of denture-base materials with metal-composite systems has been one of the approaches of prime interest.[11]

In need to have a denture resin material with improved strength, a ceramic filler like, ZrO_2 can be incorporated in acrylic powder of PMMA.[12] It is a biocompatible material that possesses high-fracture resistance and is being added to acrylic resin and other dental polymers, composites and ceramic materials to improve the fracture toughness.[13,14]

It is suggested to add zirconia to heat cure acrylic resin to increase mechanical properties of the material; impact strength, flexural strength,[15] compressive strength, fatigue strength, as well as its fracture toughness and hardness.[16,17] It may also play a role in antifungal effect.[18]

In the present study, the addition of ZrO_2 nanoparticles in different concentrations to PMMA was explored, for enhancement of values of IS and FT of PMMA resin. The null hypothesis was that

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the added ZrO_2 nanofiller has no effect on the impact strength and fracture toughness of PMMA.

Materials and Methods

The present study was conducted in the Faculty of Dentistry, Taif University, Al Huwaya, Taif, Kingdom of Saudi Arabia. Eighty specimens were prepared in total from heat-polymerized acrylic resin powder Regular Vertex (Vertex-dental B.V., Joh.v. oldenbarneveltlaan 62, 3705 HJ Zesit, The Netherlands; Lot no.: XW341P03) and liquid Regular Vertex (Vertex- dental B.V., Joh.v. oldenbarneveltlaan 62, 3705 HJ Zesit, The Netherlands; Lot no.: XW464L0). Forty specimens were prepared for each type of test—IS and FT.

Group A was for IS testing and group B for FT testing. Both groups A and B were further divided into four subgroups; GA1–GA4 and GB1–GB4, respectively. Groups GA1 and GB1 were the control groups (PMMA specimens without any additives). Groups GA2–GA4 and GB2–GB4 were the experimental groups (containing ZrO_2 nanofiller). Table 1 shows the various study groups with coding and specifications.

The average particle dimension for zirconium oxide nanofiller used was ≤100 nm (Sigma-Aldrich, St. Louis, MO, USA, Trade MKBV9830V). ZrO_2 nanofiller was incorporated in heat cure acrylic resin (PMMA) at different concentrations (0.5%, 1%, and 3%) by weight after modification of nanofiller by a silane coupling agent PlusOne™ Bind Silane (GE Healthcare- Life Sciences, Netherlands; Lot no.: L544448214620). The coupling agent (3-trimethoxysilylprpoyl methacrylate; TMSPM) aided to form reactive groups by coating the filler.[19] The powder/liquid ratio of acrylic resin was set at 2.5:1 by weight according to manufacturer's instructions, powder and liquid were mixed, packed and pressed at appropriate stage in the mold of each test used and then processed in the water bath at 78°C for 90 minutes according to manufacturer's instructions.

Table 1: Specimen groups with coding and specifications

Groups	*Specimens code*	*Description*
Group A	GA1 (Control group)	PMMA matrix without filler
	GA2	PMMA with 0.5wt% ZrO_2
	GA3	PMMA with 1wt% ZrO_2
	GA4	PMMA with 3wt% ZrO_2
Group B	GB1 (Control group)	PMMA matrix without filler
	GB2	PMMA with 0.5wt% ZrO_2
	GB3	PMMA with 1wt% ZrO_2
	GB4	PMMA with 3wt% ZrO_2

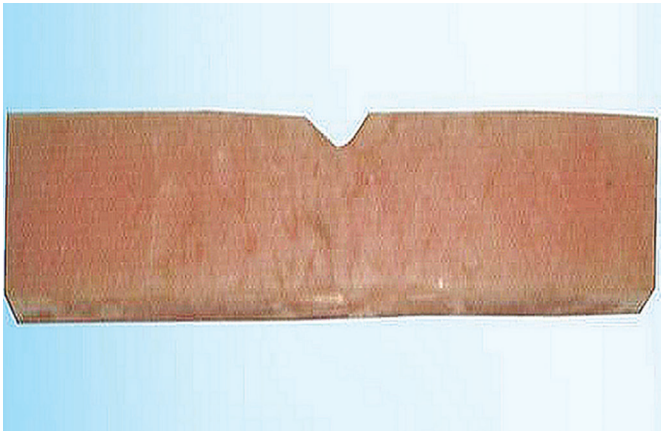

Fig 1: Specimen for impact strength testing

Impact strength (IS) Testing

The dimensions of the specimens within a flask were 80 mm × 10 mm × 4 mm, and width was 9.75 mm beneath the notch as per the requirement of the International Standards Organization (ISO) 179-A1:2005 (Fig. 1). The V-notch had a length of 0.25 mm ± 0.05 mm radius, angle notch sensitivity of 45° ±1° and the span support was 62 ± 0.5 mm.

The impact strength test (IS) was carried out by Charpy-type impact tester (Hounsfield plastic impact machine, Tensometer Ltd.) (Fig. 2). The prepared specimens were kept for 24 hours at 37°C in humidifier before IS testing. IS was calculated as loss of momentum in pendulum when the notched specimen was broken. To obtain real value, air force because of pendulum friction against air (–0.1 J) was subtracted. Prepared specimens were held in a horizontal position as a supported beam. Specimens were struck by the pendulum on the same plane of the notch but the opposite side in the middle. The specimens were tested, and the IS was calculated using equation 1:[2]

$$IS = \frac{E}{b_n d} \times 10^3$$

where, E is the energy absorbed by tested specimen on being impacted, b_n is the width of the specimen (mm) and d is the thickness of specimen (mm).

Fracture toughness (FT) Testing

Specimens were fabricated in accordance with the ISO Specification No. 13586:2000. In the flask, specimens for FT measured 100 mm length × 20 mm width × 4 mm thickness with 4 mm of notch length (Fig. 3). Support span length was 64 mm. A razor blade was tapped at the notch on the specimen to produce a crack.

The FT was determined using single edge span notch bending test (Fig. 4). Lloyd universal testing machine (model LRX plus II, Fareham, England) (Fig. 5) was used with a load cell of 5 kN and a crosshead speed of 0.5mm/minute. The prepared specimens were kept for 24 hours at 37°C in humidifier before testing. Specimens were loaded until fracture. The FT was determined in $MPa.m^{1/2}$ using the equation 2:[18]

$$FT = pc/\ bw^{1/2} \times F\ (a/w)$$

Where pc was maximum load (KN) before the crack advancement, b was the thickness of specimen (cm), w was the width of the specimen (cm), a was the length of crack (cm). F was calculated using the equation (3):[18]

$$F = \frac{(2 + a/w)\ (0.886 + a/w - 13.32\ a^2/w^2 + a^3/w^3 - 5.6\ a^4/w^4)}{(1-a/w)^{3/2}}$$

Fig 2: Charpy-type impact tester

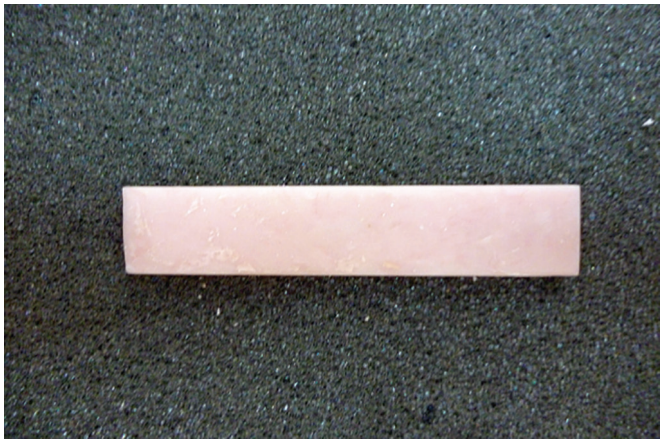

Fig 3: Specimen for fracture toughness testing

Fig 5: Universal testing machine

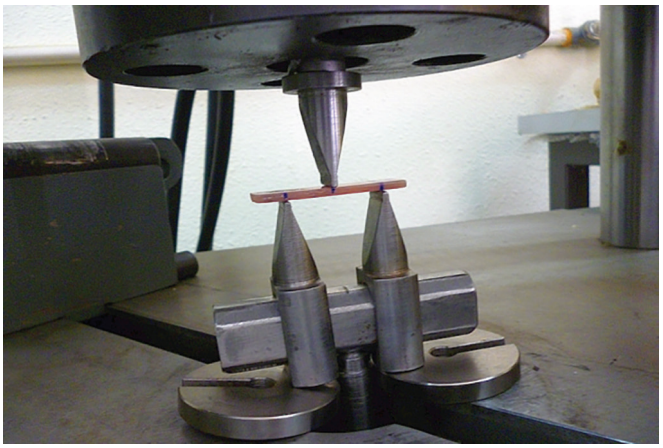

Fig 4: Fracture toughness testing on the universal testing machine

Table 2: Statistical analysis for impact strength (kJ/m^2) of control and experimental groups.

Group	*Number of specimens*	*Mean ± SD*	*p value*
GA1 (Control group)	10	$1.53^d \pm 0.76$	0.000*
GA2 (0.5 wt% ZrO_2)	10	$1.95^c \pm 0.63$	
GA3 (1 wt% ZrO_2)	10	$2.93^b \pm 0.56$	
GA4 (3 wt% ZrO_2)	10	$3.89^a \pm 1.05$	

*Significant at $p \leq 0.05$; Ranks a, b, c and d for Turkey's test refer to the significant difference among means of various groups

Results

Statistical Analysis

Values obtained for both the tests were utilized to calculate the respective means with standard deviation. ANOVA test was carried out to analyze if the difference in means between control and experimental groups was significant. Tukey's test was then applied for pairwise comparison between mean values after the results of the ANOVA test were found significant. Level of significance was set at $p \leq 0.05$. IBM statistical package for social sciences (SPSS, Inc., an IBM Company) statistics version 20 for windows was utilized for statistical analysis.

The mean, standard deviation values (SD) of IS and fracture toughness (FT) of the tested groups are summarized in Tables 2 and 3, respectively. Statistically significant differences ($p \leq 0.05$) for IS and FT were found between the control specimens without any nanofiller addition and the test specimens reinforced with ZrO_2 nanofillers at different concentrations (0.5 wt%, 1 wt%, and 3 wt%). There was a significant increase in mean IS and FT after adding ZrO_2 nanofillers at different concentrations especially with 3 wt% of ZrO_2.

For IS, the mean values obtained for groups GA1–GA4 were 1.53, 1.95, 2.93 and 3.89 respectively. The increase in IS from group GA1 to GA4 was statistically significant ($p \leq 0.05$). A comparison of the IS mean values between the control group and experimental groups showed least value of IS for the control group (GA1) which was 1.53 and the highest value of 3.89 was recorded for the group containing 3 wt% ZrO_2 nanofiller (GA4).

Similar results were obtained for FT. The mean values obtained for groups GB1–GB4 were 1.23, 1.86, 2.27 and 2.76, respectively. The increase in IS from group GB1 to GB4 was statistically significant ($p \leq 0.05$). Comparison of the FT mean values between the control group and experimental groups showed least value of FT for the control group (GB1) which was 1.23 and the highest value of 2.76 was recorded for the group containing 3 wt% ZrO_2 nanofiller (GB4).

Discussion

Since many years, PMMA resin is being utilized in making of partial and complete dentures for edentulous patients. However, acrylic dentures suffer from a high fracture rate due to inferior mechanical properties of the resin. So, several researchers tried to enhance these properties of acrylic resin material through different ways: by chemical modification of PMMA, or reinforcing PMMA with other materials. More recently, increasing the strength of PMMA resin in terms of flexural and impact strength by incorporation of nanoparticles in the resin is being tried.[2,20]

In the present study, ZrO_2 nanofillers were chosen for incorporation in the resin matrix due to several advantages: excellent biocompatibility, improved mechanical properties, white color that does not remarkably affects denture esthetics, better dispersion and the ability to eliminate plaque aggregation with improved compatibility with an organic polymer.[21,22] However, according to some authors, the percentage range of ZrO_2 nanofillers must not exceed 7% as it can lead to massive color changes in acrylic resin.[23]

The incidence of fractures in PMMA dentures is decreasing as research continues to enhance the mechanical properties of PMMA resin.[24] In the present research, the effect of adding zirconium oxide nanofiller at different concentrations on IS and FT of heat cured PMMA was studied.

The impact strength is the ability of a material to resist a sudden high-level force or "shock". High impact strength is required to avoid the fracture when the patients accidentally drop their dentures.[25] It was determined using the Charpy-type impact test.

Regarding impact strength (IS), the control group A1 had the lowest IS due to its brittle nature. There was a significant increase in IS as the concentration of ZrO_2 fillers increased as shown in Table 2. The values of IS for various experimental groups with ZrO_2 nanoparticles at different concentrations were higher than those in the control group due to the interaction between the silanized zirconium oxide nanofillers with the resin matrix. Uniform dispersion of nanofillers in polymer and cross-linking between the nanofillers and resin matrix prevents the propagation of cracks by shielding the nanofillers.[26] The smaller size of ZrO_2 nanofillers used in this study led to an increase in surface area that was utilized for dissipation of energy. Also, critical stress required for interfacial debonding increases which increases IS.[27] Similar observations were made by researchers like Alhareb[28] who incorporated silanized ceramic fillers (2.5 wt% alumina and 2.5wt% yttria stabilized zirconia) into conventional heat-cured denture base resin along with nitrile butadiene rubber particles and Al-Hiloh[29] who added 3 wt% concentration of silanized nano ZrO_2 fillers into high-impact heat-cured denture base resin.

However, researchers like Ayad[30] did not find any increase in the impact strength of the resin tested. This could be due to the difference in the particle size of the filler used. Also, the workers used higher concentrations (5 and 15 wt%) of the filler.

Fracture toughness (FT) is regarded as an important property while evaluating dental polymers.[31] It characterizes the mechanical behavior of polymer under load to determine maximum force tolerated by polymer before the fracture.[32] Varieties of tests can be used to evaluate FT. The single-edge-notched specimen test was used because of validity, reliability and ease of sample preparation.[33]

PMMA with no nanofiller had low fracture toughness compared to other formulations of PMMA with ZrO_2 nanofiller. The highest values of FT were obtained with 3 wt% of filler content. While comparing the mean values, there was a significant difference in FT values for PMMA with 3% ZrO_2 when compared to those of other study groups as shown in Table 3.

The improvement in fracture toughness can be attributed to the uniform distribution of fine nanofillers that fill the interstitial spaces in the polymer matrix, thus restricting the segmental motions of the macromolecules and causing an improvement in fracture resistance.[34] The strong interfacial bonding between silanized ZrO_2 filler particles and molecules of resin that helps cover filler particles prevents the propagation of crack and in turn, increases FT.[7] Transformation toughening is also responsible for this improvement in FT. When a crack begins to propagate under stress, ZrO_2 transforms from metastable tetragonal to stable monoclinic phase that leads to depletion of energy for the propagation of the crack. Also, there is an expansion of ZrO_2 particles that places crack in state of compressive stress and crack propagation is arrested.[35,36] Both mechanisms improve the FT of PMMA denture base under masticatory loads.

Table 3: Statistical analysis for fracture toughness ($MPa.m^{1/2}$) of control and experimental groups

Group	*Number of specimens*	*Mean ± SD*	*p value*
GB1 (Control group)	10	$1.23^{d} \pm 0.86$	0.000*
GB2 (0.5 wt% ZrO_2)	10	$1.86^{c} \pm 0.67$	
GB3 (1 wt% ZrO_2)	10	$2.27^{b} \pm 1.07$	
GB4 (3 wt% ZrO_2)	10	$2.76^{a} \pm 0.64$	

*Significant at $p \leq 0.05$; ranks a, b, c and d for Turkey's test refer to the significant difference among means of various groups

Ahmed MA[18] found the highest value of fracture toughness with 3 wt% concentration of ZrO_2 nanofiller. But the values did not improve significantly at the higher concentrations of filler (5–7 wt%). It could be attributed to over saturation of resin matrix with the filler.[37] Alhareb[28] also observed that the value of fracture toughness was maximum when the concentration of yttria-stabilized zirconia used was 2.5 wt%.

The results of the present study are in accordance with other researchers who observed that reinforcement of acrylic resins, ceramics and restorative resins with ZrO_2 nanofillers can lead to an improvement in various properties like impact strength and fracture toughness.[37,38]

However, the present study was limited to using a few concentrations (0.5 wt%, 1 wt% and 3 wt%) ZrO_2 nanofiller. Also, the effect of zirconium oxide addition was studied only on two mechanical properties (impact strength and fracture toughness) of PMMA. Also, further clinical research needs to be done to validate the results of this study.

Conclusion

Within the limitations of the present study, the conclusions drawn were:

- Addition of Zirconium oxide nanofillers at 0.5 wt%, 1 wt% and 3 wt% to heat polymerized PMMA increased impact strength and fracture toughness of PMMA.
- The best results were observed using the concentration of 3%wt of zirconium oxide nanofiller.
- When high impact strength and fracture toughness are needed, PMMA denture base reinforced with zirconium oxide nanofillers is a good choice in removable prosthodontics.

Further studies are required to evaluate the effect of zirconium oxide addition on other properties of PMMA with different concentrations.

Clinical significance

PMMA resin with improved properties obtained by addition of ZrO_2 nanofillers can serve in prosthodontic applications.

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