Original article

The effects of adding zirconium oxide Micro-particles to high impact polymethyl methacrylate on some mechanical properties

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ABSTRACT

Aim: The purpose of this study was to investigate the transverse strength and impact strength of high impact heat-cured Poly (Methyl Methacrylate) (PMMA) reinforced with zirconium oxide particles (ZrO_2).

Materials and Methods: A total of sixty specimens were fabricated for each test. The denture base materials evaluated in this study were divided equally into three groups (A), (B) and (C) for each test. group (A) control group, unmodified high impact heat cured (PMMA). Group (B) & (C) high impact heat cured (PMMA) reinforced with 5% & 10% (ZrO_2) powder respectively by volume. The transverse strength was measured using the three point bending test. The impact strength was measured using the Chrapy impact tester. The micro-structural study of the fracture surfaces of test specimens was performed using Scanning Electron Microscope.

Results: Statistical analysis were conducted on the data obtained from the experiments using one way analysis of variance(ANOVA).Both the transverse strength and impact strength of high impact heat cured PMMA reinforced with treated ZrO_2 particles were superior to the unreinforced high impact heat cured PMMA. However, the improvement in transverse strength was not directly proportional with the amount of filler particles. Scanning electron micrographs showed the degree of bonding between the filler particles and PMMA matrix.

Keywords: high-impact PMMA, transverse strength, impact strength, zirconium oxide.

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INTRODUCTION

The most commonly used material in construction of denture base is Polymethyl methacrylate (PMMA) acrylic resin due to its low cost, light weight, ease of processing and repair good adaptation and ease of application, and usage in tooth or implant-supported removable/complete prostheses and orthodontic appliances [1]. However, it is still far from an ideal in fulfilling the mechanical requirements of prosthesis [2]. Several attempts have been made to improve the mechanical properties of the denture base material to be useful in points where masticatory forces are relatively high, such as distal extensions opposing natural teeth, single complete dentures, overdentures, and implant-supported complete dentures, such as the chemical modification of PMMA, reinforcement with glass fibers, Carbone fibers, aramide fibers, metal wire, titanium oxide, silicone dioxide hydroxyapatite [3,4]. However, there are a lot of drawbacks to using these additives such as poor adhesion and corrosion of metal wires [5] and tissue irritation due to fibre reinforcement [6]. (Smith., 1961) analyzed the practical situation for fracture of dentures and showed two types of failure. Failure caused by impact forces, i.e., a high stress rate extra orally and failure usually in function; this is probably a fatigue phenomenon, i.e. a low and repetitive stress rate intra orally [7]. Aiming to improve the mechanical properties, some inorganic fillers were used to reinforce the PMMA denture base [8]. The selection of ZrO_2 as a filler in this study was based on the properties of this filler which can improve the mechanical properties of the reinforced PMMA matrix. such as impact strength and transverse strength [9-11].

MATERIAL AND METHODS

The transverse and impact strength laboratory tests for denture base materials were performed in this study. Sixty specimens were prepared for each test which were categorized equally into three groups according to the type of used denture base material. Group (A) was the control group (unmodified high impact acrylic resin - idobase specimens). Group (B) & (C) specimens made up of high impact heat curing (PMMA) reinforced with 5% & 10% ZrO_2 powder respectively by volume after their treatment with silane coupling agent 3-methacryloxypropyl-trimethoxysilane (Monobond-S). The treated ZrO_2 powder (20µm-30µm) was thoroughly mixed with acrylic powder using a mortar and pestle for initial mixing and blending, then hand tumbling in a plastic jar was performed to achieve an even color [12,13]. In the present study, 3-methacryloxypropyl-trimethoxysilane (Monobond-S) has been used as s silane coupling agent to improve the bond between the surface of ZrO_2 powder and PMMA particles for groups (B & C). Each 30 gm of ZrO_2 powder was treated with solution of 0.3 gm of coupling agent in 100 gm of acetone. A magnetic stirrer was used for stirring the metal oxide powder in the coupling agent / acetone solution for 60 min, followed by complete evaporation of acetone [14].

Transverse Strength Test:

The object of the test is to evaluate the behaviour of denture base polymer under bending loads. Metallic mold having the dimensions of flexure strength test specimens according to the American Dental Association Specification No. 12 for denture base polymer (65 mm length, 10 mm width and 2.5 mm thickness +/_0.2mm) was constructed. Sixty wax specimens representing the dimensions of test specimens were prepared from base plate wax. These sixty specimens were divided equally into three groups of twenty specimens each. The first twenty specimens were constructed from group (A), the second twenty from group (B) and the third twenty from group (C). All specimens were processed using long curing cycle. After their removal from the flask the specimens were finished using smooth sand paper. The specimens were stored in water at 37±10 C for seven days using thermostatically controlled water path before performing transverse strength test.

Testing

The specimen was mounted on the designed part of a universal testing machine, three-point loading and testing equipment (Mfd for Dayton Electric Co. Chicago 60648 USA (CEDM-4).The load was applied on the centre of the specimen with a cross head speed of 0.5 cm / min 15. The maximum load before fracture was measured. The transverse strength of the specimens was calculated using the standard relation [16].

$$S = \frac{3LP}{2WT^2} Kgs/cm^2$$

Where: S = Transverse strength, P = Maximum load before fracture, L = Distance between supports (50 mm), W = Width of the specimen (10 mm), T = Depth (thickness) of the specimen (2.5 mm).

Impact Strength Test:

Metallic mold having the dimensions of impact strength test specimens according to British Standard Institute Specification No. 771, (1948) (75 mm length, 10 mm width, and 10 mm thickness with a notch of 2 mm depth at the mid-span was constructed. The number of the test specimens, their divisions and the protocol of testing was the same as flexural strength test.

Testing

The specimen was accurately mounted in the vice of the pendulum testing machine (Charpy type impact test).The specimen was struck at the mid-span by the pendulum as it was released out of position (The force was applied to the specimen from the unnotched side)[16]. Reading was directly given and recorded on the scale by the silent arm of the double moving pointer. The impact strength of the specimen was expressed in term of J/m^2 of energy absorbed in breaking the specimens [17].

Scanning electron microscope (SEM) observation:

SEM (JEOL-JSM 5300) was used to examine the fractured surface of the randomly selected specimens. The electron voltage used in this study was set at 15 Kv. The examined specimens were gold coated using a sputtering machine (ION SPUTTERING DEVICE / JEOL – JFC – 1100E).Images were obtained with different magnifications.

RESULTS

The mean, SD and P- values of the specimens tested for transverse strength are presented in (Table 1 & Figure 1). It shows a significant increase in transverse strength values in group (B) when compared to group (A) (P 0.001) & (T= -3.769). Asignificant increase in transverse strength values was also observed in group (B) when compared to group (C) (P 0.032) & (T=2.318). However the increase in transverse strength was statistically

insignificant when group (C) and group (A) were compared (P 0.575) & (T= -0.313).

The mean, SD and P-values of the specimens tested for impact strength are presented in (Table 2 & Figure 2). It shows a significant increase in impact strength values in group (B) when compared to group (A) (P 0.006) & (T= -3.086). Also a significant increase in transverse strength values was observed in group (C) when compared to group (A) (P 0.001) & (T= -3.897). However the increase in transverse strength was statistically insignificant when group (C) and group (B) were compared (P 0.501) & (T= -686).

Scanning electron microscope (SEM) was used to view the fracture surface of different test specimens to assess the shape, size and distribution of the filler particles within the fully cured composite. The size of the ZrO_2 filler particles was 20µm to 30µm

Micrograph of fractured surface related to control group (A) (Figure 3 a & b) showed a flake like morphology and a smooth surface with small pits and cracks compared to other test specimens related to other groups. The fractured surfaces of reinforced groups (B & C) exhibited rough surface with varying morphological features of reinforcing particles which generally was round (Figure 4). The voids formed due to ZrO_2 particles that pulled out were clear (Figure 5).

Table 1: Comparison of transverse strength (Mpa) between different Groups

GROUPS	A	B	C
Mean ± SD	98.91 ± 1.096	100.75 ± 1.089	99.13 ± 1.925
T-test(P)	-3.7690.001a*	2.3180.032b*	-0.3130.575c

a. Comparing group A with Group B.
b. Comparing group B with Group C.
c. Comparing Group A with Group C.
*Significant, p ≤ 0.05.

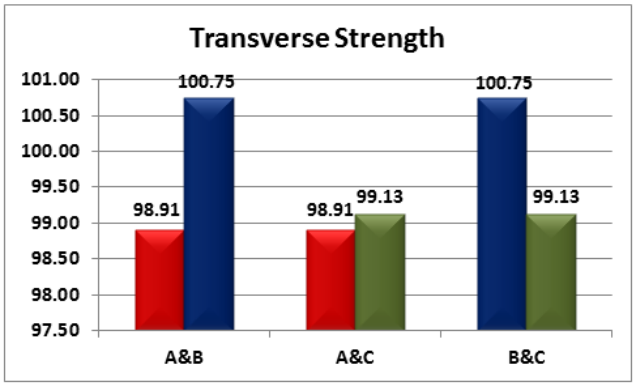

Figure 1: Comparison between the mean values of transverse strength of different groups

Table 2: Comparison between the mean values of impact strength of different groups

GROUPS	A	B	C
Mean ± SD	3493.06 ± 52.59	3591.58 ± 86.19	3618.13 ± 86.79
T-test(P)	-3.0860.006a*	-.6860.5b	-3.8970.001c*

a. Comparing group A with Group B.
b. Comparing group B with Group C.
c. Comparing Group A with Group C.
* Significant, p ≤ 0.05.

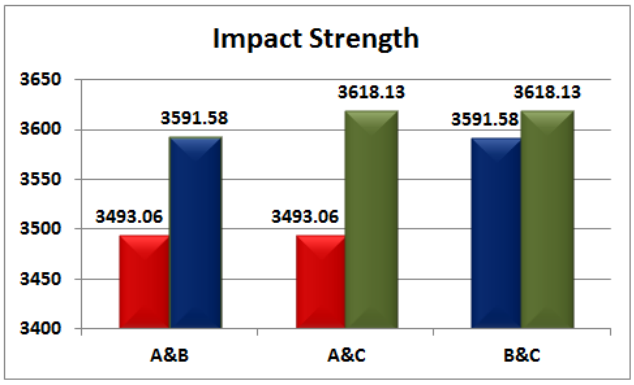

Figure 2: Comparison of impact strength (J/m2)

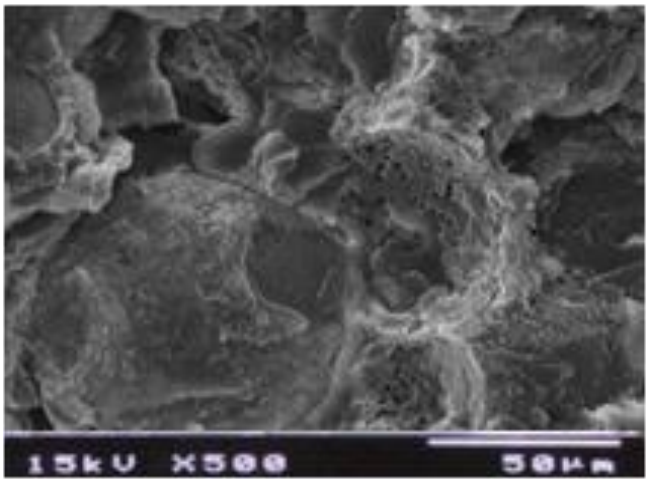

Figure 3 (a)

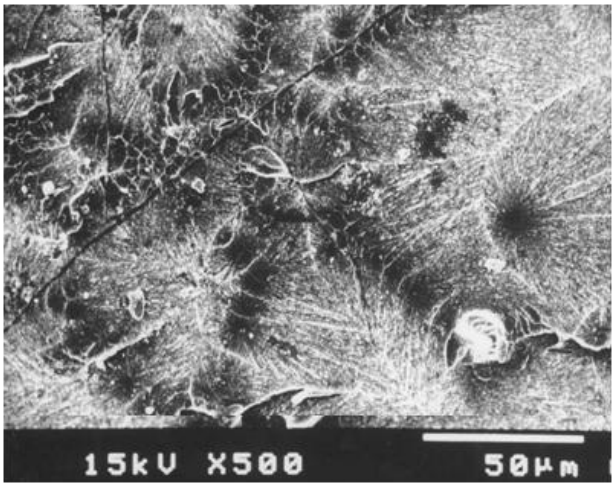

Figure.3(b)

Figure 3: SEM micrograph showing the fracture surface of PMMA specimen (Original Mag.x 500)

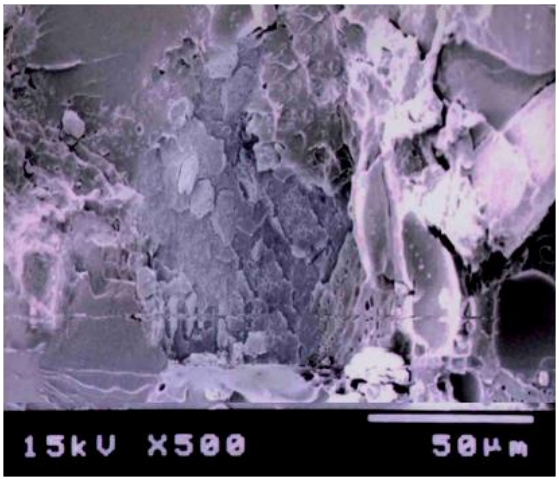

Figure 4: SEM photograph of the fractured surface of ZrO2 filled specimens. (Orig. Mag×1000)

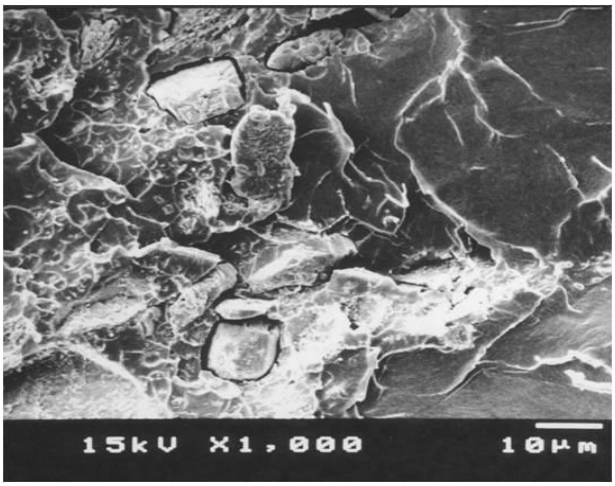

Figure 5: SEM photomicrograph showing the voids formed due to ZrO2 particles pulled out (Original Mag x 500)

DISCUSSION

In general, the PMMA denture base material is prone to fracture either intraorally or extraorally due to its poor mechanical properties. To overcome this drawback, PMMAs are reinforced with a verity of additives to improve their strength [9,18].

Only a few studies on the effect of adding ZrO_2 micro-particles in high impact heat-cured PMMA are available in the literature. In contrast, investigators have worked on improving the mechanical properties of conventional heat-cured denture base acrylic resin by incorporating different types of fillers [19,20].

The aim of the current study was to evaluate the effect of adding ZrO_2 micro-particles at low volume fractions (5% and 10%) after their treatment with silane coupling agent on the transverse and impact strength properties of high impact PMMA denture base material without adversely affecting the esthetic. All tested

specimens were stored in a water at about 37± 1 cofor seven days to simulate the wet condition inside the oral cavity. The results found that all tested groups showed increased transverse strength and impact strength. However, this improvement was not in direct proportion to the concentration of filler particles.

According to the results of the current study, a significant increase in transverse strength was found with the addition of 5% ZrO_2 particles while the improvement was not significant with the addition of 10% ZrO_2 particles. Moreover, the addition of 10% ZrO_2 resulted in decrease in transverse strength as compared with 5% ZrO_2. The amount of filler incorporated to reinforce the acrylic resin is a factor affecting the mechanical properties [21].

The percentage of reinforcing filler should be in such amount that will disperse evenly into the resin matrix without gross disruption in its continuity [22]. The improvement in the flexural strength could be due to good dispersion of the micro size ZrO_2 particles which assists in filling of the matrix interstitially [23]. Furthermore, The small particle size of the filler provides maximum surface area for bonding to resin. The employment of silane bonding Monobond-S (3-methacyloxypropyl-trimethoxysilane) in this study due to its benefit for bonding of inorganic composite fillers to organic matrix permitting better transfer stress from PMMA to fillers, thus leading to an improvement in the equivalent transverse strength of the composite [23]. It also makes the mixture more homogenous, resulting in stronger PMMA [24,25].

In this study the improvement in transverse strength decreased as the concentration of the filler particles increased which could be attributed to the agglomeration of incorporated particles, acting as stress concentration centres in the matrix and unfavourably decreasing the mechanical properties of the resin [13,26]. Moreover, the agglomerated fillers lead to formation of loose attached clusters, which affect the mode of crack propagation, reduces the transverse strength [26,27]. This finding was in agreement with some previous studies, [28,29] while in disagreement with others [10,30].

With the addition of 5% and 10% filler particles there was a significant improvement in impact strength as compared to control group. This could be due to the transfer of the applied load to the filler particles through a relatively soft ductile matrix which became the principal load – bearing constituents [31]. However, this improvement was not significant with the addition of 10% ZrO_2 particles as compared with the addition of 5% ZrO_2 filler particles which considered to be due to the large number of surface irregularities produced by the extrusion of filler particles (debonding), since a significant reduction of impact resistance of PMMA to the presence of very small surface defects is well known [31,32]. Furthermore, the distribution of ZrO_2 micro-particles in the polymer matrix particularly at high ZrO_2 concentrations 10% by volume was not homogeneous with evidence of agglomerations, which could reduce the impact strength[19].

Conclusion: with consideration to the limitations of the present study, The results of this study conclude that addition of ZrO_2 micro-particles powder 5% and 10% by volume to the high impact strength heat cure acrylic resin reveals an increase in both transverse and impact strength of reinforced resin. However, the improvement in transverse strength was not in direct proportion with the amount of filler particles.

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