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ORIGINAL RESEARCH

Influence of incorporation of ZrO_2 nanoparticles on the repair strength of polymethyl methacrylate denture bases

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Background: Repeated fracture of the denture base is a common problem in prosthodontics, and it represents a nuisance and a time sink for the clinician. Therefore, the possibility of increasing repair strength using new reinforcement materials is of great interest to prosthodontists.

Aim of the study: This study aimed to evaluate the effects of incorporation of zirconia nanoparticles (nano- ZrO_2) on the flexural strength and impact strength of repaired polymethyl methacrylate (PMMA) denture bases.

Materials and methods: One hundred eighty specimens of heat-polymerized acrylic resin were fabricated (90 for each test) and divided into three main groups: one control group (intact specimens) and two groups divided according to surface design (45° bevels and butt joints), in which specimens were prepared in pairs to create 2.5 mm gaps. Nano-ZrO₂ was added to repair resin in 2.5 wt%, 5 wt%, and 7.5 wt% concentrations of acrylic powder. A three-point bending test was used to measure flexural strength, and a Charpy-type test was used to measure impact strength. Scanning electron microscopy was used to analyze the fracture surfaces and nano-ZrO₂ distribution. The results were analyzed with a paired sample *t*-test and an unpaired *t*-test, with a *P*-value of ≤ 0.05 being significant.

Results: Incorporation of nano-ZrO₂ into the repair resin significantly increased flexural strength (P<0.05). The highest value was found in the bevel group reinforced with 7.5% nano-ZrO₂, whereas the lowest value was found in the butt group reinforced with 2.5% nano-ZrO₂. The impact strength values of all repaired groups were significantly lower than those of the control group (P<0.05). Among repaired groups, the higher impact strength value was seen in the butt group reinforced with 2.5% nano-ZrO₂. The bevel joint demonstrated mainly cohesive failure, whereas the butt joint demonstrated mainly adhesive failure.

Conclusion: Incorporation of nano- ZrO_2 into the repair resin improved the flexural strength of repaired denture bases, whereas it decreased impact strength, especially with high nano- ZrO_2 concentrations.

Keywords: denture repair, flexural strength, impact strength, PMMA, ZrO, nanoparticles

Introduction

Polymethyl methacrylate (PMMA) is the most common denture base material due to its biocompatibility, esthetics, accurate fit, stability in the oral environment, ease of fabrication and adjustment, low cost, and possibility of repair.^{1,2} However, PMMA has poor mechanical properties, which often results in denture base fractures.³ Such denture fractures may occur inside the patient's mouth usually at the midline of the denture base during mastication or outside the patient's mouth when the removable prosthesis drops suddenly.⁴⁻⁶

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Fabrication of a new removable prosthesis is highly costly and time-consuming for both patients and prosthodontists. Therefore, denture repair is required if the denture fits properly.⁷ The proper denture repair procedure should be easy, time saving, match the original color, maintain dimension accuracy, and restore original strength.^{8,9} Different materials have been used to repair fractured denture bases; these include autopolymerized, visible light-polymerized, and heat-polymerized acrylic resin.

Heat-polymerized materials have been proven to have better mechanical properties than autopolymerized resin.^{10–14} However, distortion or warpage due to reheating and timeconsuming laboratory procedures are drawbacks to their use as a repair material.^{14,15} Therefore, autopolymerized acrylic resin is preferred over heat-polymerized acrylic resin for repair.

Although autopolymerized resin is the most common material used for repair, its strength has been shown to be half that of intact heat-polymerized denture resin.¹⁶ Hence, repeated fracture of the repaired denture bases has been reported to be frequently related to the low strength of the autopolymerized repair resin.^{6,12} To achieve adequate repair strength, many attempts have been made to modify repair surface design and/or reinforce the repair resin.⁷ Different design modifications of repaired joints have been made by increasing the surface area, which therefore improves the bond strength.¹⁷ Ninety-degree butt and 45° bevel joint designs have been found to affect repair strength.^{18–21}

Zirconia (ZrO₂) is a metal oxide that has been widely used because it possesses high mechanical strength, good surface properties, and good biocompatibility and biological properties, thus making it a beneficial material for use in dental materials, such as reinforcement of denture bases and repair.^{22–25} The incorporation of zirconia nanoparticles (nano-ZrO₂) into PMMA has been suggested to improve PMMA properties.^{26,27} The addition of nano-ZrO₂ to PMMA significantly improves flexural strength and impact strength. Furthermore, the maximum increase in flexural strength and impact strength was observed in PMMA denture bases containing 5 wt% nano-ZrO₂.^{28,29}

Good adhesion and dispersion homogeneity of nano- ZrO_2 with the resin matrix effectively improve the properties of the polymer/nanoparticles composite. Therefore, surface modification of nanoparticles with a saline coupling agent (97% 3-(trimethoxysilyl)propyl methacrylate [TMSPM] solution) might reduce aggregation of nano- ZrO_2 and then enhance its compatibility with the polymer, which may result in the improvement of composite properties.^{28–30} Although flexural stresses that are counteracted by the flexural strength of the material are a constant phenomenon during mastication, impact strength is also required to prevent accidental dropping or falling of the dentures. Because dentures have frenal notches and some scratches on the denture surface that act as common stress concentrated areas and consequently reduce the strength of the dentures,³¹ a Charpy-type impact test was selected for this study, and V-shaped notches were created in the specimens in order to simulate denture borders.

The effects of nano- ZrO_2 on the repair strength of PMMA denture bases have not been well investigated in the literature. Therefore, this study was conducted to evaluate the reinforcement effect of nano- ZrO_2 on the flexural strength and impact strength of repaired PMMA denture bases. The null hypothesis is that the addition of different concentrations of nano- ZrO_2 will not improve the flexural strength and impact strength of repaired PMMA denture bases.

Materials and methods Specimen preparations

A total of 180 specimens of heat-polymerized acrylic resin (Major.Base.20; Major Prodotti Dentari Spa, Moncalieri, Italy) were prepared. Ninety specimens were prepared for the flexural strength test in dimensions of $65 \times 10 \times 2.5$ mm³ in accordance with the ANSI/ADA specification no $12.^{20}$ The other 90 specimens were prepared for the impact strength test in accordance with the ISO standard 1567:1999/Amd.1:2003(E) for denture base polymers.³² Specimens for the impact strength test were prepared with dimensions of $50 \times 6 \times 4$ mm³, and each specimen was fabricated with a V-shaped notch. The notch depth was ~0.8 mm across the entire 6 mm width of the specimen, leaving an effective depth of 3.2 mm below the notch.

Specimens for both strength tests were divided into one control group (intact specimens) and two repair groups for each strength test, and these specimens were consequently divided according to the surface design into four butt groups and four bevel groups for each test (Table 1). Specimens of repair groups were prepared in two parts (one pair) for each specimen to allow space for the repair material. To standardize the butt and 45° bevel joints, modified metal plates were customized and used to prepare the repair groups' specimens (Table 2 and Figure 1).

For control group specimens, metal molds were waxed up, and then the wax (Cavex Set Up Wax; Cavex, Haarlem, the Netherlands) specimens were invested in type III dental stone (Fujirock EP; GC, Leuven, Belgium) within a metal flask (61B Two Flask Compress; Handler Manufacturing,

Group	Code	Repair material			
Control	HC	Intact heat-polymerized specimens			
Butt joint	ВТАР	Unreinforced autopolymerized acrylic resin			
	2BTZ	2.5 wt% nano-ZrO ₂ -reinforced autopolymerized acrylic resin			
	5BTZ	5 wt% nano-ZrO ₂ -reinforced autopolymerized acrylic resin			
	7BTZ	7.5 wt% nano-ZrO ₂ -reinforced autopolymerized acrylic resin			
Bevel joint	BVAP	Unreinforced autopolymerized acrylic resin			
	2BVZ	2.5 wt% nano-ZrO2-reinforced autopolymerized acrylic resin			
	5BVZ	5 wt% nano-ZrO ₂ -reinforced autopolymerized acrylic resin			
	7BVZ	7.5 wt% nano-ZrO ₂ -reinforced autopolymerized acrylic resin			

Table I Specimens grouping and coding with specifications

Abbreviation: nano-ZrO₂, zirconia nanoparticles.

Westfild, NJ, USA). After the flasking procedure was completed, the dental wax was burned out and the mold spaces were cleaned of any wax traces by immersion in hot water and then allowed to dry. After that, a separating media (Isol Major; Major Prodotti Dentari Spa) was applied to dental stone surfaces and allowed to dry. According to the manufacturer's instructions, heat-polymerized acrylic resin was mixed and packed in the dough stage into the mold cavity; trial closures were then performed, and the flask was closed and kept under a bench press for 30 min. Acrylic resin specimens were processed for 8 h in a water bath at 74°C, and the temperature was then increased to 100°C for 1 h in a thermal curing unit (KaVo Elektrotechnisches Werk GmbH, Leutkirch, Germany). After polymerization, flasks were bench cooled at room temperature prior to deflasking. The excess resin of specimens was trimmed with a tungsten carbide bur (HM251FX-040-HP; Meisinger, Centennial, CO, USA) and polished with acrylic polisher (HM251FX-060; Meisinger, USA). For repair group specimens, each pair of respective plates was painted with petroleum jelly and subjected to the same flasking procedure as the control group. The flask was then opened, and the metal plates were removed, creating mold spaces that were cleaned, packed, processed, and finished as for intact specimens. A digital caliper (EK-1106B; China Hunan E&K Tools Inc., Changsha, China) was used to evaluate pairs of repair group specimens according to the required dimensions (Table 2). Control and repair groups were stored in distilled water at 37°C for 7 days.

Silanization of nano-ZrO₂ particles

The nano-ZrO₂ powder used (99.9% purity, 1314-23-4; Sigma-Aldrich Co., St Louis, MO, USA) had an average granularity of 90 nm and surface area of $9\pm 2 \text{ m}^2/\text{g}$. The addition of the silane coupling agent TMSPM (Shanghai Richem International Co., Ltd. Shanghai, China) to nano-ZrO₂ particles results in the creation of reactive groups on its surface, which allows for adequate adhesion between nanoparticles and the resin matrix.³³ To achieve this, 0.3 g of TMSPM was dissolved in 100 mL of acetone to ensure that it would evenly coat the surfaces of the ZrO₂ particles. Thirty grams of ZrO, particles were added to the TMSPM/acetone solution and stirred with a magnetic stirrer (Cimarec Digital Stirring Hotplates, SP131320-33Q; Thermo Fischer Scientific, Waltham, MA, USA) for 60 min. Subsequently, a rotary evaporator was used to remove the solvent under vacuum at 60°C and 150 rpm for 30 min. When the sample was dried, it was heated at 120°C for 2 h and naturally cooled to obtain the surfacetreated nano-ZrO2.26,34

PMMA/ZrO, nanocomposite preparation

The silanized nano-ZrO₂ particles were weighed using an electronic balance (S-234; Denver Instrument, Gottingen, Germany) and added in concentrations of 2.5 wt%, 5 wt%, and 7.5 wt% of autopolymerized acrylic polymer powder (Major. Repair, Major Prodotti Dentari Spa). The pre-weighted silanized nano-ZrO₂ was added to the autopolymerized

Molds	Flexural strength molds	Impact strength molds
Customized mold	65×10×2.5 mm ³	50×6×4 mm³ with V-shaped elevation and 0.8 mm height
Butt joint plate	Length =31.25 mm, width =10.0±0.01 mm, and	Length =23.75 mm, width = 6 ± 0.01 mm, and
	thickness =2.50±0.01 mm	thickness =4±0.01 mm
45 $^{\circ}$ bevel joint plate	Length of 31.25 mm for the lower surface and 30 mm for	Length of 23.75 mm for the lower surface and
	the upper surface with a 45° bevel, width =10.0 \pm 0.01 mm,	20 mm for the upper surface with a 45 $^\circ$ bevel,
	and thickness = 2.5 ± 0.01 mm	width =6 \pm 0.01 mm, and thickness =4 \pm 0.01 mm

 Table 2 Dimensions of molds and plates used for fabrication of specimens

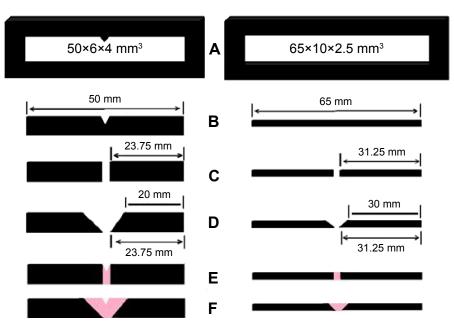


Figure I Molds (A), intact specimens (B), and modified plates (C, butt joint; D, bevel joint) used for fabrication of specimens and repair of specimens (E, butt surface design repair; F, bevel surface design repair).

acrylic polymer powder, thoroughly mixed, and stirred for 30 min to achieve an equal distribution of particles and to obtain a consistent and uniform color.^{22,35}

Repair procedures

The metal molds that were used to fabricate the control group's specimens were used to hold the reassembled specimens for repair. Repair surfaces were painted with monomer liquid and left for 3 min. Next, specimens were placed into the mold and fixed, preserving the required repair gap. The digital caliper was used to measure the proper dimensions of the repair area of each specimen and the whole length of each specimen. According to the manufacturer's recommendations, the mixed nanocomposite powder was dispersed in a methyl methacrylate monomer with a powder/liquid mass ratio of 2:1; then the subsequent material was mixed and packed into the repair area, adding an excess amount to compensate for polymerization shrinkage. Next, the molds holding the repaired specimens were placed into a pressure pot at a temperature of 37°C and subjected to 30 psi pressure for 30 min.²¹ After polymerization, the specimens were removed from the molds, and the excess acrylic resin was removed with a tungsten carbide bur at a low speed. Next, all specimens were finished, using a 600-grit abrasive paper under running water, and then stored in distilled water at 37°C for 48±2 h. Next, the specimens were subjected to the corresponding tests: flexural strength and impact strength (Table 1).

Flexural strength test

To determine flexural strength, fracture load was measured using a three-point bending test on a universal testing machine (Instron 8871; Instron Co., Norwood, MA, USA). The specimens were placed on a three-point flexure apparatus with a 50 mm distance between two supports. A 50 kgf load cell was applied at the midpoint of the repaired area with a crosshead speed of 5 mm/min until the specimen fractured. Fracture load was recorded. The formula $S = 3WL/2bd^2$ was used to calculate the flexural strength values of each specimen, where *S* is the flexural strength (MPa), *W* is the fracture load (N), *L* is the distance between the two supports, *b* is the specimen width, and *d* is the specimen thickness.^{10,32}

Impact strength test

An impact strength test was performed using a pendulum Charpy-type impact test machine (Digital Charpy Izod impact tester, XJU 5.5, Jinan Hensgrand Instrument Co., Ltd., Jinan, China). Each specimen was horizontally positioned with a distance of 40 mm between the two fixed supports. At room temperature, a drop weight of 0.5 J was applied at the mid-span of the specimen on the opposite side to the notch, and the value of the impact strength (kJ/m²) was digitally recorded.³²

Scanning electron microscope (SEM) examination

The samples were fixed on metal stubs and immersed in an ultrasonic bath of deionized water for 10 min. A sputtering

device was used to spatter the specimens with gold (one cycle of 120 sec) under vacuum. The fractured surfaces were analyzed using an SEM (Inspect S50, FEI Company; Hillsboro, OR, USA), evaluating the evenness of distribution along the interfaces between the acrylic resin matrix and the nano-ZrO₂ particles. The SEM unit operated at 20 kV, WD=15–18 mm, with a spot size range of 25–100 pA. Photomicrographs were made at ×100, ×200, ×400, ×600, ×1,000, ×1,200, ×1,600, and ×2,000 magnifications for visual inspection, and three observers noted whether the nature of the failure was cohesive (within the repair material only), adhesive (at the interface of the repair material and the repair materials).

Statistical analysis

SPSS software, version 20.0 (IBM Corporation, Armonk, NY, USA) was used for statistical data analysis. The results of the flexural strength and impact strength tests were presented as arithmetic mean and standard deviation (SD). For intragroup comparisons, in relation to the control versus various stages of flexural strength and impact strength, a paired sample *t*-test was used. For comparison between the butt and bevel joints, an unpaired *t*-test was used. A *P*-value ≤ 0.05 was considered a statistically significant result.

Results

Flexural strength

The mean and SD of flexural strength for the tested specimens are summarized in Table 3. The statistical analysis showed that the flexural strength of the control group was significantly better than all repaired groups (P<0.05), excepting the bevel group reinforced with 7.5% nano-ZrO₂ (P>0.05). Within each bevel group and butt group, there was a statistically significant difference between nano-ZrO₂-reinforced autopolymerized acrylic resin group and nano-ZrO₂-unreinforced autopolymerized group (P<0.05). Between reinforced groups, the bevel group's strength,

reinforced with 7.5% nano-ZrO₂ (91.43 MPa), was significantly higher than the other reinforced groups (P < 0.05). The lowest flexural strength value was found in the butt group reinforced with 2.5% nano-ZrO₂ (81.74 MPa). Furthermore, there were no significant differences between the butt and bevel groups reinforced with 5% nano-ZrO₂, while there was a significant difference between the 2.5% group and the 7.5% group (P < 0.05; Table 3).

Impact strength

Table 4 shows the mean and SD of impact strength for the tested groups. The mean values of all repaired groups were significantly lower than those of the control group (2.69 kJ/m²; P < 0.05). In the repaired groups, the lowest impact strength value was found in the bevel group reinforced with 7.5% nano-ZrO₂ (0.96 kJ/m²), while the highest value was seen in the butt group reinforced with 2.5% nano-ZrO, (1.70 kJ/m²). In comparison to the unreinforced autopolymerized acrylic resin group, a significant increase in the impact strength was found in the butt group reinforced with 2.5% nano-ZrO, particles, while a significant decrease was seen in the bevel group reinforced with 7.5% nano-ZrO₂ particles. Furthermore, no significant differences were found within the butt groups between the unreinforced autopolymerized group and the 5% nano-ZrO₂- or 7.5% nano-ZrO₂-reinforced autopolymerized acrylic resin group. There were no significant differences among the bevel groups between the unreinforced autopolymerized groups and the 2.5% nano-ZrO₂-reinforced resin or the unreinforced autopolymerized groups and the 5% nano-ZrO2-reinforced autopolymerized acrylic resin group.

Evaluation of SEM images

Representative SEM images showing the fractured surfaces of the control, bevel, and butt specimens are presented in Figure 2 for the flexural strength specimens and in Figure 3 for the impact strength specimens. Figure 2A (the control group)

Table 3 Mean	value and SD	of flexural	strength o	f tested s	specimens ((MPa)
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Sample	Control	nple Control Butt					Bevel			
		AP	2.5%	5%	7.5%	AP	2.5%	5%	7.5%	
Mean (SD)	92.43	53.29	81.74	85.32	84.51	54.75	86.91	87.64	91.43	
P-value		0.001ª	0.001ª	0.001ª	0.001ª	0.001ª	0.005ª	0.003ª	0.123ª	
		-	0.001 ^b	0.001 ^b	0.001 ^b	-	0.001 ^b	0.001 ^b	0.001 ^b	
		_	_	_	-	0.156°	0.020°	0.123°	0.001°	

Notes: 'Shows P-value of significance of mean effect of flexure strength of each repaired group compared to that of the control group. 'Shows P-value of significance of mean effect of flexure strength compared to that of the conventional group (AP) within each group (butt/bevel). 'Shows P-value of significance of mean effect of flexure strength compared between butt versus bevel groups.

Abbreviation: SD, standard deviation.

Sample	Control	Control Butt					Bevel			
		AP	2.5%	5%	7.5%	AP	2.5%	5%	7.5%	
Mean (SD)	2.69	1.26	1.70	1.37	1.27	1.46	1.52	0.98	0.96	
P-value		0.001ª	0.001ª	0.001ª	0.001ª	0.001ª	0.001ª	0.001ª	0.001ª	
		_	0.00 l ^b	0.063 ^b	0.938 ^b	_	0.461 ^b	0.652 [♭]	0.001 ^b	
		_	-	-	-	0.006 ^c	0.057 ^c	0.584 ^c	0.001 ^c	

Table 4 Mean value and SD of impact strength of tested specimens (J)

Notes: "Shows P-value of significance of mean effect of impact strength of each repaired group compared to that of the control group. "Shows P-value of significance of mean effect of impact strength compared to that of the conventional group (AP) within each group (butt/bevel). Shows P-value of significance of mean effect of impact strength compared between butt versus bevel groups.

Abbreviation: SD, standard deviation

shows a smooth surface with small pits displaying features of brittle fracture. Figure 2B shows fewer irregularities and dimpling. Figure 2C and D displays more irregular surfaces, and the smooth bases (non-uniform lamellar) exhibited ductile fracture features in addition to even distribution of nanoparticles filling all spaces, particularly at high concentrations. Figure 2E reveals fewer irregularities accompanied by rough surfaces, which increased as the nano-ZrO, concentration increased (Figure 2F). Figure 3A displays good surface characteristics, which changed with increasing nanoparticle concentrations, resulting in loosely bonded clusters and corresponding space formations (Figure 3B-D). Figure 3E and F shows images at high magnifications, displaying nanoparticle clusters and spaces. The fracture during the flexural strength specimens' analysis revealed that the bevel joint group suffered primarily cohesive failure, while the butt joint group presented primarily adhesive failure (Table 5).

Discussion

The choice of repair material depended primarily on the strength of the repair material, the repair surface design, and the choice of repair material reinforcement. Nano-ZrO, incorporation into a PMMA denture base resin had a significantly beneficial effect on the material's mechanical properties.^{36,37} The resin/filler interface adhesion was an important factor that affected PMMA/nano-ZrO₂ composite properties.²² Initially, it was assumed that the modification of nano-ZrO, particles with a silane coupling agent improved the bonding between reinforcement materials and the PMMA resin matrix, which consequently increased the PMMA/nano-ZrO, composite material's strength.²⁸⁻³⁰ The present in vitro study evaluated the effects of incorporation of nano-ZrO₂ particles into repair resin and the repair surface design on the flexural strength and impact strength of repaired denture base resins.

Flexural strength

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The results of this study showed that repairs using unreinforced autopolymerized resin revealed a significant decrease

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in flexural strength for both butt and bevel surface joints in comparison with the control group, with the exception of the bevel group reinforced with 7.5% nano-ZrO2. Otherwise, all the reinforced repaired groups demonstrated significantly increased flexural strength values in comparison to the unreinforced autopolymerized resin group. The present study revealed that reinforcement of a repaired denture base resin with nano-ZrO, resulted in an increase in its flexural strength. This study's findings were in agreement with the findings of a previous study that reported that the incorporation of nano-ZrO₂ into acrylic resins enhanced the flexural strength of the material.²² Similarly, Zhang et al investigated the performance of PMMA/ZrO2 nanocomposites, where they formulated a composite of nano-ZrO₂, aluminum borate whiskers, and PMMA to evaluate their effects on the flexural strength and surface hardness of the denture surface resin. They found that the composite material increased the flexural strength of the resin by >50%.³⁵ In this study, the addition of 2.5% nano-ZrO, particles, 5% nano-ZrO, particles, and 7.5% nano-ZrO, particles showed a statistically significant increase in flexural strength compared to the unreinforced autopolymerized resin. However, with the addition of 7.5% nano-ZrO₂ particles, the maximum flexural strength value was recorded, but it had no statistical significance compared to the control group. This increase in flexural strength could be attributed to nano-ZrO₂ particle sizes, their distribution within the repair material, and the silanization process, along with the joint's surface design. In addition, the transformation of ZrO₂ from the tetragonal to monoclinic phase resulted in absorbing the energy of crack propagation in a process called transformation toughening. In addition, during this process, the expansion of ZrO₂ crystals occurred and placed the crack under a state of compressive stress, which led to the arresting of crack propagation.³⁸ Based on the SEM analysis shown in Figure 2, this increase in flexural strength might be due to the good distribution of nano-size particles and interstitial filling of acrylic resin matrix with ZrO₂, which interrupted the crack propagation.27

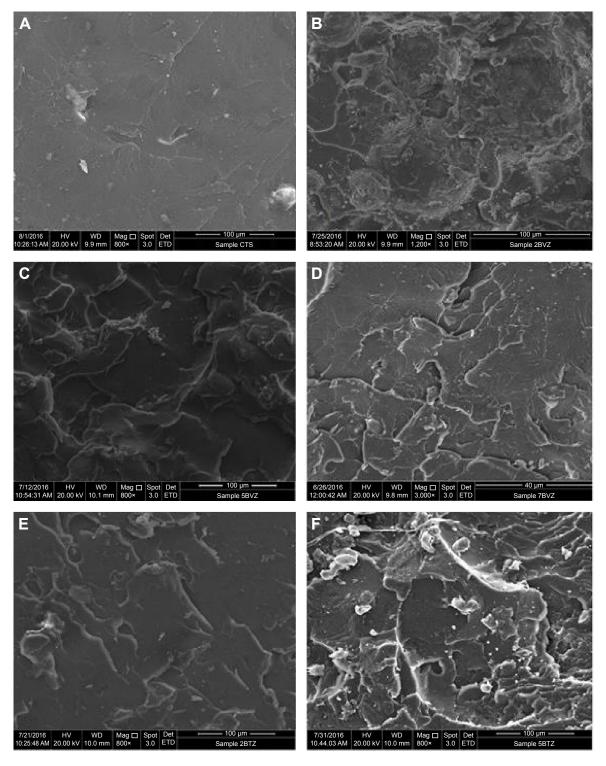


Figure 2 Representative SEM images of fractured surfaces of flexural strength specimens. Notes: (A) Control, (B) 2BVZ, (C) 5BVZ, (D) 7BVZ, (E) 2BTZ, and (F) 5BTZ. Abbreviation: SEM, scanning electron microscope.

The topography of the fractured surfaces of each specimen described by SEM was studied for changes according to nano-filler concentrations. Smooth surfaces exhibited brittle fracture characteristics, while more uniformly distributed irregularities exhibited ductile characteristics.²⁶ SEM analysis

in Figure 2B–D shows uniformly distributed irregularities with a dimpled appearance, representing ductile fractures.

The amount of filler used to reinforce acrylic resins and the filler-to-resin interactions were important factors affecting mechanical properties. The percentage of nano- ZrO_2

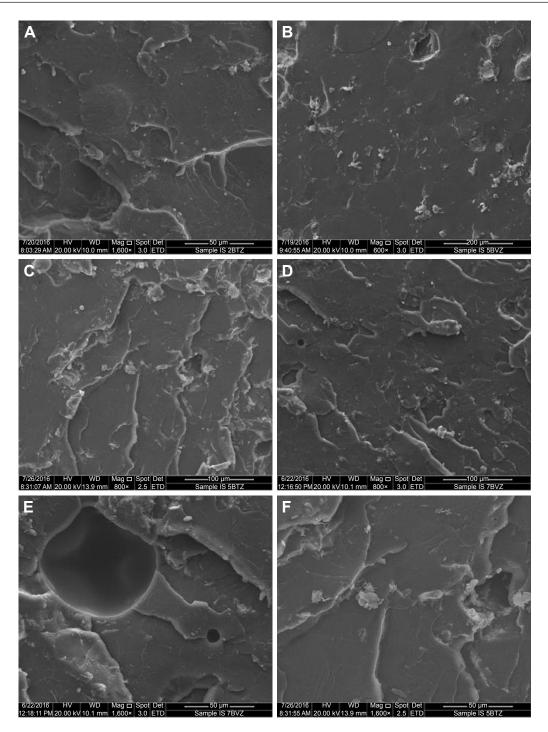


Figure 3 Representative SEM images of fractured surfaces of impact strength specimens. Notes: (A) 2BVZ, (B) 5BVZ, (C) 7BVZ, (D) 2BTZ, (E) 5BTZ, and (F) 7BTZ. Abbreviation: SEM, scanning electron microscope.

Sample	Butt			Bevel	Bevel			
Fracture mode	BTAP	2BTZ	5BTZ	7BTZ	BVAP	2BVZ	5BVZ	7BVZ
Cohesive		I	3	I.	8	8	7	6
Adhesive	9	9	7	9	2	I	I	2
Mixed	I					I	2	2

used must be dispersed evenly within the resin matrix without interrupting its continuity. Flexural strength was proportional to the nano-ZrO₂ percentage; the repair resin's flexural strength increased as the nano-ZrO₂ concentration increased. This finding was in agreement with some previous studies^{27,37} but in disagreement with others.^{28,33} These differences could be explained on the basis of studies having different methodologies or practices, such as dissimilar testing procedures, various nanoparticle concentrations, or different filler surface modifications.

Repair material and the repaired acrylic resin interface with the original material were another important factors that affected the overall repaired structure's mechanical properties. Moreover, the joint surface design has been proven to have a major impact on the strength of the repaired acrylic resin.^{18,19} Cohesive failure primarily occurred with the bevel joint design, demonstrating that beveling of repair surface design increased the flexural strength. This strength increase might be due to the 45° beveling, which increased the interface surface area and consequently provided a wide bond area. Mechanically, the 45° beveling might also shift the damaged area's tensile stress to the shear stress at the interface of the repaired specimens.^{19,21} On the other hand, the butt joint primarily experienced adhesive failure. The lack of adhesion could be attributed to the small surface area provided by the butt joint, which resulted in decreased flexural strength.³⁹

Impact strength

The results of the current study showed that all the repaired groups shared a significant decrease in their impact strength compared to the control group. Among the repaired groups, there was a significant increase in the impact strength with the 2.5% nano-ZrO₂ with a bevel-shaped repair, and there was a significant decrease with the 7.5% nano-ZrO₂ with a butt-shaped repair compared to the unreinforced autopolymerized repaired groups. This factor implied that the addition of 2.5% nano-ZrO₂ provided the nanocomposite with its maximum impact strength, but the addition of 5% nano-ZrO₂ and 7.5% nano-ZrO₂ reduced the impact strength values. The lowest mean value for impact strength occurred with the 7.5% nano-ZrO₂, which was similar to the results obtained by Asopa et al.40 This obvious reduction in impact strength may be due to the agglomeration of nano-ZrO₂ at 5 wt% and 7.5 wt%, which resulted in loosely bonded cluster formations, where crack propagation may occur and affect the impact strength.⁴¹ SEM analysis (Figure 3E-F) showed cluster formations and voids on both sides of fractures, which could explain the decreased impact strength.

Statistical analysis showed no significant difference between the reinforced bevel and butt groups, except for 7.5% nano- ZrO_2 with a bevel, which demonstrated significantly reduced impact strength. The existence of a V-notch confirmed that the specimens were broken at the same point during testing.^{31,42} Therefore, the surface design did not significantly affect the repairs' impact strength.

Based on the results of the present study, the incorporation of nano- ZrO_2 into repair resins may improve the repair strength of the material and increase the fracture resistance of the repaired denture base. This justifies the clinical importance of incorporation of nano- ZrO_2 into repair resins compared to unreinforced autopolymerized repair resins. Reinforcement materials, surface design, and repair technique were other major factors that primarily affected the repairs' strength. Therefore, the choice of repair reinforcement material in combination with the repair surface design was of major importance in obtaining the best mechanical properties for repair resins.

The limitations of this study included the fact that only two mechanical properties were investigated (flexural strength and impact strength) and only one type of nanoparticle composite material nano-ZrO₂ was evaluated as a repair material. For future research, other properties can be investigated and other nanoparticle materials can also be used for reinforcement of the repair material. In addition, the mechanical tests were not accomplished within wet environments, similar to the conditions inside the oral cavity. Future studies are required that evaluate the effects of nano-ZrO2 reinforcement on the properties of repaired denture base resins when specimens are stored in water or artificial saliva for a longer duration. In addition, clinical studies should be proposed that investigate the clinical performance of this nano-ZrO, material inside the oral cavity and its effects in cases of repeated denture fracture.

Conclusion

Within the limitations of this in vitro study, the incorporation of nano-ZrO₂ into a dental bridge repair material, combined with a bevel joint repair surface, improved the flexural strength of the repaired resin denture base. The impact strength of nano-ZrO₂-reinforced specimens increased with a low percentage (2.5%) of nano-ZrO₂ and decreased as the content of nano-ZrO₂ increased (5% and 7.5%).

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Disclosure

The authors report no conflicts of interest in this work.

References

- Meng TR, Latta MA. Physical properties of four acrylic denture base resins. J Contemp Dent Pract. 2005;6(4):93–100.
- Nejatian T, Johnson A, Van Noort R. Reinforcement of denture base resin. *Adv Sci Technol*. 2006;49:124–129.
- Raszewski Z, Nowakowska D. Mechanical properties of hot curing acrylic resin after reinforced with different kinds of fibers. *Int J Biomed Mat Res.* 2013;1:9–13.
- Beyli MS, von Fraunhofer JA. An analysis of causes of fracture of acrylic resin dentures. J Prosthet Dent. 1981;46(3):238–241.
- 5. Darbar UR, Huggett R, Harrison A. Denture fracture a survey. *Br Dent J.* 1994;176(9):342–345.
- Polyzois GL, Andreopoulos AG, Lagouvardos PE. Acrylic resin denture repair with adhesive resin and metal wires: effects on strength parameters. J Prosthet Dent. 1996;75(4):381–387.
- Seiko R, Hermana K, Neudenir J. Factors affecting the strength of denture repairs. J Prosthodont. 2007;16(4):302–310.
- Craig RG, Powers JM. Restorative Dental Materials. St Louis: Elsevier, 2001:665–666.
- Gregory L, Mary J. Fracture force, deflection at fracture, and toughness of repaired denture resin subjected to microwave polymerization or reinforced with wire or glass fiber. *J Prosthet Dent*. 2001;54:75–78.
- Alkurt M, Yeşil Duymuş Z, Gundogdu M. Effect of repair resin type and surface treatment on the repair strength of heat-polymerized denture base resin. J Prosthet Dent. 2014;111(1):71–78.
- Faot F, da Silva WJ, da Rosa RS, et al. Strength of denture base resins repaired with auto- and visible light polymerized materials. *J Prosthodont*. 2009;18(6):496–502.
- Beyli MS, von Fraunhofer JA. Repair of fractured acrylic resin. J Prosthet Dent. 1980;44(5):497–503.
- Agarwal M, Nayak A, Hallikerimath RB. A study to evaluate the transverse strength of repaired acrylic denture resins with conventional heat-cured, autopolymerizing and microwave-cured resins: an in vitro study. J Ind Prosthodont Soc. 2008;8:36–41.
- Anusavice KJ. *Philips Science of Dental Materials*. Bangalore, India: Prism Saunders, Prism book Pvt Ltd; 2002:124–136.
- Murphy WM, Huggett R, Handley RW, et al. Rigid cold curing resins for direct use in the oral cavity. *Br Dent J.* 1986;160(11):391–394.
- Kostoulas I, Kavoura VT. Fracture force, deflection, and toughness of acrylic denture repairs involving glass fiber reinforcement. *J Prosthodont*. 2008;17(4):257–261.
- Minami H, Suzuki S, Minesaki Y, Kurashige H, Tanaka T. In vitro evaluation of the influence of repairing condition of denture base resin on the bonding of autopolymerizing resins. *J Prosthet Dent.* 2004;91(2):164–170.
- Harrison WM, Stansbury BE. The effect of joint surface contours on the transverse strength of repaired acrylic resin. *J Prosthet Dent.* 1970;23: 464–471.
- Ward JE, Moon PC, Levine RA, Behrendt CL. Effect of repair surface design, repair material, and processing method on the transverse strength of repaired acrylic denture resin. *J Prosthet Dent*. 1992;67(6): 815–820.
- American dental association specification no. 12 for denture base polymers. J Am Dent Assoc. 1975;90(2):451–458.

- Hanna EA, Shah FK, Gebreel AA. Effect of joint surface contours on the transverse and impact strength of denture base resin repaired by various methods: an in vitro study. *J Am Sci.* 2010;6(9):115–125.
- Ayad NM, Badawi MF, Fatah AA. Effect of reinforcement of high impact acrylic resin with micro-zirconia on some physical and mechanical properties. *Rev Clin Pesq Odontol Curitiba*. 2008;4(3):145–151.
- Asar NV, Albayrak H, Korkmaz T, Turkyilmaz I. Influence of various metal oxides on mechanical and physical properties of heat-cured polymethyl methacrylate denture base resins. *J Adv Prosthodont*. 2013; 5(3):241–247.
- Shuai C, Feng P, Yang B, Cao Y, Min A, Peng S. Effect of nano-zirconia on the mechanical and biological properties of calcium silicate scaffolds. *Int J Appl Ceramic Technol.* 2015;12:1148–1156.
- Mallineni SK, Nuvvula S, Matinlinna JP, Yiu CK, King NM. Biocompatibility of various dental materials in contemporary dentistry: a narrative insight. *J Invest Clin Dent*. 2013;4(1):9–19.
- Yu W, Wang X, Tang Q, Guo M, Zhao J. Reinforcement of denture base PMMA with ZrO2 nanotubes. *J Mech Behav Biomed Mater*. 2014;32:192–197.
- Ahmed MA, Ebrahim MI. Effect of zirconium oxide nano-fillers addition on the flexural strength, fracture toughness, and hardness of heatpolymerized acrylic resin. *World J Nano Sci Eng.* 2014;4(2):50–57.
- Ihab NS, Moudhaffar M. Evaluation the effect of modified nano-fillers addition on some properties of heat cured acrylic denture base material. *J Bagh Coll Dentistry*. 2011;23(3):23–29.
- Ihab NS, Hassanen KA, Ali NA. Assessment of zirconium oxide nanofillers incorporation and silanation on impact, tensile strength and color alteration of heat polymerized acrylic resin. *J Bagh Coll Dentistry*. 2012; 24(4):36–42.
- Erjun T, Cheng G, Pang X, et al. Synthesis of nano-ZnO₂/PMMA composite micros sphere through emulsion polymerization and UV-Shielding property. *J Colloid Polymer Sci*. 2005;284(4):422–428.
- Radzi Z, Abu Kasim NH, Yahya NA, et al. Impact strength of an experimental polyurethane-based polymer. *Annal Dent Univ Malaya*. 2007; 14:46–51.
- ISO 1567. Dentistry Denture Base Polymer. Geneva: International Organization for Standardization; Geneva, Switzerland. 1999:1–27.
- Hameed HK, Abdul Rahman H. The effect of addition nano particle ZrO₂ on some properties of autoclave processed heat cures acrylic denture base material. *J Bagh Coll Dentistry*. 2015;27(1):32–39.
- Yoshida K, Greener EH. Effects of coupling agents on the mechanical properties of metal oxide polymethacrylate composites. *J Dent.* 1994;22(1):57–62.
- Zhang XY, Zhang XJ, Huang ZL, Zhu BS, Chen RR. Hybrid effects of zirconia nanoparticles with aluminum borate whiskers on mechanical properties of denture base resin PMMA. *Dent Mater J.* 2014; 33(1):141–146.
- Korkmaz T, Dogan A, Usanmaz A. Dynamic mechanical analysis of provisional resin materials reinforced by metal oxides. *Biomed Mater Eng.* 2005;15(3):179–188.
- Gad M, ArRejaie AS, Abdel-Halim MS, Rahoma A. The reinforcement effect of nanozirconia on the transverse strength of repaired acrylic denture base. *Int J Dentistry*. 2016;2016:1–6. Article ID 7094056.
- Kenneth JA. *Phillips' Science of Dental Materials*. Philadelphia, PA: Saunders; 2003:703–704.
- Sarac YS, Sarac D, Kulunk T, Kulunk S. The effect of chemical surface treatments of different denture base resins on the shear bond strength of denture repair. *J Prosthet Dent*. 2005;94(3):259–266.
- 40. Asopa V, Suresh S, Khandelwal M, Sharma V, Asopa SS, Kaira LS. A comparative evaluation of properties of zirconia reinforced high impact acrylic resin with that of high impact acrylic resin. *Saudi J Dental Res.* 2015;6(2):146–151.
- 41. Sun L, Ronald FG, Suhr J, et al. Energy absorption capability of nano composites: a review. *Composites Sci Technol*. 2009;69(14):2392–2409.
- 42. Shah SA, Khan S, Gulzar S, et al. A research study to compare the flexural strength and impact strength of different heat cure and chemical cure acrylic resins under various conditions. *Int J Health Sci Res.* 2015; 5(6):325–329.

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