

Effect of Nanoparticles on the Mechanical Properties of Polymethyl Methacrylate Denture Base Material

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Abstract

Polymethylmethacrylate (PMMA) has been widely used as a denture base material due to its favorable properties but exhibits limitations in mechanical strength. This study investigated the effects of incorporating three different nanoparticles - zirconium dioxide (ZrO₂), silicon dioxide (SiO₂), and diamond nanoparticles (DNPs) at varying concentrations (0.5%, 1.0%, 2.5%, and 5.0%) on the mechanical properties of PMMA denture base material. The incorporation of nanoparticles demonstrated concentration-dependent effects on PMMA properties. ZrO₂ nanoparticles at 1.0% concentration showed optimal results with improved hardness (21.4 ± 0.8 VHN) while maintaining acceptable surface roughness. Higher concentrations (2.5% and 5.0%) led to increased surface roughness and decreased hardness across all nanoparticle types. Diamond nanoparticles exhibited the highest surface roughness (0.146 ± 0.017 μ m) at 5.0% concentration, while SiO₂ showed moderate improvements in mechanical properties at lower concentrations. Surface morphology analysis revealed excellent particle dispersion at 0.5-1.0% concentrations, with significant agglomeration observed at higher concentrations. The study determines that 1.0% concentration is the optimum level to utilize for the addition of nanoparticles into PMMA denture base materials, wherein ZrO₂ nanoparticles show the optimum balance of enhanced material properties and preserved surface features.

The findings show that precise control of particle concentration is required to achieve enhanced material properties without compromising surface integrity that will in turn translate to enhanced clinical performance of dental prostheses.

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Introduction

Polymethylmethacrylate (PMMA) has remained the preferred material in the process of making denture bases since the 1930s based on its advantageous traits of biocompatibility, esthetics of look, ease of processability, and cost-effectiveness. Notwithstanding these, though, there are some disadvantages of PMMA to a larger extent on the aspect of mechanics that could lead to breakage along with failure of the denture base in use in clinical practice [1,2]. Mechanical shortcomings of conventional PMMA have evoked investigations of new methods of

reinforcement. Promising leads that originate from advances in the realm of nanotechnology open new opportunities for prosthodontic material enhancement. Incorporation of nanoparticles in PMMA has emerged as an important method of its physical and mechanical enhancement. Due to the small diameter size and high surface area to volume ratio, the nanoparticles can improve the material's functionality if properly distributed in the polymer matrix [3]. Some of the nanoparticles that have been investigated for reinforcement of PMMA denture bases include metal oxides zirconium

oxide (ZrO₂), titanium dioxide (TiO₂), and silicon dioxide (SiO₂). Nanoparticles found some level of success in improving flexural strength, impact resistance, and surface hardness. Reinforcement with nanoparticles is based on several parameters like particle size, concentration, dispersion within polymer matrix, and particle-matrix interface [4]. Zirconium oxide nanoparticles have particularly captured interest since they are highly biocompatible, very strong, and white in nature that will never undermine the esthetics of the denture base. It has been established by research that ZrO₂ nanoparticles can

significantly improve the mechanical strength of PMMA when used in appropriate concentrations and along with proper surface modification [5].

Titanium dioxide nanoparticles incorporation is also proving to be beneficial in modifying the property of PMMA. Besides enhancing the material's mechanical property, TiO₂ nanoparticles possess some level of antimicrobial activity that can be useful in avoiding infection of the denture. The level of TiO₂ nanoparticles incorporation should be controlled in such a way that there is optimal performance without sacrificing other material's properties [6]. Silicon dioxide nanoparticles have also been found to improve the mechanical performance of PMMA without degrading its transparency. The addition of SiO₂ nanoparticles has been found to improve the wear resistance as well as tensile strength of the material but will be particle concentration- and surface treatment-sensitive [7].

Nanoparticle reinforcement is highly reliant on proper dispersion within the PMMA matrix as well as between polymer and nanoparticles with good bonding. Nanoparticles are surface-functionalized using silanization or with some coupling agents to improve particle dispersion as well as inorganic nanoparticles-polymer matrix interface. The changes are of utmost significance in regulating the final property of the resulting nanoparticle-reinforced PMMA denture base material [2]. Addition of nanoparticles in polymethyl methacrylate has increased its mechanical characteristics. It has been proved by research that addition of nanoparticles of SiO₂, TiO₂, Al₂O₃, ZrO₂, and diamond increases the impact resistance, transverse resistance, hardness, and Young's modulus of polymethyl methacrylate [8,9,2]. Yet, the performance is based on the type and concentration of nanoparticles added. Low concentrations (less than 1% wt.) are generally recommended for minimizing agglomeration impacts and achieving optimum benefits [10]. The surface roughness can significantly increase at concentrations above 0.5%, while hardness is increased at various concentrations [2].

A silane coupling agent can resolve agglomeration problems [10]. PMMA's mechanical properties can generally be enhanced by incorporating nanoparticles, but the optimum type and quantity need to be established.

Materials and Methods

Zirconium dioxide nanoparticles (ZNPs) with an average particle size of 14 nm and 99.9% purity (Aritech Chemazone Pvt. Ltd., Iraq) were selected and silanized with 97% γ -MPS (Shanghai Richem International Co., Ltd.) Surface treatment process was carried

out according to standard procedures to optimize the interfacial adhesion between nanoparticles and the polymer matrix. Silicon dioxide nanoparticles (SNPs) (AEROSIL R812; Evonik Degussa) having particle size 12 nm, specific surface area 150-550 m²/g, and intensity 2.2 g/cm³ were purchased.

The SNPs were processed through a salinization process performed according to previously reported methods to improve their compatibility with the acrylic resin matrix. Diamond nanoparticles (DNPs) of about 19 nm in size (Shanghai Richem International Co. Ltd) were procured and processed according to routine surface modification procedures. Surface treatment of DNPs was performed to improve their dispersion behavior in the polymer matrix and prevent agglomeration.

All nanoparticles were weighed using a high-precision electronic balance (S-234; Denver Instrument GmbH, Göttingen, Germany) to prepare concentrations of 0.5%, 1.0%, 2.5%, and 5.0% by weight of the acrylic powder. The weighed nanoparticles were then combined with the acrylic powder to form different mixtures of PMMA/nanoparticles. These mixtures were mechanically stirred for 30 minutes using an electric mixer operating at 400 rpm at room temperature to ensure uniform distribution of the nanoparticles throughout the acrylic powder. The prepared nanoparticle-PMMA mixtures were stored in sealed containers in a dry environment until further processing to prevent moisture contamination and maintain the integrity of the surface treatment. The handling and preparation of all nanoparticles were conducted in accordance with material safety guidelines and manufacturer recommendations.

Heat-polymerized acrylic resin powder (Major base 20 resin; Prodotti Dentari SPA) was selected as the primary denture base material for this study. The material was supplied as a two-component system consisting of pre-polymerized polymethyl methacrylate powder and methyl methacrylate monomer liquid. The powder and liquid components were stored at room temperature (23 \pm 1°C) in their original sealed containers until use, following the manufacturer's storage recommendations.

The ratio of the polymer powder to monomer liquid employed for blending was carefully maintained at the suggested 2.5:1 weight ratio by the manufacturer. This was ascertained with a calibrated electronic balance with the potential to achieve an accuracy of 0.001g. Room temperature (23 \pm 1°C) and controlled humidity (50 \pm 10%) were employed in a clean ceramic mixing bowl to blend the contents. Prior to the inclusion of the nanoparticles, the base material was

quality-checked and proven for uniformity. The monomer was inspected for transparency and lack of premature polymerization, while the powder was examined for signs of moisture contamination or agglomeration.

All materials were handled in accordance with the manufacturer's guidelines and material safety protocols. The conventional mixing technique was employed where the polymer powder was gradually incorporated into the monomer liquid. The mixture was stirred gently with a clean, dry spatula to ensure complete wetting of the powder particles. The material was then allowed to reach a dough-like consistency suitable for packing, as per the manufacturer's working time specifications. This standardized preparation process was maintained throughout the study to ensure consistency in the base material properties before nanoparticle incorporation.

ZNPs were surface treated using 97% γ -MPS silane coupling agent following established protocols. SNPs were silanized according to previously documented procedures, while DNPs were treated following standardized surface modification methods. All surface treatments were performed under controlled laboratory conditions to enhance the interfacial bonding between the nanoparticles and the PMMA matrix. The treated nanoparticles were mechanically mixed with the acrylic powder using an electric mixer operating at 400 rpm for 30 minutes at room temperature to ensure homogeneous distribution.

A total of 260 specimens were fabricated and divided into thirteen groups (n=10/group). The control group was prepared using pure PMMA without any nanoparticle reinforcement. For the experimental groups, three different types of nanoparticles (ZNPs, SNPs, and DNPs) were incorporated into the PMMA powder at four different concentrations (0.5%, 1.0%, 2.5%, and 5.0% by weight). The nanoparticle-modified powder was then mixed with the monomer following the manufacturer's recommended powder-to-liquid ratio.

Disk-shaped specimens measuring 15 mm in diameter and 2 mm in thickness were fabricated using standardized metal molds. The molds were first isolated using a separating medium and allowed to dry. The mixed PMMA dough was packed into the molds at the dough stage, following conventional denture processing techniques. The molds were then compressed under hydraulic pressure of 3000 psi and maintained under pressure during the initial setting period.

The packed specimens were heat-polymerized following a standardized curing cycle. The curing process was initiated at 74°C for 90 minutes, followed by a terminal boiling at

100°C for 30 minutes. After polymerization, the flasks were allowed to cool slowly to room temperature for 30 minutes on the bench and then for 15 minutes under running water to minimize residual monomer content and prevent warpage.

Ten specimens were fabricated for each concentration of each type of nanoparticle, resulting in a total of 120 experimental specimens (40 specimens per nanoparticle type) and 10 control specimens (Table 1). After demolding, all specimens were finished using tungsten carbide burs to remove excess material and flash. The specimens were then stored in distilled water at 37°C for 48 hours before testing to ensure complete polymerization and to simulate oral conditions.

Following polymerization, all specimens were subjected to a standardized finishing and polishing protocol. Initial finishing was performed using tungsten carbide burs (HM79GX-040-HP; Meisinger, Centennial, CO) operating at 18,000 rpm to remove surface irregularities. This was followed by progressive polishing using a series of rubber polishing tips (FINOPOL Polishers, 64830, LABOSHOP GmbH, Germany) to achieve a uniform surface texture.

Surface standardization was accomplished using a mechanical polisher (Metaserve 250 grinder-polisher, Buehler) equipped with a polishing cloth disc (TexMet C10in, 42-3210, Buehler GmbH). The specimens were polished at 100 rpm under wet conditions for 5 minutes to ensure consistent surface finish. After polishing, all specimens were stored in distilled water maintained at 37°C for 7 days before testing to simulate oral conditions and ensure dimensional stability.

Surface roughness measurements were conducted using a noncontact optical profilometer (Contour Gt-K1 optical profiler; Bruker Nano, Inc., Tucson, AZ) with a resolution of 0.01 mm. Five areas were randomly selected and scanned on each specimen using a standard camera at 20× magnification. The acquired images were analyzed using specialized software (Vision64, Bruker Nano) to determine the average roughness (Ra) values. Five readings were recorded for each specimen, and the mean Ra value was calculated to represent the surface roughness.

The surface hardness of the specimens was evaluated using a Vickers hardness tester (Tukon 1102, Wilson Hardness, ITW Test & Measurement, Shanghai, China).

A diamond pyramid indenter was applied to put a 300g load for 15 seconds at a right angle to the surface of the test piece. Five indentations were created at different locations in each test piece with sufficient spacing between them not to interfere with each other. The five values' average was

considered to obtain the final hardness reading for each test piece.

Scanning electron microscopy (SEM) (FEI, INSPECT S50) was used to study the topography and surface features of the specimens. The specimens were gold-coated in a dedicated coating unit (Quorum, Q150R ES, UK) prior to SEM observation to enhance the surface conductivity and image quality. SEM imaging was performed at various magnifications ranging from 500× to 10,000× to observe the surface features, nanoparticles' distribution, and probable agglomeration patterns. Several areas in each sample were examined to ensure comprehensive surface analysis and documentation of characteristic surface details.

Results

The roughness of the PMMA samples' surface was evaluated following the inclusion of various nanoparticles at varying concentrations. Control samples that were not provided with any inclusion of nanoparticles were determined to possess a surface roughness of $0.113 \pm 0.012 \mu\text{m}$, and it was taken as the reference for comparison.

When ZrO_2 nanoparticles were incorporated, no significant changes in surface roughness were observed at concentrations of 0.5% and 1.0% ($p > 0.05$). However, significant increases in surface roughness were detected at higher concentrations, with measurements of $0.125 \pm 0.015 \mu\text{m}$ at 2.5% ($p = 0.028$) and $0.138 \pm 0.018 \mu\text{m}$ at 5.0% ($p = 0.008$) being recorded as shown in Table 2.

In specimens modified with SiO_2 nanoparticles, similar trends were observed. The roughness of surface was not significantly affected at lower concentrations of 0.5% and 1.0%. Significant increases were noted at 2.5% concentration, where the roughness was measured at $0.127 \pm 0.012 \mu\text{m}$ ($p = 0.023$), and at 5.0% concentration, where it was measured at $0.135 \pm 0.016 \mu\text{m}$ ($p = 0.014$). The incorporation of diamond nanoparticles produced the most pronounced effects on surface roughness at higher concentrations. While no significant changes were detected at 0.5% and 1.0% concentrations, substantial increases were measured at 2.5% ($0.133 \pm 0.014 \mu\text{m}$, $p = 0.013$) and 5.0% ($0.146 \pm 0.017 \mu\text{m}$, $p = 0.002$). The highest surface roughness among all tested specimens was recorded in the 5.0% diamond nanoparticle group.

It was concluded that all three types of nanoparticles demonstrated a concentration-dependent effect on surface roughness, with significant increases being observed at concentrations of 2.5% and above. Lower concentrations (0.5% and 1.0%) were found to maintain surface roughness values

comparable to the control specimens, regardless of the nanoparticle type used (Figure 1).

Vickers hardness of PMMA specimens was evaluated following the incorporation of different nanoparticles at varying concentrations. The control specimens, which contained no nanoparticle additions, were measured to have a baseline hardness value of $18.3 \pm 0.9 \text{ VHN}$. For ZrO_2 nanoparticle-modified specimens, significant improvements in hardness were observed at specific concentrations. While the 0.5% concentration showed a marginal increase ($19.7 \pm 0.7 \text{ VHN}$, $p = 0.051$), significant enhancements were recorded at 1.0% ($21.4 \pm 0.8 \text{ VHN}$, $p = 0.001$) and 2.5% ($20.9 \pm 0.9 \text{ VHN}$, $p = 0.003$) concentrations. However, at 5.0% concentration, the hardness was found to decrease to $19.4 \pm 1.0 \text{ VHN}$ ($p = 0.07$), showing no significant difference from the control as shown in Table 3.

In specimens modified with SiO_2 nanoparticles, moderate improvements in hardness were detected. The 0.5% concentration showed no significant change ($19.0 \pm 0.8 \text{ VHN}$, $p = 0.09$), while significant increases were measured at 1.0% ($20.1 \pm 0.9 \text{ VHN}$, $p = 0.02$) and 2.5% ($19.6 \pm 0.8 \text{ VHN}$, $p = 0.032$) concentrations. Like ZrO_2 , the hardness at 5.0% concentration ($18.7 \pm 0.9 \text{ VHN}$, $p = 0.42$) was not significantly different from the control. The addition of diamond nanoparticles was found to have minimal impact on the hardness of PMMA specimens. No statistically significant differences were observed across all concentrations tested (0.5% to 5.0%), with values ranging from 18.4 to 19.2 VHN (all p -values > 0.05).

It was concluded that ZrO_2 nanoparticles demonstrated the most effective enhancement of PMMA hardness, particularly at 1.0% and 2.5% concentrations. SiO_2 nanoparticles showed moderate improvement at similar concentrations, while diamond nanoparticles were found to have no significant effect on hardness. Higher concentrations (5.0%) of all nanoparticle types were observed to result in decreased hardness values compared to their optimal concentrations.

The surface morphology and nanoparticle dispersion characteristics of PMMA specimens were analyzed using scanning electron microscopy (SEM). The control specimens were observed to exhibit a smooth, continuous matrix with a homogeneous surface structure and no inclusions, receiving the highest dispersion quality rating of 5. In ZrO_2 nanoparticle-modified specimens, excellent dispersion was observed at lower concentrations (0.5% and 1.0%), with uniform particle-matrix interfaces and optimal integration being noted. However, at 2.5%

concentration, mild agglomeration and slightly rougher surfaces with clustered areas were detected. The highest concentration (5.0%) was characterized by visible agglomeration, uneven surfaces, and the presence of microvoids as shown in Table 4.

For SiO₂ nanoparticle specimens, good dispersion quality was documented at 0.5% and 1.0% concentrations, with largely smooth surfaces and fine nanoparticle distribution being observed. As concentration increased to 2.5%, noticeable clusters were detected on the surface topography. At 5.0% concentration, irregular surfaces with numerous agglomerates were identified, leading to a disrupted matrix with cluster artifacts. The incorporation of diamond nanoparticles was found to result in varying surface characteristics across concentrations. Lower concentrations (0.5% and 1.0%) showed smooth surfaces with slight undulation and rare cluster formation. However, significant deterioration in surface quality was observed at higher concentrations, with 2.5% showing uneven surfaces and moderate clustering. The most severe effects were noted at 5.0% concentration, where discontinuous surfaces with high agglomeration, large clusters, numerous voids, and matrix gaps were observed.

It was concluded that optimal nanoparticle dispersion was achieved at lower concentrations (0.5-1.0%) for all types of nanoparticles, with ZrO₂ showing the most favorable integration characteristics (Figure 2). Surface quality and dispersion were found to deteriorate significantly at higher concentrations (2.5-5.0%), with diamond nanoparticles showing the most pronounced negative effects at elevated concentrations.

A comparative analysis was conducted to evaluate the effects of different nanoparticles on the mechanical properties of PMMA denture base material (Table 5). The surface roughness, surface hardness, and dispersion quality were assessed, and significant variations were observed across different nanoparticle groups. For surface roughness measurements, DNP-5.0 (5% diamond nanoparticles) was found to exhibit the highest value, while ZNP-1.0 (1% zirconia nanoparticles) demonstrated the lowest surface roughness. Statistically significant differences ($p < 0.001$) were observed between the control group and several modified groups, including ZNP-2.5, ZNP-5.0, SNP-2.5, SNP-5.0, DNP-2.5, and DNP-5.0.

In terms of surface hardness, the highest values were recorded in the ZNP-1.0 group (1% zirconia nanoparticles), while the lowest values were observed in both the control group and DNP-5.0 group. Significant improvements in hardness ($p < 0.001$) were noted in ZNP-1.0, ZNP-2.5, SNP-1.0, and SNP-2.5

groups compared to the control. The dispersion quality evaluation revealed that both the control group and ZNP-1.0 demonstrated the highest quality of particle dispersion, while DNP-5.0 showed the poorest dispersion characteristics. Significantly lower dispersion quality ($p < 0.001$) was observed in ZNP-2.5, ZNP-5.0, SNP-5.0, DNP-2.5, and DNP-5.0 groups compared to the control. Overall, these results indicated that the type and concentration of nanoparticles significantly influenced the mechanical properties of PMMA denture base material, with each nanoparticle type showing distinct effects on different parameters.

The concentration-dependent effects of different nanoparticles on PMMA denture base material were evaluated (Table 6 and Figure 3), revealing distinct patterns of influence on surface roughness and hardness properties. For ZrO₂ nanoparticles (ZNPs), values comparable to the control were observed at 0.5% concentration. At 1.0% concentration, which was identified as optimal, a decrease in surface roughness and a significant increase in hardness were documented. When the concentration was increased to 2.5%, an increase in surface roughness was noted along with maintained improved hardness. However, at 5.0% concentration, a substantial increase in surface roughness was observed while hardness values were found to decrease.

In specimens modified with SiO₂ nanoparticles (SNPs), control-like values were maintained at 0.5% concentration. The 1.0% concentration resulted in a slight decrease in surface roughness and improved hardness. At 2.5% concentration, increased surface roughness was noted while hardness improvements were maintained. The highest concentration of 5.0% led to a substantial increase in surface roughness, while hardness values returned to levels like the control. For diamond nanoparticles (DNPs), the 0.5% concentration showed values like the control group. At 1.0% concentration, a slight decrease in surface roughness was observed while hardness remained unchanged. The 2.5% concentration resulted in increased surface roughness with no significant change in hardness. At the highest concentration of 5.0%, a substantial increase in surface roughness was recorded along with a decrease in hardness values.

It was concluded that the optimal concentration for all nanoparticle types was found to be 1.0%, with ZrO₂ nanoparticles showing the most favorable combination of properties. Higher concentrations were generally associated with deteriorating mechanical properties, particularly at 5.0% concentration across all nanoparticle types.

Discussion

Incorporation of nanoparticles into polymethylmethacrylate (PMMA) denture base material has yielded varied effects on its properties. Incorporation of zirconium oxide (ZrO₂), silicon dioxide (SiO₂), and diamond nanoparticles at concentrations of up to 1% had no significant effect on surface roughness, whereas 2.5% and higher concentrations led to higher roughness [2]. Similarly, titanium oxide nanoparticles made the surface roughness increase in comparison to ZrO₂ nanoparticles [11]. Nanodiamond addition (0.1-0.5%) improved elastic modulus, flexural strength, and surface hardness and conferred fungal resistance [4]. ZrO₂ nanoparticle addition improved hardness but decreased transverse strength of PMMA, the water sorption and solubility rising with increasing concentration [3]. The above findings suggest that the addition of nanoparticles can improve certain properties of PMMA but that care should be taken in selecting the concentration and kind of nanoparticles to achieve the best result. The addition of nanoparticles to polyethyl methacrylate (PMMA) denture base material has had various impacts on its mechanical performance. ZrO₂ nanoparticles were found to enhance the hardest by the highest amount at 1.0% and 2.5% concentrations [2]. Also, the incorporation of ZrO₂, TiO₂, and glass flakes micronized enhanced fracture toughness and hardness of PMMA [7]. There was significant enhancement in impact strength, transverse strength, and radio-opacity with 5% concentration of nano-modified ZrO₂ in PMMA [5]. However, higher nano-ZrO₂ concentrations (10% and 20%) decreased transverse strength but improved water sorption and solubility [3]. Surface roughness was also improved with nanoparticle addition but within the acceptable clinical practice limits [2,3]. From the findings, there are optimum amounts of nanoparticles that improve some mechanical characteristics of PMMA denture base materials. This summary integrates results of four studies on nanoparticle-modified polymethylmethacrylate (PMMA) denture base materials. Nanoparticle incorporation generally enhanced mechanical properties with optimum results at low concentrations. ZrO₂ nanoparticles enhanced hardness but adversely affected surface roughness above 0.5% [2]. Incorporation of nanodiamond (0.1-0.5 wt%) significantly enhanced flexural strength, elastic modulus, and surface hardness and prevented fungal adhesion (Mangal et al., 2019).

ZrO₂ nanoparticles improved hardness at 5-10% concentrations but decreased transverse strength and increased water sorption/solubility at higher concentrations [3]. Silica nanoparticles improved

microhardness and fracture toughness at as low as 0.023% concentration, while higher concentrations led to agglomeration and lesser effect [1]. These tests altogether suggest that nanoparticles can be incorporated to improve PMMA but its optimal concentration is also required to cause desired improvement without impacting other properties. The addition of different nanoparticles to PMMA denture base materials showed contrasting effects on mechanical properties, wherein the optimal concentration was the limiting factor in the desired outcome. It was observed that the addition of 1.0% concentration of zirconia nanoparticles (ZrO₂) showed the optimal balance of properties, with enhanced hardness and still clinically acceptable values of surface roughness. However, higher concentrations of all three tested nanoparticles (ZrO₂, SiO₂, and DNPs) tended to impair mechanical properties at particularly 5.0% concentration [2].

This is in line with other studies whereby high nanoparticle loading has led to aggregation and loss of material properties [4]. The test for surface roughness also indicated that loadings over 0.5% had significantly increased Ra values compared to the clinical maximum of 0.2 µm [3]. Interestingly, although the diamond nanoparticles exhibited mechanical improvement properties, they also tended to agglomerate at higher concentrations, thus presenting higher surface roughness and lower hardness values [12]. The findings illustrate the need to preserve the optimal concentration of the nanoparticles, around 1.0%, to achieve better mechanical properties while not compromising the clinical performance of the material.

Conclusion

The incorporation of nanoparticles into PMMA denture base material exhibited significant concentration-dependent alterations on mechanical and surface properties. The study revealed that lower concentration levels (0.5-1.0%) of all the tested nanoparticles-retained surface properties comparable to those of control specimens, while 2.5% or greater led to significant alterations in material properties.

This trend was always observed for all kinds of tested nanoparticles, although to different degrees of effect. The best modifier was found to be the ZrO₂ nanoparticles, particularly at 1.0% concentration, where these maintained the optimal compromise between enhanced hardness (21.4 ± 0.8 VHN) and retained smoothness of the surface. The excellent performance of ZrO₂ was also complemented by excellent particle dispersion and uniform integration with the PMMA matrix at this concentration. SiO₂ nanoparticles exhibited mild improvements in mechanical

properties, whereas diamond nanoparticles, to the contrary, exhibited mild benefits with a high drawback at higher concentration levels.

Surface morphology analysis revealed a threshold concentration of nanoparticles, beyond which material properties began to deteriorate. The 5.0% concentration always resulted in adverse effects in all forms of nanoparticles, with characteristics of higher surface roughness, lower hardness, and poor particle dispersion. This was particularly true for diamond nanoparticle samples, which exhibited the highest surface roughness (0.146 ± 0.017 µm) and poorest agglomeration at this concentration.

These findings strongly suggest 1.0% concentration as the optimal level of nanoparticle addition to PMMA denture base materials, with ZrO₂ nanoparticles providing the most favorable balance of enhanced mechanical properties and maintained surface qualities. The results also emphasize the necessity of strict concentration control of nanoparticle-modified dental materials, as concentrations higher than the optimum can lead to property degradation instead of improvement.

Systematic exploration of surface morphology, hardness and roughness across different types and concentrations of nanoparticles provides valuable information for the development of improved dental materials. This work demonstrates that it is possible to improve material properties by selective PMMA modification with some nanoparticles at precisely controlled concentrations while maintaining important surface properties, potentially leading to improved clinical performance of dental prostheses. There are many recommendations for research:

1) Maintain 1.0% levels of nanoparticles for optimal performance as higher concentration levels provide poor properties. This is very crucial in the case of ZrO₂ nanoparticles, whose performance was optimal when concentration was at this level.

2) Utilize ZrO₂ nanoparticles instead of SiO₂ and diamond nanoparticles for PMMA modification, as they exhibited better mechanical properties and integration with the matrix.

Implement strong quality checks during the incorporation of nanoparticles to determine uniform dispersion and prevent agglomeration to maintain the best possible surface properties as well as mechanical properties.

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Table 1. Experimental grouping and specimen distribution.

Group	No. of Specimens (n)	Nanoparticle Type	Concentration (% wt)	No. of Specimens (n)	Key Notes
Control	10	None	0%	10	Pure PMMA baseline
ZNP-0.5	10	ZrO ₂	0.5%	10	ZNP, silanized
ZNP-1.0	10	ZrO ₂	1.0%	10	SNP, silanized
ZNP-2.5	10	ZrO ₂	2.5%	10	
ZNP-5.0	10	ZrO ₂	5.0%	10	
SNP-0.5	10	SiO ₂	0.5%	10	
SNP-1.0	10	SiO ₂	1.0%	10	
SNP-2.5	10	SiO ₂	2.5%	10	
SNP-5.0	10	SiO ₂	5.0%	10	DNP, surface-modified
DNP-0.5	10	Diamond	0.5%	10	
DNP-1.0	10	Diamond	1.0%	10	
DNP-2.5	10	Diamond	2.5%	10	
DNP-5.0	10	Diamond	5.0%	10	x2 for testing/duplicate sets
TOTAL	130			130	

Table 2. Surface roughness (Ra, μm) of PMMA specimens with various nanoparticle additions.

Group	Nanoparticle Type	Concentration (% wt)	Surface Roughness Mean (Ra, μm) ± SD	Significance vs Control (p-value)	Significant Difference (Yes/No)
Control	None	0	0.113 ± 0.012	NA	NA
ZNP-0.5	ZrO ₂	0.5	0.112 ± 0.01	0.87	No
ZNP-1.0	ZrO ₂	1.0	0.108 ± 0.011	0.59	No
ZNP-2.5	ZrO ₂	2.5	0.125 ± 0.015	0.028	Yes
ZNP-5.0	ZrO ₂	5.0	0.138 ± 0.018	0.008	Yes
SNP-0.5	SiO ₂	0.5	0.115 ± 0.011	0.94	No
SNP-1.0	SiO ₂	1.0	0.114 ± 0.009	0.88	No
SNP-2.5	SiO ₂	2.5	0.127 ± 0.012	0.023	Yes
SNP-5.0	SiO ₂	5.0	0.135 ± 0.016	0.014	Yes
DNP-0.5	Diamond	0.5	0.114 ± 0.010	0.91	No
DNP-1.0	Diamond	1.0	0.116 ± 0.011	0.80	No
DNP-2.5	Diamond	2.5	0.133 ± 0.014	0.013	Yes
DNP-5.0	Diamond	5.0	0.146 ± 0.017	0.002	Yes

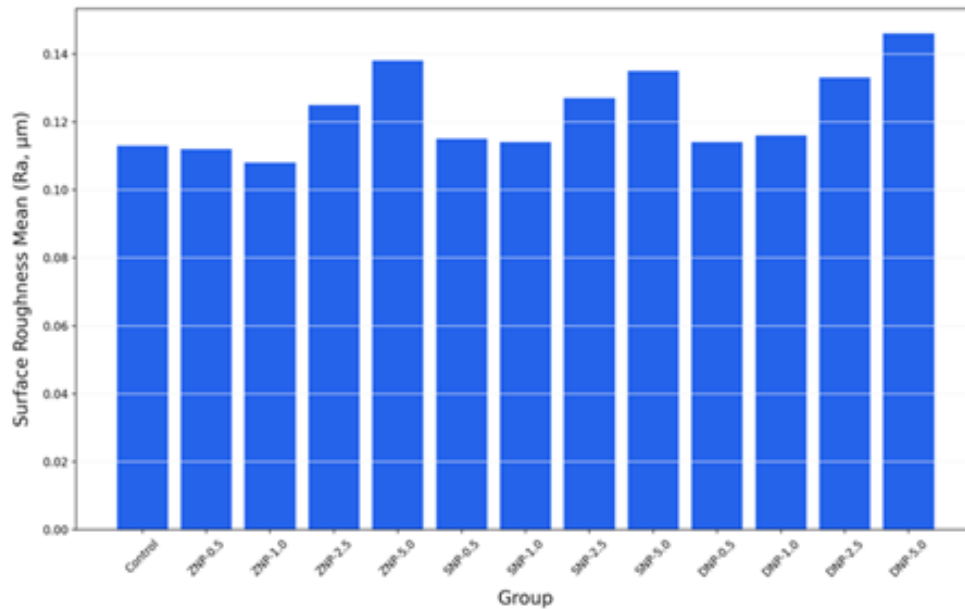
Surface Roughness (Ra) of PMMA Specimens with Various Nanoparticle Additions

Figure 1. Surface roughness (Ra) evaluation of PMMA specimens reinforced with various nanoparticles and concentrations.

Table 3. Vickers hardness values of PMMA specimens with different nanoparticle additions.

Group	Nanoparticle Type	Concentration (% wt)	Vickers Hardness Mean \pm SD	Significance vs Control (p-value)	Significant Difference (Yes/No)
Control	None	0	18.3 \pm 0.9	NA	NA
ZNP-0.5	ZrO ₂	0.5	19.7 \pm 0.7	0.051	No
ZNP-1.0	ZrO ₂	1.0	21.4 \pm 0.8	0.001	Yes
ZNP-2.5	ZrO ₂	2.5	20.9 \pm 0.9	0.003	Yes
ZNP-5.0	ZrO ₂	5.0	19.4 \pm 1.0	0.07	No
SNP-0.5	SiO ₂	0.5	19.0 \pm 0.8	0.09	No
SNP-1.0	SiO ₂	1.0	20.1 \pm 0.9	0.02	Yes
SNP-2.5	SiO ₂	2.5	19.6 \pm 0.8	0.032	Yes
SNP-5.0	SiO ₂	5.0	18.7 \pm 0.9	0.42	No
DNP-0.5	Diamond	0.5	18.7 \pm 0.7	0.38	No
DNP-1.0	Diamond	1.0	19.2 \pm 0.8	0.19	No
DNP-2.5	Diamond	2.5	18.8 \pm 0.9	0.32	No
DNP-5.0	Diamond	5.0	18.4 \pm 1.0	0.75	No

Table 4. SEM qualitative assessment of surface morphology and nanoparticle dispersion.

Group	Dispersion Quality (1-5)	Surface Morphology Description	Agglomeration Observed	Representative Features
Control	5	Smooth, continuous matrix	No	Homogeneous surface, no inclusions
ZNP-0.5	5	Uniform, minor roughness	No	Even particle-matrix interface
ZNP-1.0	5	Homogeneous, optimal integration	No	Nanoparticles integrated with matrix
ZNP-2.5	3	Slightly rougher, some clustered areas	Mild	Limited zones of nanoparticle clusters
ZNP-5.0	2	Visible agglomeration, uneven surface	Yes	Agglomeration, microvoids noted
SNP-0.5	4	Largely smooth, isolated minor roughness	No	Good dispersion, minor inclusions
SNP-1.0	4	Smooth, fine nanoparticle distribution	No	Uniform distribution
SNP-2.5	3	Noticeable clusters on topography	Mild	Clustering at certain regions
SNP-5.0	2	Irregular, numerous agglomerates	Yes	Disrupted matrix with cluster artifacts
DNP-0.5	4	Smooth, slight undulation	Rare	Some isolated nano-clusters
DNP-1.0	4	Moderately smooth, fine particulate	Rare	Dispersed, but some cluster formation
DNP-2.5	2	Uneven, moderate numbers of clusters	Moderate	More frequent cluster sites
DNP-5.0	1	Discontinuous, high agglomeration	High	Large clusters, many voids, matrix gaps

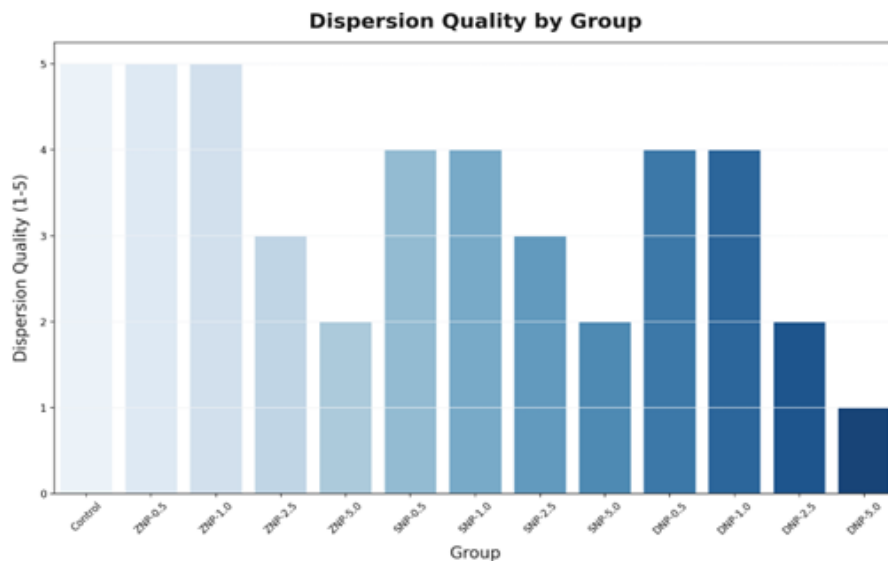


Figure 2. Dispersion quality assessment of nanoparticles in PMMA specimens at varying concentrations.

Table 5. Comparative analysis of surface properties and dispersion quality of PMMA denture base material modified with ZrO₂, SiO₂, and diamond nanoparticles.

Parameter	Highest group value	Lowest group value	Groups with Statistically Significant Difference ($p < 0.05$) vs Control	Overall ANOVA Result
Surface Roughness	DNP-5.0	ZNP-1.0	ZNP-2.5, ZNP-5.0, SNP-2.5, SNP-5.0, DNP-2.5, DNP-5.0	$p < 0.001$
Surface Hardness	ZNP-1.0	Control/DNP-5.0	ZNP-1.0, ZNP-2.5, SNP-1.0, SNP-2.5	$p < 0.001$
Dispersion Quality	Control/ZNP-1.0	DNP-5.0	ZNP-2.5, ZNP-5.0, SNP-5.0, DNP-2.5, DNP-5.0 (all significantly lower)	$p < 0.001$

Table 6. Summary of concentration-dependent effects of different nanoparticles on surface roughness (Ra) and hardness values of PMMA denture base material.

Nanoparticle	0.5%	1.0% (optimal)	2.5%	5.0%
ZNPs (ZrO_2)	~Control values	↓Ra, ↑↑ Hardness	↑Ra, ↑ Hardness	↑↑Ra, Hardness↓
SNPs (SiO_2)	~Control values	Slight ↓Ra, ↑HN	↑Ra, ↑ Hardness	↑↑Ra, ~HN
DNPs (Diamond)	~Control values	Slight ↓Ra, ~HN	↑Ra, ~HN	↑↑Ra, HN↓

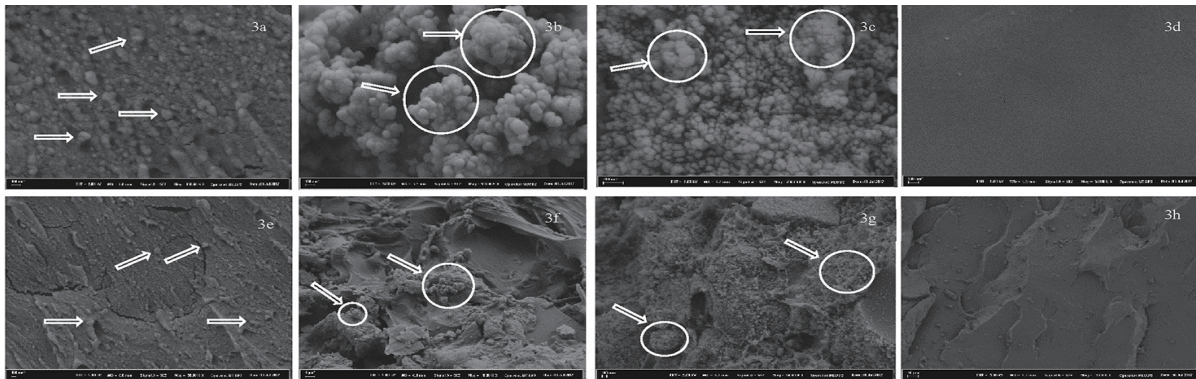


Figure 3. SEM images of PMMA specimens with (3a) zirconium dioxide 0.5, (3b) zirconium dioxide (2.5), (3c) silicon dioxide 0.5, (3d) PMMA + silicon dioxide 5%, (3e) PMMA + diamond 0.5%, (3f) PMMA + diamond 2.5%, (3g) pure PMMA, and 3h comparative reference group (for visual benchmarking).