Journal of Scientific and Innovative Research 2026; 15(2): 20-24
Available online at: www.jsirjournal.com

Journal of Scientific & Innovative Research

Research Article

ISSN 2320-4818
JSIR 2026; 15(2): 20-24

Received: 05-04-2026
Accepted: 11-08-2026
Published: 31-08-2026
DOI: 10.31254/jsir.2026.15202

Effect of TiO_2, Al_2O_3, and ZrO_2 Nanoparticles on the Flexural Strength, Surface Hardness, and Surface Roughness of PMMA Denture Base Resin: A Comparative *in vitro* Study

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Abstract

Background: PMMA is widely used in dentistry but has suboptimal mechanical properties. Nanoparticle reinforcement may enhance its performance. **Objective:** To evaluate the effect of 3 wt.% and 7 wt.% TiO_2, Al_2O_3, and ZrO_2 nanoparticles on flexural strength, surface hardness, and surface roughness of heat-cured PMMA. Study setting and design: An *in vitro* study **Materials and Methods:** A total of 105 PMMA specimens were divided into seven groups (n = 15): one control and six nanoparticle-reinforced groups (3 wt.% and 7 wt.% of each oxide). Flexural strength was tested using a universal testing machine, surface hardness was measured with Vickers hardness tester, and surface roughness was assessed using a profilometer; statistical analysis was performed using Bonferroni post hoc tests. **Results:** Flexural strength was highest in the 7 wt.% ZrO_2 group, followed by 3 wt.% ZrO_2, while the control group showed the lowest strength. Surface hardness was greatest in the 7 wt.% ZrO_2 group, followed by the 3 wt.% ZrO_2 and TiO_2 groups. Surface roughness was highest in the 7 wt.% TiO_2 group, whereas the control and ZrO_2 groups exhibited the lowest roughness values. **Conclusion:** Nanoparticles significantly improved PMMA's mechanical properties. The 3 wt.% ZrO_2 group offered the best balance of strength, hardness, and minimal roughness, suggesting it as the optimal nanofiller for clinical use.

Keywords: PMMA Denture Base Resins, TiO_2, Al_2O_3, ZrO_2 Nanoparticles, Flexural Strength, Surface Hardness, Surface Roughness.

INTRODUCTION

Complete and removable partial dentures remain reliable options for replacing missing teeth and restoring oral function, improving aesthetics, speech, and mastication. Over time, denture base materials have evolved, with polymethylmethacrylate (PMMA) becoming the most commonly used due to its favorable aesthetics, ease of handling, cost-effectiveness, and acceptable performance [1].

However, PMMA has inherent limitations, including brittleness and low impact resistance, which may lead to fractures during use or accidental drops [2]. Various reinforcements, such as metal wires, plates, fibers, and elastomers, have been used to overcome these issues but often result in drawbacks like poor bonding, corrosion, high cost, or tissue irritation [2].

Nanoparticle reinforcement has emerged as a promising alternative to enhance PMMA's mechanical properties. Due to their increased surface area and interaction with polymer chains, nanoparticles can significantly improve strength, wear resistance, and surface characteristics [3,4]. The performance of nanocomposites depends on particle type, size, dispersion, and concentration [2,5,6].

Among potential fillers, titanium dioxide (TiO_2) offers biocompatibility, chemical stability, and antimicrobial properties, along with improved strength and surface characteristics [2]. Aluminium oxide (Al_2O_3) improves hardness and thermal stability with minimal color interference, and has been shown to enhance flexural strength [6]. Zirconium dioxide (ZrO_2), a strong and aesthetic bioceramic, enhances fracture toughness and reduces water sorption, particularly at 5 wt.% concentration [7].

Surface roughness also plays a critical role in hygiene and comfort, as smoother surfaces limit microbial adhesion and plaque accumulation [8]. While individual effects of these nanoparticles have been studied, direct comparative data under standardized conditions is limited.

This study aims to evaluate and compare the effects of incorporating 3 wt.% and 7 wt.% of TiO_2, Al_2O_3, and ZrO_2 nanoparticles on the flexural strength, surface hardness, and surface roughness of PMMA.

MATERIALS AND METHODS

This *in-vitro* study was conducted at the Department of Prosthodontics, Vydehi Institute of Dental Sciences and Research Centre, Bangalore, Karnataka, India (Certificate No. OC/IEC/PG/APP/2023/59, dated 15/105/2023). The study aimed to compare and evaluate the flexural strength, surface hardness, and surface roughness of heat-cured acrylic resin reinforced with different concentrations of titanium dioxide (TiO_2), aluminium oxide (Al_2O_3), and zirconium oxide (ZrO_2) nanoparticles.

A metal mold (65 × 10 × 2.5 mm) was used for specimen fabrication. Materials included heat-cured acrylic resin (DPI), modeling wax, Type III dental stone (Kalabhai), and nanoparticles of TiO_2, Al_2O_3, and ZrO_2 (30–50 nm) with 99.56% purity. A silane coupling agent (Silane-X) was used to treat the nanoparticles. Equipment used for testing included a Universal Testing Machine (UTM), Digital Vickers Microhardness Tester, and a non-contact optical interferometric profilometer.

The sample distribution consisted of a control group and several experimental groups. The control group included 15 specimens. The experimental groups were divided based on type and concentration of nanoparticles added: titanium dioxide (TiO_2) at 3% and 7%, aluminum oxide (Al_2O_3) at 3% and 7%, and zirconium dioxide (ZrO_2) at 3% and 7%, with each group containing 15 specimens. Each set of 15 specimens was further subdivided for testing three different properties: flexural strength, surface hardness and surface roughness.

For sample preparation, a metal mold with internal dimensions of 65 mm × 10 mm × 2.5 mm was fabricated in accordance with ISO 1567 standards to ensure specimen standardization.

A total of 105 wax patterns were prepared by pouring molten modeling wax into a lubricated metal mold. After cooling, defect-free patterns were selected. These were invested in dental plaster using a mechanical vibrator to minimize air bubbles. Following plaster setting, dewaxing was performed at 100 °C for 5 min, and residual wax was removed with boiling water. The mold was air-cooled, and a separating medium (Cold Mould Seal; DPI) was applied.

For specimen fabrication, fifteen control specimens were produced using heat-cured acrylic resin without nanoparticle reinforcement. The resin powder and monomer were mixed in a 3:1 ratio and packed into molds during the dough stage. After trial closure, the flasks were left to bench-cure for 1 h, followed by polymerization using a short curing cycle at 74 °C for 2 h with a final boiling phase lasting 1 h. After bench cooling, the specimens were deflasked, trimmed, finished, and polished.

Ninety experimental specimens were fabricated by incorporating TiO_2, Al_2O_3, and ZrO_2 nanoparticles into acrylic resin at 3 wt.% and 7 wt.% concentrations (Figure 1). Nanoparticles were pre-treated with 1 wt% silane to enhance dispersion and bonding. The modified powders were mixed with monomer in a 3:1 ratio and processed like the control group. Specimens were inspected for defects, finished with sandpaper, polished with pumice, and measured using a Vernier caliper. The accepted specimens were stored in distilled water at 37 °C for one week to enhance the release of residual monomer and minimize cytotoxicity.

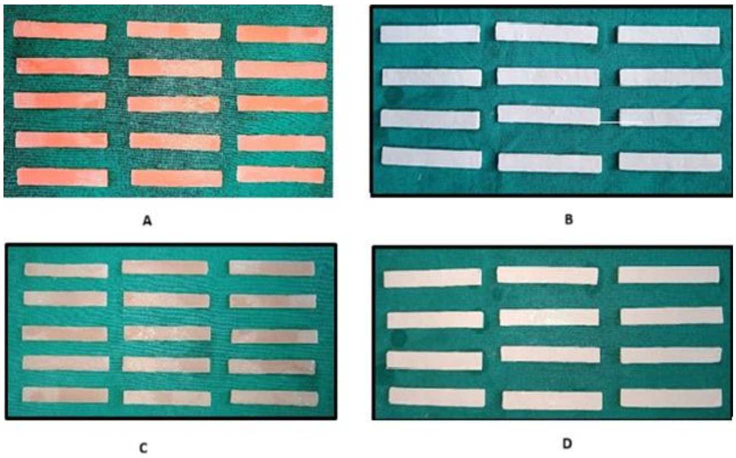


Figure 1: A (Heat cured samples), B (TiO_2 nano particle reinforced specimens), C (Al_2O_3 nano particle reinforced specimens), D (ZrO_2 nano particle reinforced specimens)

The testing procedures involved assessing flexural strength, surface hardness, and surface roughness. Flexural strength was measured using a Universal Testing Machine (UTM) with a three-point bending configuration at a crosshead speed of 5 mm/min. The fracture load was recorded digitally, and the strength was calculated using the machine's software (Figure 2). Surface hardness was evaluated with a Digital Vickers Microhardness Tester, applying a 50-gf load for 5 s; Vickers Hardness Numbers (VHN) were recorded digitally (Figure 3). Surface roughness was measured using a non-contact optical profilometer, with each specimen scanned at three different points to obtain an average Ra value in micrometres (Figure 4).

Figure 2: Testing of samples using Universal testing machine

Figure 3: Testing of samples using Vickers hardness testing machine

Figure 4: Testing of samples using Profilometer

Statistical Analysis

All statistical analyses were performed using SPSS Version 26.0, with the level of significance set at $p < 0.05$. Group comparisons were carried out using one-way ANOVA followed by Bonferroni post hoc tests.

RESULTS

Table 1 summarizes the mean and standard deviation values of flexural strength, surface hardness, and surface roughness for PMMA and nanoparticles reinforced PMMA groups.

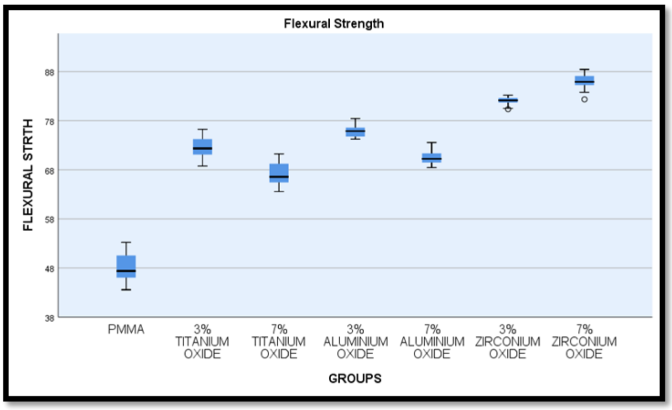
Table 1: Comparison of flexural strength, surface hardness and surface roughness between control group and reinforced groups

		Flexural Strength		Surface Hardness		Surface roughness	
	N	Mean	SD	Mean	SD	Mean	SD
PMMA	15	48.14	3.143	18.29	0.168	4.478	0.17033
3% Titanium Oxide	15	72.42	2.387	25.60	1.433	6.551	0.22925
7% Titanium Oxide	15	67.20	2.366	27.99	0.923	7.254	0.16881
3% Aluminium Oxide	15	75.91	1.178	21.31	0.515	5.342	0.22713
7% Aluminum Oxide	15	70.62	1.590	23.10	0.659	6.419	0.23505
3% Zirconium Oxide	15	81.96	0.884	31.09	0.667	4.557	0.19274
7% Zirconium Oxide	15	85.96	1.655	32.74	0.472	5.421	0.17908

Flexural Strength

One-way ANOVA showed a statistically significant difference in flexural strength among the groups ($F = 552.62$, $p = 0.0001$). The highest mean value was observed in 7% Zirconium Oxide (85.96 MPa), followed by 3% Zirconium Oxide (81.96 MPa), 3% Aluminium Oxide (75.91 MPa), 3% Titanium Oxide (72.42 MPa), 7% Aluminium Oxide (70.62 MPa), 7% Titanium Oxide (67.20 MPa), and PMMA (48.14 MPa).

Post hoc analysis confirmed that all nanoparticle-reinforced groups had substantially higher flexural strength than PMMA ($p = 0.0001$), with the most pronounced improvement seen in 7% Zirconium Oxide (mean difference = -37.82 MPa). Among reinforced groups, zirconium oxide consistently outperformed others, and its effect was concentration-dependent. The difference between 3% Titanium Oxide and 7% Aluminium Oxide was not significant ($p = 0.351$), indicating that both nanoparticle type and concentration play critical roles in enhancing flexural strength.

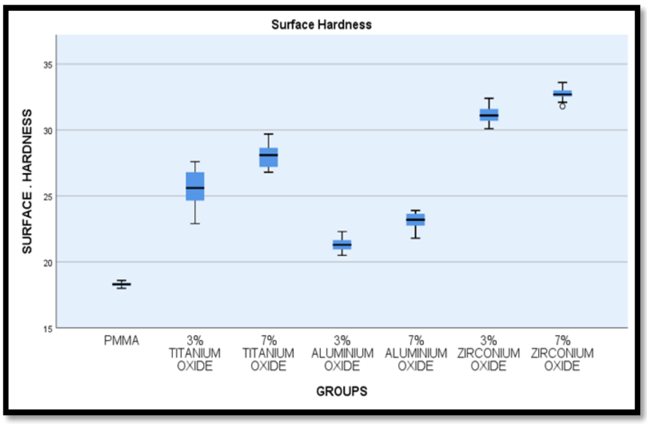


Graph 1: Boxplot of flexural strength by group

Surface Hardness

One-way ANOVA showed a statistically significant difference in surface hardness among groups ($F = 671.28$, $p = 0.0001$). Mean values in ascending order were: PMMA (18.29), 3% Aluminium Oxide (21.31), 7% Aluminium Oxide (23.10), 3% Titanium Oxide (25.60), 7% Titanium Oxide (27.99), 3% Zirconium Oxide (31.09), and 7% Zirconium Oxide (32.74).

Post hoc analysis showed all group differences were statistically significant ($p = 0.0001$). The greatest improvement was seen in 7% Zirconium Oxide (mean difference from PMMA = -14.453). Zirconium Oxide demonstrated the highest hardness overall, with a significant concentration-dependent increase ($p = 0.0001$). Similar trends were observed for Titanium and Aluminium Oxides. These findings confirm that both the type and concentration of metal oxide nanoparticles significantly increase the surface hardness of PMMA, with 7% Zirconium Oxide showing the most substantial effect.

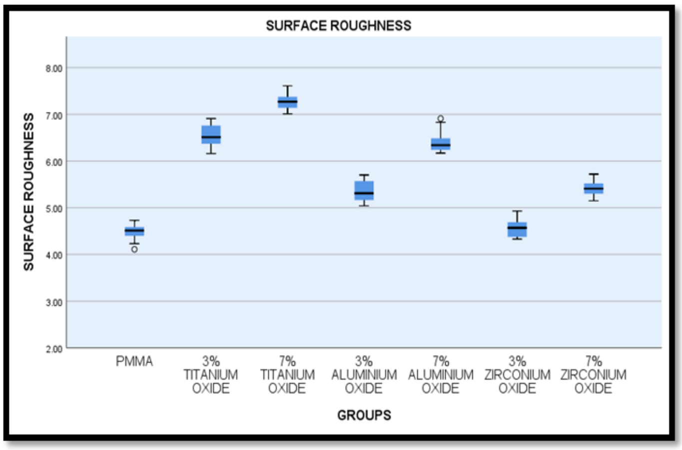


Graph 2: Boxplot of surface hardness by group

Surface Roughness

One-way ANOVA showed a statistically significant difference in surface roughness among groups ($F = 407.27$, $p = 0.0001$). Mean surface roughness values in ascending order were: PMMA (4.478 ± 0.170), 3% Zirconium Oxide (4.557 ± 0.193), 3% Aluminium Oxide (5.342 ± 0.227), 7% Zirconium Oxide (5.421 ± 0.179), 7% Aluminium Oxide (6.419 ± 0.235), 3% Titanium Oxide (6.551 ± 0.229), and 7% Titanium Oxide (7.254 ± 0.169).

Post hoc analysis showed that PMMA had significantly lower surface roughness than all other groups, except 3% Zirconium Oxide ($p = 1.000$). 7% Titanium Oxide had the highest roughness and was significantly different from all other groups ($p < 0.0001$). Significant differences were observed between concentrations of each oxide type, confirming that both nanoparticle type and concentration influence surface roughness, with higher concentrations generally increasing it.



Graph 3: Boxplot of Surface Roughness by Group

DISCUSSION

Polymethyl methacrylate (PMMA) has traditionally been the preferred material for denture bases owing to its advantageous properties, including handling properties, biocompatibility, acceptable esthetics, and cost-effectiveness. Despite its widespread use, PMMA has intrinsic mechanical limitations, particularly low impact and flexural strength, which leads to fractures during functional or accidental stresses such as mastication or denture dropping.

Flexural strength, surface hardness, and surface roughness are essential parameters for evaluating denture base material performance. Flexural strength, in particular, is vital as dentures must withstand repeated masticatory loading without deformation or fracture. Surface hardness contributes to wear resistance and ease of polishing, while surface roughness affects microbial adhesion, plaque accumulation, and esthetics.

To enhance the performance of PMMA, the incorporation of nanoparticles such as titanium dioxide (TiO_2), aluminium oxide (Al_2O_3), and zirconium dioxide (ZrO_2) has gained increasing attention. Nanoparticles, due to their high surface area-to-volume ratio, have the potential to bond more effectively with polymer matrices, resulting in superior mechanical performance.

This study showed that flexural strength was significantly improved in all nanoparticle-reinforced groups compared to unmodified PMMA. The group reinforced with 7% ZrO_2 exhibited the highest flexural strength (85.96 MPa), followed by 3% ZrO_2, 3% Al_2O_3, and 3% TiO_2. The enhanced strength in ZrO_2 groups can be attributed to its small particle size and uniform dispersion, allowing the nanoparticles to fill intermolecular spaces in the PMMA matrix. This reduces chain mobility, increases stiffness, and restricts segmental motion of the macromolecules, ultimately resulting in higher fracture resistance.

Supporting studies by Ayad *et al* and Asopa *et al* have shown that ZrO_2 added in 5–15% concentrations significantly improves transverse and flexural strength of PMMA. Azmy *et al* also found that 3% and 7% ZrO_2 increased the flexural and impact strength significantly compared to control PMMA, mirroring the current study's findings [2].

In contrast, while TiO_2 and Al_2O_3 at 3% concentrations improved flexural strength, their 7% counterparts showed a decline. This was likely due to nanoparticle agglomeration, where high filler loading exceeds the polymer's capacity to distribute particles evenly. Such clusters can act as stress concentration zones, creating weak points that promote crack initiation and propagation under load. Harini *et al*. reported similar observations, noting that TiO_2 nanoparticles increased flexural strength only up to 5% concentration, beyond which the material became brittle due to disruption of the polymer matrix [11]. Pokuri *et al*. observed the same trend with Al_2O_3, where 2.5% loading yielded the highest flexural strength, while higher concentrations caused weakening [12].

With respect to surface hardness, all nanoparticle-reinforced PMMA groups outperformed the control group. Notably, 7% ZrO_2 again demonstrated the highest Vickers Hardness Number (VHN), indicating superior wear resistance. This improvement can be linked to the high hardness and strong ionic bonding of ZrO_2, which increases the energy required to deform the polymer matrix. Titanium dioxide and aluminium oxide also enhanced surface hardness in a concentration-dependent manner, with 7% TiO_2 outperforming 3% TiO_2, and similarly for Al_2O_3. Surface hardness is critical as it affects the wear resistance, long-term appearance, and hygiene of the denture.

Studies by Pokuri *et al*. corroborate these findings, reporting that nano-Al_2O_3 and nano-TiO_2 reinforcement significantly improved hardness when incorporated in optimal concentrations [12]. Similarly, Alwan and Alameer observed improved hardness and strength upon adding silanized 3% TiO_2 [13].

The surface roughness outcomes showed a less favorable trend, with all nanoparticle additions increasing roughness compared to the control. The most significant increase was observed in the 7% TiO_2 group (7.254 ± 0.169 µm), followed by 3% TiO_2 and 7% Al_2O_3, whereas the 3% and 7% ZrO_2 groups showed the least roughness increase, closely matching the control. Increased surface roughness is likely due to nanoparticle agglomeration at higher concentrations and microstructural heterogeneity between the fillers and the polymer matrix. Moreover, during polishing, loosely bound or poorly integrated particles may dislodge, leaving behind microvoids or rough patches.

Clinically, rough surfaces can retain more plaque and food debris, and offer ideal conditions for Candida albicans adhesion and microbial colonization, potentially leading to denture stomatitis. According to Bollen *et al*., a surface roughness above 0.2 µm significantly increases plaque retention, making it essential to maintain smoothness within clinically acceptable limits. Although TiO_2 offers mechanical benefits, its tendency to increase roughness at higher concentrations necessitates caution. Similar conclusions were drawn by Chowdhury *et al*. and Aljafery *et al*., who observed that TiO_2 and Al_2O_3 nanoparticles significantly increased roughness when not uniformly dispersed or when added at higher concentrations [7,14].

Overall, this study reveals that ZrO_2 nanoparticles are the most effective in enhancing PMMA's performance, showing substantial improvements in both flexural strength and surface hardness while minimally impacting surface roughness. This makes ZrO_2 a promising candidate for clinical applications in areas of high functional stress such as single complete dentures opposing natural teeth, distal extension bases, long-term interim prostheses, and implant-retained dentures. Titanium and aluminium oxides also improve mechanical properties but require optimal concentrations ($\leq$3%) to avoid drawbacks related to roughness and decreased strength.

Despite their value, the present study had some limitations. Only a single type of heat-cured PMMA resin was evaluated, and all experiments were conducted under *in vitro* conditions, thus lacking the complexity of the oral environment, including thermal fluctuations, pH variations, enzymatic activity, and microbial interactions. Additionally, long-term durability, color stability, and biocompatibility of the modified materials under intraoral conditions require further investigation.

Future studies should examine multiple commercial PMMA brands, varied nanoparticle combinations, and include *in vivo* or in situ testing. Simulated aging protocols, such as thermocycling, water sorption, and fatigue loading, should also be incorporated to better reflect the clinical scenario. Finally, evaluating cytotoxicity and inflammatory responses to these nanoparticles will be crucial before widespread clinical adoption.

CONCLUSION

Based on the methodology and results, the following conclusions were drawn:

1. Nanoparticles improved PMMA's flexural strength, with 7% ZrO_2 showing the highest values, followed by 3% ZrO_2 and 3% Al_2O_3. ZrO_2 was the most effective overall.
2. All reinforced groups had higher surface hardness than the control, with 7% ZrO_2 being the highest. Hardness depended on both nanoparticle type and concentration.
3. Surface roughness increased with nanoparticle addition, highest in 7% TiO_2. 3% ZrO_2 had minimal impact, closest to the control.

Overall, 3% ZrO_2 offered the best balance, enhancing strength and hardness while maintaining low roughness, making it the most suitable for clinical use.

Author Contributions

AKP contributed to the conceptualization and design of the study, investigation, methodology, data analysis and processing, and drafting of the original manuscript. SR and AK contributed to review and editing, validation, and supervision of the study. AP contributed to the investigation and data analysis. All authors reviewed and approved the final version of the manuscript and agreed to be accountable for all aspects of the work.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Use of AI in Drafting of Manuscript

The authors declare that they have not used any generative AI/AI-assisted technologies in the writing of this manuscript.

Conflict of interest

The authors declare no conflict of interest.

Financial Support

None declared.

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