



ORIGINAL RESEARCH

INFLUENCE OF THE ADDITION OF NANO CERIUM OXIDE/CHITOSAN COMPOSITE ON THE MECHANICAL CHARACTERISTICS OF POLYMETHYLMETHACRYLATE RESIN

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ABSTRACT

Background: Cerium oxide and chitosan (Ce/CS) nanoparticles have garnered significant interest due to their remarkable properties, including antioxidant activity, biocompatibility, antibacterial effectiveness and anti-inflammatory activities. This study evaluates the impact of incorporating a Ce/CS nanocomposite into polymethylmethacrylate (PMMA) resin on its mechanical properties. The composite consisted of 30% cerium oxide and 70% chitosan by weight (wt), with varying percentages incorporated into the PMMA resin selected according to a pilot study.

Material and Methods: Ninety PMMA specimens were fabricated and categorized into three groups according to the Ce/CS nanocomposite percentage in the PMMA resin: control (0 wt%), group A (1 wt%), and group B (5 wt%). Transverse strength, impact strength, and surface hardness were assessed, with 10 specimens for each test. Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM) were used to examine the chemical and surface alterations of PMMA resin. The collected data were analyzed using one-way ANOVA and Tukey HSD test, with a significance level of $P \leq 0.05$.

Results: The resulting data revealed a significant difference among all groups ($P \leq 0.05$). Group A exhibited the highest values in transverse and impact strength compared with the other groups. Group B showed the highest values in surface hardness compared with group A and the control group. The FTIR graph indicated changes in the material's chemical structure, suggesting the formation of new functional groups. SEM image showed an even distribution of Ce/CS nanocomposite inside the PMMA matrix of group A compared with group B.

Conclusion: The study concludes that incorporating 1 wt% of a nano cerium oxide/chitosan composite into PMMA denture base resin significantly enhances its transverse and impact strength compared with 5 wt%. Furthermore, both 1 wt% and 5 wt% Ce/CS nanocomposites resulted in a notable improvement in the surface hardness of the PMMA resin.

Keywords: Cerium oxide; Chitosan; Polymethylmethacrylate ; Transverse strength; Impact strength; Surface hardness.

INTRODUCTION

The Denture base, typically made of polymethylmethacrylate (PMMA), is a non-toxic and inexpensive material with low water absorption and aesthetically pleasing features. It is easy to process, finish, polish and repair¹. However, despite fulfilling cosmetic requirements for dental prostheses, PMMA resin lacks the mechanical performance needed for long-term durability². Its primary drawback lies in its poor mechanical properties, including low flexural

strength, hardness, and limited fracture and impact strength. Therefore, improving PMMA resin involves enhancing its mechanical properties.

In recent years, nanotechnology has been increasingly applied in dental materials, with numerous studies exploring its benefits and clinical applications³.

Polymeric nanocomposites, composed of nanoscale fillers dispersed within a polymer matrix, have emerged as promising candidates for this purpose⁴. One of the most

practical and cost-effective strategies for improving the mechanical, thermal, chemical, and physical properties of PMMA is the incorporation of suitable nanofillers⁵. These nanoscale reinforcing agents can impart unique physical, mechanical, and biological properties, resulting in advanced nanocomposites with superior performance⁶.

Previous studies have explored various filler materials, including glass ceramics, alumina (Al_2O_3), hydroxyapatite, yttria-stabilized zirconia, nitrile rubber, polyethylene fibers, fiberglass, and stainless steel, all of which have significantly enhanced the properties of PMMA⁷.

Chitosan (CS), a linear polysaccharide, offers exceptional biological properties, including antimicrobial activity, biodegradability, non-toxicity, and biocompatibility⁸. Similarly, Cerium oxide (CeO_2) nanoparticles have garnered considerable interest due to their remarkable characteristics, such as redox activity, biofilm inhibition, antibacterial efficacy, and anti-inflammatory effects⁹. Furthermore, incorporating nano CeO_2 into denture base resins has improved their mechanical properties¹⁰.

This study evaluates the effect of incorporating cerium oxide and chitosan (Ce/CS) nanocomposite into PMMA on its mechanical properties. It uses a fixed Ce/CS ratio applied at different percentages designated by a pilot study.

MATERIALS AND METHODS

Specimen grouping

A total of 90 specimens of PMMA material (PROCRYLA® / Fast Heat-Polymerizing Acrylic Resin, PD, Germany) were prepared and categorized into three groups (30 specimens each) based on the percentage composition of the Ce/CS nanocomposite. A composite ratio of 30 wt% Ce and 70 wt% CS was incorporated into the PMMA resin, selected from a pilot study that included both transverse strength and surface hardness tests. For each test, nine PMMA specimens were prepared with varying percentages of CS/Ce fillers into the PMMA: 1 wt%, 3 wt% and 5 wt%, and three control specimens without filler. The study comprised three groups: a control group (C) consisting of PMMA specimens without filler, group (A) with PMMA containing 1 wt% of Ce/CS nanocomposite and group (B) with PMMA containing 5 wt% of Ce/CS nanocomposite. Three mechanical tests were conducted: transverse strength, impact strength, and surface hardness. For each test, 10 PMMA specimens were used.

Specimen patterns preparation

Plastic patterns were fabricated using laser-cutting machines, with dimensions specified according to the requirements of each mechanical test. For the transverse strength test, the specimens measured $65 \times 10 \times 2.5$ mm in length, width and thickness,

respectively, following ADA (1999) guidelines¹¹. For the impact strength test, the specimens were measured $80 \times 10 \times 4$ mm in length, width, and thickness, respectively, following ISO 179-1:2000 standards¹². For the surface hardness test, the specimens measured $35 \times 35 \times 6$ mm in length, width and thickness, respectively, prepared according to ISO 868:2003 standard¹³, as shown in Fig. 1.

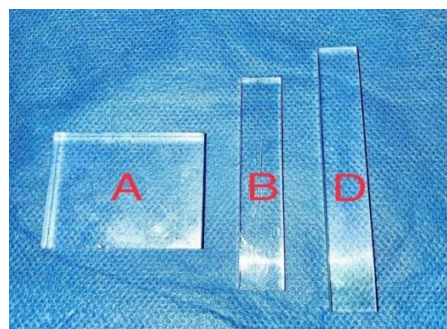


Figure 1. Transparent plastic patterns. A: For hardness test, B: For Transverse strength test, D: For impact strength test.

The plastic patterns were coated with a separating medium and invested in a metal flask using Type 4 dental stone (Zhermack®, Italy), prepared according to the manufacturer's instructions at a water-to-powder ratio of 25 mL/100 g. After the dental stone had set, the two halves of the flask were separated and the patterns were removed to form mold cavities for the PMMA resin.

Calculating the mixing amount of PMMA and Ce/CS nanocomposite

The PMMA resin was prepared according to the manufacturer's instructions by mixing 21 grams of polymer with 10 ml of monomer. The quantities of polymer and Ce/CS nanocomposite used in this study are detailed in Table 1. The volume of monomer used was consistently 10 ml for all groups.

Table 1. Mixing amount of Ce/CS nanocomposite and PMMA resin powder.

| Groups | Amount of Ce+CS (gm) | Amount of Polymer (gm) |
|---------|------------------------|------------------------|
| Control | 0 | 21 |
| A | $0.21 = 0.063 + 0.147$ | 20.79 |
| B | $1.05 = 0.315 + 0.735$ | 19.95 |

Mixing and packing of Ce/CS nanocomposite and PMMA resin

A probe sonication machine was used to mix Ce/CS nanocomposite and PMMA powders with the monomer, utilizing a clean and dry glass mixing bowl to ensure uniform dispersion of the fillers and resin powders within the monomer. The mixing process continued for five minutes, using 120 watts of power at a frequency of 60

kilohertz. These parameters were selected based on a pilot study, demonstrating their effectiveness in breaking the fillers into individual nanocrystals and achieving a homogeneous mixture. The mixture was left for five minutes until it reached the dough stage. The PMMA resin was then packed and inserted into the mold cavity within the flask. A polyethylene sheet was placed over the resin to achieve a smooth surface. The two halves of the flask were sealed and placed under a hydraulic press at 100 psi to ensure uniform distribution of the PMMA resin inside the mold. Excess resin was removed, and the flask was placed in a water bath for curing. Following the manufacturer's recommendations, a short curing cycle with a two-step process was initially carried out for 1.5 hours at 74°C, followed by an additional 0.5 hours at 100°C.

Transverse strength test

A three-point bending test was proposed to evaluate the transverse strength of PMMA specimens using an Instron universal testing machine. Each specimen was placed on two opposing support wedges, separated by 50 mm. A load was then applied at the center of the specimen using a loading rod positioned midway between the two supports (Fig. 2). The transverse strength was calculated according to the following equation:

$$\text{Transverse strength} = \frac{3FL}{2bd^2}$$

Where: P = Maximum force exerted on specimen (N), L = Distance between the supports (mm), b = The specimen width (mm) and d = specimen depth (mm). The load was applied with a crosshead speed of 1 mm/min, bending the specimens until fracture occurred. The fractured surfaces were examined using a scanning electron microscope (SEM).

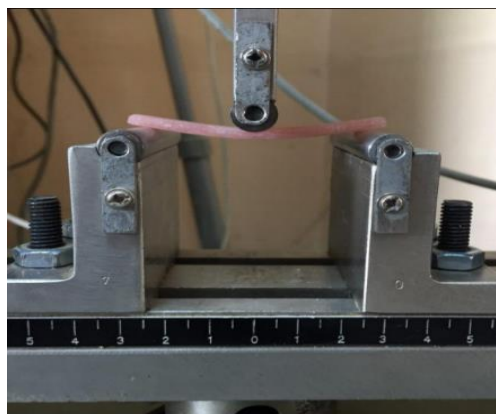


Figure 2. Instron universal testing machine and the specimen is under test.

Impact strength test

The impact strength test was conducted using a Charpy impact testing device, which applies a pendulum to calculate the impact load on the PMMA specimen (13). The impact strength was calculated using the following formula:

$$\text{Impact strength} = \frac{E}{bd10^3}$$

Where: E = absorption of impact energy in joules, b = width of the specimen (mm) and d = thickness of the specimen (mm).

Surface hardness test

The Shore D hardness device measures the depth of penetration of the durometer indenter foot into the specimen surface. For each PMMA specimen, hardness measurements were taken at five locations: one in the center and four in the margins. The surface hardness value for each specimen was determined by calculating the mean of these five measurements, following the ASTM D2240 and ISO 868:2005 (14).

Fourier transform infrared spectroscopy (FTIR) and SEM were used to examine the chemical and surface alterations of PMMA resin.

Statistical analysis

Data description, analysis, and presentation were conducted using the Statistical Package for the Social Sciences (SPSS, version 22; Chicago, IL, USA). The Shapiro–Wilk test was applied to assess the normality of the variables, confirming that all were normally distributed across the groups. A one-way analysis of variance (ANOVA) was conducted, followed by Tukey's post hoc test for pairwise comparisons among the groups, with a significance level of $P \leq 0.05$.

RESULTS

Cross-sectional examination of the fractured PMMA specimens using SEM at 1000× magnification revealed a smooth and homogeneous resin matrix in the control group (Fig. 3A). In contrast, specimens from group A exhibited a uniform dispersion of Ce/CS nanoparticles within the resin matrix (Fig. 3B), while in group B, filler particles were unevenly distributed, forming aggregated clusters in the matrix (Fig. 3C).

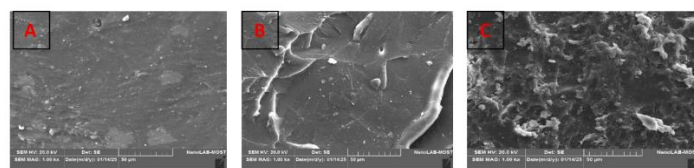


Figure 3. Control (A), 1% by vol. (B), 5% by vol. group (C) specimen under the scanning electron microscope at a magnification scale of 50µm.

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum study reveals chemical interaction between cerium oxide/chitosan nanofiller powder and PMMA, indicating the formation of new functional groups (Fig. 4A, 4B, and 4C). From a chemical perspective, these interactions suggest a reaction between the acrylic resin and the cerium oxide/chitosan nanofiller, as demonstrated by observable changes in the FTIR spectra.

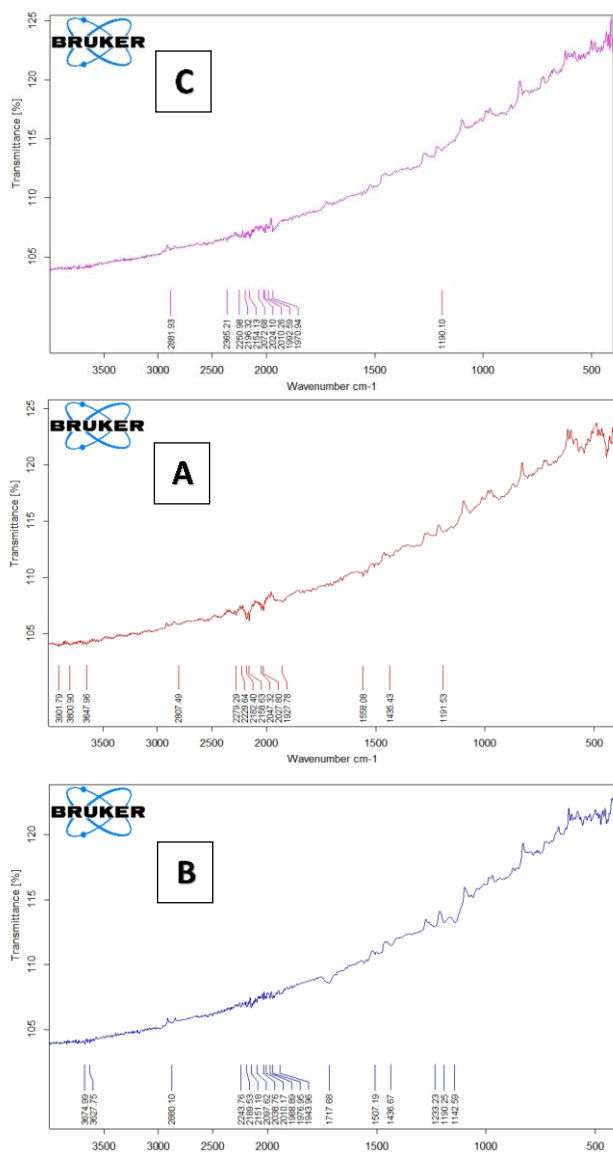


Figure 4. FTIR graph for the control (C) and experimental group (A) and (B).

Transverse strength statistics

Descriptive analysis of transverse strength values showed that group A exhibited the highest mean value (104.54 N/mm²), followed by the control group (93.15 N/mm²) and group B (89.76 N/mm²). One-way ANOVA revealed a statistically significant difference among the tested groups (P value ≤ 0.05), as shown in Table 2. Post hoc comparisons using Tukey's HSD test indicated a significant difference at $P \leq 0.05$ between group A and the control group; however, no significant difference between the control group and group B. Moreover, a significant difference ($P \leq 0.001$) was observed between groups A and B.

Table 2. The descriptive statistics and One-way ANOVA test of the transverse strength value in (N/mm²).

| Groups | N | Mean | ± SD | F | Sig. $P \leq 0.05$ |
|---------|----|--------|------|--------|--------------------|
| Control | 10 | 93.15 | 2.69 | | |
| A | 10 | 104.54 | 2.66 | 103.22 | 0.00 |
| B | 10 | 89.76 | 1.74 | | |

Impact strength statistics

The mean value of group A exhibited the highest impact strength value (11.33 KJ/m²), followed by the control group (10.04 KJ/m²), while group B displayed the lowest value (9.63 KJ/m²). Furthermore, the one-way ANOVA test revealed a significant difference at $P \leq 0.05$ among the tested groups, as shown in Table 3. The Tukey's HSD test showed a significant difference between groups A and the control group and group B. However, no significant difference was observed between Group B and the control group.

Table 3. Descriptive statistics and One-way ANOVA test of the impact strength test of all experimental groups in (KJ/m²).

| Groups | N | Mean | ±SD | F | Sig. $P \leq 0.05$ |
|---------|----|-------|------|-------|--------------------|
| Control | 10 | 10.04 | 0.68 | | |
| A | 10 | 11.33 | 0.87 | 11.20 | 0.00 |
| B | 10 | 9.63 | 0.93 | | |

Surface hardness statistics

Group A exhibited the highest mean value (88.07), followed by group B (85.80), while the control group had the lowest value (84.46). A one-way ANOVA revealed a statistically significant difference among the groups ($P \leq 0.05$), as shown in Table 4. Post hoc analysis using Tukey's test indicated significant differences between the experimental groups A and B compared to the control group and between groups A and B.

Table 4. The descriptive statistics and One-way ANOVA test for the surface hardness test of all groups.

| Groups | N | Mean | ±SD | F | Sig. $P \leq 0.05$ |
|---------|----|-------|------|-------|--------------------|
| Control | 10 | 84.46 | 0.60 | | |
| A | 10 | 88.07 | 0.75 | 49.99 | 0.00 |
| B | 10 | 85.80 | 1.03 | | |

DISCUSSION

Numerous researchers have proposed that the mechanical and physical properties of PMMA resin can be improved by incorporating various additives¹⁵. The null hypothesis of the current study was rejected. A mixture of nanosized cerium oxide and chitosan particles was added to the PMMA resin in a specific amount, determined through a pilot study, to improve its mechanical and physical properties. The filler particles are significantly smaller than the PMMA resin powder, allowing them to fill the interstitial spaces within the PMMA polymer matrix. This leads to a heterogeneous composite without disturbing the segments of the polymer chain. Furthermore, the filler proportion must remain minimal to ensure proper interlocking with the PMMA resin and maintain the PMMA matrix's structural integrity¹⁵.

The transverse strength test evaluates a material's resistance to bending and its stiffness, providing insight into its mechanical behavior under flexural stress¹⁶. High flexural strength is critical for denture base materials, as they face repeated flexing during mastication, resulting in fatigue and potential failure¹⁷. The three-point bending test is often used to replicate the functional stresses experienced by dentures during chewing and other oral activities¹⁸.

In this study, incorporation of 1 wt% Ce/CS nanocomposite into PMMA increased the transverse strength value to 104.54 N/mm², compared with adding 5 wt%, which exhibited the lowest value at 89.76 N/mm². This improvement is attributed to the uniform distribution of Ce/CS nanoparticles within the PMMA matrix, as shown in Fig. 4, which shows the incorporation of 1 wt% Ce/CS nanoparticles into PMMA. Furthermore, the enhanced transverse strength is attributed to the formation of interconnections across the linear macromolecular chains, promoted by the nanoparticles. These findings are consistent with the observations of Alamel and Mudhaffer, who reported that transverse strength diminished with increased concentrations of SiO₂ nanoparticles, reinforcing the importance of optimizing filler content for mechanical performance¹⁹.

Several studies have reported that incorporating carbon-based nanomaterials, such as graphene oxide, into PMMA resins enhances their mechanical properties²⁰ and reduces the degree of polymerization shrinkage. This reduction in shrinkage restricts the segmental motion of the macromolecular chains, increasing the material's strength and rigidity, thereby improving both fracture resistance and transverse strength²¹.

The tendency of nanoparticles to agglomerate can lead to a reduction in transverse strength when 1 wt% Ce/CS nanocomposites are incorporated, as this aggregation induces stress concentrations at the agglomerated sites, thereby diminishing mechanical properties²². Once the PMMA resin reaches its filler saturation point, it cannot incorporate additional nanoparticles without compromising the integrity of the resin matrix, resulting in reduced performance. This alteration is statistically significant and will influence the performance of the PMMA-Ce/CS nanocomposite. Xia et al. (2008) emphasized the significance of uniform dispersion and reported that transverse strength improved with an optimal distribution of nanofiller particles (100 nm)²³.

Impact strength test measures the energy required per unit area to fracture a material under sudden force, such as the impact from a falling denture. Therefore, denture base materials must possess sufficient impact

strength to prevent breakage in such situations²⁴. In this study, a Charpy impact testing machine was employed, one of the most commonly used methods for assessing a material's toughness²⁵. High-impact denture base resins are especially useful for individuals at a high risk of dropping their dentures, such as patients with physical limitations or cognitive impairments. This study demonstrated that incorporating a 1 wt% Ce/CS nanocomposite significantly increased the impact strength value compared with the control group. Statistically, no significant differences were observed among the other tested groups. The interfacial shear strength between the nanofiller and the matrix significantly enhances the impact strength due to the formation of cross-links or supramolecular bonds. The nanofillers act as a shield, preventing the propagation of fractures. Furthermore, ensuring strong bonding between the nanofiller and the resin matrix can effectively alter the fracture propagation path²⁶.

Additionally, the critical stress value for particle/matrix debonding increases as particle size decreases, which may also contribute to enhanced impact strength. This improvement results from the increased total particle/matrix interfacial surface area available for energy dissipation²⁷. The decrease may be attributed to the agglomeration of nanoparticles, which possess high surface energy and tend to adhere to one another, forming clumps. Agglomeration likely results from difficulties dispersing nanofillers uniformly in the polymer matrix using conventional methods such as probe sonication apparatus, leading to inhomogeneity and promoting nanoparticle aggregation as illustrated in Fig. 4. Consequently, increasing in nanoparticle concentration to 5 wt% correlates with a decrease in impact strength, likely due to poor distribution within the acrylic matrix (28). This clarifies why the impact strength of the 5 wt% nanoparticles group is inferior to that of the 1 wt% group.

Another explanation is inadequate bonding at the interface between the inorganic CeO₂ nanoparticles and the organic polymer matrix in the PMMA-containing nanoparticle fillers. This weak interfacial bonding leads to stress concentration at the filler–matrix interface. The ineffective transmission of stress across the filler–matrix interface results in localized stress concentrations, which facilitate crack initiation and ultimately lead to material failure. The PMMA matrix demonstrates brittle behavior, characterized by rapid crack propagation and sudden fracture of the specimen. This behavior indicates a low resistance to crack growth and suggests that only a small amount of energy is required to initiate and propagate fractures within the material²⁹.

Surface hardness refers to a material's resistance to surface penetration or indentation. The Shore D hardness test was employed in this study because it is more suitable for PMMA resins³⁰. The results showed a significant improvement in surface hardness with the addition of

Ce/CS nanocomposite compared to the control group. This enhancement is attributed to the reduced interparticle distance within the polymer matrix facilitated by Ce/CS nanoparticles. The decreased spacing promotes stronger interparticle bonding, leading to nanoparticle aggregation and the accumulation of hard Ce/CS nanoparticles on the surface of the PMMA matrix, thereby improving the material's hardness performance³¹.

Furthermore, a high concentration of nanocomposite group (5 wt%) resulted in a considerable increase in hardness due to the denser network of reinforcing particles. In contrast, the low concentration group (1 wt%) produced a modest increase in hardness, probably due to the lower network density and reduced accumulation of hard particles within the PMMA resin matrix³².

The current study recommends that future research be conducted to investigate the effects of the Ce/CS nanocomposite on the color, polishability, and clinical performance of denture bases fabricated from PMMA resin.

CONCLUSION

Within the limitations of this study, the 1 wt.% of a nano cerium oxide/chitosan composite incorporated into PMMA denture base resin significantly enhances transverse and impact strength compared with 5 wt.%. Furthermore, both 1 wt.% and 5 wt.% Ce/CS nanocomposites led to a notable improvement in the surface hardness of the PMMA resin.

DECLARATIONS

Competing interest

The authors declare that there are no competing interest.

Funding

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Ethical Approval

None declared.

Competing interests

The authors declare no competing interests.

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