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Titanium dioxide nanoparticle-reinforced PMMA denture base resin: An *in vitro* study

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Abstract:

Heat-polymerized PMMA denture base resin is widely used but remains susceptible to fracture because of its limited mechanical strength. Therefore, it is of interest to evaluate the effect of 2% and 4% titanium dioxide nanoparticles on the tensile, impact and flexural strengths of heat-polymerized PMMA. Ninety specimens were fabricated and tested using standardized mechanical testing methods. PMMA reinforced with 2% TiO₂ nanoparticles showed significantly higher tensile, impact and flexural strengths than the control and 4% TiO₂ groups ($p < 0.001$). Thus, data shows that 2% TiO₂ nanoparticle reinforcement effectively enhances the mechanical performance of heat-polymerized PMMA denture base resin.

Keywords: Polymethyl methacrylate, titanium dioxide (TiO₂) nanoparticles, denture base resin, flexural strength, impact strength, tensile strength, heat-polymerized acrylic resin, nanocomposites

Background:

Edentulism continues to be a significant oral health concern worldwide, affecting mastication, speech, facial appearance and quality of life. Complete dentures remain one of the most widely used treatment options because of their affordability, accessibility and clinical effectiveness. Heat-polymerized polymethyl methacrylate (PMMA) has been the preferred denture base material for decades owing to its favorable esthetics, ease of processing, biocompatibility and cost-effectiveness [1–3]. Despite these advantages, PMMA exhibits limited flexural strength, low impact resistance and susceptibility to fatigue-related fracture, making denture fracture one of the most common complications in removable prosthodontics [4]. Various approaches, including copolymerization, cross-linking and reinforcement with fibers, metal insert and particulate fillers have been investigated to improve its mechanical properties [5–7]. Although these methods have shown varying degrees of success, limitations related to processing complexity, esthetics and inconsistent mechanical improvement remain [8]. Recent advances in nanotechnology have introduced titanium dioxide (TiO₂) nanoparticles as a promising reinforcing agent because of their biocompatibility, chemical stability and high surface area-to-volume ratio, which enhances interaction with the PMMA matrix [9]. However, the optimal concentration of TiO₂ nanoparticles required to maximize mechanical properties without compromising material integrity remains uncertain [10]. Therefore, it is of interest to report the effect of 2% and 4% titanium dioxide nanoparticle reinforcement on the tensile, impact and flexural strengths of heat-polymerized PMMA denture base resin.

Materials and methods:

This *in vitro* experimental study was conducted in the Department of Prosthodontics and Crown & Bridge, Seema Dental College and Hospital, Rishikesh, Uttarakhand, in collaboration with Pyrax Polymers, Roorkee, after institutional ethics committee approval. The study evaluated and compared the tensile, flexural and impact strengths of conventional heat-polymerized PMMA denture base resin and PMMA reinforced with titanium dioxide (TiO₂) nanoparticles at concentrations of 2% and 4%. Ethical was obtained from Institutional Ethics Committee of Seema Dental College and Hospital [Reference No.: SDCH/MDS-PROSTHO/IEC/12/2024] dated 14 February 2024. The sample size was calculated using the formula $n = 2(Z\alpha$

+ $Z\beta)^2 s^2 / d^2$, where $Z\alpha = 1.96$ (5% type I error), $Z\beta = 0.84$ (80% power) and $s = 10.2$ (pooled standard deviation). The calculated sample size was approximately 11 specimens per group; for standardization, 10 specimens were included in each subgroup. A total of 90 specimens were fabricated and divided into three groups: Group A (control, conventional heat-polymerized PMMA; $n = 30$), Group B (PMMA reinforced with 2% TiO₂; $n = 30$) and Group C (PMMA reinforced with 4% TiO₂; $n = 30$). Each group was further subdivided into tensile, impact and flexural strength subgroups ($n = 10$ each). The materials used were heat-polymerized PMMA denture base resin (Pyrax® Heat Cure Acrylic; Pyrax Polymers, India), titanium dioxide nanoparticles (30–80 nm; Techinstro, Nagpur, India), Type II dental stone, petroleum jelly and cold mould seal. Custom-made stainless-steel moulds were fabricated according to ADA Specification No. 12 and ISO 1567 standards, with specimen dimensions of $65 \times 10 \times 3$ mm for impact testing, $60 \times 12 \times 4$ mm for tensile testing and $60 \times 10 \times 3$ mm for flexural testing. For the control group, PMMA polymer and monomer were mixed per the manufacturer's instructions and processed using the conventional compression-moulding technique. For the experimental groups, TiO₂ nanoparticles were incorporated into the methyl methacrylate monomer at 2% and 4% concentrations and the nanoparticle-monomer mixture was probe-sonicated for 15 minutes to ensure uniform dispersion. The modified monomer was mixed with PMMA powder in a 3:1 polymer-to-monomer ratio by volume until the dough stage. The material was packed into the mould cavities, trial packed and subjected to final closure under hydraulic pressure. Polymerization was performed in an acrylizer using a long curing cycle at 71°C (160°F) for 90 minutes. After bench cooling, specimens were retrieved, finished and polished using standardized procedures.

Outcome measures:

The three primary outcomes were flexural strength, impact strength and tensile strength. Flexural strength was evaluated by a three-point bending test on a universal testing machine (Hounsfield Test Equipment Ltd., UK), with load applied at the midpoint at a crosshead speed of 1 mm/min until fracture and calculated as $FS = 3PL/2bd^2$. Impact strength was measured on a Charpy impact testing machine (CIT-21-09437) by recording the energy absorbed at fracture and calculated as $IS = E/A$. Tensile strength was measured on a universal testing machine fitted with a 200-kg load cell at a crosshead speed of 1 mm/min until

fracture and calculated as $TS = P/A$. All measurements were recorded by a single calibrated operator.

Statistical analysis:

Data were analysed using SPSS version 21.0 (IBM Corp., Armonk, NY, USA). Descriptive statistics, including mean and standard deviation, were calculated for each group. One-way analysis of variance (ANOVA) was used to compare mean values among the study groups and Tukey's post-hoc test was applied for multiple pairwise comparisons. The level of statistical significance was set at $p < 0.05$.

Results:

The mean tensile, impact and flexural strength values of the three study groups are summarized in **Table 1** and the post hoc pairwise comparisons are presented in **Table 2**. Across all three mechanical properties, the 2% TiO₂ group (Group B) recorded the highest mean values and the 4% TiO₂ group (Group C) the lowest, with the control group (Group A) intermediate. The

highest mean tensile strength was observed in the 2% TiO₂ group (58.89 ± 0.99 MPa) and the lowest in the 4% TiO₂ group (52.86 ± 1.06 MPa) ($F = 65.556$, $p < 0.001$). A similar pattern was seen for impact strength, which was greatest in the 2% TiO₂ group (9.83 ± 0.20 kJ/m²) and least in the 4% TiO₂ group (6.97 ± 0.16 kJ/m²) ($F = 605.587$, $p < 0.001$) and for flexural strength (95.71 ± 0.90 MPa versus 81.86 ± 0.81 MPa; $F = 650.124$, $p < 0.001$). One-way ANOVA revealed statistically significant differences among the groups for all tested properties ($p < 0.001$). Post-hoc comparison showed that the 2% TiO₂ group differed significantly from both the control and the 4% TiO₂ group for every property ($p < 0.001$), whereas the difference between the control and 4% TiO₂ group was significant for impact strength ($p < 0.001$) but not for tensile strength ($p = 0.455$). Overall, reinforcement with 2% TiO₂ nanoparticles significantly improved the tensile, impact and flexural strengths of heat-polymerized PMMA, while increasing the concentration to 4% reduced them below control values.

Table 1: Comparative evaluation of tensile, impact and flexural strength among the study groups (mean $\pm$ SD)

Mechanical property	Control (A)	2% TiO ₂ (B)	4% TiO ₂ (C)	F	P
Tensile strength (MPa)	53.56 ± 1.69	58.89 ± 0.99	52.86 ± 1.06	65.556	<0.001*
Impact strength (kJ/m ²)	8.42 ± 0.19	9.83 ± 0.20	6.97 ± 0.16	605.59	<0.001*
Flexural strength (MPa)	88.21 ± 0.87	95.71 ± 0.90	81.86 ± 0.81	650.12	<0.001*

Each subgroup $n = 10$. One-way ANOVA; * = statistically significant ($p < 0.05$).

Table 2: Post-hoc (Tukey) pairwise comparison of tensile and impact strength

Pairwise comparison	Tensile strength (P)	Impact strength (P)
Control (A) vs 2% TiO ₂ (B)	<0.001*	<0.001*
Control (A) vs 4% TiO ₂ (C)	0.455 (NS)	<0.001*
2% TiO ₂ (B) vs 4% TiO ₂ (C)	<0.001*	<0.001*

* = statistically significant ($p < 0.05$); NS = not significant.

Discussion:

The present study investigated the influence of titanium dioxide (TiO₂) nanoparticle reinforcement on the mechanical properties of heat-polymerized PMMA denture base resin. Incorporation of TiO₂ significantly affected the tensile, impact and flexural strengths of the material: specimens reinforced with 2% TiO₂ exhibited the highest values for all tested properties, whereas performance declined when the concentration was increased to 4%. These findings indicate that the reinforcing effect of TiO₂ is concentration dependent and that an optimum level of incorporation is necessary to achieve maximum enhancement of PMMA [11]. The results agree with previous investigations reporting improvements in the mechanical properties of PMMA following nanoparticle reinforcement, in which appropriate concentrations of reinforcing nanoparticles improved the strength and fracture resistance of denture base resins by enhancing their ability to withstand functional stresses [6, 12]. Comparable concentration dependent behaviour and the negative effect of higher filler loading have been described for TiO₂ and other metal-oxide nanoparticles added to acrylic resins [12]. The superior performance of the 2% TiO₂ group may be attributed to improved interaction between well-dispersed nanoparticles and the PMMA matrix, which facilitates more effective stress distribution and increases resistance to crack initiation and propagation [13, 14]. In contrast, the reduction

observed at 4% may be associated with particle aggregation at higher filler levels, which creates localized areas of weakness within the resin matrix and reduces the effectiveness of reinforcement. Differences among studies may also relate to variations in nanoparticle size, concentration, mixing procedures, polymerization methods and testing conditions [15, 16]. Clinically, fracture of acrylic dentures remains a common complication that leads to patient discomfort, treatment delays and additional cost. The improved tensile, impact and flexural strengths of the 2% TiO₂-reinforced PMMA suggest that such reinforcement may enhance the durability and longevity of complete dentures, particularly in patients prone to fracture because of high occlusal loads, parafunctional habits, unfavourable ridge conditions, or accidental dropping of the prosthesis. TiO₂ nanoparticles are also of interest because, in addition to reinforcement, they may confer antimicrobial and favourable surface characteristics on PMMA [17, 18]. Nevertheless, caution is warranted when extrapolating laboratory findings to clinical situations, as the oral environment presents numerous variables that cannot be fully reproduced *in vitro*.

Conclusion:

TiO₂ nanoparticles significantly affected the mechanical properties of heat-polymerized PMMA denture base resin. The

2% TiO₂ group showed the highest tensile, impact and flexural strengths, while 4% TiO₂ reduced these properties. Thus, 2% TiO₂ is a promising concentration for reinforcing PMMA denture base resin.

Limitations:

This study was conducted under controlled laboratory conditions and therefore could not replicate the complex biological, mechanical and environmental factors of the oral cavity, such as thermal cycling, moisture absorption, fatigue loading, pH fluctuation and long-term ageing. Only two nanoparticle concentrations were evaluated and scanning electron microscopic assessment of nanoparticle distribution was not performed; consequently, the proposed mechanisms of dispersion and agglomeration are based on previously reported evidence. Other important properties, including surface roughness, colour stability, water sorption and biocompatibility, were beyond the scope of this investigation. Larger studies incorporating a broader range of concentrations, simulated oral conditions and long-term ageing are needed to confirm these findings.

Conflict of interest:

The authors declare no conflict of interest.

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