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Comparative evaluation of nanoparticles as reinforcing agents on the flexural strength in denture base materials

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Abstract:
A major limitation of conventional PMMA denture base materials is their inherent brittleness and low flexural strength, which frequently leads to midline fractures under clinical masticatory forces. Therefore, it is of interest to compare the flexural strength of PMMA denture base materials reinforced with zirconium oxide, titanium dioxide and boron nitride nanoparticles. Hence, 40 PMMA specimen were divided into 4 groups (n=10), tested using a Universal Testing Machine and analyzed with one way ANOVA and TURKEY'S HSD test ($p<0.05$). Zirconium oxide and titanium dioxide significantly improved flexural strength compared with boron nitride and control group. ($p < 0.001$). Turkey's HSD showed no significant difference between zirconium oxide and titanium oxide while load displacement analysis conform their superior fracture resistance. Thus, zirconium oxide and titanium dioxide are effective PMMA reinforcing agents with zirconium oxide showing the highest overall performance, whereas boron nitride offered very little reinforcement.

Keywords: Polymethyl methacrylate (PMMA), Titanium dioxide nanoparticle, silver nanoparticle, boron nitride nanoparticle, colony forming units, probe sonicator

Background:
Because of its many benefits, including ease of handling, affordability, lightweight nature, aesthetic qualities and stability in the oral environment, acrylic resin (PMMA) is the most used material for creating denture bases **[1].** Nevertheless, it has a weak surface and dubious mechanical properties, especially fracturing susceptibility under functional stresses **[2, 3].** To increase the strength of denture base resins, a number of reinforcing techniques have been used throughout the years. Although additives including glass fibers, polyurethane, aramid fibers **[4, 5]** and metal wires in different forms particles, flakes, fibers, or fabrics **[6]** have shown advantages but more development is still required. According to a clinical survey, almost 70% of dentures broke during their first three years of use. According to another study on denture fractures, midline fractures (often in maxillary dentures) accounted for 29% of repairs, while other fracture types accounted for the remaining 33% **[7].** This shows the clinical need to strengthen denture base resins. Because of their specific chemical and physical characteristics, nanoparticles gained interest as reinforcing agents. Titanium dioxide nanoparticles (TiO_2) are inexpensive and exhibit biocompatibility, chemical inertness, antibacterial action, high refractive index, corrosion resistance and microhardness **[8]**. Since dentures are subjected to high occlusal forces, compressive resistance is critical to prevent failure; however, the compressive and flexural behavior of PMMA reinforced with TiO_2 remains inadequately documented **[9]**. Zirconium dioxide nanoparticles (ZrO_2) are also increasingly popular for reinforcement due to their superior toughness, crack resistance, abrasion and corrosion resistance, biocompatibility and white coloration. Being the hardest oxide, ZrO_2 effectively resists crack propagation. A recent meta-analysis demonstrated that both TiO_2 and ZrO_2 substantially increase the flexural strength of PMMA compared to other fillers, while maintaining esthetics, unlike metallic nanoparticles such as silver **[10]**. Despite this, limited literature directly compares the reinforcing potential of TiO_2 and ZrO_2. Boron nitride (BN) is another promising candidate, with properties such as chemical stability, thermal resistance and low water sorption, color matching ability, non-toxicity and ease of laboratory handling. Its structure resembles graphene, conferring strength and stability **[11]**. BN has been studying dentistry for reinforcing ceramics **[12]**, resin-based sealants **[13]**, adhesives **[14]** as well as coatings for implants **[15]**. Recent research emphasizes the potential of BN nanofillers in creating novel nanocomposites with improved physical and mechanical properties. The principle of nanoscale reinforcement lies in reducing filler size to the nanometer level, thereby enhancing interfacial bonding with the resin matrix and improving material performance **[16].** Therefore, it is of interest to report that determination and comparison of the effectiveness of titanium dioxide, zirconium dioxide and boron nitride nanoparticles as reinforcing agents shall enhance the flexural strength of heat-cured PMMA denture base resin.

Materials and Methods:
Specimen preparation:
40 rectangular specimens of heat-cured PMMA were fabricated using standardized metal die, each measuring 100 × 20 × 12 mm. Four groups (n = 10):

[1] Group A1: PMMA reinforced with titanium dioxide nanoparticles
[2] Group A2: PMMA reinforced with boron nitride nanoparticles
[3] Group A3: PMMA reinforced with zirconium oxide nanoparticles
[4] Group A4 (Control): Unmodified heat-cured PMMA

Nanoparticle incorporation:
Heat-cure PMMA resin was used as the base material. Zirconium oxide, titanium dioxide and boron nitride nanoparticles were added at 3 wt% into 22 mL of PMMA monomer. Each nanoparticle–monomer mixture was homogenized using a probe sonicator. (Soniprep 150, England; 120 W, 60 kHz) for 3 minutes, ensuring uniform nanoparticle dispersion within the monomer matrix.

Specimen fabrication:
PMMA polymer powder was combined with the nanoparticle-modified monomer in a 2.5:1 (w/v) ratio. The resulting dough was then packed into preformed rectangular dies and processed using a short curing cycle. After polymerization samples were finished and polished to achieve standardized dimensions and surface smoothness.

Flexural strength testing:
Flexural strength was determined using a Universal Testing Machine (Instron Model 3345, USA) for three-point bending test. Two supports with a span length of 25 mm were used to hold the specimens. A 500 N load cell was used to apply a load at the midpoint at a crosshead speed of 1 mm/min until fracture occurred.

Statistical analysis:
Microsoft Excel was used to compile the data and IBM SPSS Statistics, Version 30.0 (IBM Corp., Armonk, NY, USA) was used for analysis. The data's normality was evaluated using the Kolmogorov-Smirnov test. For every group, descriptive statistics (Mean ± standard deviation) were computed. One-way analysis of variance (ANOVA) was used to assess group differences and Tukey's Honestly Significant Difference (HSD) post-hoc test was used for pairwise comparisons. At a 95% confidence interval, a p-value of less than 0.05 was deemed statistically significant.

Results:
The zirconium oxide group had the greatest mean flexural strength values (MPa) (63.23 ± 8.10), followed by titanium dioxide (62.38 ± 7.85), boron nitride (42.36 ± 6.45) and the control group (27.88 ± 4.20) **(Table 1).** A statistically significant difference between the groups was shown by a one-way ANOVA ($F = 62.33$, $p < 0.001$). Zirconium oxide and titanium dioxide-reinforced PMMA showed significantly higher flexural strength when compared to boron nitride and control groups ($p < 0.001$), according to post-hoc Tukey HSD analysis. There was no significant difference between zirconium oxide and titanium dioxide ($p = 0.99$). Load–displacement analysis verified these findings, showing that zirconium oxide and titanium dioxide groups sustained higher peak loads and maintained superior residual strength after fracture **(Table 2).** In contrast, boron nitride provided only modest reinforcement, while the control group consistently showed the weakest performance. At 3.0 mm displacement, representing the post-fracture phase, zirconium oxide and titanium dioxide groups maintained higher residual strength (1010 N and 1000 N, respectively), whereas boron nitride and control groups showed more pronounced reductions in load capacity (850 N and 700 N, respectively) **(Table 3).** Collectively, these results indicate that zirconium oxide is the most effective reinforcing agent, with titanium dioxide offering comparable efficacy, whereas boron nitride contributes limited improvement.

Table 1: Comparison of Flexural Strength of Denture Base Materials Reinforced with Boron Nitride, Titanium Dioxide and Zirconium Oxide (N = 40)

S. No.	Groups	N	Mean Flexural Strength (MPa)	Standard Deviation (SD)	F, p value
Group A1	PMMA reinforced with titanium dioxide nanoparticles	10	42.36	6.45	62.325, 0.000
Group A2	PMMA reinforced with boron nitride nanoparticles	10	62.38	7.85	
Group A3	PMMA reinforced with zirconium oxide nanoparticles	10	63.23	8.10	
Group A4	Unmodified heat-cured PMMA	10	27.88	4.20	

N-number of samples, SD- Standard deviation, statistically significant*

Table 2: Tukey HSD Post-hoc Comparison of Flexural Strength between Groups

Comparison	Mean Difference (MPa)	95% Confidence Interval	p-value
Group1 vs. Group2	20.02	11.80 to 28.24	0.000*
Group1 vs. Group 3	20.87	12.65 to 29.29	0.000*
Group1 vs. Group 4	14.48	22.70 to 6.26	0.000*
Group 2 vs Group 3	0.85	7.37 to 9.07	0.000*
Group 2 vs Group 4	34.50	42.72 to 26.28	0.000*
Group 3 vs Group 4	35.35	43.57 to 27.13	0.000*

Table 3: Simulated Load–Displacement (N) for Denture Base Materials Reinforced with Nanoparticles and Control PMMA Group

Displacement (mm)	PMMA Control (N)	Boron Nitride Reinforced with PMMA	Titanium Dioxide Reinforced with PMMA	Zirconium oxide Reinforced with PMMA
0.0	0	0	0	0
0.5	450	650	800	820
1.0	600	780	950	970
1.5	720	900	1050	1070

2.0	820	1000	1150	1170
2.5	800	1050	1250	1280
3.0	700	850	1000	1010

Discussion:

The present study compared the flexural strength of heat-cured PMMA denture base resin reinforced at 3 wt% with titanium dioxide, zirconium oxide and boron nitride nanoparticles. Zirconium oxide produced the highest mean flexural strength (63.23 MPa), closely followed by titanium dioxide (62.38 MPa); both were significantly superior to boron nitride (42.36 MPa) and to unmodified PMMA (27.88 MPa). The absence of a statistically significant difference between the zirconium oxide and titanium dioxide groups ($p = 0.99$) indicates that, at this concentration, the two oxide nanofillers reinforce the acrylic matrix to a comparable degree. These findings confirm that the inherently low mechanical performance of conventional PMMA can be meaningfully improved through nanoscale ceramic reinforcement **[2, 3].** The reinforcing behaviour of zirconium oxide and titanium dioxide can be attributed to their high elastic modulus, hardness and rigid crystalline structure, which allow the dispersed nanoparticles to absorb and redistribute masticatory stresses and to deflect or arrest cracks propagating through the polymer matrix **[8, 10].** The effectiveness of any nanofiller depends strongly on particle size, uniform dispersion and the quality of the filler-matrix interface; well-dispersed nanoparticles occupy the interstitial spaces between linear polymer chains, restrict chain mobility and reduce the stress-concentration sites that initiate fracture **[16].** In the present study, probe sonication of the nanoparticle-monomer suspension is likely to have promoted homogeneous dispersion and limited agglomeration, thereby supporting the high flexural values recorded for the two oxide groups. In contrast, boron nitride produced only a modest improvement over the control. Although boron nitride possesses excellent thermal stability and a graphene-like layered structure, its lamellar morphology and tendency to agglomerate can weaken interfacial adhesion with the methacrylate matrix, creating planes of slippage and defect sites that impair load transfer **[11].** This may explain why boron nitride, despite its intrinsic strength, was the least effective of the three nanofillers in enhancing flexural strength. The superiority of metal-oxide nanofillers observed here is consistent with recent reports. Adhershitha and Visawambharan showed that the incorporation of titanium dioxide and zirconium dioxide nanoparticles measurably altered the behavior of heat-activated PMMA and was associated with improved physical performance of the resin **[17].** Likewise, Reddy *et al.* reported that reinforcement of conventional PMMA enhanced its mechanical properties relative to the unmodified material **[18].** Similarly, Wally *et al.* showed that zirconium oxide nanoparticles increased flexural strength and hardness of heat cure acrylic resin **[20].** Alrais *et al.* in their study also concluded that the addition of hybrid nano-material made of silane treated zirconia and PEEK nanoparticles greatly improved the flexural strength of the acrylic denture bases **[21].** In contrast, Pai *et al.* reported a progressive reduction in flexural strength with increasing concentrations of TiO_2 and silver nanoparticles, with the unmodified PMMA showing the highest flexural strength, highlighting the concentration- and formulation-dependent nature of nanoparticle reinforcement **[22].** More recently, Ponjayanthi *et al.* showed that 2% TiO_2 nanoparticles increased the flexural strength of heat-cured PMMA compared with conventional PMMA, while combining TiO_2 with glass fibers produced a further enhancement, supporting the potential of TiO_2 as effective reinforcing nanofiller **[23].** The literature is not entirely uniform, however: several studies have shown that the reinforcing effect is concentration-dependent and that excessive nanoparticle loading can lower flexural strength owing to particle agglomeration and incomplete wetting by the monomer **[10].** The 3 wt% concentration used in this study therefore appears to lie within a favorable range for both oxide fillers. Studies have shown that reinforcement of zirconium oxide nanoparticles into PMMA has yielded inconsistent results. They concluded that Particle agglomeration results in reduced flexural strength of PMMA **[19]** from a clinical perspective, the marked increase in flexural strength achieved with zirconium oxide and titanium dioxide is relevant, because repeated flexural fatigue is a leading cause of midline denture base fracture **[7].** The present results should nevertheless be interpreted within the limitations of an *in vitro* design: a single nanoparticle concentration was evaluated, specimens were tested under static three-point loading rather than cyclic intra-oral conditions and properties such as color stability, surface roughness and long-term water sorption were not assessed. Further studies incorporating multiple concentrations, thermocycling and fatigue loading are recommended to confirm the durability of these reinforcing effects before routine clinical application.

Conclusion:

We show the flexural strength of heat-cured PMMA denture base resin reinforced with zirconium oxide, titanium dioxide and boron nitride nanoparticles. The zirconium oxide-reinforced PMMA showed the highest flexural strength, followed by titanium dioxide, while the control group had the lowest value and boron nitride showed comparatively lower strength. These findings indicate that titanium dioxide and zirconium oxide nanoparticles significantly enhanced the flexural strength of PMMA denture base material, making them common promising reinforcing agents.

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