

EFFECT OF METAL OXIDE NANOPARTICLE REINFORCEMENT ON SURFACE ROUGHNESS AND MECHANICAL PROPERTIES OF HEAT CURE ACRYLIC RESINS

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ABSTRACT

1.Introduction: Poly(methyl methacrylate) (PMMA) remains the most widely used denture base resin due to its costeffectiveness and tissue-mimicking properties. PMMA is prone to fractures resulting from fatigue or accidental impact. Enhancing the properties of surface roughness, flexural strength, and impact strength are critical in improving clinical performance and longevity of the resin. Surface roughness influences plaque retention and infection risk, while flexural and impact strength determine resistance to masticatory and accidental stresses. Incorporating nanoparticles like silver (AgNP), copper oxide (CuO), and zinc oxide (ZnO) can improve these properties, though challenges remain regarding dispersion and concentration effects.

2.Objective: The aim of this in vitro study was to evaluate and compare the surface roughness, flexural strength, and impact strength of two high-impact heat-cure PMMA resins—Ivoclar and Lucitone—when reinforced with 2.5% and 5% concentrations of CuO, ZnO, and AgNP nanoparticles, processed using compression moulding technique.

3.Materials and Methods: Two heat-cured Hi impact PMMA resins—Ivoclar (PMMA Type I) and Lucitone (PMMA Type II)—were used to prepare one control (no nanoparticles) and six experimental subgroups incorporating 2.5% and 5% concentrations of CuO, ZnO, and AgNP. A total of 840 specimens (420 per resin) were fabricated and equally assigned to surface roughness, flexural strength, and impact strength tests (140 specimens each). Each test group comprised seven subgroups (n = 20). Surface roughness, flexural strength, and impact strength were measured using a surface roughness tester, universal testing machine, and Charpy impact tester, respectively. Data were analyzed in SPSS v26 using ANOVA, Tukey's post hoc, unpaired t-tests, and the Shapiro–Wilk test, with significance set at $p < 0.05$.

4.Results: The highest surface roughness was recorded in Ivoclar with 5% AgNP, while the lowest was seen in Lucitone with 2.5% AgNP. Flexural strength was highest for Ivoclar with 5% AgNP and for Lucitone with 5% ZnO. Both Ivoclar and Lucitone achieved their highest impact strength values when reinforced with 5% AgNP. Statistical analysis confirmed significant Indian differences among several subgroups ($p < 0.05$), particularly with higher nanoparticle concentrations affecting mechanical outcomes.

5.Conclusion: Reinforcement of HI-PMMA resins with nanoparticles notably influenced their mechanical properties. 5% silver nanoparticles significantly enhanced flexural and impact strength, while copper oxide and zinc oxide nanoparticles contributed to improved surface smoothness. These results suggest that tailored nanoparticle integration can optimize denture base resins for enhanced durability, strength, and resistance to microbial colonization.

KEYWORDS: Poly methyl methacrylate (PMMA); Nanoparticles; Silver nanoparticles (AgNP); Zinc oxide nanoparticles (ZnO); Copper oxide nanoparticles (CuO); Denture base resin; Surface roughness; Flexural strength; Impact strength; Ivoclar; Lucitone.

INTRODUCTION

Polymethyl methacrylate (PMMA) has been considered as the material of choice for denture base fabrication due to its aesthetic qualities, ease of manipulation, and cost-effectiveness. Despite these advantages, conventional heat-cured acrylic resins exhibit certain limitations such as low impact and flexural strength, as well as susceptibility to surface wear and microbial colonization, which can compromise the longevity and hygiene of prosthetic appliances [1-3]. In response to these challenges, reinforcement of PMMA with various fillers, including metal and metal oxide nanoparticles, has emerged as a promising strategy to enhance its mechanical and surface properties.

Nanoparticles such as copper oxide (CuO), zinc oxide (ZnO), and silver (Ag) have garnered significant interest due to their unique physicochemical characteristics. Their high surface area-to-volume ratio and antimicrobial potential make them suitable candidates for reinforcing denture base resins. Studies have shown that the incorporation of such

nanoparticles at controlled concentrations can improve the material's flexural strength, impact resistance, and reduce surface roughness, thus enhancing its clinical performance [4-8].

Among various commercially available high-impact heat-cured resins, Ivoclar and Lucitone are commonly used due to their improved toughness and fracture resistance. However, limited data exists comparing their performance when reinforced with metal oxide nanoparticles at different concentrations [9-11].

Therefore, this in vitro study aims to evaluate and compare the surface roughness, flexural strength, and impact strength of two high-impact heat-cured denture base resins, Ivoclar and Lucitone, when reinforced with 2.5% and 5% concentrations of copper oxide, zinc oxide, and silver nanoparticles. The findings of this study may offer valuable insights into the potential of nanoparticle reinforcement in enhancing the physical and mechanical properties of denture base resins

MATERIALS AND METHODS

This in-vitro experimental study was conducted at the Department of Prosthodontics, KIMS Dental College and Hospital to evaluate the effects of incorporating copper oxide (CuO), zinc oxide (ZnO), and silver nanoparticles (AgNP) at 2.5% and 5% concentrations into two brands of heat-polymerized polymethyl methacrylate (HI-PMMA) denture base resins: Type I - Ivoclar SR Triplex® Hot Vivadent Marketing (India) Pvt Ltd and Type II - Lucitone 199® Dentsply Marketing YORK DIVISION Pvt Ltd. The impact of nanoparticle addition was assessed based on surface roughness, flexural strength, and impact strength.

Standardized stainless steel metal dies were fabricated using CNC milling for specimen preparation: 64×10×3.3 mm (flexural strength), 55×10×10 mm (impact strength), and 12×12×3 mm (surface roughness), adhering to ISO 20795-1:2013, ASTM D-256, and BSI 771 (1984) standards, respectively. The dies were made from EN8-grade stainless steel. Nanoparticles (30–70 nm) were sourced from Techinstro (CuO, ZnO) and Nano Research Lab (AgNP). PMMA was prepared using a 3:1 polymer-to monomer ratio. For nanoparticle incorporation, 0.829 g (2.5%) and 1.659 g (5%) were added to 21 g polymer + 10 mL monomer mixtures and thoroughly mixed. Wax patterns were fabricated using silicone molds of the dies, prepared with Silliform Type 2 duplicating silicone. Wax was melted at 50°C and poured to form patterns, which were then invested and processed using standard curing cycles (74°C for 90 min + 100°C for 30 min). All samples were finished and polished uniformly, and dimensional accuracy was confirmed using a digital vernier caliper before storage in distilled water at 37°C for 48 ± 2 hours.

The study included two resin types, each tested with and without nanoparticles, used a statistical power (G*Power value) of 0.80. A total of 840 samples were prepared—420 per resin type—divided equally among surface roughness, flexural strength, and impact strength tests (140 each). Each test group had 7 subgroups (n=20): control (no additive), 2.5% and 5% CuO, ZnO, and AgNP.

Surface roughness was measured using an SRT 620 tester with a diamond stylus (2 µm tip, 0.7 N force). Flexural strength was determined using a 3-point bending test on a universal testing machine, with strength (M) calculated as:

$$M = 3WI/2bd^2$$

(W = fracture load, I = span, b = width, d = thickness)

Impact strength was tested using a Charpy Impact Tester (Model AMT-3) per ASTM D-256, where a 0.5 J pendulum struck the specimen midpoint. Energy absorbed was recorded in KJ/m². All data were statistically analyzed to compare nanoparticle-reinforced and control groups.

Statistical Analysis

Data analysis was done using the Statistical Package for Social Sciences (SPSS version 26). Basic descriptions were presented in the form of mean and Standard deviation. The data were assessed for normality using the Shapiro-Wilk test (P>0.05). Analysis of variance (ANOVA) test was used to analyse the overall difference for surface roughness, Impact strength, flexural strength and Tukey post hoc were used to assess the pair wise comparisons. Unpaired 't' test was used to analyse the difference between the groups. The level of significance was set at p < 0.05 for all tests.

RESULTS

The incorporation of CuO, ZnO, and AgNP nanoparticles into high-impact PMMA denture base resins (Ivoclar and Lucitone) demonstrated statistically significant variations in surface roughness, flexural strength, and impact strength across the test groups.

Surface Roughness

Among the Ivoclar subgroups (Group A), values ranged from 2.28 µm (A2 – 2.5% CuONP) to 6.99 µm (A7 – 5% AgNP), with the highest roughness in the 5% AgNP group. For Lucitone subgroups (Group D), values ranged from 4.60 µm (D6 – 2.5% AgNP) to 6.85 µm (D7 – 5% AgNP), again highest in the 5% AgNP group. Overall, Group A7 (Ivoclar/5% AgNP) exhibited the highest surface roughness among all 14 subgroups at baseline. One-way ANOVA revealed statistically significant differences within Ivoclar subgroups (F = 12.253, p < 0.001), whereas differences among Lucitone subgroups were not statistically significant (F = 2.050, p = 0.063).

Graph 1 presents the pairwise comparison of surface roughness between corresponding Ivoclar and Lucitone subgroups. Statistically significant differences were observed between the control groups (A1 vs D1, p < 0.001) and between the CuONP-2.5% groups (A2 vs D2, p < 0.001), indicating higher surface roughness in Ivoclar for these subgroups. All other comparisons, including those with 5% concentrations of CuO, ZnO, and AgNP, showed no statistically significant differences (p > 0.05). These results suggest that material type and nanoparticle concentration influence surface roughness variably, with significant differences noted primarily in the control and CuONP-2.5% groups.

Flexural Strength

Among Ivoclar subgroups, Ivoclar/AgNP–5% (B7) showed the highest flexural strength (63.53 MPa), while in Lucitone, Lucitone/ZnONP–5% (E5) recorded the highest value (65.49 MPa). Overall, Lucitone/ZnONP–5% demonstrated the

greatest flexural strength among all groups. However, statistical analysis revealed no significant differences in flexural strength within Ivoclar ($F = 0.595$, $p = 0.734$) or Lucitone ($F = 1.643$, $p = 0.140$) groups.

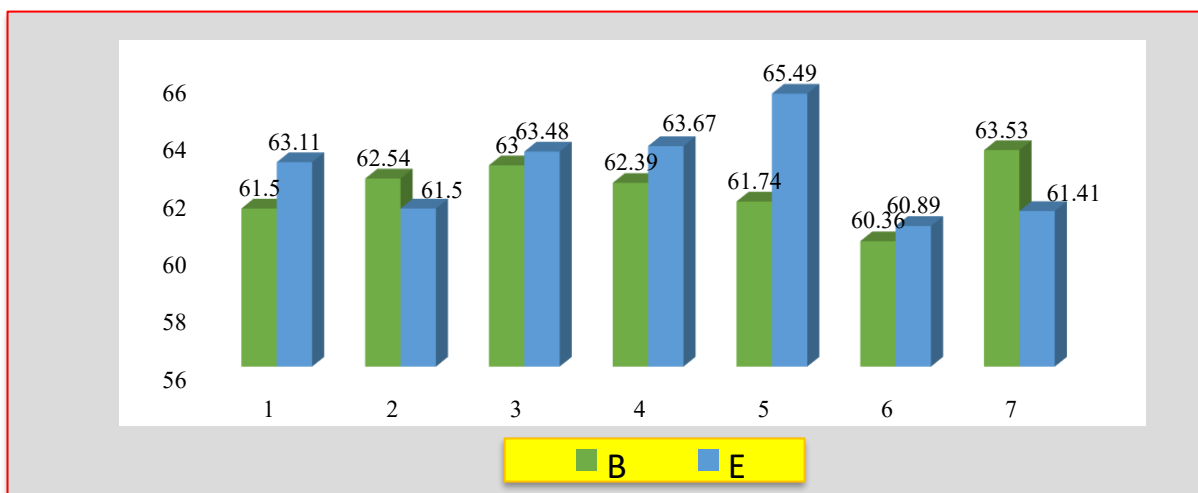
Graph 2 compares the flexural strength between corresponding Ivoclar and Lucitone subgroups. Most pairs showed no statistically significant differences ($p > 0.05$), indicating comparable performance between materials across various nanoparticle reinforcements. However, a significant difference was observed between Ivoclar/ZnONP-5% (B5) and Lucitone/ZnONP-5% (E5) with a mean difference of 3.749 ($p = 0.042$), suggesting Lucitone/ZnONP-5% exhibits superior flexural strength in this comparison.

Impact Strength

Among Ivoclar subgroups (C1–C7), Ivoclar/AgNP-5% (C7) showed the highest impact strength (4.68) with statistically significant difference ($p < 0.001$; $F = 10.450$). Similarly, among Lucitone subgroups (F1–F7), Lucitone/AgNP-5% (F7) had the highest value (4.49). Overall, Ivoclar/AgNP-5% (C7) exhibited the highest impact strength across all 14 groups with significant difference ($p < 0.001$; $F = 7.916$).

Graph 3 shows the pairwise comparison of flexural strength between corresponding Ivoclar and Lucitone subgroups. All comparisons, including conventional and nanoparticle-reinforced groups (CuONP, ZnONP, AgNP at 2.5% and 5%), revealed no statistically significant differences ($p > 0.05$). This indicates that both Ivoclar and Lucitone exhibit comparable flexural strength across all tested nanoparticle concentrations.

Graph 1: A and D [Pairwise comparison of mean difference of surface roughness between Ivoclar (A) and lucitone (D) groups].



Graph 2: B and E [Pairwise comparison of mean difference of flexural strength between Ivoclar (B) and lucitone (E) groups].



DISCUSSION

The present study investigated the effect of incorporating copper oxide (CuO), zinc oxide (ZnO), and silver nanoparticles (AgNPs) at 2.5% and 5% concentrations on the surface roughness, flexural strength, and impact strength of two high-impact heat-cured denture base resins, Ivoclar and Lucitone. The findings demonstrate that nanoparticle reinforcement significantly influences both surface and mechanical properties in a material- and concentration-dependent manner, which is consistent with the reports (Rama Krishna et al., 2020; Ansarifard et al., 2023; Hasan et al., 2023)[4,6,12].

To interpret these results it is important to consider the composition of the denture base resin and the physicochemical characteristics of the nanoparticles. Both Ivoclar and Lucitone are polymethyl methacrylate (PMMA)-based, high-impact heat-cured resin systems; however, they differ in copolymer formulation, molecular weight distribution, crosslinking density, and the type and amount of impact-modifying additives (for example, rubber or rubber-like graft copolymers, or core-shell reinforcements) incorporated into the matrix. These compositional differences influence the polymer's free volume, chain mobility, and capacity to wet and bond with dispersed inorganic phases. Likewise, the nanoparticles used (CuO, ZnO, Ag) differ in crystal structure, surface energy, typical particle size distribution, tendency to aggregate, and surface chemistry (hydrophilicity, presence of oxide layers), all of which govern their interaction with the PMMA matrix and any coupling agents used.

Overall, Ivoclar exhibited greater sensitivity to nanoparticle incorporation than Lucitone, particularly with respect to surface roughness and impact strength. The highest surface roughness was observed in Ivoclar reinforced with 5% AgNPs, while the lowest roughness occurred with 2.5% CuO nanoparticles. Increased surface roughness at higher silver nanoparticle concentrations has been attributed to nanoparticle agglomeration and uneven dispersion within the PMMA matrix (Mohammed et al., 2022; Rama Krishna et al., 2020)[12,13]. From a compositional standpoint, AgNPs tend to nucleate and form clusters if their surface is not sterically or chemically stabilized; in a PMMA matrix with lower compatibility (poorer wetting or weaker interfacial interactions), these clusters protrude to the surface during polymerization and polishing, increasing topographic irregularity. By contrast, CuO nanoparticles can exhibit stronger interfacial adhesion when smaller and better dispersed, and when their surface chemistry (for example, hydroxylated oxide surfaces) facilitates bonding or hydrogen-bonding interactions with residual monomer or with surface-modified PMMA, thereby producing smoother finished surfaces at lower concentrations.

Despite this increase in roughness, silver nanoparticles provide significant antimicrobial benefits, as Zhe Li et al. (2016) demonstrated reduced *Candida albicans* adhesion and biofilm formation on AgNP-modified PMMA surfaces[14]. This antimicrobial effect arises from ionic silver release and surface contact killing; however, it is balanced against compositional trade-offs where higher Ag loading increases the probability of agglomeration and local stiffness heterogeneities.

In contrast, Lucitone demonstrated a more stable surface response to nanoparticle reinforcement, with reduced surface roughness observed following incorporation of CuO and ZnO nanoparticles at both concentrations. This suggests improved compatibility between these nanoparticles and the Lucitone resin matrix. The lucitone formulation's specific copolymer content or higher degree of matrix crosslinking may promote better wetting or mechanical interlocking with oxide nanoparticles and reduce nanoparticle migration to the surface during polymerization and heat curing. Similar findings were reported by Hasan et al and Ansarifard et al. (2023), who observed no significant increase in surface roughness with CuO nanoparticle incorporation at lower concentrations[6,7].

With respect to flexural strength, the effects of nanoparticle reinforcement varied between the two resin systems. Ivoclar exhibited its highest flexural strength with 5% AgNPs, whereas Lucitone demonstrated superior flexural performance when reinforced with 5% ZnO nanoparticles. Improvements in flexural strength depend on effective stress transfer across the particle-matrix interface, which requires good dispersion, strong interfacial adhesion (chemical bonding or mechanical interlocking), and appropriate particle size (nanoscale dimensions large enough to interact with polymer chains but small enough to avoid stress-concentrating defects). Hasan et al. (2023) reported comparable improvements in flexural strength with ZnO nanoparticle incorporation, attributing the enhancement to improved stress transfer between the resin matrix and nanoparticles[6]. ZnO often has a higher surface area to volume ratio and, when well dispersed, can impede crack propagation and increase the energy required for bending failure. Kamonkhantikul et al. (2017) similarly emphasized the role of nanoparticle-matrix interaction in improving flexural properties.[15]

However, lower concentrations of AgNPs in Ivoclar were associated with reduced flexural strength, possibly due to inadequate dispersion or weak interfacial bonding. At sub-optimal loadings, nanoparticles can act as defects rather than reinforcements—insufficient particle content or poor interfacial coupling fails to produce a reinforcing network and instead creates stress-concentration sites where microvoids and debonding initiate under load. Koroglu et al. (2016) also reported that silver nanoparticles may adversely affect transverse strength and elastic modulus at certain concentrations[16]. The balance between matrix toughness and particle stiffness is therefore composition dependent: a matrix with lower crosslink density may accommodate particle inclusion better by allowing polymer chain rearrangement around particles, whereas a stiffer matrix may promote interfacial debonding if bonding is weak. Overall, Lucitone reinforced with ZnO nanoparticles demonstrated the highest flexural strength among all groups, indicating its suitability for applications requiring increased resistance to bending stresses.

Impact strength results revealed that nanoparticle incorporation generally improved resistance to sudden forces in both Ivoclar and Lucitone. The highest impact strength was observed in Ivoclar reinforced with 5% AgNPs, while the lowest values were recorded in the control group of Lucitone. Enhanced impact resistance can be explained by crack-arresting mechanisms where well-dispersed nanoparticles deflect or blunt crack tips and dissipate impact energy through interfacial debonding and particle pull-out. Suganya et al. (2021) and Hasan et al. (2023) reported similar enhancements with silver and zinc oxide nanoparticles. The improved impact resistance may be attributed to energy dissipation within the

nanoparticle-modified resin matrix when the interphase is engineered to allow controlled debonding rather than brittle fracture [6,17].

Nevertheless, higher concentrations of CuO and ZnO nanoparticles did not consistently improve impact strength, which may be related to particle agglomeration and stress concentration within the resin matrix (Venkatachalapathy et al., 2023)[18]. Agglomerates behave as micron-sized inclusions that reduce toughness and act as initiation sites for catastrophic crack growth. Additionally, increasing AgNP concentration beyond 2.5% in Lucitone did not result in further enhancement of impact strength, indicating a saturation threshold, as also suggested by Koroglu et al. (2016). This saturation reflects the compositional limit where additional rigid inclusions no longer improve energy absorption but instead increase brittleness or reduce cohesive matrix volume. Moreover, newer fabrication techniques like CAD/CAM have shown even greater improvements in mechanical performance, as noted by Kirad et al. (2021); these techniques change composition at the macroscopic level (densely polymerized, fewer residual monomers, more homogeneous microstructure) and reduce variability arising from hand processing[19].

Collectively, the findings of the present study indicate that silver nanoparticles are most effective in enhancing impact strength, zinc oxide nanoparticles significantly improve flexural strength— particularly in Lucitone—and copper oxide nanoparticles contribute to improved surface smoothness without compromising mechanical performance. These performance trends reflect complex compositional interactions: particle size and surface chemistry, matrix copolymer composition and crosslink density, presence of impact modifiers, and the quality of the particle–matrix interface. However, excessive nanoparticle loading may negatively influence surface characteristics and mechanical stability by promoting agglomeration, interfacial debonding, and heterogeneous mechanical domains. Therefore, careful selection of nanoparticle type, surface treatment (for example, silane coupling agents or polymer grafting to improve compatibility), and concentration is essential to achieve an optimal balance between surface quality, mechanical strength, and long-term clinical performance of high-impact denture base resins. Further long-term in vivo studies are needed to evaluate oral environment especially regarding wear, cleanser exposure, and color stability.

Limitations of the Study

This in vitro study may not fully reflect the oral environment, as it only tested short aging periods, ignored the effects of denture cleaners, and did not assess properties like color stability or wear resistance, so future research should include more PMMA types, varied nanoparticle concentrations, longer aging periods, and evaluation of additional properties such as color, wear, and durability.

CONCLUSION

This study showed that adding nanoparticles to denture base resins affects their surface roughness, flexural strength, and impact strength in different ways. Ivoclar resin had the highest surface roughness with 5% silver and the lowest with 2.5% copper oxide, while Lucitone was smoothest with 2.5% silver and roughest with 5% silver. Flexural strength was best in Lucitone with 5% zinc oxide and weakest in Ivoclar with 2.5% silver. Both copper and zinc oxide generally improved flexural strength in both resins. Impact strength was highest in Ivoclar with 5% silver and lowest in the Lucitone control group, with all three nanoparticles enhancing impact resistance at both concentrations. Overall, silver nanoparticles (5%) significantly boosted impact strength, while zinc oxide (5%) improved flexural performance, suggesting that nanoparticle reinforcement can enhance the durability of denture base resins.

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