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Comparative Evaluation of Flexural Strength and Impact Strength of Heat Cure Acrylic Denture Base Reinforced with Nanoparticles of Zirconium Oxide, Peek and Glass Fibers- An In-Vitro Study

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ABSTRACT

Aim: The aim of this study is to compare the Flexural Strength and Impact Strength of PMMA with PMMA reinforced with nanoparticles of Zirconium Oxide, Polyetheretherketone and Silanized E-Glass Fibres.

Materials and Methods: A total of one hundred and twenty specimens were fabricated. Out of 120 specimens, sixty specimens were fabricated to evaluate Flexural Strength (dimensions: 65mm x 10mm x 2.5mm) and another sixty specimens to evaluate Impact Strength (dimensions: 80mm x 10mm x 4mm). Flexural strength testing conducted using the three-point bending test in a universal testing machine. Impact Strength Testing conducted using Charpy and Izod testing machine. A one-way analysis of variance (ANOVA) was performed to evaluate statistically significant differences among the groups for both flexural and impact strength. Following this, Tukey's post hoc test was employed for intra-group comparisons.

Results: PMMA reinforced with PEEK nanoparticles (Group P) exhibited the highest mean flexural strength (55.08±12.91), followed closely by PMMA reinforced with Glass Fibers (Group G) (52.62±11.26). The control group (PMMA) had the highest mean impact strength (34.95±10.62), followed closely by the Glass Fibers group (33.96±11.64), and then the PEEK group (31.54±11.31).

Conclusion: PEEK and glass fibers are more effective reinforcements for improving the mechanical performance of PMMA, particularly in applications requiring greater flexural strength.

INTRODUCTION

The history of complete dentures dates back to 700 BC, with early prostheses made from materials like bone, wood, ivory, and rubber. In 1937, Walter Wright introduced PMMA as a denture base material, revolutionizing prosthodontics due to its superior aesthetic qualities, ease of processing, low cost, and repairability. By the 1940s, PMMA had become the dominant material for denture bases, remaining widely used despite the introduction of alternatives such as polystyrene and light-cured urethane dimethacrylate.

However, PMMA is not without limitations. It has low thermal conductivity, is brittle, and exhibits a high coefficient of thermal expansion and water sorption. These mechanical drawbacks lead to common clinical failures such as midline fractures from flexural

fatigue and accidental breakage from impact. Flexural strength (FS) and impact strength (IS) are key parameters influencing denture longevity. Hence, improving PMMA's mechanical performance is a primary research focus.

Several methods have been explored to strengthen PMMA. One chemical strategy is modifying the polymer matrix by incorporating cross-linking agents or rubber copolymers, which may increase impact strength but often reduce flexural strength and fatigue resistance. A more promising physical approach involves the addition of inorganic fillers, such as nanoparticles and fibres, to enhance the material's structural integrity.

Nanoparticles like aluminium oxide (Al_2O_3), silicon dioxide (SiO_2), titanium dioxide (TiO_2), and zirconium dioxide (ZrO_2) have shown to improve PMMA properties due to their nanoscale size, shape, and high surface area. Nano-ZrO_2, in particular, is favored for its excellent toughness, white color (maintaining aesthetics), corrosion resistance, and biocompatibility. However, an excessive concentration may lead to particle agglomeration and reduced mechanical performance.

Polyetheretherketone (PEEK), a high-performance polymer initially used in aerospace and medical implants, has emerged as a viable alternative material for denture bases. PEEK exhibits superior dimensional stability, low water absorption, no polymerization shrinkage, excellent mechanical strength, and biocompatibility. Its flexural modulus is close to that of human bone, making it suitable for durable and lightweight dentures with minimal wear on opposing teeth.

Fibre reinforcement has also been extensively investigated. Materials such as carbon fibres, nylon, and glass fibres can significantly improve flexural and impact strength if properly embedded in the PMMA matrix. Glass fibres, especially when silanized, offer an ideal combination of strength, bonding ability, aesthetics, and resilience, making them highly effective in denture reinforcement.

Mechanical testing techniques like three-point bending tests (for FS) and Charpy/Izod tests (for IS) are standard for evaluating material performance. Research has demonstrated that reinforcing heat-cured PMMA with 1 wt% of nano-ZrO_2, nano-PEEK, or silanized E-glass fibres can enhance its mechanical properties. This study aimed to assess and compare the flexural and impact strengths of PMMA reinforced with these materials. The null hypothesis proposed that such fillers would not significantly influence PMMA's mechanical performance—an assumption tested through an in-vitro investigation.

Materials and Methods: A total of one hundred and twenty specimens were fabricated. Out of 120 specimens, sixty specimens were fabricated to evaluate Flexural Strength (dimensions: 65mm x 10mm x 2.5mm) and another sixty specimens to evaluate Impact Strength (dimensions: 80mm x 10mm x 4mm). For each test four groups were considered containing one control group and three experimental groups as follows:

1. Group **C** -PMMA (Control group)
2. Group **Z** - PMMA reinforced with Zirconium Oxide nanoparticles
3. Group **P** - PMMA reinforced with PEEK nanoparticles
4. Group **G** - PMMA reinforced with Glass fibres

Each group contain 15 specimens.

Preparation of the Specimens

Flexural strength test specimens: Sixty specimens from four denture base resin groups (15 specimens for each of above mentioned four groups) were processed to get specimens having dimensions of 65 mm length × 10 ± 0.01 mm width × 2.50 ± 0.01 mm thickness according to ISO 20795-1:2008 and ANSI/ADA Specification no. 12. A custom-made three-piece stainless steel metal mould having ten rectangular cavities of dimensions 65 mm × 10 mm × 2.5 mm in the middle part was used. This resulted in ten specimens at a time.

Impact strength test specimens: Sixty specimens from four denture base resin groups (15 specimens for each of above mentioned four experimental groups) were processed to get specimens having dimensions of 80 mm (l) × 10 ± 0.01 mm (w) × 4 ± 0.01 mm (h), in according to ISO 20795-1:2008. A custom-made three-piece stainless steel metal mould having ten rectangular cavities of dimensions 80 mm × 10 mm × 4 mm in the middle part was used. This resulted in ten specimens at a time.

Specimen processing

Control group (Group C) processing: Each metal mould was coated with a thin layer of cold mould seal and white petroleum jelly to facilitate easy retrieval of the heat-polymerized specimens. All samples were prepared using the conventional compression moulding technique, following the manufacturer's recommendations. For the control group (Group C), polymer powder (DPI Heat Cure Denture Base Material) was weighed using a precision digital scale, and the monomer was measured using a syringe, maintaining a polymer-to-monomer ratio of 1.6:1 (75g of polymer to 47ml of monomer). The materials were hand-mixed with a stainless steel spatula in a silicone mixing jar until the mixture reached a dough stage. Once the dough stage was achieved, the material was kneaded thoroughly and packed into the mould space. Trial closure was performed at 1500 psi to remove flash, followed by final closure at 3500 psi using a hydraulic bench press. The moulds were left under pressure for 30 minutes for bench curing, after which the screws were tightened, and the moulds were removed from the press.

Polymerization was carried out by immersing the custom mould assembly in a water bath for 9 hours, first for 7 hours at 74°C (±3°C) and then for 2 hours at 95°C (±3°C). After completing the curing cycle, the moulds were allowed to cool at room temperature for 30 minutes and then under running tap water for 15 minutes before retrieving the specimens. The specimens were finished by removing excess acrylic with trimming burs and polished with silicone rubber polishing burs, followed by sanding with 120 and 180 grit sandpapers. Final polishing was done using pumice cake and pumice powder to achieve a smooth surface.

Experimental groups processing

Processing of Group Z (PMMA reinforced with zirconium oxide nanoparticles): Zirconium group (Z) specimens were fabricated with 1wt% Zirconium Oxide nanoparticles (Vedayukt India Pvt. Ltd., Jamshedpur, Jharkhand, India) is added to heat cure acrylic powder before adding the liquid component in ratio of 0.750g ZrO_2: 74.250g polymer: 47ml monomer using a vaccum mix machine (MIX Vacuum mixer, Dentalfarm, Torino, Italy).

Processing of Group P (PMMA reinforced with PEEK nanoparticles): PEEK specimens were fabricated with 1wt% PEEK nanoparticles (Vedayukt India Pvt. Ltd., Jamshedpur, Jharkhand, India) is added to heat cure acrylic powder before adding the liquid component in ratio of 0.750g PEEK:74.250g polymer:47ml monomer using a vacuum mix machine (MIX Vacuum mixer, Dentalfarm, Torino, Italy).

Processing of Group G (PMMA reinforced with Silanized E-Glass fibers): To enhance chemical bonding between the acrylic resin matrix and E-glass fibers, the fiber surfaces were treated with a silane coupling agent (3-Trimethoxysilyl Propyl Methacrylate, 3-TPM). Ethanol (99%) served as a solvent, with the pH adjusted to 4.5–5.5 using acetic acid to ensure particle surface cleanliness before coating. A solution of 0.25 g of 3-TPM in 5 mL of ethanol was stirred magnetically at 500 rpm for 5 minutes and hydrolyzed at room temperature for 15 minutes. Separately, 5 g of E-glass fibers were dispersed in 25 mL of solvent using ultrasonic processing (40 KHz, 5 minutes), followed by mixing with the silane solution for 5 minutes. After 24 hours of room-temperature storage to promote hydrogen bonding, the treated fibers were washed with ethanol (centrifuged at 1500 rpm for 15 minutes) and dried at room temperature for 24 hours, then at 100°C for 2 hours to convert hydrogen to covalent bonding. The monomer-to-polymer ratio of 47 mL to 75 g was used to incorporate the silanized E-glass fibers into PMMA. Initially, 40 mL of MMA was used to moisten the compacted chopped fibers, which were manually redistributed for thorough saturation. Then, 74.25 g of PMMA powder was mixed with the liquid in six short cycles (10 seconds each), followed by a 2-minute stirring to ensure uniform fiber distribution. Finally, the remaining 7 mL of MMA was added to complete the mixture.

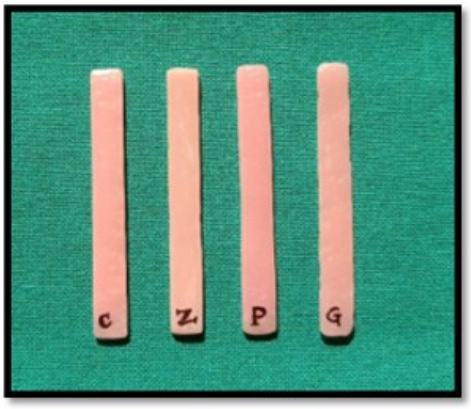

Figure 1- Fabricated specimens

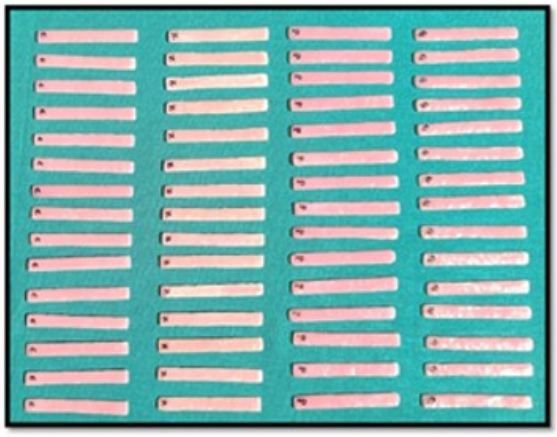

Figure 2 - Flexural strength test specimens

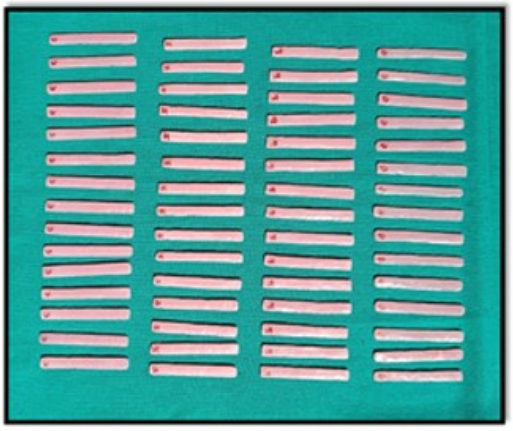

Figure 3- Impact strength test specimens

Testing of Specimens

Flexural strength testing: Flexural strength test was performed according to ISO 20795-1:2008. Fifteen specimens from each group were subjected to flexural strength testing under three-point loading (Fig. 31) with a crosshead speed of 5 mm/min in a universal testing machine (Tinius Olsen, Model 25ST – 25 kN, Noida, Uttar Pradesh, India). The flexural testing device consisted of a central loading plunger and two polished cylindrical supports, 3.2 mm in diameter and 10.5 mm long. The distance between the canters of the supports was 50 mm. This dimension represents the space between the maxillary molars in a complete denture. The load was applied perpendicular to the centre of specimen strips until the deviation of the load-deflection curve and fracture of specimen occurred.

Impact strength testing: Impact strength test was performed according to ISO 20795-1:2008. Fifteen specimens from each group were subjected to impact strength testing using digital Izod type impact testing machine (PRESTO Izod Tester, Presto Stantest Pvt. Ltd., Faridabad, Haryana, India). For this, a specimen of dimension 80mm × 10mm × 4mm was kept on the jig. A 5.5J pendulum hammer was used to impart the energy at centre of the specimen. After deducting the attrition value (0.04 J), the net energy absorbed was obtained for each specimen. The Izod impact test (kJ/m2) utilized a universal pendulum impact testing machine. The pendulum load cell was 0.5 J and directly faced the centre of the specimen. When the test will be started, the pendulum will be released to strike the specimen, and the impact energy absorbed was recorded in joules (J).

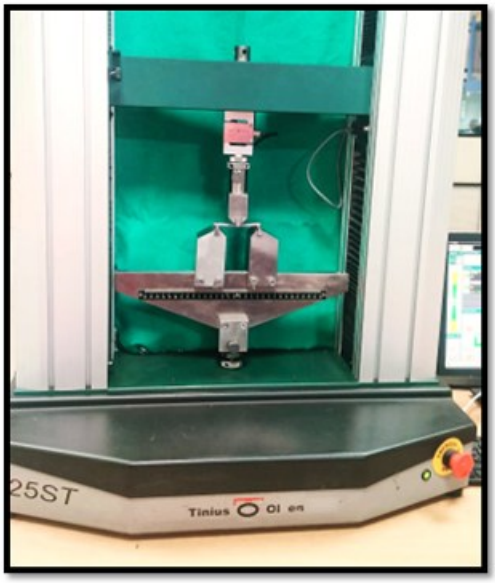

Figure 4- Specimen subjected to flexural strength testing under three-point loading

Figure 3 - Specimen subjected to Impact strength testing

Storage: For each group, 15 specimens were prepared for flexural strength testing and another 15 for impact strength testing. These specimens were stored separately in water-filled containers at room temperature for 4 weeks as 30 days of storage in water resulted in a significant decrease in dimensional changes and compensated the polymerization shrinkage.

Statistical Analysis: The data obtained were subjected to statistical analysis using the Statistical Package for the Social Sciences (IBM Corp. Released 2011. IBM SPSS Statistics for Windows, Version 20.0, Armonk, NY: IBM Corp.). The study aimed to compare the flexural strength and impact strength of PMMA with PMMA reinforced with nanoparticles of Zirconium Oxide, Polyetheretherketone (PEEK), and Silanized E-glass fibers. A one-way analysis of variance (ANOVA) was performed to evaluate statistically significant differences among the groups for both flexural and impact strength. Following this, Tukey's post hoc test was employed for intra-group comparisons. A p-value of less than 0.05 was considered statistically significant, with a 95% confidence interval.

Results: This study aimed to compare the flexural and impact strengths of unmodified PMMA and PMMA reinforced with Zirconium Oxide nanoparticles, PEEK nanoparticles, and silanized E-glass fibers. Descriptive statistics revealed that PMMA reinforced with PEEK showed the highest flexural strength (mean = 55.08±12.91 MPa), followed by E-glass fiber reinforcement (52.62±11.26 MPa), while the control group and Zirconium Oxide groups had significantly lower values (30.06±9.51 and 22.04±5.41 MPa, respectively). ANOVA indicated a statistically significant difference in flexural strength among the groups ($p=0.001$), with post-hoc tests confirming that PEEK and E-glass fiber reinforcements differed significantly from the control and Zirconium Oxide groups ($p<0.05$). In terms of impact strength, the control group (34.95±10.62 J) and E-glass fiber group (33.96±11.64 J) performed better than the Zirconium Oxide group (24.51±8.04 J), while the PEEK group had intermediate values (31.54±11.31 J). ANOVA showed a significant difference in impact strength ($p=0.03$), but post-hoc analysis revealed that only the comparison between the control and Zirconium Oxide group was statistically significant ($p=0.04$). Overall, PEEK and E-glass fiber reinforcements significantly improved the flexural strength of PMMA, while only minimal differences were observed in impact strength enhancement. (Table 1-3)

Table 1: Descriptive statistics for flexural strength among various groups

Variables	N	Min value	Max value	Mean	SD
PMMA Control Group	15	2.50	44.60	30.0667	9.51928
PMMA Reinforced with Zirconium Oxide Nanoparticles	15	12.20	29.10	22.0400	5.41121
PMMA Reinforced with PEEK Nanoparticles	15	19.20	71.40	55.0800	12.91230
PMMA Reinforced with Glass Fibers	15	34.80	68.30	52.6200	11.26919

Table 2: Descriptive statistics for impact strength among various groups

Variables	N	Min value	Max value	Mean	SD
PMMA Control Group	15	24.65	60.20	34.9507	10.62880
PMMA Reinforced with Zirconium Oxide Nanoparticles	15	12.38	39.22	24.5180	8.04146
PMMA Reinforced with PEEK Nanoparticles	15	20.74	50.28	31.5420	11.31003
PMMA Reinforced with Glass Fibers	15	19.68	51.37	33.9640	11.64196

Table 3: Intra-group comparison of various materials used

Variables		p-value
PMMA Control Group	PMMA Reinforced with Zirconium Oxide Nanoparticles	0.04 (s)
	PMMA Reinforced with PEEK Nanoparticles	0.81 (n.s)
	PMMA Reinforced with Glass Fibers	0.99 (n.s.)
PMMA Reinforced with Zirconium Oxide Nanoparticles	PMMA Reinforced with PEEK Nanoparticles	0.27 (n.s.)
	PMMA Reinforced with Glass Fibers	0.07 (n.s.)
PMMA Reinforced with PEEK Nanoparticles	PMMA Reinforced with Glass Fibers	0.921(n.s.)

DISCUSSION

PMMA (polymethyl methacrylate) has been the most widely used denture base material for decades due to its affordability, lightweight nature, insolubility in oral fluids, excellent aesthetics, and reparability. However, its mechanical limitations—such as low surface hardness, poor impact and flexural strength, brittleness, and polymerization shrinkage—limit its long-term durability. To address these weaknesses, this study investigated the reinforcement of PMMA with 1 wt% of zirconia nanoparticles, PEEK (polyether ether ketone), and E-glass fibers. These materials were selected due to their known mechanical strength and compatibility with PMMA. The study evaluated the flexural and impact strength of the reinforced materials and found statistically significant improvements, justifying the rejection of the null hypothesis.

Denture fractures commonly result from impact (e.g., accidental drops) or flexural fatigue from mastication. Flexural strength, which reflects a material's resistance to bending and fracture, and impact strength, which measures resistance to sudden forces, are critical indicators for evaluating denture durability. The incorporation of zirconia (ZrO_2) nanoparticles into PMMA has shown variable results in past studies. While some reported increased flexural strength and fracture toughness, others noted decreased strength due to particle agglomeration. This clustering may act as stress concentrators, reducing the mechanical integrity of the composite. Notably, Zidan et al. found a 15% increase in flexural strength at 3 wt% zirconia, but strength declined at higher concentrations. Zhang et al. and Chęcińska et al. also confirmed improvements with 1–2 wt% zirconia, supporting the idea that optimal filler concentration is crucial.

Zirconia reinforcement has also been linked to improved thermal conductivity, potential antifungal effects, and reduced water sorption and solubility. SEM analysis has shown that zirconia enhances particle dispersion and decreases porosity, although this depends on proper surface treatment and filler integration. Salinized zirconia nanoparticles were particularly effective in improving tensile strength, fatigue resistance, and hardness while decreasing surface roughness and porosity. However, in the present study, zirconia-reinforced PMMA showed a 20% decrease in flexural strength compared to the control group, possibly due to nanoparticle clustering.

PEEK, on the other hand, demonstrated the highest improvement in flexural strength—rising by 57.17% from 35.8 MPa in the control group to 55.2 MPa. PEEK is known for its excellent tensile strength and resistance to notch sensitivity. Muhsin et al. confirmed its superior fracture resistance, especially in conditions involving notched frameworks or anatomical variations. The uniform mixing of PEEK into PMMA using a blender helped distribute the filler evenly, minimizing agglomeration and enhancing mechanical bonding. Moreover, no polishing difficulties were observed, and the final specimens had acceptable surface quality and aesthetics.

The use of E-glass fibers as reinforcement has been explored since the 1960s. E-glass, derived from alumina-lime-borosilicate, offers good strength, fracture resistance, chemical stability, and thermal tolerance. These fibers are biocompatible and enhance both flexural and impact strength, especially when used in silanized form. In this study, E-glass fibers increased flexural strength by 48.57% (from 35.8 MPa to 52.2 MPa). The effectiveness of E-glass fibers depends on their position within the denture base: when placed near the surface (compressive stress zone), overall strength improves significantly. However, poorly bonded or improperly impregnated fibers can act as voids and reduce the mechanical strength of the composite. Surface treatments like plasma spraying or silanization improve fiber-matrix adhesion and water resistance.

Fiber concentration is another critical variable. Gutteridge suggested 1 wt% as ideal for polyethylene fibers, with higher amounts complicating manipulation. Similarly, Ladizesky et al. found diminishing returns and handling difficulties beyond 4 wt% fiber content. Clarke et al. recommended 2 wt%. In this study, 1 wt% was used across all reinforcements, balancing performance and workability. Aesthetic evaluation revealed that zirconia imparted a slightly whiter hue, PEEK a pinkish tone, and E-glass fibers retained a shade similar to unmodified PMMA—all acceptable for clinical use.

Despite promising results, this study has several limitations. It was conducted in vitro using simplified bar-shaped specimens rather than actual denture geometries. Moreover, oral conditions such as temperature fluctuations, moisture, microbial presence, and mechanical wear were not simulated. Aging processes like thermal cycling, which affect long-term material behavior, were not incorporated. These omissions limit the extrapolation of results to real-world performance. Additionally, the study did not explore the optimal filler concentration beyond 1 wt%, and higher or mixed ratios may yield different outcomes. The influence of long-term exposure to oral fluids, masticatory forces, and cleaning agents remains unknown.

Future studies should aim to replicate oral environments more closely through thermal cycling and long-term aging tests. They should also evaluate the biocompatibility of the composites, as well as conduct SEM analysis to assess filler distribution, porosity, and agglomerate formation at fracture sites. Moreover, identifying the ideal concentration of each filler type could help balance mechanical enhancement with ease of fabrication and cost-effectiveness. While the current findings highlight the potential of these reinforcements—especially PEEK and E-glass fibers—in improving denture base durability, their clinical application requires further validation through comprehensive in vivo studies.

In conclusion, this study supports the reinforcement of PMMA with PEEK, zirconia, and E-glass fibers to enhance flexural strength and potentially reduce denture fracture risk. PEEK demonstrated the highest improvement in flexural strength, followed by E-glass fibers, with zirconia showing mixed results due to possible agglomeration. All reinforcements maintained acceptable aesthetics and handling properties. However, due to the limitations of in-vitro testing and unverified long-term behavior, further research is essential before adopting these materials in routine clinical practice.

CONCLUSION

Within the scope and limitations of this study, it was concluded that reinforcing PMMA with PEEK nanoparticles and silanized E-glass fibers significantly improved its flexural strength, with PEEK yielding the most substantial enhancement. In contrast, the addition of zirconium oxide nanoparticles resulted in the lowest flexural strength, falling below that of the unreinforced control group. Impact strength was generally unaffected by reinforcement, except for a notable decrease observed with zirconium oxide. Overall, PEEK and glass fibers emerged as more effective reinforcements for enhancing the mechanical performance of PMMA, particularly in applications demanding higher flexural strength.

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