

Comparative Evaluation of Flexural Strength of Heat-Cured PMMA Reinforced with Nano-Zirconium Oxide and Nano-Zirconium Oxide–Glass Fiber Combination: An In-Vitro Study

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ABSTRACT: Polymethylmethacrylate (PMMA) is widely used for denture bases but has limited flexural strength. This in-vitro study aimed to compare the flexural strength of heat-cured PMMA reinforced with 5 wt% nano-zirconium oxide (ZrO₂) and a combination of 2.5 wt% nano-ZrO₂ and 2.5 wt% E-glass fibers. Forty standardized specimens were equally divided into two groups (n = 20), fabricated using heat-cured PMMA, surface treated with TMSPM, and polymerized under controlled conditions. After water storage, flexural strength was evaluated using a three-point bending test in a universal testing machine and expressed in MPa. The study provides comparative information on the reinforcing effects of nano-ZrO₂ alone and its combination with glass fibers on the flexural strength of PMMA denture base resin.

KEYWORDS: Polymethylmethacrylate; PMMA; Nano-zirconium oxide; Glass fibers; E-glass fibers; Denture base resin; Flexural strength; Nanocomposite; Fiber reinforcement.

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I. INTRODUCTION

Polymethyl methacrylate (PMMA) is widely used as a denture base material because of its acceptable esthetics, biocompatibility, ease of processing, and cost-effectiveness. However, its relatively low flexural and impact strength can contribute to denture base fracture during functional and accidental loading.^{1, 2}

Several methods have been investigated to improve the mechanical properties of PMMA, including the incorporation of fibers, fillers, and nanoparticles. Zirconium dioxide (ZrO₂) nanoparticles have been explored as reinforcing agents because of their favorable mechanical properties and potential to improve the strength of PMMA.^{2, 3} Similarly, glass fibers can reinforce PMMA by facilitating stress transfer and restricting crack propagation.^{1, 4}

The use of combinations of reinforcing materials has gained attention because different reinforcement mechanisms may act within the polymer matrix. Gad et al. investigated the reinforcement of PMMA using a combination of ZrO₂ nanoparticles and glass fibers, suggesting the potential of hybrid reinforcement for improving denture base properties.³

However, the relative effect of ZrO₂ nanoparticle reinforcement alone compared with a combination of ZrO₂ nanoparticles and glass fibers at an equivalent total reinforcement concentration requires further evaluation. Therefore, the present in-vitro study was undertaken to compare the selected mechanical property of PMMA denture base resin reinforced with 5% ZrO₂ nanoparticles and 2.5% ZrO₂ nanoparticles combined with 2.5% glass fibers.

This comparison aims to determine whether replacing part of the ZrO₂ nanoparticle reinforcement with glass fibers produces a different mechanical response in the PMMA denture base material.

II. MATERIALS AND METHODOLOGY

Study Design

The present study was an in-vitro comparative study designed to evaluate and compare the flexural strength of heat-cured polymethylmethacrylate (PMMA) denture base resin reinforced with nano-zirconium oxide (ZrO₂) and a 1:1 combination of nano-ZrO₂ and glass fibers.

A total of 40 specimens were fabricated and equally divided into two groups, with 20 specimens in each group (n = 20).

The total concentration of reinforcement was maintained at 5 wt% of the acrylic polymer powder in both groups. This enabled comparison of the effect of reinforcement composition while maintaining the same total reinforcement concentration.

Grouping of Specimens

Group Composition Number of specimens

Group I - ZrO₂ 5 wt% nano-ZrO₂ + 95 wt% PMMA = 20

Group II - ZrO₂ + GF 2.5 wt% nano-ZrO₂ + 2.5 wt% glass fibers + 95 wt% PMMA = 20

In Group I, the entire 5 wt% reinforcement component consisted of nano-ZrO₂. In Group II, the 5 wt% reinforcement component consisted of equal proportions of nano-ZrO₂ and glass fibers.

Materials

Heat-cured PMMA denture base resin was used as the resin matrix.

The nano-ZrO₂ used in the study had a purity of 99.9%, with a nominal particle size of 100 nm and an average measured particle size of approximately 40 ± 3 nm.

The glass fibers used were E-glass fibers, approximately 3 mm in length and 12 µm in diameter.

A silane coupling agent, 3-trimethoxysilyl propyl methacrylate (TMSPM), was used for surface treatment of the reinforcing materials.

Specimen Preparation

Standardized bar-shaped specimens were fabricated for the evaluation of flexural strength.

The dimensions of each specimen were:

65 mm × 10 mm × 2.5 mm ± 0.2 mm

These dimensions were based on the flexural-strength specimen dimensions described in the applicable standard/protocol used for the study.

A metal mold of the required dimensions was used to prepare wax patterns. The wax patterns were subsequently invested in dental stone to obtain the mold spaces.

After the dental plaster had set, the flasks were placed in a wax elimination unit for removal of the wax. The mold spaces were then cleaned with hot running water to remove residual wax. A separating medium was applied to the stone surfaces before packing of the acrylic resin.

Surface Treatment of Glass Fibers

The glass fibers were treated with the silane coupling agent before incorporation into the PMMA resin. The required quantity of glass fibers was weighed using a precision electronic balance. The fibers were immersed in TMSPM silane coupling agent for 1 minute at room temperature and subsequently dried at 60°C for 24 hours.

No glass fibers were incorporated into Group I.

Surface Treatment of Nano-ZrO₂

The required amount of nano-ZrO₂ was weighed according to the respective group allocation.

The nano-ZrO₂ was surface treated with TMSPM to improve its interaction with the PMMA resin matrix. The TMSPM treatment was performed according to the protocol of the parent study to facilitate adhesion between the filler particles and the resin matrix.

Preparation of Reinforcement Mixtures

The total reinforcement concentration was maintained at 5 wt% of the acrylic polymer powder for both groups.

For Group I, 5 wt% nano-ZrO₂ was incorporated with 95 wt% PMMA polymer powder.

For Group II, 2.5 wt% nano-ZrO₂ and 2.5 wt% glass fibers were incorporated with 95 wt% PMMA polymer powder.

The predetermined quantities of nano-ZrO₂ and glass fibers were accurately weighed and incorporated into the PMMA polymer powder by thorough manual mixing using a stainless-steel spatula until a visually homogeneous mixture was obtained. The same mixing procedure and duration were employed for both groups to ensure uniformity and standardization.

Packing and Polymerization

The polymer and monomer were mixed according to the manufacturer's recommended 3:1 polymer-to-monomer ratio by volume. The mixture was allowed to reach the dough stage before packing into the prepared mold spaces.

The flasks were subjected to hydraulic pressing at approximately 30 MPa for 30 minutes.

Polymerization was carried out using a thermostatically controlled water bath. Starting from cold water, the temperature was raised to 70°C and maintained for 90 minutes, followed by 100°C for 30 minutes.

After completion of the polymerization cycle, the flasks were allowed to cool to room temperature for approximately 1 hour before deflasking.

Finishing and Polishing

After polymerization and cooling, the specimens were carefully retrieved from the molds.

The specimens were finished using a fine cross-cut tungsten carbide bur. Subsequent polishing was performed using coarse and fine rubber polishing burs, followed by final polishing with a soft-bristle brush and fine pumice mixed with an equal volume of water.

The dimensions of each specimen were verified using a digital caliper with an accuracy of 0.01 mm. Specimens that did not conform to the predetermined dimensions were excluded and replaced.

Storage of Specimens

Following finishing and polishing, the specimens were stored in distilled water at 37°C for 48 hours before flexural-strength testing.

Evaluation of Flexural Strength

Flexural strength was evaluated using a three-point bending test in a universal testing machine.

Each specimen was positioned on two supporting pins with a 50-mm span between the supports. A load was applied at the center of the specimen using the universal testing machine.

A crosshead speed of 5 mm/min was used, and the load at fracture was recorded for each specimen.

The flexural strength was calculated using the following equation:

$$S = 3WL / 2bd^2$$

where:

S = Flexural strength in MPa

W = Load at fracture in Newtons

L = Distance between supports (50 mm)

b = Width of specimen (10 mm)

d = Thickness of specimen (2.5 mm)

The resulting flexural-strength values were expressed in MPa.

Statistical Analysis

The flexural-strength values obtained from all 40 specimens were recorded and expressed as mean and standard deviation (SD) for each group.

The flexural-strength values of Group I and Group II were compared statistically to determine whether there was a significant difference between the two reinforcement compositions. The level of statistical significance was set at $p < 0.05$.

III. Results

The comparison of flexural strength between the two study groups showed that the 5% ZrO₂ + 95% PMMA group exhibited a mean flexural strength of 82.60 ± 6.30 , whereas the 2.5% ZrO₂ + 2.5% glass fiber + 95% PMMA group demonstrated a higher mean flexural strength of 98.41 ± 6.74 .

The difference between the two groups was statistically significant according to the independent *t* test ($t = -7.661$, $p < 0.001$). The negative *t* value reflects the direction of subtraction based on the order in which the groups were entered into the analysis and does not indicate a negative flexural strength.

Numerically, the mean flexural strength of the 2.5% ZrO₂ + 2.5% glass fiber + 95% PMMA group was approximately 15.81 units higher, representing about a 19.1% increase compared with the 5% ZrO₂ + 95% PMMA group.

IV. Discussion

PMMA is the most commonly used material for denture-base fabrication because of its acceptable esthetics, ease of manipulation, low cost, and favorable clinical handling properties. However, conventional PMMA has relatively low flexural and impact strength, which may contribute to denture-base fracture during functional or accidental loading.¹

To overcome these limitations, different reinforcing materials such as fibers, fillers, metal oxides, and nanoparticles have been incorporated into PMMA. These modifications aim to improve the mechanical properties of the denture base and increase its resistance to functional stresses.²

Zirconium dioxide (ZrO₂) nanoparticles have been investigated as reinforcing fillers for PMMA because of their favorable mechanical properties and ability to modify the behavior of the polymer matrix. When adequately dispersed, ZrO₂ nanoparticles may restrict deformation of the polymer matrix and interfere with crack development.¹²

The effectiveness of ZrO₂ reinforcement depends on factors such as concentration, particle distribution, interaction with the polymer matrix, and specimen thickness. Albasarah et al. demonstrated that the incorporation of nano-ZrO₂ influenced the flexural properties of PMMA and that its effect varied with concentration and denture-base thickness.¹²

Glass fibers are another important reinforcing material used with PMMA. Unlike nanoparticles, which are dispersed throughout the polymer matrix, glass fibers act as load-bearing reinforcing elements. They can facilitate stress transfer through the resin and restrict the propagation of cracks.¹

The effectiveness of glass-fiber reinforcement depends on factors such as fiber concentration, orientation, distribution, impregnation, and bonding with the PMMA matrix. John et al. demonstrated that incorporation of glass fibers can improve the flexural strength of heat-polymerized PMMA denture resin.⁴

The combination of ZrO₂ nanoparticles and glass fibers provides two different forms of reinforcement. ZrO₂ nanoparticles primarily reinforce the polymer matrix, whereas glass fibers provide a load-bearing structure that can facilitate stress transfer. Therefore, combining these materials may provide complementary reinforcement. Gad et al. specifically investigated different combinations of ZrO₂ nanoparticles and glass fibers in PMMA and demonstrated that the proportion of the two reinforcing materials influenced the mechanical properties of the resin.³

Based on this concept, the present study compared two PMMA denture-base formulations with the same total reinforcement concentration of 5%: Group I-5% ZrO₂ nanoparticles + 95% PMMA, and Group II-2.5% ZrO₂ nanoparticles + 2.5% glass fibers + 95% PMMA.

The mean flexural strength of Group I was 82.60 ± 6.30 MPa, whereas Group II demonstrated a higher mean flexural strength of 98.41 ± 6.74 MPa. The difference between the groups was statistically significant, with a *t* value of -7.661 and $p < 0.001$.

Thus, the hybrid reinforcement demonstrated a mean increase of 15.81 MPa, corresponding to approximately 19.1% higher flexural strength compared with the 5% ZrO₂ group.

The higher flexural strength observed in Group II indicates that, under the conditions of the present study, 2.5% ZrO₂ combined with 2.5% glass fibers produced a greater flexural-strength response than 5% ZrO₂ alone. Since both groups contained an identical total reinforcement concentration of 5%, the difference is more appropriately attributed to the type and combination of reinforcing materials rather than simply to the quantity of reinforcement.

This finding is particularly relevant when compared with the study by Gad et al., which evaluated different combinations of ZrO₂ nanoparticles and glass fibers while maintaining the total reinforcement concentration at 5%. Their findings demonstrated that the 2.5% ZrO₂ + 2.5% glass-fiber combination produced favorable flexural properties.³ The present study therefore supports the observation that changing the composition of reinforcement can influence the mechanical performance of PMMA.

The higher flexural strength of the hybrid group may be explained by the complementary roles of the two reinforcing materials. ZrO₂ nanoparticles may reinforce the PMMA matrix, while glass fibers may function

as load-bearing structures and facilitate transfer of stresses through the resin. The combination may consequently provide a more effective distribution of applied stress and delay crack initiation or propagation. The importance of fiber type and concentration in determining the reinforcing effect has also been demonstrated by Yu et al.¹

The contribution of glass fibers may explain the additional improvement observed in Group II. John et al. reported increased flexural strength following incorporation of glass fibers into heat-polymerized PMMA.⁴ Similarly, Vallittu demonstrated improved fatigue resistance of acrylic resin reinforced with continuous glass fibers.¹⁰ Although fatigue resistance was not evaluated in the present study, these findings support the role of glass fibers as effective reinforcing elements within acrylic resin.

The findings of Im et al. also demonstrated improved fracture resistance of glass-fiber-reinforced complete dentures under dynamic fatigue loading.⁹ Although their experimental design differed from the present study, their findings provide further support for the ability of glass fibers to contribute to the mechanical resistance of acrylic denture bases.

The present findings should not, however, be interpreted as indicating that ZrO₂ nanoparticles alone are ineffective. The 5% ZrO₂ group demonstrated a mean flexural strength of 82.60 ± 6.30 MPa, indicating that ZrO₂ provided substantial reinforcement to the PMMA matrix. Previous research has demonstrated improvements in the mechanical properties of PMMA following incorporation of ZrO₂ nanoparticles.¹⁴

The difference observed in the present study therefore indicates that the hybrid reinforcement produced a greater flexural-strength response than an equivalent 5% concentration of ZrO₂ alone. This may be because the glass fibers introduce a different load-bearing mechanism that complements the matrix-reinforcing effect of ZrO₂ nanoparticles.

The findings of Neset et al. also support the potential of metal-oxide modification to alter the mechanical properties of heat-cured PMMA.⁵ However, differences in material composition and testing methodology should be considered when comparing their results with those of the present study.

Similarly, Moreno-Maldonado et al. demonstrated that incorporation of fibers into PMMA can modify its mechanical behavior.⁷ This supports the present observation that introducing glass fibers into the ZrO₂-reinforced PMMA system can produce an additional mechanical effect.

Jagger et al. emphasized that the reinforcing behavior of denture-base materials depends on factors such as reinforcement type, distribution, concentration, and interaction with the polymer matrix.¹¹ This is relevant to the present study because both groups contained the same total reinforcement concentration, yet the group containing two different reinforcing phases demonstrated higher flexural strength.

More recent research continues to investigate modification of denture-base polymers through combinations of reinforcing materials. Majeed et al. highlighted the continuing development of reinforced denture-base materials, particularly with newer polymer technologies.⁸ Ponjyanthi et al. also reported differences in flexural and impact properties following reinforcement of PMMA with nanoparticles and glass fibers.¹³ Although their nanoparticle material and experimental formulation differed from those used in the present study, their findings support the continued investigation of hybrid reinforcement systems.

The standardization of specimen preparation and mechanical testing is also important when evaluating denture-base polymers. The ISO 1567 standard provides requirements for denture-base polymers and is relevant to standardized evaluation of these materials.⁶

From a prosthodontic perspective, the increased flexural strength observed with the hybrid formulation may be beneficial because denture bases are subjected to repeated bending stresses during mastication. However, the present study was conducted under in-vitro conditions, and flexural strength alone cannot establish long-term clinical performance. Factors such as fatigue, water absorption, thermal cycling, aging, porosity, fiber orientation, and fiber-matrix bonding may influence the behavior of reinforced PMMA.²

The proposed explanation involving improved stress transfer and restriction of crack propagation should also be considered a possible mechanism, rather than a mechanism directly demonstrated by the present experiment. Microscopic examination of the fracture surface and evaluation of the fiber-matrix interface would be required to confirm these mechanisms.

Overall, the present study demonstrated that incorporation of 2.5% ZrO₂ nanoparticles and 2.5% glass fibers into PMMA resulted in significantly greater flexural strength than incorporation of 5% ZrO₂ nanoparticles alone. The flexural strength increased from 82.60 ± 6.30 MPa to 98.41 ± 6.74 MPa, with a statistically significant difference ($p < 0.001$). The approximately 19.1% increase suggests that the hybrid reinforcement provided a more favorable response to flexural loading under the conditions of the present study.

Therefore, the 2.5% ZrO₂ + 2.5% glass-fiber formulation demonstrated higher flexural strength than 5% ZrO₂-reinforced PMMA, supporting the potential of hybrid reinforcement for improving the mechanical performance of denture-base resin.

Limitations

The present study had some limitations. As an in-vitro study, the findings may not fully reflect the behavior of reinforced PMMA under actual oral conditions. The investigation was limited to flexural strength and two reinforcement formulations: 5% ZrO₂ nanoparticles and 2.5% ZrO₂ nanoparticles + 2.5% glass fibers. Other properties, reinforcement concentrations, and long-term aging conditions were not evaluated. Further studies with different formulations, aging protocols, and clinical evaluation are recommended to confirm the long-term performance of the materials.

V. Conclusion

Within the limitations of the present in-vitro study, PMMA denture-base resin reinforced with a combination of 2.5% ZrO₂ nanoparticles and 2.5% glass fibers demonstrated significantly higher flexural strength than PMMA reinforced with 5% ZrO₂ nanoparticles alone. The hybrid-reinforced group showed a mean flexural strength of 98.41 ± 6.74 MPa, compared with 82.60 ± 6.30 MPa for the 5% ZrO₂ group, with the difference being statistically significant ($p < 0.001$).

The approximately 19.1% increase in flexural strength suggests that combining ZrO₂ nanoparticles with glass fibers may provide a more favorable reinforcement effect than ZrO₂ nanoparticles alone at the same total reinforcement concentration. Therefore, the 2.5% ZrO₂ and 2.5% glass-fiber combination may be considered a promising approach for improving the flexural strength of PMMA denture-base resin, although further studies evaluating long-term aging, fatigue, fracture resistance, and clinical performance are required.

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Figure 1: Heat cure PMMA



Figure 2: E-Glass fibres



Figure 3: Zirconium dioxide nanoparticles



Figure 4: Silane coupling agent



Figure 5: Wax blanks invested in flask for packing of heat cure

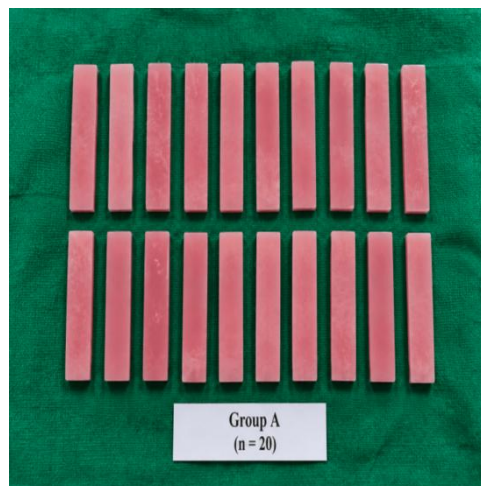


Figure 6: Group A samples (95% PMMA+5% ZrO₂)

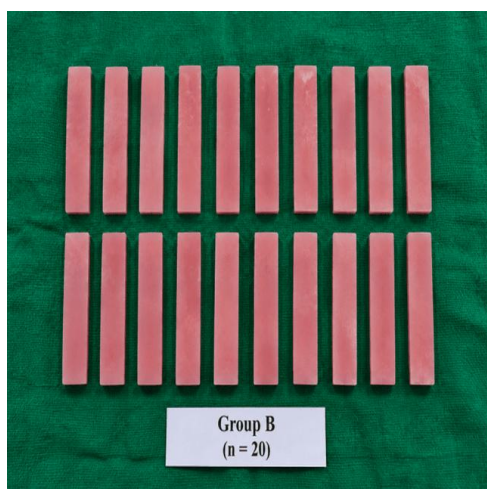
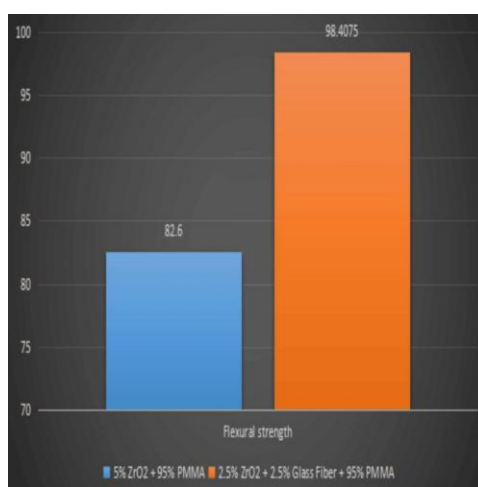


Figure 7: Group B samples (95% PMMA + 2.5% ZrO₂ + 2.5% E Glass Fibers)



Figure 8: Flexural strength testing on Universal testing machine



Bar Diagram 1 : Comparison Of Flexural Strength Among Study Groups

Groups	Mean	Std. Deviation	Std. Error Mean	t-value	p-value
5% ZrO ₂ + 95% PMMA	82.6000	6.30052	1.40884	-7.661	0.000*
2.5% ZrO ₂ + 2.5% Glass Fiber + 95% PMMA	98.4075	6.74178	1.50751		

Graph 1 : Comparison Of Flexural Strength Among
Study Groups