Hindawi
International Journal of Dentistry
Volume 2023, Article ID 4245801, 8 pages
https://doi.org/10.1155/2023/4245801

Research Article

Polymethylmethacrylate-Based Nanocomposites for Denture Base Fabrication: Impact of Nanoparticle Type and Concentration on the Color Change In Vitro

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Received 12 January 2023; Revised 25 March 2023; Accepted 7 April 2023; Published 29 April 2023

Academic Editor: Carlos A. Munoz-Viveros

Background. Although the mechanical behaviors of PMMA were improved with nanoparticles addition, there is a lack of study on the color changes of nanocomposite denture base resin. This study aimed to assess and compare the color of nanocomposite denture base resin modified with different nanoparticles and concentrations. *Materials and Methods*. Three nanoparticles (zirconium dioxide (ZNP), titanium dioxide (TNP), and silicon dioxide (SNP)) were added to heat-polymerized acrylic resin in 3 and 7 wt% concentrations. A total of 70 acrylic discs ($20 \times 2 \pm 0.03$ mm) specimens were prepared while one without addition (control) and three main groups according to nanoparticles and two subgroups according to % (3ZNP, 7ZNP, 3TNP, 7TNP, 3SNP, and 7SNP) with total 70 acrylic discs ($n = 10$). Spectrophotometer was used for color change (ΔEab) followed by value conversion to National Bureau of Standards units (NBS) to relate the color alterations (ΔEab) to the clinical environment which aids in determining a threshold for clinical acceptance of the color change. ΔEab data were analyzed and compared using one- and two-way ANOVA tests followed by Bonferroni's post hoc test ($\alpha = 0.05$). *Results*. Two-way ANOVA showed that filler type (regardless of filler concentration) had a statistically significant effect on mean ΔEab ($P < 0.001$). Filler concentrations (regardless of filler type) showed a significant effect on mean ΔEab while the filler type and concentration interaction showed no significant effect on mean ΔEab ($P < 0.001$). One-way ANOVA in terms of filler types results showed a significant difference between mean ΔEab ($P < 0.001$), where TNP group showed the highest mean ΔEab followed by ZNP and SNP. Pair-wise comparison revealed that 3% concentration showed a significant lower mean ΔEab than 7% concentration ($P < 0.001$). *Conclusion*. Modification of heat-polymerized denture base resin with ZNP, TNP, or SNP causes clinically unacceptable color change. TNP produced the highest color change followed by ZNP and SNP, and the color change is concentration dependent; the color change increases as the concentration increases.

1. Introduction

Polymethylmethacrylate (PMMA) materials are most commonly used for the rehabilitation of partially and completely edentulous patients because of their advantages; for instance, aesthetics, low cost, ease of manipulation and use, and stability in oral environments [1]. Despite the popularity and advantages of PMMA, low mechanical performance was reported to lead to denture fractures which affected denture longevity [2, 3].

Numerous trials have been suggested to overcome the drawbacks of PMMA and enhance its physiomechanical properties by introducing alternative resins, modifying chemical structures, and/or incorporating reinforcement materials [3, 4]. Recently, the

incorporation of nanofillers is the most common approach to improve antifungal activities and biocompatibility [5] flexural strength, impact strength, wear resistance and hardness [6], and decrease the water sorption and solubility of modified resins [7]. Nanoparticles (NPs) gain their advantages from their nanosized, high specific area, and their ability of bonding and interfacial interaction with resin matrix resulting in good properties compared with the unmodified one [5]. It was reported that the nanocomposite resin was affected by NP type, size, and concentrations in addition to strong bonding of NP with the organic resin matrix [5–7].

Amongst commonly used NPs are, zirconium dioxide (ZNP), titanium dioxide (TNP), and silicon dioxide (SNP). Azmy et al. [6] found that these NPs might improve the strength and wear resistance of modified PMMA denture base resins.

ZNP is a white crystalline dental ceramic; its incorporation into PMMA has been advised in the literature to enhance its properties [3]. It has advantageous properties such as high mechanical properties, low surface roughness, and excellent biocompatibility [5]. Due to these advantages, previous studies suggested incorporation of ZNP into PMMA denture base resin and reported that this nanocomposite could be used as an alternative to the conventional resin [3, 6–9]. According to many literature, the effect of ZNP on the denture base resins is concentration dependent [6–9]. Albasarah et al. [10] reported an increase in flexural strength and elastic modulus of denture base resin when modified with 5% ZNP. Also, Zidan et al. [11] used ZNP concentration up to 10% and reported an improvement in the flexural strength, modulus, fracture toughness, and hardness with an optimum concentration of 3–5 wt% ZNP. Recent study suggested 3%–7% and demonstrated that both concentrations increased the strength of PMMA denture base resin [6].

TNP is one of the most commonly used NPs due to its merits such as biocompatibility, antimicrobial activity, high stability, and low cost with high strength [12, 13]. Several studies reported an amazing effect of TNP on the improvement of impact, transverse strength, and surface hardness as well as can decrease water sorption and solubility of TNP-modified heat polymerized acrylic resin [6, 14, 15]. Abdelraouf et al. [16] found that the addition of 5 wt% TNP to the acrylic resin improved its flexural strength and reduced its water-sorption without impairing its surface microhardness and roughness [16]. Many investigations suggested the addition of 5% TNP was considered suitable to raise the surface hardness of conventional and high-impact heat-cured acrylic resin to significant values [17, 18]. While another study reported that up to 7% suggested increasing the strength of PMMA denture base resin [6]. Previous studies found that the addition of TNP to polymers enhanced their antimicrobial activities due to the photocatalytic ability of TNP [18, 19]. Azmy et al. [19] reported that addition of 3%–7% TNP can improve the antimicrobial activity of soft liners as well as the flexural strength, impact strength, and wear resistance of PMMA denture base resins [6, 19]. Hashem et al. [18] found 1%–3% TNP improved the wetting properties, reduced surface tension, increased strength and hardness in addition to the enhancement in the thermomechanical properties in terms of glass transition temperature (Tg, 118.6°C). Nazirkar et al. [20] and Sodagar et al. [13] explored the effect of addition of 0.5%–1% TNP into heat cure acrylic resin on antimicrobial properties and reported an adverse effect on the flexural strength of nanocomposite.

SNP is one of the most popular NPs with a lot of applications and usage in dental materials. This is due to its inherent characteristics high strength, high wear resistance, biocompatibility, and thermal stability, which make it a suitable nanofiller for denture base resin reinforcement [21]. The incorporation of SNP into PMMA denture base resin improved the physical and mechanical properties of the modified resin enhancing its durability as reported in previous studies [13, 22, 23]. The addition of SNP to PMMA denture base was recommended in low concentrations [21, 23] and this confirmed the study done by Karci et al. [22] who investigated 1%–5% SNP and reported that low concentrations improved the flexural strength of nanocomposite. However, Azmy et al. [6] suggested increasing the concentration of SNP up to 7% and reported an increase in the strength and wear resistance of SNP-modified resin.

One of the most common problems seen regarding the reinforcement of heat-cured acrylic resin via nanoparticles is the color instability [24, 25]; the color change of the acrylic prosthesis may cause esthetic problems which affects patient satisfaction and acceptance of the prosthesis [26]. Accordingly, reinforcement of PMMA with different NPs to improve its mechanical properties without adversely affecting the esthetics is mandatory to achieve optimal esthetics and serviceability [26, 27]. Color stability of PMMA is correlated with other properties that could affect the color changes such as water sorption, hydrophilicity, and surface roughness in addition to intrinsic factors associated with polymerization techniques like the degree of monomer conversion and chemical degradation of resin materials [28, 29].

Generally, PMMA reinforced with inorganic fillers with proper mechanical properties and accepted esthetics are favored. Therefore, this study aimed to evaluate and compare the color stability of PMMA denture base resin modified with different concentrations of ZNP, TNP, and SNP. The null hypothesis of this study was that the addition of 3 and 7 wt% of ZNP, TNP, and SNP has no significant effect on the color change of heat-polymerized PMMA.

2. Materials and Methods

Sample size was calculated based on previous studies [30–32] and revealed that to conduct the current study, 70 specimens ($n = 10$) were required. The specimens were fabricated by one investigator for standardization and to minimize possible researcher-related variability. All materials used in the current study, their types, chemical compositions, and manufacturer's specifications were summarized in Table 1. Disc-shaped specimens were prepared according to ISO 1999 Specification no. 1567, with $20 \times 2 \pm 0.03$ mm; diameter and thickness, respectively [33]. Specimens were divided into four main groups according to NPs, one control without additions, and three NP-modified groups, ZNP, TNP, and SNP. Furthermore,

Table 1: The study's materials.

Trade name	Manufacturer	Specifications
Vertex	Vertex Dental, Netherlands	Powder: polymethyl methacrylate, 500 g Liquid: phthalyl butyl glycolate, ethanol, 250 ml
ZrO_2 nanoparticles	NanoGATE, Ciro, Egypt	Spherical, white, and tetragonal particles (12 ± 3 nm; purity >99%)
TiO_2 nanoparticles		Spherical, white, and anatase particles (15 ± 3 nm; purity >99%)
SiO_2 nanoparticles		Spherical, white, and amorphous particles (21 ± 3 nm; purity >99%)
Silane coupling agent (3-trimethoxysilyl-propyl-methacrylate (TMSPM))	Sigma–Aldrich Chemie GmbH Riedstrasse2, Germany	Purity 98%, ethanol 99.7%; lot no. 440159

Table 2: Grouping and coding of different subgroups according to NP type and concentrations.

NP	Code	Description
Control	C	Unreinforced heat cured acrylic resin
ZrO_2	3ZNP	Heat polymerized acrylic resin reinforced with 3 wt% of ZNP
	7ZNP	Heat polymerized acrylic resin reinforced with 7 wt% of ZNP
TiO_2	3TNP	Heat polymerized acrylic resin reinforced with 3 wt% of TNP
	7TNP	Heat polymerized acrylic resin reinforced with 7 wt% of TNP
SiO_2	3SNP	Heat polymerized acrylic resin reinforced with 3 wt% of SNP
	7SNP	Heat polymerized acrylic resin reinforced with 7 wt% of SNP

each modified group was divided into two subgroups according to NP concentrations, 3% and 7% as described in Table 2.

Silanization of NPs was performed using a silane coupling agent to form reactive groups on NPs surfaces for strong bonding between NPs and organic resin matrix. The silanization process was completed as described in previous studies [8, 34]. After silanization, NPs were added separately to heat-polymerized acrylic resin. An electronic balance (Denver Instrument, Göttingen, Germany) was used to weigh each NP to be added in two concentrations, 3 and 7 wt% of acrylic resin powder. Then NP was added to the resin powder and mixed using a magnetic stirrer for 30 min to ensure homogenous distribution of NP within the resin powder.

According to the manufacturer's recommendations, NP/powder mixtures were added to the monomer, mixed, and then packed in dough stage in the prepared mold spaces within a metal flask. The nanocomposite specimens were prepared simulating laboratory procedures used for denture fabrication with specific dimensions as detailed in previous studies [8, 33, 35]. After polymerization, resin specimens were retrieved from the flask and finished using a tungsten carbide bur. For surface standardization, all specimens were polished using silicon carbide paper with different grits (600-, 800-, 1,000- and 1,200-grit). Then, polishing of the specimens was completed using a cloth wheel and pumice (TexMet C, PSA, 10 in, Buehler GmbH) with a mechanical polisher (Metaserve 250 grinder polisher, Buehler) for 5 min at 100 rpm in wet conditions [36]. A digital caliper was used to confirm the accurate dimension of the polished specimens followed by visual examination for any voids or defects, and then the approved specimens were stored in distilled water at 37°C for 48 hr [37].

Table 3: NBS rating system for expressing color system.

NBS units	Remarks on color difference
0.0–0.5	Trace change
0.5–1.5	Slight change
1.5–3	Noticeable change
3–6	Marked change
6–12	Extremely marked change
12 or more	Change to other color

A spectrophotometer (SpectraMagic NX, RM2002QC, Konica Minolta Corp., Ramsey, Japan) was used for Color measurement. The Spectrophotometer was calibrated and then used according to the manufacturer's instructions. All specimens were tested against a standard white background plate. While the specimens were set on the port of the spectrophotometer, the measurement of L^*, a^*, and b^* values was recorded and repeated three times per specimen followed by an average calculation per coordinate parameter.

The color difference (ΔEab) between unmodified (control) and nanomodified PMMA (experimental) was calculated by the following equation:

$$\Delta\text{Eab} = \left[\left(\Delta L\left(L_{\text{exp}} - L_{\text{control}}\right)\right)^2 + \left(\Delta a\left(a_{\text{exp}} - a_{\text{control}}\right)\right)^2 + \left(\Delta b\left(b_{\text{exp}} - b_{\text{control}}\right)\right)^2\right]^{1/2}. \quad (1)$$

Finally, the levels of color change (ΔEab) have been quantified by the NBS units of color difference (Table 3) using the following formula to relate the color alterations (ΔEab) to the clinical environment and allow for qualitative

Table 4: Two-way ANOVA results for the effect of different variables on mean ΔEab of acrylic resin specimens.

Source of variation	Type III sum of squares	*df*	Mean square	*F*-value	*P*-value	Effect size (partial η^2)
Filler type	1,978.976	2	989.488	302.576	<0.001*	0.918
Filler concentration	116.869	1	116.869	35.737	<0.001*	0.398
Filler type × filler concentration interaction	12.735	2	6.367	1.947	0.153	0.067

df, degrees of freedom = $(n-1)$. *Significant at $P \leq 0.05$.

Table 5: Mean, SD values, and results of one-way ANOVA test for comparison between ΔEab of filler types regardless of filler concentration.

ZNP Mean ± SD	TNP Mean ± SD	SNP Mean ± SD	*P*-value	Effect size (Partial η^2)
10.7 ± 2.6^{B}	18 ± 2.7^{A}	3.9 ± 1.3^{C}	<0.001*	0.918

*Significant at $P \leq 0.05$. Different superscripts are statistically significantly different.

Table 6: Mean, SD values, and results of one-way ANOVA test for comparison between ΔEab of filler concentrations regardless of filler type.

3% Mean ± SD	7% Mean ± SD	*P*-value	Effect size (partial η^2)
9.5 ± 5.9	12.3 ± 6.3	<0.001*	0.398

*Significant at $P \leq 0.05$.

presentation of ΔEab discrepancy beside its importance for comparison of the color changes and quality control functions [38, 39].

$$\text{NBS} = \Delta\text{Eab}^* \times 0.92. \quad (2)$$

The collected data were statistically analyzed using IBM SPSS Statistics. Normality test was performed using Shapiro–Wilk and Kolmogorov–Smirnov; all data showed (normal) distribution, so a parametric test was used. One-way and two-way ANOVA tests were performed for overall analysis between groups. In case of significant value, Bonferroni's post hoc test was used for pairwise comparison. $P \leq 0.05$ was considered significant.

3. Results

Two-way ANOVA results (Table 4) showed that filler types (regardless of filler concentration) showed a statistically significant difference in ΔEab ($P<0.001$). Regardless of filler type, filler concentrations had a statistically significant effect on ΔEab ($P<0.001$). The interaction between filler types and concentrations showed no significant effect on ΔEab ($P = 0.153$), so the two variables are independent.

Mean ΔEab, standard deviation (SD), and pairwise comparisons are summarized in Tables 5–7. The effects of both concentrations of different nanoparticles on the color of PMMA specimens are shown in Figure 1. Results showed that respective to nanoparticle types, with 3 or 7 wt% concentrations, a significant difference in ΔEab was found between different filler types ($P<0.001$). Pair-wise comparisons revealed that TNP group showed the statistically significant highest mean ΔEab followed by ZNP, while SNP showed the statistically significant lowest mean ΔEab value.

Regarding nanoparticle concentrations, there was a statistically significant difference between the mean ΔEab of three filler types (ZNP, TNP, and SNP) (*P*-value < 0.001, effect size = 0.32), (*P*-value < 0.006, effect size = 0.132) and (*P*-value < 0.018, effect size = 0.1) respectively. Pair-wise comparisons revealed that 3% concentration showed statistically significant lower mean ΔEab than 7% concentration (*P*-value < 0.001, effect size = 0.398).

In concern NBS findings within NPs-modified groups (Table 7), ZNP groups ranged within extremely marked changes (6–12) and TNP group changes to other colors (12 or more). While SNP showed a noticeable change with 3SNP which changed with 7SNP with a marked change.

4. Discussion

Ideally, incorporating nanoparticles into PMMA denture base resin should enhance the properties of PMMA nanocomposite while maintaining its original color for esthetic purposes and patient satisfaction [28]. Accordingly, different NPs with two concentrations were selected in the current study to evaluate and compare the color change of nanocomposite denture base resins after NPs addition. Two concentrations (3 and 7 wt%) of nanoparticles were chosen based on previous studies; it was reported that 7% and less concentrations are recommended, otherwise above this concentration, an obvious color change occurred [11, 17, 31]. The null hypothesis was rejected as the addition of ZNP, TNP, and SNP at both concentrations affected the color of the heat-polymerized PMMA denture base resin.

All samples were fabricated as routinely used in dental practice by one operator for standardization and to minimize possible researcher-related variability as well as to simulate clinical manipulation of the acrylic dentures. Moreover, the manufacturer's instructions for storage, mixing time, and manipulation were carefully followed to avoid any faults during the preparation of specimens [6, 8]. Nanoparticles were treated with silane coupling agent and then added individually to PMMA powder by mechanical mixing to improve

Table 7: Mean, SD values, and results of one-way ANOVA test for comparison between ΔEab of filler types with each filler concentration.

Concentration	ZNP Mean ± SD	TNP Mean ± SD	SNP Mean ± SD	*P*-value	Effect size (partial η^2)
3%	8.7 ± 0.6^{B}	16.8 ± 2.1^{A}	3 ± 0.6^{C}	<0.001*	0.847
NBS	8.04	15.45	2.76		
7%	12.8 ± 2.3^{B}	19.2 ± 2.9^{A}	4.9 ± 1^{C}	<0.001*	0.852
NBS	11.77	17.66	4.5		
P-value	<0.001*	0.006*	0.018*		
Effect size (partial η^2)	0.32	0.132	0.1		

*Significant at $P \leq 0.05$. Different superscripts in the same row indicate statistically significant difference between filler types.

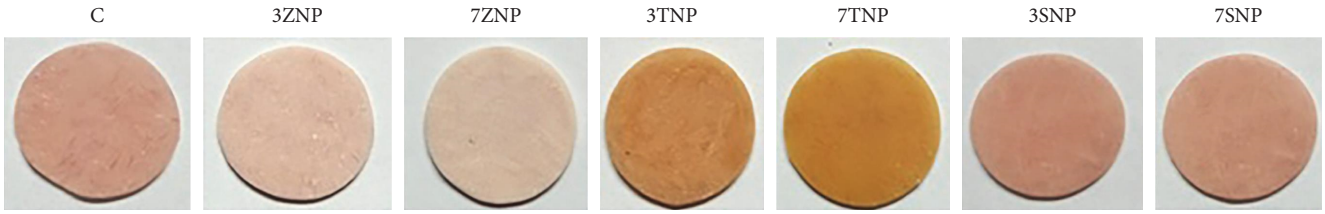

Figure 1: Representative images for specimens' color without (C) and with nanoparticles (NPs).

their binding, prevent agglomeration, and allow a homogenous mix [34, 40]. Spectrophotometers are commonly used to evaluate color changes in dental materials as they are sensitive to minor color alterations, repeatable, and can eliminate subjective interpretations [41].

According to the results of this study, incorporation of 3 and 7 wt% of ZNP into PMMA resulted in much and very much color changes, respectively. Based on ΔEab values, the present study found significant color differences between modified and unmodified specimens with different nanoparticle types and concentrations; TNP had the highest mean ΔEab followed by ZNP. while SNP showed the lowest mean ΔEab value and the color changes were increased with the increasing of nanoparticles concentrations which was clinically unacceptable. In agreement with the current results, Ihab et al. [9] and Fathey et al. [42] reported significant color differences between the control group and ZrO_2-modified specimens at different concentrations which were clinically unacceptable. Moreover, previous studies agreed with the present findings and reported that PMMA color was changed to pale pinkish white with ZNP addition [43, 44] and as the concentration of ZNP increased, the color change was increased [24, 45]. On the other hand, our findings are in disagreement with other studies which concluded that the addition of ZNP displayed insignificant color changes of PMMA [27, 46].

The incorporation of 3 and 7 wt% of TNP resulted in very much color change and change of specimens' color to another one, respectively. In agreement with the present findings, Shakir and Abdul-Ameer [47] concluded that the addition of TNP adversely affected the color of maxillofacial elastomer. The color changes may be attributed to that, nanocomposites formed of a mixture of two materials (resin and NPs) which have different refractive indices. Therefore, the differences in refractive index could be the reason for the change in color with the addition of ZNP and TNP. The presence of NPs affects the refraction and reflection of light at the interface between resin and NPs. It was reported that the higher the refractive index difference between the two phases, the greater the opacity of the nanocomposite with decreasing in their translucency producing opaque color. The refractive index of both TNP and ZNP is higher than that of PMMA [42, 48, 49].

SNP showed noticeable color change which was clinically acceptable with 3 wt% and unacceptable with 7 wt%. These results come in agreement with Kotanidis et al. [26] who found that SNPs slightly affected the optical properties of PMMA resin with significantly lower color changes and attributed that SNP could effectively reduce the water absorption and solubility of acrylic resin improving its color stability. Moreover, the refractive index of SNP is closer than that of PMMA, which could be considered as another reason for good color stability of SNP, which is consistent with Gad et al. [25] study.

Regardless of the nanoparticles type, low concentration (3 wt%) showed lower ΔEab. This result comes in agreement with Ihab et al. [9] who compared the color differences after ZNP addition and showed a significant difference between control and modified specimens and reported that ΔEab was increased with the increasing of ZNP concentration. On the other hand, this finding is in disagreement with previous studies [27, 46]. The discrepancies between the results of this study and other articles may be explained by the differences in experimental methodology, concentrations of tested NPs as well as the usage of different types of PMMA material.

From the clinical point of view, the application of nanotechnology in the improvement of dental materials greatly

influences the success of many dental procedures, so modification of PMMA with ZNP, TNP, or SNP could lead to improving their mechanical properties and strength. However, the balance between the strength and esthetics of the final nanocomposite should be considered. These findings proved the fact that the selection of NP type and concentrations are the main factors affecting the esthetics of removable prostheses fabricated from this nanocomposite denture base resin. Considering this fact, proper selection of NP type and concentrations that could enhance the strength and at the same time has no adverse effect on the esthetics and patient satisfaction is recommended.

One strength of this study is the comparison between different nanoparticles; however, using only two concentrations per nanoparticle add study limitation. Using one type of PMMA with a simple disc-shaped specimen and lack of clinical simulation such as human saliva and different cleansing protocols that can affect the results were considered as study limitations, in addition to thermocycling was not performed. In terms of color evaluation method, recently CIEDE2000 is recommended due to its reliability compared CIELab ΔE. Accordingly, clinical and long-term studies to support these results, further research using different denture base materials, and different nanoparticle types with different concentrations are recommended for color stability assessment using CIEDE2000 method.

5. Conclusion

Within the limitations of the present study, it could be concluded that

(1) Addition of ZNP, TNP, or SNP causes clinically unacceptable color changes of heat polymerized PMMA; TNP produces the highest color change followed by ZNP, while SNP produces the least color change.

(2) The color change increases with the increase of nanoparticle concentration from 3 to 7 wt%.

(3) Careful selection of both types and concentration of reinforcing fillers to achieve a balance between mechanical properties and esthetic concerns was recommended.

Data Availability

The data are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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