Original Article

Impact Strength and Dimensional Accuracy of Heat-Cure Denture Base Resin Reinforced With ZrO_2 Nanoparticles: An *In Vitro* Study

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Abstract

Background: Polymerization shrinkage and fracture are the two common trouble shoots with denture base resins. Polymerization shrinkage affects the dimensional accuracy and fit of the prosthesis. The effect of zirconia (ZrO_2) nanoparticles on polymerization shrinkage is not documented yet. **Purpose:** The aim and objective of this study were to evaluate the impact strength and dimensional accuracy of heat-cured poly methyl methacrylate (PMMA) on reinforcement with ZrO_2 nanoparticles. **Materials and Methods:** Conventional heat-cure denture base resin (control) and the polymer reinforced with 3, 5, and 7 wt% of ZrO_2 nanoparticles were prepared and used in this study. Forty bar-shaped specimens were prepared and tested for impact strength using Charpy's type impact tester. Forty denture bases were fabricated and checked for dimensional accuracy by measuring the distance between the denture base and the cast in two different sections using the travelling microscope. **Results:** The impact strength decreased with increased concentration of ZrO_2 and found to be least at 7 wt% concentration (2.01 ± 0.26 J/mm^2). The distance between the denture base and the cast significantly decreased both in the posterior palatal seal region (0.060 ± 0.007 cm) and mid-palatine section region (0.057 ± 0.006 cm) with ZrO_2 nanoparticles reinforcement and was found to be least at 7 wt% concentration. **Conclusion:** Reinforcement of heat-cured PMMA with ZrO_2 nanoparticles significantly increased the dimensional accuracy and decreased the impact strength.

Keywords: *Dimensional accuracy, impact strength, PMMA, travelling microscope, ZrO_2 nanoparticles*

Introduction

The development of material science over time has led to a slow and steady increase in the quality of materials used for dental prostheses. Search for materials that are biocompatible, readily available, cost effective, easy to manipulate, less technique sensitive, functionally efficient, and esthetically pleasing is a persistent process.[1]

Poly methyl methacrylate (PMMA) has been the most commonly used denture base material, because of its positive properties such as ease of working, processing, intraoral fit, stability, and aesthetics.[2] Regardless of these advantages, there are certain shortcomings pertaining to its strength properties and polymerization shrinkage. The denture base made of PMMA is exposed to different types of stresses such as compressive, tensile, shear, and impact stresses.

Skinner stated two major disadvantages of PMMA resins. They are large curing shrinkage during processing and its property of high water sorption. The heat produced during polymerization that is exothermic in

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turn produces an internal heat especially in the thickest portion of the denture, which is the posterior palatal seal (PPS) region in the maxillary denture and hence polymerization shrinkage is high in this region.[3]

To overcome fracture susceptibility and polymerization shrinkage of PMMA, numerous modifiers have been used to reinforce the resin polymer. The various materials that have been used for the reinforcement include woven glass fiber, polystyrene fibers, silver, aluminum, and copper and titanium oxide powders.[4] These particles are reinforced in the form of nanoparticles due to better handling characteristics and even distribution.[4-6] They tend to increase the impact strength (IS) considerably. Zirconia (ZrO_2) is a noncytotoxic metal oxide that is insoluble in water and lacks the bacterial adhesion property.[7] Recently, extensive research has been conducted on reinforcement of PMMA with ZrO_2 to modify the mechanical properties such as transverse strength, flexural strength, and IS.[8] Despite the reinforcements of PMMA, the success of the complete denture in terms of retention and stability is closely related to its accurate fit, which in turn, depends on a series of factors, which include the clinical expertise of the dentist, accuracy of all the laboratory procedures of denture preparation, the type of materials used, and the dimensional stability of the mucosal tissues.[9] Evaluation of dimensional accuracy with ZrO_2 reinforcements has not been yet documented in the literature. Hence, the IS and dimensional accuracy of heat-cure acrylic resins have been investigated with the focus on incorporation of ZrO_2 nanoparticles at varying concentrations.

Materials and Methods

Zirconia Powder (30–50 nm; Nano Research Lab, Jamshedpur, Jharkhand, India) of 99.5% purity was selected as filler. The ZrO_2 filler and PMMA (DPI, Bombay Burmah Trading Corp Ltd, Mumbai, India) were pre-weighed using an electronic balance (Avery India Ltd, Ballabgarh, India) in order to ensure a filler concentration of 3%, 5%, and 7% by weight.[10-14] ZrO_2 particles were treated with 1 wt% of silane coupling agent before the mixing of filler particles and heat-cure PMMA resin polymer.[15] Mixing and blending was carried out thoroughly using ceramic ball milling, which rotates at a rate of 850 rpm to obtain a uniform mix.

A total of 40 bar-shaped specimens of dimensions 80 × 7 × 4 mm (ISO specification No: 1567) were prepared out of vacuum-formed thermoplastic sheet dies of 4-mm thickness.[15,16] For fabricating the specimens of group A, the control (subgroup: A1), pre-weighed ZrO_2-reinforced acrylic resins of concentrations 3% (subgroup: A2), 5% (subgroup: A3), and 7% (subgroup: A4) were mixed with monomer in a ratio of 3:1 and packed into mold space in the dough stage.[17] Trial closure was conducted and compressed with hydraulic press for 1 hour at 1200 psi.[18] The flasks were bench cured for 20 minutes and heat cured at 74°C for 2 hours and 100°C for 1 hour.[17] After curing, the flasks were bench cooled to room temperature. The specimens of each subgroup were finished and polished after retrieval. The specimens with internal or external porosities, warpage, broken edges, and surface defects were excluded from the study.

A total of 40 heat-cure denture bases were fabricated on dental stone casts. Thermoplastic sheets of 2-mm thickness were used as die. For fabricating the specimens of group B, the control (subgroup: B1), pre-weighed ZrO_2-reinforced acrylic resins of concentrations 3% (subgroup: B2], 5% (subgroup: B3), and 7% (subgroup: B4) were mixed with monomer in a ratio of 3:1 and packed into mold space in the dough stage. The trial closure and processing regimens were same as that of group A. The specimens of each subgroup were finished and polished. All the specimens were immersed in distilled water at 37°C for 7 days.[13]

The IS of the 40 bar-shaped specimens were tested using Charpy's impact tester (Modern Metallurgical and Scientific Services, Chennai, Tamil Nadu). The specimens were prepared by marking three lines. Two lines were drawn at a distance of 10 mm from the borders of the specimen. The third midline was marked at 30 mm away from the two lines. These two lines correspond to the location of supporting arm in the testing machine and conform to the span length of 60 mm. At the midline, a V-shaped notch of 1.2 mm was prepared with a notch cutter (Hounsfield notching machine, Tensometer Ltd., Croydon, UK).[19] The pendulum of the testing machine, which has an impact capacity of 164 J and a striking velocity of 5.6 m/s, would come and impact the specimen from the other side. The pendulum hit the specimen to fracture and this maximum load before fracture (F) was displayed in the machine. This value was recorded as the IS of the specimens in joule per square millimeter.

Dimensional accuracy was measured in terms of the distance between the denture base and the cast at the PPS and mid-palatine section (MPS) regions. At MPS, the distance between the casts and the denture bases was measured after sectioning the cast–denture base assembly anteroposteriorly using a diamond disk. The distance was measured with the help of travelling microscope (INCO, Ambala, India) with an accuracy of 0.001 cm. The distance between the denture base and

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cast was measured in three regions of the PPS namely hamular notches on either side of cast and in the midline using the travelling microscope and the average was calculated in centimeter.[3,20-24] The distance between the cast and the denture base were measured in three regions in MPS using the travelling microscope. The regions selected were incisive papilla, palatal vault, and posterior border of the denture base.[22,23] The average of the three readings was calculated in centimeter. The obtained values of both group A and B were subjected to statistical analysis using one-way analysis of variance and Bonferroni multiple comparison tests.

Results

The mean and standard deviation IS of subgroups A1, A2, A3, and A4 were 3.93 ± 0.17, 3.73 ± 0.19, 3.24 ± 0.35, and 2.01 ± 0.26 J/mm^2, respectively [Table 1 and Figure 1]. The IS had decreased considerably from A1 to A4 and the least IS was with subgroup A4. While comparing the mean IS of the subgroups, a statistically significant difference ($P < 0.001$) existed. In Table 2, the Bonferroni multiple comparison tests were conducted at 95% confidence interval to compare the mean IS within the subgroups. The difference in the mean IS between A1 and A2 was statistically insignificant. However, statistically significant difference in mean IS existed when comparing between other sub-groups.

The mean distance and standard deviation between the denture base and the cast of subgroups B1, B2, B3, and B4 were 0.148 ± 0.031, 0.116 ± 0.017, 0.090 ± 0.016, and 0.060 ± 0.007 cm, respectively [Table 3 and Figure 2]. The mean had decreased considerably from B1 to B4 and the least distance was at subgroup B4. While comparing the mean dimensional accuracy in relation to the distance between the denture base and the cast at PPS of the subgroups, a statistically significant difference ($P < 0.001$) existed. In Table 4, the Bonferroni multiple comparison tests were conducted to compare the mean dimensional accuracy in terms of distance between the denture base and the cast at PPS section within the subgroups. The difference in the mean values on comparing between the subgroups was statistically significant.

The mean distance and standard deviation between the denture base and the cast at MPS of subgroups B1, B2, B3, and B4 were 0.128 ± 0.025, 0.097 ± 0.008, 0.076 ± 0.010, and 0.057 ± 0.006 cm, respectively [Table 5 and Figure 3]. The mean had decreased considerably from B1 to B4 and the least distance was at subgroup B4. While comparing the mean dimensional accuracy in relation to the distance between the denture base and the cast at MPS of the subgroups, a statistically significant difference ($P < 0.001$) existed. In Table 6, the Bonferroni multiple comparison tests were conducted to compare the mean dimensional accuracy in terms of distance between the denture base and the cast at MPS within the subgroups. The difference in the mean values on comparing between subgroups was statistically significant.

Discussion

Several studies have been carried out to improve the properties of PMMA, which include addition of reinforcing material as fibers, fillers, hybrid reinforcement, and recently, nanoparticles. However, the most effective reinforcement is not apparent, and research scholars are confused about designing such reinforcements. Reinforcement has two important purposes on prosthesis. The initial purpose is to improve the strength and prevent fracture, and the

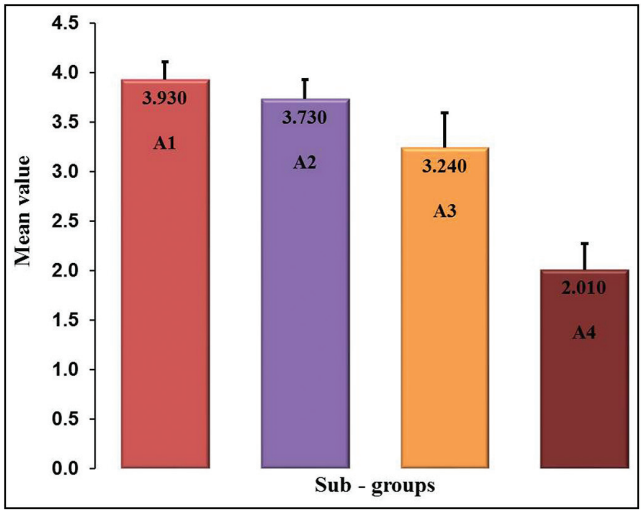

Figure 1: Mean impact strength

Table 1: One-way analysis of variance to compare mean IS values between sub-groups

Variables	Subgroups	*N*	Mean	Standard Deviation	*F* value	*P* value
IS Group A	A1	10	3.9300	0.17670	112.597	<0.001
	A2	10	3.7300	0.19465		
	A3	10	3.2400	0.35340		
	A4	10	2.0100	0.26437		

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Table 2: Bonferroni multiple comparisons of mean IS values

Dependent variable	Subgroup	Subgroup	Mean difference	Standard error	*P* value	95% confidence interval	
						Lower bound	Upper bound
IS Group A	A1	A2	0.20000	0.11487	0.541	−0.1207	0.5207
		A3	0.69000*	0.11487	0.000	0.3693	1.0107
		A4	1.92000*	0.11487	0.000	1.5993	2.2407
	A2	A3	0.49000*	0.11487	0.001	0.1693	0.8107
		A4	1.72000*	0.11487	0.000	1.3993	2.0407
	A3	A4	1.23000*	0.11487	0.000	0.9093	1.5507

*The mean difference is significant at the 0.05 level

Table 3: One-way analysis of variance to compare mean distance at PPS

Variables	Subgroups	*N*	Mean	Standard deviation	*F* value	*P* value
Group B: Dimensional accuracy in relation to distance between denture and cast at PPS	B1	10	0.14890	0.031494	35.280	<0.001
	B2	10	0.11690	0.017266		
	B3	10	0.09000	0.016350		
	B4	10	0.06060	0.007306		

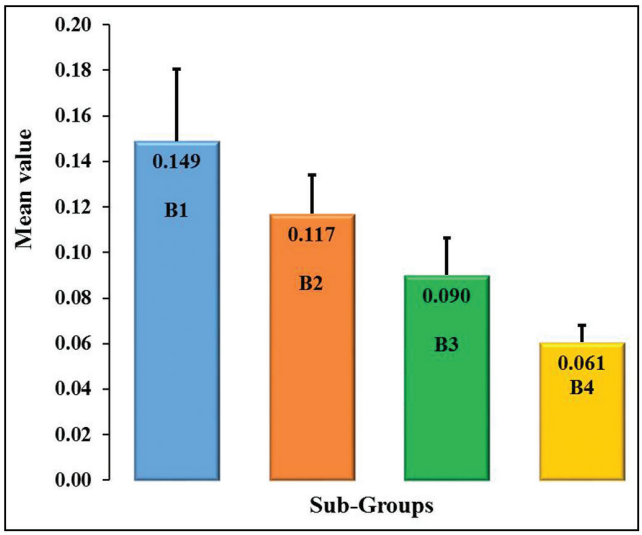

Figure 2: Mean distance between denture base and cast at posterior palatal seal

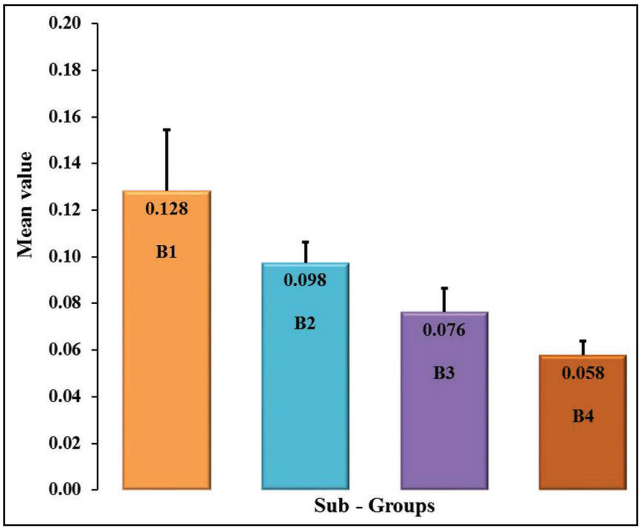

Figure 3: Mean distance between denture base and cast at mid-palatine section

Table 4: Bonferroni multiple comparisons of mean dimensional accuracy in relation to the distance between the denture base and the cast at PPS

Dependent variable	Subgroup	Subgroup	Mean difference	Standard error	*P* value	95% confidence interval	
						Lower bound	Upper bound
Group B in relation to distance between the denture and cast at PPS	B1	B2	0.032000*	0.008974	0.006	0.00694	0.05706
		B3	0.058900*	0.008974	0.000	0.03384	0.08396
		B4	0.088300*	0.008974	0.000	0.06324	0.11336
	B2	B3	0.026900*	0.008974	0.029	0.00184	0.05196
		B4	0.056300*	0.008974	0.000	0.03124	0.08136
	B3	B4	−0.029400*	0.008974	0.014	−0.05446	−0.00434

*The mean difference is significant at the 0.05 level

Table 5: One-way analysis of variance to compare the mean distance at MPS

Variables	Subgroups	*N*	Mean	Standard Deviation	*F* value	*P* value
Group B in relation to distance between denture and cast at MPS	B1	10	0.12840	0.025902	41.173	<0.001
	B2	10	0.09750	0.008860		
	B3	10	0.07630	0.010264		
	B4	10	0.05780	0.006125		

Table 6: Bonferroni multiple comparisons of mean dimensional accuracy in relation to the distance between the denture base and the cast at MPS

Dependent variable	Subgroup	Subgroup	Mean difference	Standard error	*P* value	95% confidence interval	
						Lower bound	Upper bound
Group B in relation to distance between denture and cast at MPS	B1	B2	0.030900*	0.006679	0.000	0.01225	0.04955
		B3	0.052100*	0.006679	0.000	0.03345	0.07075
		B4	0.070600*	0.006679	0.000	0.05195	0.08925
	B2	B3	0.021200*	0.006679	0.018	0.00255	0.03985
		B4	0.039700*	0.006679	0.000	0.02105	0.05835
	B3	B4	0.018500	0.006679	0.053	−0.00015	0.03715

*The mean difference is significant at the 0.05 level

second purpose is to improve the dimensional accuracy in order to prevent residual ridge resorption of the associated structures.[25] Currently, reinforcements are carried out at the nanoparticles level.[4-6,10-14,25-36] The properties of resin reinforced by nanofillers depend highly on the factors that include size, shape, type, and concentration of the reinforced material.[35] In this study, ZrO_2 nanoparticles were selected to evaluate the effect of reinforcement on the IS and dimensional accuracy of heat-cure denture base acrylic resin.

Studies about the ZrO_2 reinforcement on the IS of the acrylic resin are very few in the dental literature. Charpy's impact test was chosen for this study in which V-shaped notches were made in the specimens to act as areas of stress concentration.[13] In this *in vitro* study with respect to IS, the mean IS value decreased with increasing concentration of ZrO_2 nanoparticles. However, the difference in the mean IS values between A1 (control) and A2 (3%) was not statistically significant ($P = 0.541$). This result was in agreement with the previous experiments.[13,15,37] Silanation of ZrO_2 nanoparticles further enhanced and improved the IS. However, in this *in vitro* study, despite the silanation of ZrO_2 nanoparticles, the IS decreased significantly with increase in ratio of reinforcement.

Accurate fit of the dentures is very important for maintaining healthy and stable tissues and helps in reducing the degree of tissue changes.[21] There are many studies conducted by various authors, which have evaluated the dimensional accuracy of the denture bases, and have concluded that processing technique and water sorption does have an influence on dimensional accuracy.[3] There are no studies in the dental literature evaluating the dimensional accuracy of heat-cure denture base acrylic resin reinforced with ZrO_2 nanoparticles.

Hence, in this study, the fit was measured in PPS[3] and MPS.[21] Polymerization shrinkage tends to draw the denture flanges inwards and as a result the denture gets slightly elevated in the MPS. The lesser the distance, the better is the dimensional accuracy of the denture base. The distances between the denture base and the cast at PPS and MPS were significantly lesser with 7% ZrO_2 nanoparticles reinforcement than the control. Thus, the dimensional accuracy or fit of the dentures improved significantly with increase in ratio of reinforcement of ZrO_2 nanoparticles.

This is an *in vitro* study and hence, an exact clinical implication of the test results is questionable. The effect of water sorption, which has an influence on the polymerization shrinkage, is not taken into consideration. This study used only simulations of the oral environment. However, it could not accurately reproduce all the oral factors such as thermal fluctuations, masticatory load, masticatory cycles, salivary pH, its buffering capacity, and flow rate.

Conclusion

Within the limitations of this *in vitro* study, the following conclusions were deduced:

1. The reinforcement of ZrO_2 nanoparticles with heat-cure denture base resin decreased the IS of the resin.
2. The reinforcement of ZrO_2 nanoparticles with heat-cure denture base resin increased the dimensional accuracy and fit at both PPS and MPS.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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