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Influence of the Addition of Nano Cerium Oxide/Chitosan Composite on the physical Characteristics of Polymethylmethacrylate Resin

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Abstract:

Cerium oxide and chitosan (Ce/CS) nanoparticles' antioxidant, biocompatible, antibacterial, and anti-inflammatory properties have garnered interest. Integration of a Ce/CS nanocomposite into polymethylmethacrylate (PMMA) resin affects its physical properties. Based on an initial investigation, the composite consisted of 30% cerium oxide and 70% chitosan by weight, blended with PMMA resin in varying proportions. Data analysis showed significant differences among all groups ($P \leq 0.05$). Surface roughness did not change between the control and experimental groups employing 1% and 5% CS/Ce ($P > 0.05$). Water sorption and solubility were lowest in group B compared to A and the control group. The FTIR graph showed changed chemical structure, suggesting new functional groups. Ce/CS nanocomposite was evenly distributed in group A's PMMA matrix by SEM compared to group B. The study found that adding 5 wt.% nano cerium oxide/chitosan composite to PMMA denture base resin reduces its water sorption and solubility. The surface roughness of PMMA resin improved non-significantly using 1 and 5 wt.% Ce/CS nanocomposites.

Keywords: Cerium oxide; Chitosan; Polymethylmethacrylate; surface roughness; and water sorption solubility.

Introduction:

Polymethyl methacrylate (PMMA) acrylic resin is frequently employed as a denture foundation material due to its cost-effectiveness, flexibility, and adaptability in a variety of applications, such as complete denture bases, denture teeth, implant-supported dentures, and orthodontics (Maji et al., 2016). The most frequently used material in the fabrication of partial, removable, and complete denture bases and facings for fixed bridges is acrylic resin, which enhances the aesthetic appeal of the restoration (Noori et al., 2023). The long-term service life of acrylic resin material is challenging to predict due to the numerous environmental factors that influence its strength (Abdullah, 2023). Water sorption and release are among the characteristics

of acrylic polymers, which can lead to dimensional instability and internal stresses. These features can lead to the formation of cracks and, in the long term, denture fractures. Water is gradually absorbed by acrylic polymers over an extended period. The resin molecules' polar properties are the primary cause of this imbibition. The sorption of material is the quantity of water that is adsorbed on the surface and incorporated into the material's body during fabrication or while the restoration is in service. Generally, the term "sorption" is employed to encompass the complete phenomenon, as both adsorption and absorption are involved. A high percentage of water sorption usually results in warpage and a major dimensional change in the material (Malacarne et al., 2006). The mass of the soluble materials from polymers is denoted by the term for solubility. Initiators, plasticisers, and free monomers are the sole soluble components of denture base resins. The solubility of the specimen is determined by any observed decrease in resin weight. Consequently, the durability is significantly impacted by the solubility and water sorption (Phillips, 1973). Takahashi et al. (Takahashi et al., 1998) discovered that water molecules diffuse between the macromolecules of the material, thereby causing them to separate. The dimensional behaviour and denture stability are influenced by this behaviour; as a result, the water absorption and solubility of these materials should be minimised. Polymer networks should be insoluble materials that exhibit relatively high chemical and thermal stability. Nevertheless, the majority of the monomers utilised in dental resin materials have the ability to incorporate water and substances from the environment, as well as release components into the encompassing environment. The roughness of the denture base surface is known to induce halitosis and is considered more susceptible to discolouration than smooth surfaces, thereby diminishing patient comfort (Al-Dwairi et al., 2019) (Rasan and Farhan, 2023). Denture stomatitis, an irritation of the mucosa beneath the denture, can be exacerbated by rough surfaces. However, it is crucial due to its impact on the dental health of tissues that are in close proximity to prostheses. To overcome these drawbacks, various types of fillers and fibers, such as zirconia, glass fiber, alumina, tin, and copper, have been employed in an attempt to enhance and modify the properties of PMMA (Messersmith and Giannelis, 1994). By incorporating the modified nanoparticles of zirconium dioxide (ZrO2) into acrylic resin, the abrasive wear resistance, tensile, and fatigue strength were enhanced, while the water sorption, solubility, and porosity of the heat-cured denture base resin were reduced (Mohammed and Mudhaffar, 2012). Chitosan (CS), a linear polysaccharide, possesses extraordinary biological properties, such as biodegradability, biocompatibility, non-toxicity, and antimicrobial activity (Chandrasekaran et al., 2020). In the same time, Cerium oxide (CeO_2) nanoparticles have attracted significant attention as a result of their exceptional properties, including biofilm inhibition, redox activity, antibacterial efficacy, and anti-inflammatory effects (Jairam et al., 2023). Additionally, the mechanical properties of denture base resins have been enhanced by the incorporation of nano CeO_2 (CHU et al., 2015). This investigation assesses the physical properties of PMMA following the incorporation of a cerium oxide and chitosan (Ce/CS) nanocomposite. It employs a fixed Ce/CS ratio that is implemented at varying percentages as determined by a pilot study.

Materials and Methods:

Specimen grouping:

Sixty specimens of PMMA material (PROCRYLA® / Fast Heat-Polymerizing Acrylic Resin, PD, Germany) were manufactured and classified into three groups (control, group A and B) each of one containing 20 specimens, according to the percentage composition of the Ce/CS nanocomposite. A composite ratio of 30 wt.% Ce and 70 wt.% CS was integrated into the PMMA resin, chosen from the pilot study. The investigation included three groups: a control group (C) with PMMA specimens devoid of filler, group (A) with PMMA incorporating 1 wt.% of Ce/CS nanocomposite, and group (B) with PMMA including 5 wt.% of Ce/CS nanocomposite. two physical tests were performed: water sorption and solubility, and surface roughness test. Ten PMMA specimens were used for each test.

Specimen patterns preparation:

A circular stainless-steel mould and lid were fabricated in compliance with ISO standards (20795-1, 2013) for the assessment of surface roughness, water sorption, and solubility. (Figure 1) delineates the proportions of the mould and cover. A lathe machine was used to fabricate them in the specified shape and dimensions (Figure 2.5). ISO (20795-1, 2013) criteria were followed to create specimens with the following dimensions: 50 ± 1 mm diameter, 0.5 ± 0.1 mm thickness. All specimens were stored in distilled water at 37° C for 48 hours before testing (ADA standard No. 12, 1999).

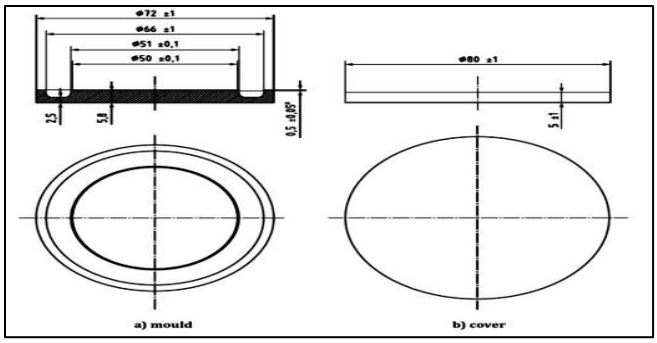

Figure 1: Dimensions of the stainless-steel mould and cover for surface roughness and water sorption and solubility test (International standard 20795-1, 2013)

Figure 2: Finished stainless steel A: Mold, B: Cover.

Acrylic specimens were produced using the same methodology as in the fabrication of acrylic dentures. The stainless-steel mould and a lid were coated with a separating agent and invested in a metal flask using Type 4 dental stone (Zhermack®, Italy), made in accordance with the manufacturer's specifications at a water-to-powder ratio of 25 mL/100 g. after the setting of the dental stone, the two parts of the flask were detached, and ready for the PMMA resin as (Figure 3) shows.

Figure 3: Stainless steel mold after investment.

PMMA-Ce/CS nanocomposite mixing calculation:

The PMMA resin was synthesised following the manufacturer's guidelines by combining 21 grammes of polymer with 10 millilitres of monomer. The amounts of polymer and Ce/CS nanocomposite used in this work are specified in Table 1. The amount of monomer used was uniformly 10 ml across all groups.

Table 1: Mixing ratio of acrylic resin powder and Chitosan-Cerium Oxide (CS & Ce) powder.

Groups	Amount of CS/Ce(g)	Quantity of polymer (g)	Quantity of monomer (ml)
Control group (C)	0	21	10
1wt%. CS/Ce	0.21	20.79	10
70%CS / 30%Ce	0.147 CS / 0.063 Ce		
5wt% CS/Ce	1.05	19.95	10
70%CS / 30%Ce	0.735 CS / 0.315 Ce		

Mixing and packing of PMMA resin and Ce/CS nanocomposite:

The monomer was mixed with Ce/CS nanocomposite and PMMA powders in a clean and dried glass by using a probe sonication machine to ensure that the fillers and resin powders were uniformly dispersed within the monomer. The blending process persisted for five minutes, utilising 120 watts of power at a frequency of 60 kilohertz. The effectiveness of these parameters in separating the additives into individual nanocrystals and attaining a homogeneous

mixture was demonstrated in a pilot study. Consequently, they were chosen. The dough stage was achieved by allowing the mixture to sit for five minutes. The PMMA resin was subsequently packed and inserted into the mould cavity within the flask. In order to obtain a uniform and smooth surface, a polyethylene sheet was affixed to the resin inside the flask. The PMMA resin was uniformly distributed within the mould by sealing the two halves of the flask and placing them under a hydraulic press at 100 psi. The flask was cured in a water immersion after the excess resin was removed. A brief curing cycle with a two-step procedure was implemented in accordance with the manufacturer's instructions. The initial phase lasted for 1.5 hours at 74°C, and an additional 0.5 hours was implemented at 100°C.

Surface roughness test:

A portable roughness tester (TR220-China) was utilized to analyze surface roughness to ANSI/ADA standard, No.12, 1999. The apparatus has a diamond-sharp stylus for surface irregularity analysis. For each specimen, the stylus was set to make three standardized contacts and gather three readings (Figure 4).

Figure 4: A profilometer was used to analyse the roughness of the diamond surface analyser.

The specimen was positioned on a rigid surface, and the stylus was let to contact the first point. The device scale exhibited the data, and the average of three measurements (one at the centre and two at each extremity) established the mean Ra values were calculated in micrometres (μm).

Water sorption and solubility test:

The specimens were dehydrated in a chamber with new silica gel, as seen in Figure 5. The discs were then placed in an incubator at a temperature of 37°C ± 2°C for 24 hours.

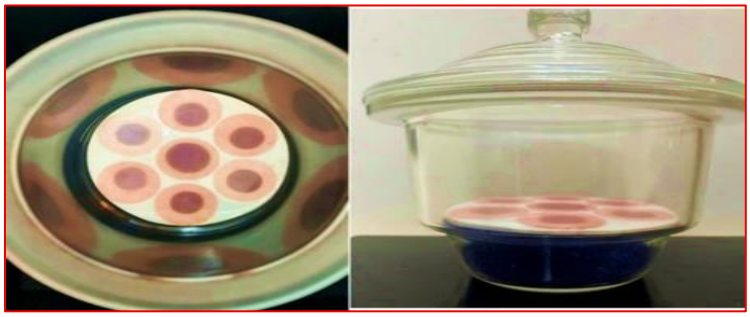

Figure 5: The specimens were dehydrated in a desiccation chamber filled with a new, fresh silica gel.

The specimens were then transferred to room temperature and placed in a separate desiccator containing newly dried silica gel for a period of (60 ± 10) minutes. The readings were later acquired using an electronic balance with an accuracy of (0.0001g), as seen in (Figure 6). The technique was performed every day at a fixed time until a stable mass, referred to as conditioned mass (m1), was achieved after five days, during which the mass decrease for each specimen did not exceed 0.2 mg (0.0002 g).

Figure 6: Measurement of specimen mass using an electronic balance with an accuracy of (0.000l g).

Specimens were submerged in distilled water at 37°C ± 2°C for a duration of seven days. Subsequent to the specified duration, each specimen was extracted from the water using tweezers and dried using a pristine towel. Thereafter, the specimen was exposed to ambient air for 15 seconds and weighed on a digital scale, resulting in a measurement referred to as (m_2). After weighing, specimens were reconditioned in desiccators at 37°C ± 2°C until a constant mass was achieved. The mass acquired after reconditioning was recorded as (m_3).

The water sorption and solubility measurement (WS) for each specimen was calculated using the subsequent equations:

$WSP = \frac{m_2 - m_3}{V}$ Equation of water sorption (Sideridou et al., 2003)

$WS = \frac{m_1 - m_3}{V}$ Equation of water solubility (Sideridou et al., 2003)

Statistical analysis:

The Statistical Package for the Social Sciences (SPSS, version 22; Chicago, IL, USA) was used for data description, analysis, and presentation. The groups were compared using a one-way analysis of variance (ANOVA) and then Tukey's post hoc test, with a significance threshold of $P \leq 0.05$.

Results:

Scanning electron microscopy:

SEM cross-sectional analysis of the fractured PMMA specimens at 1000× magnification demonstrated a uniform and smooth resin matrix in the control group (Fig. 7A). In contrast, the Ce/CS nanoparticles were uniformly dispersed within the resin matrix of the specimens from group A (Fig. 7B). Conversely, the filler particles in group B were erratically distributed, resulting in aggregated clusters within the matrix (Fig. 7C).

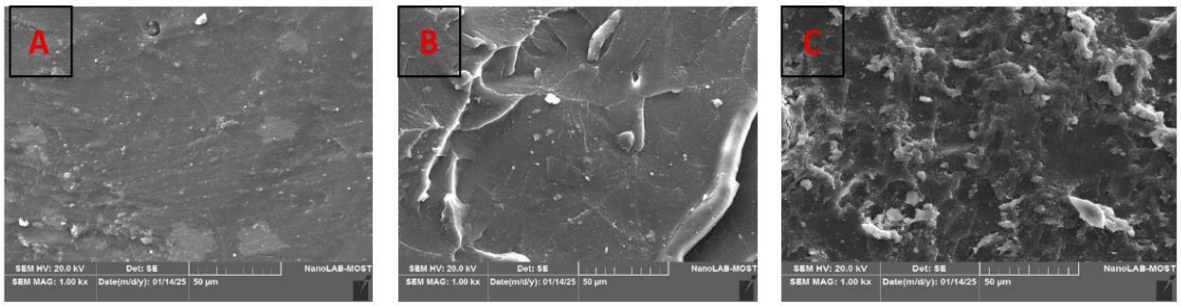

Figure 7: Control (A), 1% by vol. (B), 5% by vol. group (C) specimen under the scanning electron microscope at a magnification scale of 50µm.

FTIR, or Fourier Transform Infrared Spectroscopy:

The FTIR spectrum analysis demonstrates the chemical interaction between PMMA and cerium oxide/chitosan nanofiller powder, which implies the formation of new functional groups (Fig. 8A, 8B, and 8C). In terms of chemistry, these interactions indicate a reaction between the acrylic resin and the cerium oxide/chitosan nanofiller, as demonstrated by the discernible changes in the FTIR spectra.

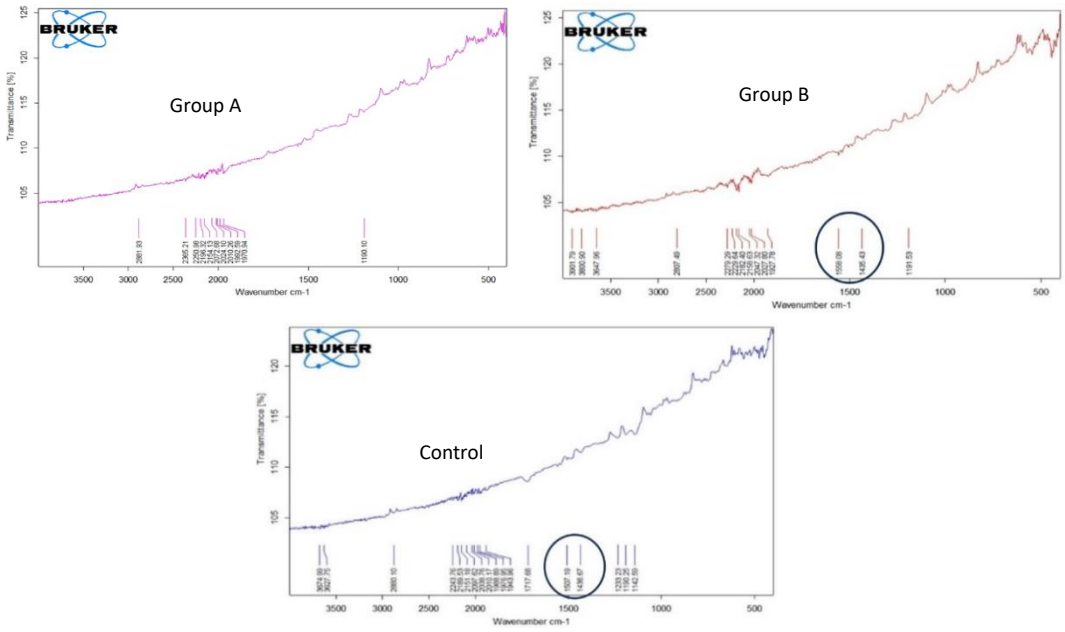

Figure 8. FTIR graph for the control and experimental group

Surface roughness statistics:

Descriptive data from the study indicates that Group B (5% CS/Ce) had the highest average value (0.9204µm). Next, Group A (1% CS/Ce) had an average value of 0.9087µm. The control group had the lowest mean value, 0.8954µm. A one-way ANOVA showed non-significant difference across tested groups (P value > 0.05), as indicated in Table 2.

Post hoc analysis using Tukey's test indicated non-significant differences between the experimental groups A and B compared to the control group and between groups A and B.

Table 2: Single-way ANOVA regarding Surface roughness testing.

	Sum of squares	df	Mean square	F	P values
Between the Groups	0.00	2	0.00	0.01	0.98
Within the Groups	3.60	27	0.13		
Totals	3.60	29			

Water sorption statistics:

Descriptive analysis shows that the control group had the highest mean reading (0.41 mg/cm²), followed by Group A (1 wt.% CS/Ce) (0.34 mg/cm²) and Group B (0.29 mg/cm²). A one-way ANOVA showed highly significant difference across tested groups (P value < 0.05), as indicated in Table 3.

Table 3: One-way of ANOVA test regarding water sorption testing.

	Sum squares	df	Mean square	F	P value
Between Groups	0.07	2	0.03	10.85	0.00
Within Groups	0.08	27	0.00		
Total	0.15	29			

Tukey's HSD post hoc comparisons showed a significant difference between the control and group B (P < 0.05). Significant statistical difference (P < 0.05) was discovered between the control and group A. However, the A and B groups had no significant difference (P > 0.05).

Solubility test statistics:

Descriptive analysis shows that the control group had the highest mean value (0.45 mg/cm2), followed by 1% CS/Ce (Group A) at 0.25 mg/cm2 and 5% (Group B) at 0.22 mg/cm2. A one-way ANOVA showed highly significant difference across tested groups (P value < 0.05), as indicated in Table 4.

Table 4: One-way ANOVA regarding solubility testing.

	Sum squares	df	Mean squares	F	P values
Between Groups	0.313	2	0.156	40.463	0.00
Within Groups	0.104	27	0.004		
Total	0..417	29			

The Tukey's HSD test showed significant difference ($P < 0.05$) was found between the experimental and control groups. when utilising 1% and 5% CS/Ce. The 1% and 5% CS/Ce groups had no significant difference ($P > 0.05$).

Discussion:

The use of a wide variety of nano additives has been suggested by a number of researchers as a means of enhancing the mechanical and physical characteristics of PMMA resin (Asar et al., 2013). In the current investigation, the null hypothesis was found to be incorrect. For the purpose of enhancing the mechanical and physical characteristics of the PMMA resin, a combination of nanosized cerium oxide and chitosan particles was added to the resin in a certain quantity, which was determined through pilot research. The filler particulates are considerably smaller than the PMMA resin granules, which enables them to occupy the interstitial spaces within the PMMA polymer matrix (Alnamel and Mudhaffer, 2014). This results in a heterogeneous composite that does not disrupt the segments of the polymer chain (Salman et al., 2017). Furthermore, the filler proportion must be kept to a minimum to guarantee that the PMMA resin is properly interlocked and that the structural integrity of the PMMA matrix is preserved (Asar et al., 2013).

Surface roughness:

Gaining a smooth surface with minimal surface scratches has always been an important objective in resin repair, since an increase in surface roughness detracts from the denture's appearance. The anti-plaque, stain-resistant properties of the acrylic resin are enhanced by its smooth, glossy surface (Arslan et al., 2025). As shown in this study there are non-significant change in response to variable concentrations of CS/Ce nanoparticles. The extremely small size of the CS/Ce nanoparticles may be the reason for this. Additionally, the surface roughness test is limited to the external surface of the nanocomposite, rather than its internal structure. Consequently, when a low percentage of nanoparticles is embedded in the acrylic matrix, only a small number of particles interact with the specimens' surface (Khairi and Naji, 2023). So that the addition of a lesser fraction of nanoparticles to the acrylic resin will have minimal impact on the surface irregularity. As a consequence, the CS/Ce nanoparticles will not significantly impact surface irregularity in comparison to the control group. Safi in 2011(Safi, 2011) determined that the roughness of PMMA did not considerably increase with the incorporation of ZrO2 nanoparticles.

Water sorption and solubility:

Water sorption is the term used to describe the quantity of water that is incorporated into the structure of a material and adsorbs onto its surface during the production or reconstruction process. The solubility of a material is the maximum concentration that can be dissolved in a particular solvent at specific temperatures (Tafeeq and Al-Khafaji, 2024). The results refuted the null hypothesis, as the solubility and water sorption of PMMA denture base resin were influenced by the addition of variable concentrations of CS/Ce nanoparticles. Polar carbonyl chemicals included in PMMA resins control the development of hydrogen bonds with water and impact the design of the network (Malacarne et al., 2006). The material's macromolecules can be

slightly separated by molecular water diffusing between them. These separations are then transmitted to the mass of PMMA, where they rest between the molecules of the polymer (Saini et al., 2016). Water is absorbed by polymers when unsaturated molecular bonds or unbalanced intermolecular interactions are present (Tuna et al., 2008). According to the current research, Ce/CS nanoparticles considerably decreased the heat-polymerized PMMA's sorption and water solubility. One important contributing aspect might be the presence of nanoscale CS/Ce particles inside the interstitial spaces of polymerised PMMA resin. Additionally, it could have drawn molecular resin, which eliminates the area for water sorption during the curing process and produces more intricate network architectures (Tekale et al., 2019).

There is also another explanation: perhaps the use of CS/Ce nanoparticles in place of hydrophilic resin results in less water sorption (Alwan and Alameer, 2015). The inclusion of CS/Ce nanoparticles enhanced the sorption and solubility of a resin material for a number of reasons, including the fact that metal oxide decreases the solubility of acrylic resin and that nanofillers are insoluble in water (Dehis et al., 2018). These findings are consistent with those of Alwan & Alameer (2015), who found that adding 3 weight percent titanium oxide nanocomposite to PMMA significantly decreased its water sorption and solubility values (Alwan and Alameer, 2015). The results also concur with Asar et al. (2013), who discovered that heating polymerised acrylic resin with 1% ZrO2 and 1% TiO2 microparticles decreased solubility and water sorption (Asar et al., 2013).

Conclusion:

1. Both 1 wt.% and 5 wt.% CS/Ce nanocomposites show non-significant differences in the surface roughness.
2. 1 wt.% and 5 wt.% CS/Ce nanocomposites significantly reduce water solubility and sorption the of heat-polymerized PMMA.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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