RESEARCH PAPER

Effect of Zinc Oxide and Aluminium Oxide Nanoparticles on Flexural Strength and Surface Roughness of Provisional Restorative Materials: An In Vitro Study.

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ABSTRACT

Background & Objective: Provisional restorations are indispensable in contemporary prosthodontic practice. However, their performance in clinical practice is limited by inadequate flexural strength and suboptimal surface characteristics. This in vitro study aimed to evaluate the effects of incorporating zinc oxide (ZnO) and aluminium oxide (Al_2O_3) nanoparticles at concentrations of 3% and 5% by weight, on the flexural strength and surface roughness DPI Tooth Moulding Material (polymethyl methacrylate [PMMA]-based) and Protemp 4 (bis-acryl composite resin–based).

Materials and Methods: Forty eight rectangular specimens (25 × 2 × 2 mm) were fabricated per material and divided into four experimental groups incorporating 3% ZnO, 5% ZnO, 3% Al_2O_3, and 5% Al_2O_3 nanoparticles, with six specimens per subgroup. After standardised finishing, polishing, and conditioning in artificial saliva for seven days, the flexural strength and surface roughness was assessed. Data were analysed using one-way ANOVA and post hoc independent t-tests.

Results: Incorporation of nanoparticle significantly improved flexural strength in both materials ($p < 0.001$). In DPI, 5% ZnO achieved the highest mean flexural strength (84.98 ± 0.74 MPa), followed by 5% Al_2O_3 (82.87 ± 0.81 MPa), 3% ZnO (78.97 ± 0.79 MPa), and 3% Al_2O_3 (76.97 ± 0.74 MPa). Corresponding values for Protemp 4 were 74.98 ± 0.75, 72.87 ± 0.79, 68.97 ± 0.82, and 66.97 ± 0.76 MPa. Surface roughness increased proportionally with nanoparticle concentration, with 5% ZnO yielding the highest Ra values in both materials ($p < 0.001$).

Conclusion: Incorporation of ZnO and Al_2O_3 nanoparticles significantly enhanced the flexural strength of provisional restorative materials. ZnO nanoparticles at 5% concentration yielded the greatest mechanical improvement.

Keywords: .Provisional restorations; zinc oxide nanoparticles; aluminium oxide nanoparticles; flexural strength; surface roughness; PMMA; bis-acryl composite resin; nanotechnology; prosthodontics

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INTRODUCTION

Provisional restorations hold a key position in the field of prosthodontics. They serve as the critical interface between tooth preparation and definitive prosthesis placement.[1] Beyond their transitional role, they perform essential clinical functions such as preservation of prepared enamel and dentinal surfaces, maintenance of arch integrity and occlusal stability, restoration of phonetics and aesthetics, facilitation of periodontal conditioning, and provide diagnostic information regarding occlusal scheme and phonetic requirements.[2] With the rise of complex prosthodontic rehabilitations, which includes full-mouth reconstruction, implant-supported prostheses, and extended-span restorations, provisional materials which endure prolonged intraoral service, often spanning several months are largely sought after.[3]

Polymethyl methacrylate (PMMA)-based resins and bis-acryl composite resins are two material categories that currently dominate available provisional materials in clinical practice.[4] Powder-liquid PMMA resins are affordable and easy to handle and have good esthetic properties. Unfortunately, they suffer from drawbacks such as shrinkage, cytotoxicity, pulpal irritation due to heat generation during polymerization, and microcracking.[5] Bis-acryl composite resins, including Protemp 4, offer better dimensional stability, lower exotherm, and better aesthetics.

Despite all their benefits, bis-acryl composites are highly fragile in situations that require higher functionality or longer spans.[6]

Flexural strength and surface roughness are extremely consequential properties of provisional materials in clinical practice. Flexural strength demonstrates the resistance to bending forces which is a key determinant of longevity in restorations under cyclical masticatory loads.[7] Surface roughness (Roughness Average Ra) directly influences plaque biofilm adhesion, microbial colonization, and periodontal tissue health. Rougher surfaces attract bacterial retention and increase the risk of gingival irritation.[8] It is well established that Ra values exceeding 0.2 µm serve as a threshold above which adhesion of bacteria significantly increases.[9]

Nanotechnology has emerged as a compelling choice for augmenting the properties of resin-based dental materials.[10] Nanoparticles are particles that have at least one dimension lying between 1 and 100 nm and offer improved mechanical, thermal, and biological properties owing to their unusually large surface area-to-volume ratios and ability to enable close polymer-filler coupling. Of the various nanoparticles available, ZnO and Al_2O_3 nanoparticles are especially significant in the dental field. The former have gained popularity on account of their proven antimicrobial properties resulting from reactive oxygen species formation and zinc ion release along with their mechanical reinforcing properties.[11] Al_2O_3 nanoparticles are well known for their exceptional hardness, chemical inertness, and capacity to improve flexural strength and wear resistance in composite systems.[12]

Previous studies have demonstrated the efficacy of various nanoparticles in enhancing provisional PMMA-based materials. Comparative studies specifically examining ZnO and Al_2O_3 nanoparticles in clinically relevant provisional systems, particularly bis-acryl composite resins, remain limited. The present study was therefore designed to evaluate and compare the effect of incorporating ZnO and Al_2O_3 nanoparticles at concentrations of 3% and 5% by weight into DPI Tooth Moulding Material and Protemp 4, assessing their influence on flexural strength and surface roughness under simulated intraoral conditioning.

MATERIALS AND METHODS

Study Design

This investigation follows the design of an in vitro comparative experimental study. No human participants or animal subjects were involved. Therefore, institutional ethics committee approval was not mandated. The study was conducted in accordance with the CRIS (Conducting and Reporting of In vitro Studies) guidelines.[13]

Sample Size Determination

Sample size was calculated using OpenEpi software (Version 3.04) with a 95% confidence interval, α error of 5%, β error of 20%, and power of 80%. A minimum of six specimens per subgroup was determined to be statistically adequate. Each material comprised four subgroups, yielding 24 specimens per material and a total of 48 specimens across both materials.

Materials

Two provisional restorative materials were selected: DPI Tooth Moulding Material (Dental Products of India, Mumbai), a self-curing PMMA powder–liquid system; and Protemp 4 Temporization Material (3M ESPE, USA), an automixed bis-acryl composite system. ZnO nanoparticles and Al_2O_3 nanoparticles with particle sizes of 30–50 nm were procured for incorporation at 3% and 5% concentrations by weight. Artificial saliva was prepared by dissolving KCl (0.4 g L^{-1}), NaCl (0.4 g L^{-1}), $CaCl_2 \cdot 2H_2O$ (0.906 g L^{-1}), $Na_2HPO_4 \cdot 2H_2O$ (0.690 g L^{-1}), $Na_2S \cdot 9H_2O$ (0.005 g L^{-1}), and urea (1 g L^{-1}) in distilled water, and the pH was adjusted to 6.528.

Group Allocation

Specimens were allocated into the following groups (n = 6 per subgroup):

DPI groups: Group A (3% ZnO); Group B (5% ZnO); Group C (3% Al_2O_3); Group D (5% Al_2O_3).

Protemp 4 groups: Group A1 (3% ZnO); Group B1 (5% ZnO); Group C1 (3% Al_2O_3); Group D1 (5% Al_2O_3).

Specimen Fabrication

Rectangular specimens measuring 25 mm × 2 mm × 2 mm were fabricated in accordance with ISO 10477:2018.[14] Standardised wax patterns of these dimensions were invested in polyvinyl siloxane impression material to produce uniform moulds. For DPI specimens, polymer powder and monomer liquid were combined in a 3:1 ratio by volume. In experimental groups, nanoparticles were incorporated into the polymer powder using a calibrated digital balance prior to mixing and homogenised manually for 60 seconds to promote uniform dispersion. For Protemp 4 control specimens, the material was dispensed via an automixing gun. In experimental Protemp 4 groups, nanoparticles were incorporated into the base paste prior to dispensing and mixed manually. All specimens were allowed to polymerise per manufacturer instructions before demolding.

Finishing, Polishing, and Conditioning

All specimens were finished using 600-grit wet abrasive paper with a standardised number of strokes per surface, followed by polishing with pumice slurry on a low-speed buffing wheel for a fixed duration. All finishing and polishing was done by one operator only in order to minimize errors due to inter-operator variability. Specimens were immersed in distilled water for 24 hours first, followed by artificial saliva for seven days at 37 °C.

Surface Roughness Assessment

The surface roughness measurement was done via a digital contact profiler (Surface Roughness Tester

SJ Series, Mitutoyo, Japan). The stylus scanned along the long axis of the specimens, and three consecutive measurements were made for each sample. The value of Roughnes Average (Ra) in microns (µm) was computed and analyzed statistically.

Flexural Strength Testing

Three-point bending tests were conducted using a universal testing machine (UNITEST 10; Acme Engineers, India) at a crosshead speed of 1 mm/min. The load was applied at the midpoint of each specimen, supported at two points 20 mm apart, until fracture. Flexural strength (σ, MPa) was calculated using the formula:

$\sigma = 3WL / 2bd^2$

where W = maximum load at fracture (N), L = span length (mm), b = specimen width (mm), and d = specimen thickness (mm).

Statistical Analysis

The data was entered in Microsoft Excel spreadsheets and was analyzed by using IBM SPSS Version 25.015[15]. Normality of distribution was tested using the Kolmogorov-Smirnov test and Shapiro-Wilk test. Mean value ± Standard deviation was estimated for all the groups. Differences among groups were assessed by using one-way analysis of variance (ANOVA). Paired comparison among the groups was assessed by independent sample t-test using 95% confidence interval (CI).

RESULTS

Flexural Strength – DPI Tooth Moulding Material

Descriptive statistics for DPI flexural strength are presented in Table 1. Group B (5% ZnO) demonstrated the highest mean flexural strength (84.98 ± 0.74 MPa), followed by Group D (5% Al_2O_3) at 82.87 ± 0.81 MPa, Group A (3% ZnO) at 78.97 ± 0.79 MPa, and Group C (3% Al_2O_3) at 76.97 ± 0.74 MPa. The low standard deviation values (< 1 MPa) across all groups indicated high intragroup consistency. One-way ANOVA revealed a highly significant intergroup difference (F = 114.79, $p < 0.001$) (Table 2). Post hoc pairwise comparisons confirmed statistically significant differences between all group pairs ($p < 0.05$), with 95% confidence intervals not crossing zero in any comparison (Table 3).

Table 1. Descriptive statistics of flexural strength (MPa) among experimental groups of DPI Tooth Moulding Material

Group	n	Mean (MPa)	SD (MPa)	Rank
A (3% ZnO)	6	78.97	0.79	3rd
B (5% ZnO)	6	84.98	0.74	1st
C (3% Al_2O_3)	6	76.97	0.74	4th
D (5% Al_2O_3)	6	82.87	0.81	2nd
ANOVA: F = 114.79, p < 0.001				

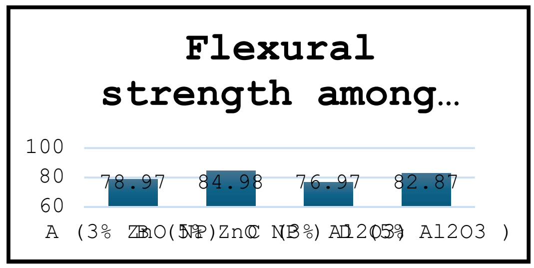

Figure 1: Flexural strength among different groups of DPI tooth moulding material

Surface Roughness – DPI Tooth Moulding Material

Surface roughness data for DPI are shown in Table 2. Group B (5% ZnO) yielded the highest mean Ra value (94.98 ± 0.75 µm), followed by Group D (92.87 ± 0.79 µm), Group A (88.97 ± 0.82 µm), and Group C (86.97 ± 0.76 µm). One-way ANOVA revealed highly significant intergroup differences (F = 118.32, $p < 0.001$). All pairwise post hoc comparisons were statistically significant ($p < 0.05$), with confidence intervals excluding zero, confirming the reliability of findings.

Table 2. Descriptive statistics of surface roughness (µm) among experimental groups of DPI Tooth Moulding Material

Group	n	Mean Ra (µm)	SD (µm)	Rank
A (3% ZnO)	6	88.97	0.82	3rd
B (5% ZnO)	6	94.98	0.75	1st
C (3% Al_2O_3)	6	86.97	0.76	4th

D (5% Al_2O_3)	6	92.87	0.79	2nd
ANOVA: F = 118.32, p < 0.001				

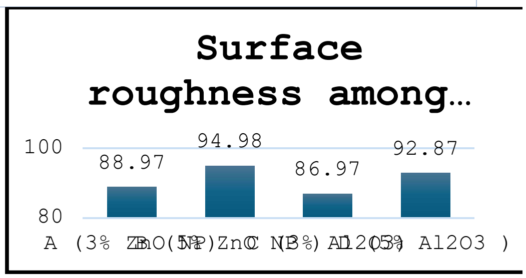

Figure 2: Surface roughness (µm) among different groups of DPI tooth moulding material

Flexural Strength – Protemp 4

The flexural strength for Protemp 4 is presented in Table 3. Group B1 (5% ZnO) recorded the highest mean value (74.98 ± 0.75 MPa), followed by Group D1 (72.87 ± 0.79 MPa), Group A1 (68.97 ± 0.82 MPa), and Group C1 (66.97 ± 0.76 MPaSignificant group differences were found using one-way ANOVA ($F = 116.95$, $p < 0.001$). Statistical differences were obtained between all pairs of groups ($p < 0.05$), as noted in the results of DPI. The results of absolute values of flexural strength for DPI were higher than those of Protemp 4.

***Table 3.** Descriptive statistics of flexural strength (MPa) among experimental groups of Protemp 4*

Group	n	Mean (MPa)	SD (MPa)	Rank
A1 (3% ZnO)	6	68.97	0.82	3rd
B1 (5% ZnO)	6	74.98	0.75	1st
C1 (3% Al_2O_3)	6	66.97	0.76	4th
D1 (5% Al_2O_3)	6	72.87	0.79	2nd
ANOVA: F = 116.95, p < 0.001				

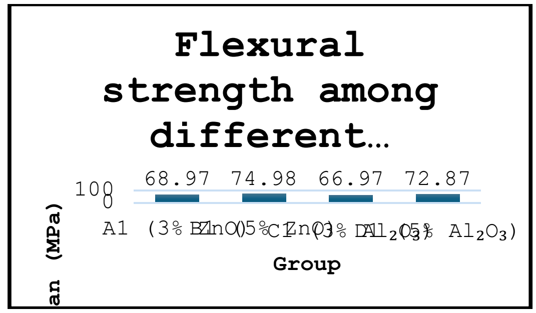

***Figure 3:** Flexural strength among different groups of Protemp4 material*

Surface Roughness – Protemp 4

Surface roughness for Protemp 4 is summarised in Table 4. Group B1 (5% ZnO) recorded the highest Ra (64.98 ± 0.75 µm), followed by Group D1 (62.87 ± 0.80 µm), Group A1 (58.97 ± 0.83 µm), and Group C1 (56.97 ± 0.76 µm). The one-way ANOVA test showed extremely significant differences between the groups ($F=117.88$, $p<0.001$). A post-hoc test revealed statistically significant results for all pairs ($p<0.05$), with none of the confidence intervals overlapping zero. The ranking of surface roughness was similar to that of flexural strength.

Table 4. Descriptive statistics of surface roughness (µm) among experimental groups of Protemp 4

Group	n	Mean Ra (µm)	SD (µm)	Rank
A1 (3% ZnO)	6	58.97	0.83	3rd
B1 (5% ZnO)	6	64.98	0.75	1st
C1 (3% Al_2O_3)	6	56.97	0.76	4th
D1 (5% Al_2O_3)	6	62.87	0.79	2nd
ANOVA: F = 117.88, p < 0.001				

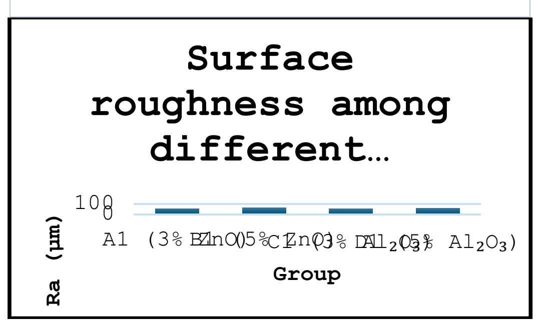

Figure 4: Surface roughness among different groups of Protemp4 material

DISCUSSION

The present study provides substancial evidence that the incorporation of ZnO and Al_2O_3 nanoparticles exerts a measurable influence on the mechanical and surface properties of provisional restorative materials. The results of this study are interpreted in the context of material chemistry, nanoparticle reinforcement mechanisms, and comparative data from the existing literature.

A statistically significant improvement was observed in flexural strength across all nanoparticle-reinforced groups. This is consistent with the established mechanisms of particulate reinforcement in polymer systems. Nanoparticles, due to their high surface area-to-volume ratio, improve the interfacial adhesion within the polymer matrix, redistribute applied stress homogeneously, reduce the incidence and propagation of internal microcracks, and impede crack growth under loading.[17] These synergistic effects translate to enhanced resistance to bending fracture.

The superiority of ZnO nanoparticles over Al_2O_3 at equivalent concentrations in this study is owed to the higher degree of polymer chain interaction and reportedly superior dispersion characteristics of ZnO nanoparticles within resin matrices.[18] This finding is in agreement with the work of **Vikram et al. (2020)**[15], who demonstrated that ZnO nanoparticle incorporation into PMMA at the concentration of 5% produced the greatest enhancement in flexural strength. This effect is attributed to reinforced interfacial bonding and reduced internal defect density. Similarly, **Jehan et al. (2023)**[19] studied PMMA reinforced with ZrO_2, TiO_2, and Al_2O_3 nanoparticles (2.5%) and reported that all three nanoparticle types improved flexural strength and surface hardness significantly, though the magnitude of improvement varied in terms of nanoparticle chemistry, parallel to the present study where ZnO consistently outperformed Al_2O_3.

A concentration-dependent improvement in flexural strength was observed in this study, with 5% yielding higher values than 3% for both nanoparticle types. This supports the findings of **Thakur et al. (2019)**[20], who demonstrated that higher concentrations of nano-zirconia and nano-titanium dioxide in heat-cured PMMA produced greater transverse strength proportionally. However, their study also cautioned that concentrations exceeding optimal thresholds may trigger particle agglomeration and compromise the reinforcing effect. In the present study, 5% appeared to represent the optimal concentration, though concentrations beyond this limit were not evaluated.

It is apparent that the greater absolute flexural strength value of DPI when compared to Protemp 4 can be attributed to the inherent difference in their composition. The PMMA polymer chain structure of DPI, along with the use of benzoyl peroxide in the polymerisation process, makes it easier for the nanoparticles to integrate into the three-dimensional structure. Protemp 4 is a bis-acryl resin based material with a brittle matrix of BIS-GMA, as outlined by Rajput et al. (2022)[21], who reported that DPI exhibited superior initial flexural strength compared to Protemp 4 in direct testing. Their study noted that bis-acryl materials, despite dimensional advantages, are more prone to fracture under bending loads.

The findings are also favourable with the work of **Vojdani et al. (2012)**[22], who observed that the incorporation of Al_2O_3 nanoparticle to acrylic resin improved both flexural strength and surface hardness significantly. They concluded that alumina nanoparticles increase stiffness and resistance to deformation. The present study affirms these findings and situates Al_2O_3 as an effective, if less potent, alternative to ZnO for provisional material reinforcement. **Kaurani et al. (2017)**[23], in their systematic review on nanoparticle-reinforced PMMA denture base materials, an impressive improvement in flexural strength and fracture toughness can be attained by employing nanoparticles. However, this should be done under conditions where uniform distribution of nanoparticles is ensured, which in turn was achieved in the current study through manual homogenization of fillers before mould packing.

The increased surface roughness seen in both the materials on addition of nanoparticles is a clinically important point that requires judicious consideration. Increased surface roughness is related to particle agglomeration of nanoparticles at high concentrations, formation of surface discontinuities during curing and remaining surface defects, despite following a standardized surface treatment regimen.[24]

This pattern is in agreement with **Sokhal et al. (2025)**[4], who reported that bis-acryl materials show greater surface roughness than PMMA-based materials after polishing and water immersion. This underscores the material-specific capacity for surface refinement. **Melo-Soares et al. (2025)**[25] investigated the incorporation of TiO_2 nanoparticle into PMMA. They reported that particle agglomeration at higher concentrations adversely influenced surface smoothness, an observation that aligns with the trend identified in the present study. In an evaluation of ZnO nanoparticle-reinforced PMMA, **Fouda et al. (2019)**[26] found that lower concentrations showed that at low concentration, there were some improvements in the strength along with minimal surface modifications. However, high concentrations resulted in significant surface modification.

However, a key consequence of higher surface roughness clinically is the possibility of increased plaque biofilm formation and adherence. In this regard, **Quirynen and Bollen**[9] found that Roughness Average (Ra) values greater than 0.2 μm indicate significantly higher microbial retention, emphasizing the biological importance of surface characteristics in provisional restorations. Nevertheless, the antibacterial effects inherent in ZnO nanoparticles, due to their ability to produce reactive oxygen species and release zinc ions, can partly compensate for the biological drawbacks of rougher surfaces. As demonstrated by **Kassaee et al. (2008)**[27] and corroborated by **Fardiaz et al. (2024)**[28], who found that inclusion of ZnO nanoparticles enhanced the mechanical properties of the PMMA/PEG blend and could provide antimicrobial benefits. Experimental confirmation of this hypothesis in vivo is a crucial topic for further research.

These findings indicate the potential of nanoparticle modification to enhance provisional restorative materials, especially in high-stress conditions that require prolonged provisionalization, such as integration stages of implants, rehabilitation of complete arches, or reconstructive treatment of the occlusion. The inclusion of 5% ZnO nanoparticles is the optimal choice for improving flexural strength, resulting in increased fracture resistance during occlusal function.

A limitation of this laboratory-based study is that factors present within the oral cavity, such as thermal stress, mastication-induced fatigue, enzymatic degradation by salivary enzymes, pH changes, or long-term aging effects cannot be replicated. The current study investigated only two concentrations of nanoparticles without considering their wear resistance, shade retention, fracture resistance, marginal integrity, or toxicity. Future research should aim to overcome these limitations.

CONCLUSION

Within the limitations of this in vitro investigation, it was found that incorporation of ZnO and Al_2O_3 nanoparticles at 3% and 5% concentrations significantly enhanced the flexural strength of both DPI Tooth Moulding Material and Protemp 4, with all intergroup differences reaching statistical significance ($p < 0.001$). ZnO nanoparticles demonstrated superior reinforcing efficacy compared to Al_2O_3 at equivalent concentrations, and 5% concentration produced greater mechanical improvement than 3% for both nanoparticle types. DPI consistently exhibited higher flexural strength than Protemp 4 across all experimental conditions, consistent with the known mechanical characteristics of PMMA-based versus bis-acryl systems. Surface roughness increased proportionally with nanoparticle concentration; however, the antimicrobial properties of ZnO may partially mitigate the potential biological consequences of increased Ra. The findings support nanoparticle reinforcement particularly with 5% ZnO as a clinically viable strategy for enhancing provisional restorative materials in complex prosthodontic rehabilitation.

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Conflicts of Interest: The authors declare no conflicts of interest.

Data Availability: The datasets generated during this study are available from the corresponding author upon reasonable request.

Ethical Approval: This study was purely in vitro and did not involve human participants or animal subjects. No ethical committee approval was required.

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