Translucency of nanoparticle-reinforced PMMA denture base material: An in-vitro comparative study

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The aim of this study was to assess the translucency of denture base acrylic resin reinforced with zirconium dioxide (ZrO2NPs), silicon dioxide (SiO2NPs), and diamond (DNPs) nanoparticles. A total of 130 heat-polymerized acrylic discs (15×2.5 mm) were fabricated conventionally and divided into control and experimental groups according to nanoparticle type and concentration (0.5, 1, 1.5, and 2.5 wt%). Unmodified acrylic resin specimens served as control. All specimens were thermocycled (5,000 cycles). Translucency was measured using a spectrophotometer. ANOVA and post-hoc Tukey’s test were used for data analysis at α=0.05. The translucency of modified PMMA was significantly lower than control (p<0.05) except 0.5% ZrO2NPs and SiO2NPs (p>0.05) which exhibited the highest translucency values among modified groups. As the NPs concentration increased, the translucency decreased and the lowest value was seen with 2.5% DNPs (1.18±0.10). The addition of ZrO2NPs, SiO2NPs, and DNPs into denture base resin decreased the translucency.

Keywords: Esthetics, Nanoparticle, Optical properties, Polymethyl methacrylate, Removable dental prostheses

INTRODUCTION

Although polymethyl methacrylate (PMMA) is considered the common material for denture base fabrication, it has its drawbacks. PMMA’s poor physical and mechanical properties could affect the denture’s overall longevity. Reinforcement with fibers, macro- or nanofillers, chemical modification, or the development of an alternative material to PMMA have all been explored.

Significant attention has been paid towards incorporating nanoparticles into PMMA. Their addition was reported to positively enhance the final PMMA/nanocomposite properties in comparison to unmodified PMMA versions. The final characteristics of the nanocomposites are dependent on the properties of added nanoparticles, such as size, shape, concentration, and their interaction with the polymer matrix. Superior characteristics of nanoparticles were linked to their nano-scale and large specific surface relative to their volume which made them appropriate to be used as fillers for the polymers. These unique properties of the nanoparticles permit a strong interfacial interaction with the organic polymers, which result in a nanocomposite with new mechanical, chemical, and optical properties.

Getting a functioning prosthesis that is also highly esthetic is a usual request of prosthodontic patients. Matching the color and appearance of underlying soft tissue is an important requirement of acrylic denture base. Similar to pure PMMA, acceptable visual properties of the modified versions are also imperative. Since PMMA is very versatile and accepts the addition of fillers, that may affect the translucency, attention should be paid to the resulting denture esthetics. Translucent material has the ability to pass light through its structure, and allows the background to show through.

Commonly used nanoparticles include silver (AgNP), zirconium dioxide (ZrO2NPs), silicon dioxide (SiO2NPs), and diamond (DNPs) nanoparticles. ZrO2NPs became popular owing to their superior scientific, technological, and medical aspects. It possesses remarkable antimicrobial effects, and the potential to enhance modified nanocomposites’ mechanical properties of the modified material. Similar results were reported with SiO2NP addition. At low concentrations, SiO2NPs improved the mechanical properties and permitted the fabrication of thin, strong, and durable denture bases. DNPs have excellent interfacial bonds with PMMA due to the multiple reactive groups (NH2, OH) on the surface, therefore, they are considered a compatible filler material. Additionally, DNPs has...
confirmed antibacterial activity\textsuperscript{22}, as well as positive effects on mechanical properties\textsuperscript{23,24}. A recent study concluded that 0.1–0.5 wt% DNP\textsubscript{s} added to auto-polymerized PMMA acrylic resin significantly improved the mechanical and surface properties in addition to the antimicrobial effect\textsuperscript{26}.

Superior mechanical properties and acceptable esthetics are required for removable prostheses. Although the effects of adding nanoparticle on different aspects of PMMA resin material have been performed, their effect on optical properties including translucency was not thoroughly investigated. Consequently, the present in-vitro study was planned to evaluate the effects of ZrO\textsubscript{2}NPs, SiO\textsubscript{2}NPs, and DNP\textsubscript{s} addition on the translucency of PMMA and compare between those nanofillers. The null hypothesis stated that addition of ZrO\textsubscript{2}NPs, SiO\textsubscript{2}NPs, and DNP\textsubscript{s} to PMMA will not affect the translucency of the resulting nanocomposites.

### MATERIALS AND METHODS

Table 1 summarizes the materials and nanoparticles used in the current study. First, the shape, size and reinforcing nanomaterials’ structure; ZrO\textsubscript{2}NPs, SiO\textsubscript{2}NPs, and DNP\textsubscript{s} were microscopically evaluated before incorporation into the PMMA using transmission electron microscope (TEM; Morgagni 268, FEI, Brno, Czech Republic). TEM analysis showed the characteristics and specifications of the three types of nanoparticles (Fig. 1). TEM examination results confirmed that ZrO\textsubscript{2}NPs were spherical in shape, and averaged ~40 nm in size with crystalline structure (Figs. 1A and B). The selected area electron diffraction (SAED) pattern revealed the existence of nanoparticles in monoclinic and tetragonal states, which were similar to standard cards (JCPDS PDF no. 37-1484 and JCPDS PDF no. 49-1642)\textsuperscript{25,26}. The average size of SiO\textsubscript{2}NPs was ~15 nm, spherical in shape with some degree of agglomeration. The particles displayed non-crystalline structure as confirmed by

| Material                                           | Manufacturer                                |
|----------------------------------------------------|---------------------------------------------|
| Zirconium dioxide nanoparticles (ZrO\textsubscript{2}NPs, Av Size: 40 nm, 99.9% purity) | Shanghai Richem International, Shanghai, China |
| AEROSIL R812, silicon dioxide nanoparticles (SiO\textsubscript{2}NPs, Av Size: 15 nm, >99.8% purity) | Evonik, Essen, Germany                       |
| Diamond nanoparticles (DNP\textsubscript{s}, Size: 20–150 nm, 98–99% purity) | Shanghai Richem International               |
| Silane coupling agent (3-trimethoxysilyl propyl methacrylate, 97%) (γ-MPS) | Shanghai Richem International               |
| Major.Base.20, heat-polymerized acrylic resin      | Major Prodotti Dentari, Moncalieri, Italy    |

**Table 1** Materials used in the present study

![Fig. 1](image-url)  
**Fig. 1** Transmission electron microscopy (TEM) images of the three reinforcing nanofillers along with corresponding SAED patterns. (A, B) ZrO\textsubscript{2}NPs, (C, D) SiO\textsubscript{2}NPs and (E, F) DNP\textsubscript{s}. The size of the nanofillers is found between 15–150 nm.
blurred diffraction rings of the SAED pattern (Figs. 1C and D). The DNPs showed various morphologies and shapes, i.e., irregular and flake-like, with size range ~20–150 nm and few tens of nanometers in thickness. The SAED pattern of DNPs exhibited well separated dotted rings, the first seven distinct characteristic diffraction planes of the nanocrystalline diamond (starting from the inner ring) were confirmed as (111), (200), (220), (311), (222), (004) and (331). In summary, the size of the three nanoparticles (nanofillers) used in this study was in the range of 15–150 nm.

Next, the nanoparticles surface was treated by a silane coupling reagent, as described in previous studies for ZrO$_2$NPs, SiO$_2$NPs, and DNPs, to create functional groups and improve bonding to resin matrix. In order to inspect the nanoparticle distribution within the PMMA polymer before polymerization, samples of three mixtures; PMMA/ZrO$_2$NPs, PMMA/SiO$_2$NPs and PMMA/DNPs were examined at 20 kV under a scanning electron microscope (SEM; Inspect S50, FEI) (Fig. 2). For comparison, the powder of pure PMMA resin was also analyzed (Fig. 2A), and the average size of the PMMA spheres was found to be nearly 25 µm.

To get the balance between the achieved mechanical and esthetic properties, the concentrations used ranged between 0.5–2.5% based on a pilot study that resulted in extreme color change (whitish, blackish) when ZrO$_2$NP or DNP were more than 2.5%. Therefore, the tested concentrations were unified (0.5%–2.5%) for all nanoparticles for the sake of comparison. Digital scale (WENSAR Mab Dab Series Analytical Balance, DAB 220, IndiaMART InterMESH, Uttar Pradesh, India) was used to weigh the treated nanoparticles to produce mixtures with 0.5, 1, 1.5 and 2.5 wt% of acrylic powder. Nanoparticles were mixed separately with PMMA polymer for 30 min at 400 rpm to reach homogeneity. A total of 130 heat-polymerized acrylic resin discs (15×2.5 mm) were fabricated and separated into 4 main groups; control and three experimental groups. Experimental groups were categorized into 4 subgroups (n=10) based on nanoparticle concentration 0.5, 1, 1.5 and 2.5 wt%.

Investment of the wax discs with stone was done within a metal flask. After the stone has set, wax was eliminated and a separating medium was painted on the mold spaces (Isolmajor, Major Prodotti Dentari, Moncalieri, Italy) then permitted to cool to room temperature. After mixing the acrylic resin, following the manufacturer’s instructions, the mix was packed in its doughy consistency, then the acrylic was pressed for 30 min using pneumatic press. Following that, the acrylic was polymerized using thermal curing device (KaVo Elektrotechnisches Werk, Leutkirch, Germany). The water temperature was gradually raised from room temperature to 74°C and for 8 h, then to 100°C for ½ h.

After polymerizations, flasks were permitted to cool down before specimen retrieval. They were then finished with tungsten carbide burs to remove the flashes (HM79GX-040-HP, Meisinger, Centennial, CO, USA) at 18,000 rpm and polished by means of gradually increasing grits of silicon carbide papers, starting with 320 then 400, and eventually with 600, slurry pumice with soft bristle brush was used for the final polish (Steribim Super, Bego, Wilhelm-Herbst-Strabe, Germany). The specimens were kept in a 37°C distilled water container for 72 h. Samples were subjected to a total of 5,000 thermo-cycles using a thermoclycling unit (Thermocycler THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany) at an alternating temperature between 5°C and 55°C set at 30-s dwell time.

A spectrophotometer (Color-Eye® CE-7000A, X-rite, Carlstadt, NJ, USA) measured the 3 dimensions of color ($L^*$, $a^*$, and $b^*$) of the Commission Internationale de l’Eclairage (CIE) system. Standard calibration procedure as instructed by the manufacturer was done before testing. The specimens were individually held against the port, backed up by reference white or black background before initiating the reading. The data was charted and following equation was used to calculate the translucency value: $Tr = [(L^*_{\text{white}} - L^*_{\text{black}})^2 + (a^*_{\text{white}} - a^*_{\text{black}})^2 + (b^*_{\text{white}} - b^*_{\text{black}})^2]^{1/2}$. Higher value indicated more light reflectance; hence the measured material had a higher translucency level and vice versa.

SPSS-20.0 (IBM, Chicago, IL, USA) was used for statistical analysis. The data were tested for normality using Shapiro-wilk test where insignificant results indicated normal distribution; this is why parametric tests were used for analysis. Variation in translucency averages due to change in concentration was analyzed.
using one-way analysis of variance (ANOVA). The pairwise comparisons of different concentration levels were checked with Tukey’s post-hoc test. To analyze the joint effect of concentration and nanoparticle, two-way ANOVA was used. The significance level was set to 0.05.

Table 2 Two-way ANOVA results showing the effect of each variable and the combined effect of both (concentration and nanoparticle type)

| Source                  | Type III sum of squares | df  | Mean square | F          | p-value |
|-------------------------|-------------------------|-----|-------------|------------|---------|
| Concentration           | 588.492                 | 3   | 196.164     | 880.553    | <0.001* |
| Nanoparticle            | 123.556                 | 2   | 61.778      | 277.313    | <0.001* |
| Concentration* Nanoparticle | 65.288                | 6   | 10.881      | 48.845     | <0.001* |
| Error                   | 24.060                  | 108 | 0.223       | —          | —       |
| Total                   | 2,706.305               | 120 | —           | —          | —       |
| Corrected total         | 801.395                 | 119 | —           | —          | —       |

*indicates significant value at α=0.05.

Table 3 One-way ANOVA results for effect of nanofiller concentration on translucency

| Source  | Sum of squares | df | Mean square | F     | Sig.  |
|---------|----------------|----|-------------|-------|-------|
| ZrO2NPs | Between Groups | 1,075.797 | 4 | 268.949 | 773.890 | <0.001* |
|         | Within Groups  | 15.639   | 45 | 0.348  | —      | —     |
|         | Total          | 1,091.436 | 49 | —      | —      | —     |
| SiO2NPs | Between Groups | 1,285.165 | 4 | 321.291 | 1,334.261 | <0.001* |
|         | Within Groups  | 10.836   | 45 | 0.241  | —      | —     |
|         | Total          | 1,296.001 | 49 | —      | —      | —     |
| DNs     | Between Groups | 1,381.410 | 4 | 345.353 | 1,328.722 | <0.001* |
|         | Within Groups  | 11.696   | 45 | 0.260  | —      | —     |
|         | Total          | 1,393.106 | 49 | —      | —      | —     |

Table 4 Mean±SD of translucency, and significances for pairwise comparisons between different concentrations of tested groups

| Concentration (%) | ZrO2NPs Mean±SD | SiO2NPs Mean±SD | DNs Mean±SD |
|-------------------|-----------------|-----------------|-------------|
| 0 (Control)       | 15.28±0.72a     | 15.28±0.72a     | 15.28±0.72  |
| 0.5               | 7.93±0.57b,c,h  | 9.38±0.67p,s,e  | 4.90±0.61h,A |
| 1.0               | 4.78±0.88 p,s,A | 5.78±0.42c,f    | 2.60±0.46l,c,k |
| 1.5               | 3.72±0.26d      | 2.45±0.13s,R,E  | 1.50±0.42d,F,H |
| 2.5               | 2.24±0.21p,c,d  | 1.33±0.24p,F,G  | 1.18±0.10k,g,H |

ZrO2NPs: Zirconium dioxide nanoparticles; SiO2NPs: Silicon dioxide nanoparticles; DNs: Diamond nanoparticles. SD: Standard deviation.

Same small letters in the column per respective nanoparticle (element) denote no statistically significant differences between groups while same capital letters denote no statistically significant differences between all nanoparticle groups (p>0.05).
concentrations, one-way ANOVA showed no significant difference between 1% ZrO$_2$NPs vs 0.5% DNPs, $p=1.000$. Statistically, there were no significant differences between 2.5% ZrO$_2$NPs vs (1.5% SiO$_2$NPs, ($p=0.999$); 1% DNPs, ($p=0.956$); and 1.5% DNPs, ($p=0.093$)). Moreover, translucency values of 1.5% and 2.5% SiO$_2$NP and DNPs were lower than those of ZrO$_2$NPs counterparts. However, no significant differences were found between 1.5% SiO$_2$NPs vs 1% DNPs, ($p=1.000$); 2.5% SiO$_2$NPs vs 1.5% DNPs, ($p=0.999$); and 2.5% DNPs, ($p=1.000$)). The lowest translucency value of nanomodified specimens were recorded with 2.5% DNPs (1.18±0.10).

Variation in translucency caused by change in concentration was statistically significant ($p<0.001$) in all three nanofillers (Table 3). Mean and standard deviation of translucency at given levels of concentration for each nanofiller was presented in Table 4. Figure 3 shows the variation in translucency between specimens for each nanofiller was presented in Table 4. Figure 3 shows the variation in translucency between specimens with different nanoparticles at each concentration level. Pairwise comparisons of concentration levels within each nanofiller was done through post-hoc analysis. Translucency values for all nanofillers showed downward trends as the filler concentration increased with 0.5% and 2.5% showing the highest and lowest translucency values, respectively, for their respective nanofiller (Table 4). Changes in the means in all consecutive pairs of concentration levels within each nanofiller was found statistically insignificant ($p>0.05$) (Table 4). Capital letter alphabets denotes insignificant changes in the means for all subgroups of the three nanofillers using post-hoc analysis ($p>0.05$). It was found that statistical insignificance was detected when the difference between the means was less than 0.75.

**DISCUSSION**

Heat-polymerized PMMA denture base material has a highly esthetic translucent pink color$^{4,6}$. Variant nanoparticles were added to enhance the mechanical properties; however, this addition compromised the denture esthetics$^6$. Therefore, the study was implemented to investigate the impact of different nanoparticles addition at various concentrations and compare their effects on the translucency degree of the resulting modified nanocomposite denture base material. The results of the current research confirmed that all the tested nanoparticles (ZrO$_2$NPs, SiO$_2$NPs, and DNPs) adversely affected the translucency of the final nanocomposite denture base material. Therefore, the null hypothesis was rejected.

In the process of improving PMMA denture base material’s mechanical properties, nanoparticles shape, size, and distribution within the polymer matrix all play very important roles in the resulting properties$^{17}$. Additionally, refractive index of both materials—nanoparticles and PMMA—will affect the optical properties$^{4,6}$. It was shown that a good distribution and homogenous mixtures could be prepared with small-sized (nano-sized) particles$^{59}$. In the present study, the particles used ranged between 15–150 nm in size and were added to 25 µm acrylic powder (an average size of the PMMA spheres). The small size (a nano range) of the nanoparticles allowed them to fill the cracks and gaps between the polymer chains and produce homogenous PMMA/nanoparticles matrix after heat-polymerization. Therefore, the proportion of nanoparticles should be maintained at minimum to guarantee adequate distribution within the resin without agglomerations$^{17}$.

ZrO$_2$NPs decreased the translucency at all concentrations except 0.5%. The translucency was inversely related to ZrO$_2$NPs concentration, with 2.5% showing the lowest translucency value. The results of the current study were in accordance with Gad et al., who studied the effect of adding 2.5%–7.5% ZrO$_2$NPs to PMMA and reported a significant reduction in translucency with all concentrations$^4$. Lower translucency may be associated with the inherent optical properties of the ZrO$_2$NPs themselves and their distribution within the resin matrix. ZrO$_2$NPs being crystalline in nature causes higher opacity with a reduction in light transmission, thereby reducing the translucency. Additionally, the same effect is observed if clusters of ZrO$_2$NPs are formed$^4$. However, zirconia powder is white in color and therefore has minimal compromising effect on the esthetic appearance, unlike metallic equivalents, such as, copper, aluminum or silver$^{4,31}$.

Similar to the effect of ZrO$_2$NPs, SiO$_2$NPs reduced the translucency of the modified acrylic resin specimens except for 0.5%. The lowest translucency value was reported with 2.5% nanofiller. One advantage of SiO$_2$NPs over ZrO$_2$NPs is their translucency. However, the reduction in nanocomposite translucency may be due to the volume of added nanoparticles. ZrO$_2$NPs and DNPs were heavier in weight than SiO$_2$NPs resulting in
larger volume of SiO\textsubscript{2}NPs addition compared to ZrO\textsubscript{2}NPs and DNP\textsubscript{s} for the same concentration. Additionally, ZrO\textsubscript{2}NPs and DNP\textsubscript{s} exhibited higher crystallinity resulting in higher degree of opacity than SiO\textsubscript{2}NPs\textsuperscript{6}. On the other hand, SiO\textsubscript{2}NPs showed great biocompatibility, and excellent optical property\textsuperscript{6,19,30,32}.

DNPs showed different effects, where all concentrations significantly reduced the translucency. This effect was due to the nature of DNP\textsubscript{s} rather than concentration. Crystalline structure, gray color, and surface treatment other than silanization of the other nanoparticles could have contributed to these adverse effects on the translucency of PMMA/DNP\textsubscript{s}. Also, the morphology of the nanoparticles, (spheres as in ZrO\textsubscript{2}NPs or SiO\textsubscript{2}NPs; and sheet-like in the case of DNP\textsubscript{s}) may have prevented the passage of light or caused more light reflection than transmission making the nanocomposite more opaque\textsuperscript{33}.

The specimens were thermally stressed for 5,000 times to simulate 6 months of clinical use\textsuperscript{34}. With thermocycling, there is a chance for increased water sorption especially with warm temperature\textsuperscript{34,35}. Therefore, absorbed water could disrupt the passage of UV beam and produce lower readings indicating less translucency\textsuperscript{6}. Aggregation of NPs resulted in almost complete blockage of light transmission and drastic change in color or translucency\textsuperscript{6}. However, silanization enhanced the uniform dispersion of NPs within the PMMA matrix due to the interaction between -OH groups of the NPs and the hydrolysable groups of the silane coupling agent, thus increasing the distance between the NPs and allowing more light to pass\textsuperscript{33}.

Optical property differences of nanocomposites originated from the variation in types and amounts of fillers within\textsuperscript{6}. To add to that, the difference amongst the refractive indices of nanoparticles and PMMA matrix phases affect light refraction and reflection at the interface and the translucency thereafter\textsuperscript{36}. It was found that the refractive index of ZrO\textsubscript{2}NPs (2.1750\textsuperscript{7}) and DNP\textsubscript{s} (2.8610\textsuperscript{8}) were higher than that of the PMMA (1.4813\textsuperscript{37}). Nanocomposite becomes more opaque if the difference between the refractive indices of the two phases is great (Figs. 4A and B). Although the refractive index of SiO\textsubscript{2}NPs (1.475)\textsuperscript{8} was close to PMMA, the agglomeration seen with SiO\textsubscript{2}NPs affected the nanocomposite translucency. In the present study and compared to control, translucency was not affected at low concentration (0.5\%) for ZrO\textsubscript{2}NPs and SiO\textsubscript{2}NPs in contrast to higher concentrations; where agglomeration tendency of the nanoparticles was great. SiO\textsubscript{2}NPs exhibited lower density compared to ZrO\textsubscript{2}NPs and DNP\textsubscript{s}\textsuperscript{17}, resulting in excessive particle amount per unit area that exceeded those of the two other nanoparticles.

From a clinical perspective, selection of the optimal concentration of NPs to be incorporated into PMMA to enhance the mechanical properties without adverse effects on esthetics is crucial\textsuperscript{33}. The addition of different concentrations of NPs exhibits unpredictable change in translucency. This phenomenon is a shortcoming of nano-metal oxide reinforcement of PMMA. Therefore, the translucency of nanocomposite denture base materials should be considered during nanoparticles selection as a reinforcement material otherwise denture bases with adequate mechanical performance but lack the appropriate esthetics will lead to patient dissatisfaction.

The limitations of this study include the use of one type of acrylic resin, and the lack of oral environment simulation. Therefore, the authors recommend testing these nanocomposites in-vivo with clinical studies. Further researches to assess the biological effects are recommended. Thus, the results of this study should be looked at carefully and analyzed further for use in the future.

CONCLUSION

The incorporation of more than 0.5\% of ZrO\textsubscript{2}NPs or SiO\textsubscript{2}NPs into denture base material significantly decreased the translucency and could be detrimental to denture esthetics. While the inclusion of any DNP\textsubscript{s} concentration into the PMMA denture bases resulted in adverse effects on translucency. Nanoparticle type and concentration should be selected with caution to get a balance between the desirable properties and the adverse effects of these fillers.

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