

Impact of ZrO₂ nanoparticles addition on flexural properties of denture base resin with different thickness

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Received May 23, 2021 / Last Revision July 27, 2021 / Accepted August 9, 2021 **PURPOSE.** This study aimed to evaluate the effect of incorporating zirconium oxide nanoparticles (nano-ZrO₂) in polymethylmethacrylate (PMMA) denture base resin on flexural properties at different material thicknesses. MATERIALS AND METHODS. Heat polymerized acrylic resin specimens (N = 120) were fabricated and divided into 4 groups according to denture base thickness (2.5 mm, 2.0 mm, 1.5 mm, 1.0 mm). Each group was subdivided into 3 subgroups (n = 10) according to nano-ZrO₂ concentration (0%, 2.5%, and 5%). Flexural strength and elastic modulus were evaluated using a three-point bending test. One-way ANOVA, Tukey's post hoc, and two-way ANOVA were used for data analysis (α = .05). Scanning electron microscopy (SEM) was used for fracture surface analysis and nanoparticles distributions. RESULTS. Groups with 0% nano-ZrO₂ showed no significant difference in the flexural strength as thickness decreased (P = .153). The addition of nano-zirconia significantly increased the flexural strength (P < .001). The highest value was with 5% nano-ZrO₂ and 2 mm-thickness (125.4) \pm 18.3 MPa), followed by 5% nano-ZrO $_2$ and 1.5 mm-thickness (110.3 \pm 8.5 MPa). Moreover, the effect of various concentration levels on elastic modulus was statistically significant for 2 mm thickness (P = .001), but the combined effect of thickness and concentration on elastic modulus was insignificant (P = .10). **CONCLUSION.** Reinforcement of denture base material with nano-ZrO₂ significantly increased flexural strength and modulus of elasticity. Reducing material thickness did not decrease flexural strength when nano-ZrO₂ was incorporated. In clinical practice, when low thickness of denture base material is indicated, PMMA/nano-ZrO₂ could be used with minimum acceptable thickness of 1.5 mm. [J Adv Prosthodont 2021;13:226-36]

KEYWORDS

Nano-fillers; PMMA denture base; Reinforcement; Flexural strength; Denture base thickness

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INTRODUCTION

Polymethylmethacrylate (PMMA) is the material of choice for complete denture fabrication due to its ease of handling, light weight and good aesthetics.1 It has been recommended to fabricate dentures at a minimum thickness of 2.5 mm,² as denture base thickness may influence the mechanical properties of PMMA.³ Denture thickness commonly causes patient discomfort, especially for first-time wearers, as it may cause tongue restriction, distorted speech, and activation of the gag reflex.4 This results in frequent patients' complaints that contribute to the limited overall satisfaction with removable dentures. Decreasing the denture base thickness may improve patient's comfort and improve adaptation. However, it decreases the flexural strength of PMMA and therefore increases the risk of fracture.⁵

Tooth- and implant-supported overdentures are commonly used to rehabilitate the edentulous mandible. However, fracture is a common clinical occurrence⁶ that usually occurs after repeated flexing of the denture under small loads. Abutments occupy the prosthetic space inside the denture resin base causing the surrounding acrylic resin to be thin and susceptible to fracture.8 Furthermore, the addition of denture teeth to simulate clinical conditions has been shown to further decrease the strength of the denture base resin.9 Metal reinforcements have been proposed to increase overdenture resistance to fracture. 6,10-12 However, they have poor adhesion to acrylic resin and may produce stress concentration, which could actually weaken rather than reinforce overdentures.12

Nanoparticles have been used to improve some of the mechanical properties of PMMA.¹³ Amongst nanoparticles, zirconium oxide nanoparticles (nano-ZrO₂) have particularly been shown to improve mechanical properties.^{14,15} The incorporation of nano-ZrO₂ into a heat-polymerized denture base material increases the material's flexural strength, tensile strength, hardness, and fracture toughness, especially when incorporated in the high concentrations.¹⁴⁻¹⁶ Moreover, the properties of the resulting nanocomposite are dependent on the concentration of the nanofillers.¹³ It has been reported that a concentra-

tion of 5 wt% ZrO₂ nanoparticles resulted in the maximum increase in the physical and mechanical properties; impact strength and fracture toughness are significantly improved, with a noticeable reduction in water sorption and solubility. Additionally, good adhesion and homogenous distribution of the nano fillers within the resin matrix effectively improve the nanocomposite properties. The use of silane coupling agent to treat the nano-ZrO₂ surface may reduce the nano filler agglomeration and improve the distribution within the polymer matrix. 17,18

To the authors' knowledge, no study has investigated the effect of incorporating nano-ZrO₂ on denture base resins with reduced thickness, and the minimum acceptable denture base thickness with appropriate flexural properties is unknown. Therefore, this *in vitro* study aimed to evaluate the effect of nano-ZrO₂ on the flexural strength and elastic modulus of denture base material with different thickness. The null hypothesis was that the addition of nano-ZrO₂ to PMMA denture base material would have no effect on the flexural strength and elastic modulus of different denture base thicknesses.

MATERIALS AND METHODS

Nano-ZrO₂ particles (Shanghai Richem International Co., Ltd., Shanghai, China) were treated with a 3-(Trimethoxysilyl)propyl methacrylate silane (TMSPM, Sigma Aldrich, St. Louis, MO, USA) silane coupling agent to form a reactive group that facilitates bonding with a resin matrix as described in previous studies.^{17,19-24} The treated nano-ZrO₂ was weighed using an electronic balance (Motorized Analytical Balance Scale, Denver Instrument, Bohemia, NY, USA) and placed in 2.5% or 5% concentration within a heat-polymerized acrylic powder. A vigorous mix was performed using an electronic mixer to achieve an even distribution of the nanoparticles within the acrylic powder.

The sample size was determined by World Health Organization formula, 25 which revealed that 10 specimens per group would be sufficient to detect the postulated effect size. As shown in Fig. 1, the control group specimens were fabricated with dimensions according to ADA No.12 (65 \times 10 \times 2.5 mm) 2 , while the other three groups were fabricated with the simi-

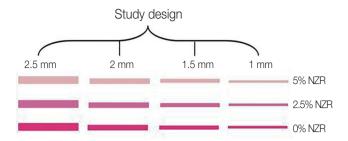


Fig. 1. Illustrated figure for specimen thickness and nano- ZrO_2 concentrations.

lar dimensions but with varying thicknesses (2.0 mm, 1.5 mm, or 1.0 mm). Each group was further subdivided into three subgroups with different nano-ZrO₂ concentrations (0%, 2.5% and 5%). Heat-polymerized acrylic resin (Major.Base.20, Major Prodotti Dentari, Moncalieri, Italy) specimens were fabricated by investing wax specimens in dental stone within flasks, followed by wax elimination (WAPO-Ex 12 II, Wassermann, Hamburg, Germany) creating mold spaces. According to manufacturer's instructions, the polymer and monomer were mixed and packed at the dough stage after applying separating medium (Isolmajor, Major Prodotti Dentari, Moncalieri, Italy) over the stone surfaces. Polymerization was done in a water bath curing unit (KaVo Elektrotechnisches Werk, Leutkirch, Germany) at 74°C for eight hours followed by one hour at 100°C. After complete polymerization, specimens finishing was done using a straight handpiece with a tungsten carbide bur (HM251FX-040 HP, Meisinger, Centennial, CO, USA). Polishing was done using a Polishing Compact Unit (WP-Ex 2000 II, Wassermann, Hamburg, Germany) with pumice and green wax. A caliper was used to evaluate the proper specimens' dimensions. Approved specimens were stored in distilled water at 37°C for one week prior to testing.

The flexural strength was evaluated using a three-point bending test that was performed at room temperature using a universal testing machine (Instron, ElectroPlus™, E3000, Buckinghamshire, UK). Each specimen was placed horizontally on a three-point flexure apparatus with a 50 mm distance between two vertical supports. The vertical load was applied midway between the supports at a 5 mm/min cross-

head speed until the specimen fractured. The fracture load was recorded. The following formula was used to calculate the flexural strength values of each specimen²⁶:

$$FS = 3FL / 2bh^2$$

where FS indicates flexural strength (MPa), F is fracture load (N), L is the distance between the two supports, b is specimen width, and h is specimen thickness.

The results from the flexural strength test were used to calculate the elastic modulus using the formula²⁶

$$E = FL3 / 4bh3d$$

where the variables consist of the following: E is the elastic modulus (MPa), F is the load (N) at a convenient point (p) in the straight line of the tension/deformation curve (elastic deformation), L is the distance between the two supports, b is specimen width, h is specimen thickness, and d is the deflection at the point (p).

After the flexural experiments, the fractured specimens were gold coated for SEM (VEGA3, TESCAN, Brno, Czech Republic) analysis at an accelerating voltage of 20 kV with different magnifications (i.e. ×200, ×500 and ×1000). The details of SEM preparation are described in previous studies. 17,27 The results were displayed at a representative magnification of ×1000 for 0%, 2.5% and 5% nanofiller for specimens of 2.5 mm, 1.5 mm, and 1 mm thickness. Prior to their addition into the PMMA for heat polymerization, the size, shape and structure of the nano-ZrO₂ powder were also analyzed by using SEM and TEM (Morgagni 268, FEI, Brno, Czech Republic) at 80 kV. To draw the size histogram, several particles were chosen from the TEM images, their sizes were measured, and the average size of the particles was calculated (Fig. 2). After mixing of the nanofiller, the PMMA/nano-ZrO₂ mixture was observed via SEM to verify the presence of the nanofiller and its distribution into the PMMA powder prior to heat polymerization. For comparison, the pure PMMA powder was also examined and displayed along with the mixture specimen (Fig. 3). In addition, energy-dispersive X-ray (EDX) spectroscopy was performed via SEM to further confirm the presence of the Zr-element in the PMMA matrix.

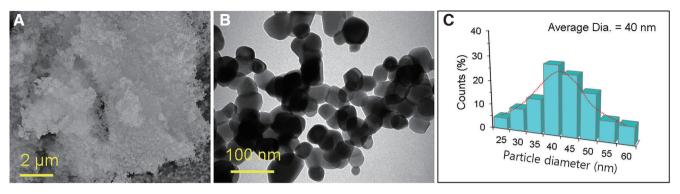


Fig. 2. (A) SEM micrograph (\times 25,000) of nano-ZrO₂ powder, (B) TEM image (\times 180,000) of nano-ZrO₂ powder, and (C) size histogram of nano-ZrO₂ particles.

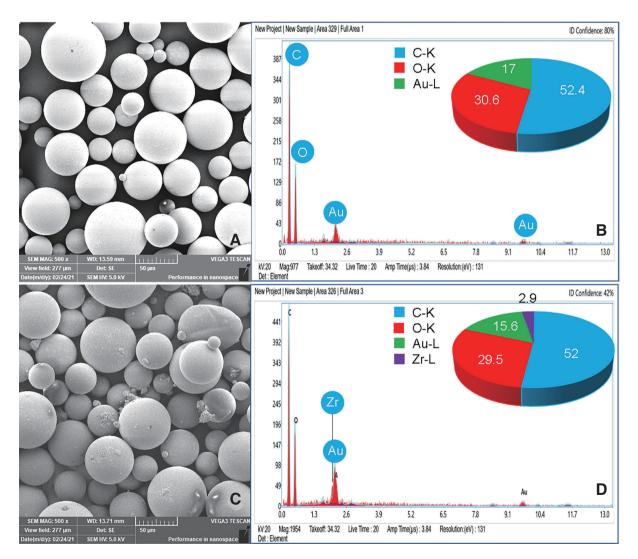


Fig. 3. A-D. SEM image (×500) and corresponding EDX spectrum of the pure PMMA and the mixtures. (A, B) Pure PMMA powder and (C, D) PMMA/nano-ZrO₂ mixture. Note: Au peaks appeared from gold coating.

Fourier-transform infrared spectroscopy (FTIR, Nicolet 6700, Thermo Fisher Scientific, Waltham, MA, USA) was performed to analyze the interaction and bonding between the nano-ZrO₂ particles and the PMMA resins reinforced with 0%, 2.5%, and 5% nano-ZrO₂. The heat-polymerized specimens were placed in the infrared cell, and the spectra were acquired at the entire infrared region (4000 - 500 cm⁻¹). The plots are presented in Fig. 4.

The Statistical Package for the Social Sciences (SPSS v.23, IBM, Chicago, IL, USA) was used for analysis. Means and standard deviations were computed under descriptive analysis. Wilk test was used to check normality and insignificant results revealed that the data were normally distributed. Hence, one-way ANO-VA was used to study the variation in means due to changes in thickness, or concentration of nano-ZrO₂ (separately) on flexural properties. Tukey's post hoc test was used with significant results from one-way ANOVA. Furthermore, two-way ANOVA was used to study the combined effect of thickness and concentration on the flexural strength and elastic modulus. The level of significance was set at 0.05.

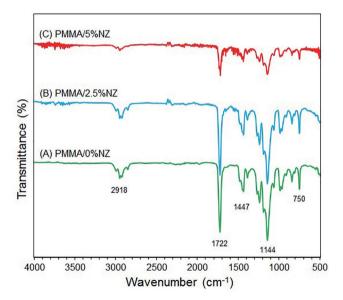


Fig. 4. FTIR of heat-polymerized PMMA resins reinforced with nano-ZrO₂ (0, 2.5 and 5 % NZ) in the spectral range of 4000 - 500 cm⁻¹. The main bands are labelled.

RESULTS

The mean, SD, and significance among groups regarding the flexural strength and elastic modulus of tested groups are summarized in Table 1 and Table 2. During flexural strength testing, specimens of the 1.0 mm thickness group did not fracture within the end limits of the penetrant's possible movement; thus, the values of flexural strength for this group could not be measured. Variation in flexural strength due to change in denture base thickness was found to be statistically insignificant among groups with 0% na $no-ZrO_2$ incorporated (P = .153), while a significant difference was noted at nano-ZrO2 concentrations of 2.5% (P = .02) and 5% (P < .01). Pairwise comparison showed that at the 2.5% level of concentration, differences in means in pairs (2.5 mm vs 2 mm; 2 mm vs 1.5 mm) were statistically insignificant (P = .210 and P = .210.422, respectively). In groups with nano-ZrO₂ concentration of 5%, the difference between each pair was significant (P < .001) Similarly, the effect of variation in concentration levels was tested at a given level of thickness (Table 1). The effect of various concentration levels on flexural strength for all thicknesses was statistically significant (P < .001). However, pairwise comparison provided an insignificant difference between 2.5% and 5% concentration at 2.5 mm thickness (P = .98) and between 0% and 2.5% at 2.0 mm thickness (P = .068).

Variation in elastic modulus due to change in denture base thickness was statistically insignificant at 5% concentration, while variation in thickness had a significant effect on flexural strength at the 0% and 2.5% concentration levels. All pairwise comparisons yielded statistically significant results. The effect of variation in concentration levels was tested at a given level of thickness (Table 2). The effect of various concentration levels on elastic modulus among the 2.0 mm thickness groups was significant (P = .001) while for the 2.5 mm and 1.5 mm groups, the difference was insignificant. The pairwise comparison for the 2.0 mm thickness specimens showed a significant difference, except between the groups with 0% and 2.5% concentration (P = .954).

Table 3 shows the combined effect of thickness and concentration on flexural strength and elastic mod-

Table 1. Mean, SD, and significance between groups regarding flexural strength of tested groups

NZR concentration		<i>P</i> value		
	2.5 mm	2 mm	1.5 mm	P value
0% NZR	84.5 (4.4)	74.7 (8.4)	68.6 (9.4)	.153
2.5% NZR	93.1 (2.7)a,A	97.9 (8.6)a,b,	101.4 (5.7) ^b	.020*
5% NZR	93.4 (2.8) ^A	125.4 (8.3)	110.3 (8.5)	.000*
<i>P</i> value	.000*	.000*	.000*	

^{*}statistically significant at 0.05 level of significance.

Same small alphabetical letter showed insignificant difference in each row. Same capital alphabetical letter showed insignificant difference in each column.

Table 2. Mean, SD, and significance between groups regarding elastic modulus of tested groups

NZR concentration		<i>P</i> value		
	2.5 mm	2 mm	1.5 mm	P value
0% NZR	8011 (672.6)	5640.6 (808.2) ^A	6858.9 (1049.5)	.000*
2.5% NZR	8105 (380.4)	5507.3 (625.2) ^A	6666.5 (900.8)	.000*
5% NZR	8265.1 (573.9)a	7322.1 (1444.6) ^a	7477.7 (1867.3) ^a	.289
<i>P</i> value	.594	.001*	.382	

^{*}statistically significant at 0.05 level of significance.

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Table 3. Two-way ANOVA analysis for combined effect of thickness and concentration over flexural strength

Property	Source	Sum of square	df	Mean square	F-value	<i>P</i> value
Flexural strength	Thickness	2285.3	2	1142.6	14.6	.000*
	Concentration	11065.5	2	5532.7	70.6	.000*
	Thickness*concentration	3437.3	4	859.3	10.9	.000*
	Error	6343.9	81	78.3		
	Total	862495.6	90			
Elastic Modulus	Thickness	58666886.9	2	29333443.4	27.9	.000*
	Concentration	15924695.0	2	7962347.5	7.7	.001*
	Thickness*concentration	8458629.3	4	2114657.3	2.0	.10
	Error	85119405.5	81	1050856.8		
	Total	4698803050	90			

^{*}statistically significant at 0.05 level of significance.

ulus was tested via two-way ANOVA. The effect of thickness and concentration on flexural strength was found to be statistically significant (P < .001). However, for elastic modulus, the combined effect of thickness and concentration was statistically insignificant (P = .10)

The FTIR spectra of the heat-polymerized specimens are shown in Fig. 4; the specimens are rein-

forced with 0%, 2.5%, or 5% nano-ZrO₂ filler. The FTIR spectra showed the main characteristics bands associated with the PMMA resins: an asymmetric stretching band of the methyl (CH₃) group (~ 2918 cm⁻¹), the deformation modes of the CH₃ group (1447 and 1387 cm⁻¹), and CH₃ twisting (1144 cm⁻¹). Furthermore, the spectra revealed the characteristic ester carbonyl (C=O) stretching vibrations of PMMA (an intense band

at ~1722 cm⁻¹), C–O stretching modes at ~1239 cm⁻¹ and in- and out-of-plane bending of C=O at 750 cm⁻¹. The addition of nano-ZrO₂ particles into the PMMA matrix brought no shifting in the band position of the characteristic PMMA bands, only the intensity of the certain bands varied with the addition of nano-ZrO₂. The bands with minimum peak intensity were observed for the PMMA/5% nano-ZrO₂ specimen. The FTIR spectra confirmed the incorporation of nano-ZrO₂ nanoparticles in the PMMA matrix, as well as their interaction with PMMA molecules.

According to the SEM findings, the pure PMMA specimens displayed faint lamellae with smooth back-

ground (Fig. 5A, B) and these lamellae diminished with a mirror-like appearance in 1.5 mm specimens (Fig. 5C), representing a brittle fracture type. The behavior of all thicknesses (2.5 mm, 2 mm, and 1.5 mm) with the addition of nano-ZrO₂ (2.5% and 5%) displayed the same features as shown in Fig. 5. The obvious difference was the presence of the nanofiller on the surface for the specimens those reinforced with nano-ZrO₂. As expected, the density of the particles was greater for 5% nano-ZrO₂ compared to 2.5% nano-ZrO₂ specimens (Fig. 6, Fig. 7). Dramatic changes in the surface topography, such as an irregular and rough surface and multiple small sharp lamellae,

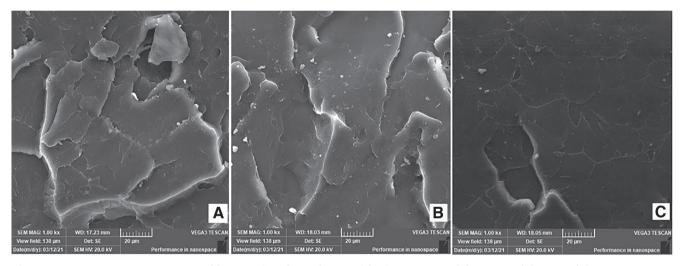


Fig. 5. Representative SEM images of fractured surface of unmodified specimens. (A) 2.5 mm, (B) 2 mm, (C) 1.5 mm.

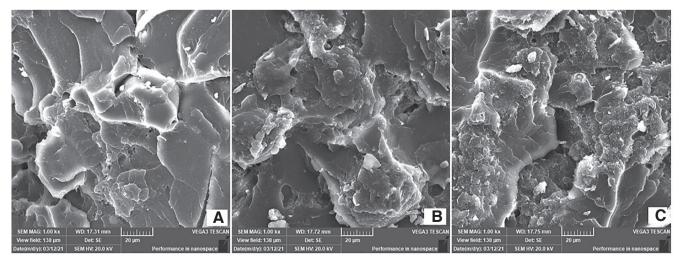


Fig. 6. Representative SEM images of fractured surface of 2.5% nano-ZrO₂-modified specimens. (A) 2.5 mm, (B) 2 mm, (C) 1.5 mm.

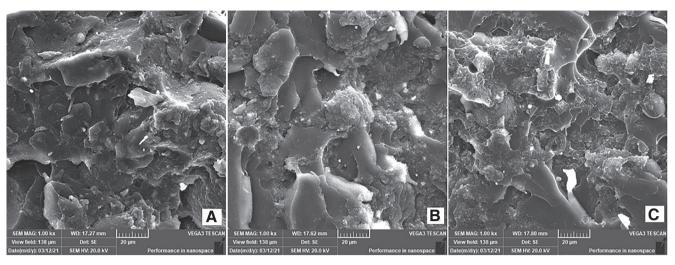


Fig. 7. Representative SEM images of fractured surface of 5% nano-ZrO₂-modified specimens. (A) 2.5 mm, (B) 2 mm, (C) 1.5 mm.

were noted for 2.5% and 5% nano-ZrO₂ specimens in comparison to pure PMMA specimens, representing intermediate to ductile fracture in addition to the good distribution of nano-ZrO₂ and the absence of voids or clusters formation.

DISCUSSION

The aim of this study was to evaluate the effect of nano- ZrO_2 reinforcement on PMMA denture base material made with different thicknesses in an attempt to determine the minimum acceptable denture base thickness without adverse effects on flexural strength. The null hypothesis was rejected as the addition of nano- ZrO_2 to the PMMA denture base material with different denture base thicknesses affected the flexural strength and elastic modulus.

The denture base is exposed to flexural stress during function as it is typically supported by unequal alveolar ridges caused by the different patterns of bone resorption. Hence, the denture base should have acceptable flexural strength to prevent fracture during flexural loading to prolong its performance.²⁷ Reducing the denture base thickness to less than 2.0 mm has been reported to adversely affect flexural strength as the load bearing capacity decreases.^{9,28,29} In this study, the addition of nano-ZrO₂ to PMMA denture base material was shown to provide significant

improvement in the flexural strength even when the specimen's thickness was decreased. Incorporating nano-ZrO₂ has been previously reported to improve flexural strength values, 17,30 which could be attributed to the fact that nanoparticles may fill the spaces between polymeric chains. Bonding nano-ZrO₂ with the resin matrix may also have a silanization effect, thus increasing the interfacial shear strength between the polymeric chains. Furthermore, the transformation toughening process of nano-ZrO₂ may help in the absorption of crack propagation energy resulting in greater fracture resistance.¹⁷ These positive effects were directly proportional to the concentration of nano-ZrO₂ up to 5%, which is in agreement with previous studies.14,31 A higher concentration of nanoparticles could have a contrasting effect possibly due to the creation of clusters that cause discontinuity of the resin matrix.14

A previous study reported a substantial decrease in denture base strength when the thickness is less than 2.0 mm.⁹ The results of the present study show that a denture base material of 2 mm thickness has the highest flexural strength when it is reinforced with 5% nano-ZrO₂. However, specimens with 1.5 mm thickness and nano-ZrO₂ reinforcement had a significantly higher flexural strength than that of the control specimens. When thickness was reduced to 1.0 mm, all specimens were bent during 3-point bending test

until they came in contact with the base between the two supports with no fracture; therefore, the flexural strength values could not be recorded. These specimens may have undergone plastic deformation rather than fracture due to the decreased thickness, as it has been previously reported that reducing thickness of PMMA increases fracture toughness³² but decrease resistance to deformation.⁹

Maxillary dentures frequently have midline fractures due to continuous flexion of the denture. 6 Thus, the denture base should have satisfactory elastic modulus and flexural properties to avoid fracture and prevent permanent deformation of the denture base material caused by continuous stress or strain during mastication.³³ The elastic modulus of the denture base resin is expected to decrease as the denture base thickness decreases. The addition of 2.5% nano-ZrO₂ did not significantly alter the elastic modulus when compared with the control group, and the reduction in thickness resulted in a significant decrease. However, with 5% nano-ZrO₂ concentration, the elastic modulus was similar regardless of specimen thickness. This finding is in agreement with a previous study that reported an increase in elastic modulus value as the reinforcement material increases.34 It is worth mentioning that elastic modulus values decreased when the thickness was reduced to 2 mm, then increased at 1.5 mm thickness. Although all values were within the clinically acceptable limit, this warrants further study to investigate the mechanical behavior of PMMA at variable thicknesses.

Some situations indicate a thin denture base, such as tooth- or implant-supported overdenture, where the part over the abutment is weak and likelier to break. 9,28 Also, in cases where the patient has a sensitive gag reflex, the reflex will be stimulated by the thick maxillary denture base at the posterior area. 4,26 Moreover, some patients may encounter tongue restriction and distorted speech due to the thick denture base. 26 Therefore, the results of this study may be significant in daily clinical practice. When a minimal thickness of the denture base material is indicated, the PMMA/nano-ZrO₂ resin could be fabricated with minimum acceptable thickness of 1.5 mm. This can also be helpful for patients with overdentures who repeatedly have their dentures broken due to limited

prosthetic space.

The limitations of the present study include *in vitro* design, which inherently lacks the challenges of the oral cavity such as fluids, masticatory stress, and irregular configuration of dentures following the underlying ridges. The materials tested in this study were also not subjected to ageing procedures such as thermal or pH cycling. Future studies should be directed towards testing different materials, evaluating other properties and exposing materials to artificial ageing to investigate long-term outcomes.

CONCLUSION

The present study showed that decreasing the thickness of the denture base material significantly decreased flexural strength. Reinforcement with nano-ZrO₂ increased the flexural strength of PMMA resin, even with decreased denture base thickness. Decreasing the denture base thickness had a minimal impact on elastic modulus when the thickness was reduced from 2.5 mm to 1.5 mm. The results are promising for PMMA/nano-ZrO₂ to be used clinically with dentures at a reduced thickness.

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