

## An in-vitro study to compare the flexural strength, flexural modulus, and impact strength of heat-cure PMMA, heat-cure PMMA with reinforcement, polyamide, and polyamide with nano-additive

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### ABSTRACT

**Introduction:** All denture base materials require sufficient flexural and impact strength to withstand functional and para-functional forces. Conventional PMMA offers high flexural strength but limited impact resistance, whereas polyamide provides flexibility and higher impact resistance but lower flexural strength. Reinforcement with aluminium oxide micro-particles and nanoparticles has been suggested to improve mechanical properties. This study aimed to evaluate the effect of aluminium oxide microparticles and nanoparticles on the flexural and impact strength of PMMA and polyamide denture base materials.

**Methods:** Four groups of denture base materials were tested: heat-cure PMMA (1A–1B), PMMA reinforced with 2.5% aluminium oxide microparticles (2A–2B), polyamide (3A–3B), and polyamide with 0.5% aluminium oxide nanoparticles (4A–4B). Ten specimens per group were fabricated for flexural and impact strength testing according to ADA No. 12 and ASTM D-256 specifications, respectively. Flexural testing was performed using an Instron Universal Testing Machine, and impact strength was measured with a CEAST Izod impact tester. Data were analyzed using one-way ANOVA with post hoc multiple comparisons;  $p < 0.05$  was considered statistically significant.

**Results:** Heat-cure PMMA (1A) exhibited the highest flexural strength ( $91.69 \pm 13.66$  MPa), which was significantly greater than that of reinforced PMMA and polyamide groups ( $p < 0.001$ ). PMMA reinforced with aluminium oxide microparticles (2A) showed improved flexural strength compared to unmodified PMMA. Polyamide groups (3A, 4A) exhibited higher impact strength than PMMA groups, with no significant difference between polyamide with and without nano-additives.

**Conclusion:** Aluminium oxide microparticle reinforcement improved the flexural strength of PMMA, whereas aluminium oxide nanoparticles in polyamide did not significantly enhance flexural properties. PMMA is preferable when high flexural resistance is required, while polyamide offers superior impact resistance and flexibility. Clinicians should select denture base materials according to functional requirements and esthetic needs.

**Key words:** Additives, Denture, PMMA, Polyamide, Reinforcement

*Conflict of Interest: None*

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### INTRODUCTION

Poly(methyl methacrylate) (PMMA) was first introduced in the 1930s and has remained the primary material for denture bases due to its ease of manipulation, low cost, satisfactory aesthetics, and acceptable biocompatibility<sup>1,2</sup>. For decades, PMMA has served as the benchmark

for evaluating newer denture base materials. However, its major limitations—particularly low impact resistance, limited flexural strength, and susceptibility to fatigue and midline fractures—have been consistently reported in clinical practice<sup>3,4</sup>. These shortcomings often necessitate repeated repairs or remakes, causing inconvenience for patients and clinicians alike.

To address these issues, polyamide (nylon) resins were introduced as flexible alternatives. Their flexibility, resistance to impact fracture, and ability to engage undercut areas without metal clasps make them useful in specific clinical situations.<sup>5,6</sup> Nevertheless, polyamide resins also have drawbacks, including increased water sorption, susceptibility to discoloration, microbial colonization, and difficulties in relining or rebasing, which limit their widespread adoption.<sup>5,7</sup>

Since neither PMMA nor polyamide fully meets the mechanical and biological requirements for denture base applications, there has been growing interest in reinforcing these materials. Strategies have included the incorporation of glass fibers, polyethylene fibers, carbon nanotubes, zirconia, and other nanofillers to enhance flexural strength, impact resistance, wear resistance, and dimensional stability.<sup>8-11</sup> Such modifications aim to overcome inherent material weaknesses while preserving desirable characteristics such as aesthetics and biocompatibility. This study aimed to evaluate and compare the effects of reinforcement on the mechanical properties of PMMA and polyamide denture base materials. Research hypothesis was reinforcement of denture base resins with aluminium oxide ( $\text{Al}_2\text{O}_3$ ) additives will significantly improve their mechanical properties compared to unreinforced resins.

## METHODS

Four materials coded 1A-1B, 2A-2B, 3A-3B, and 4A-4B were tested for flexural and impact

strength. Group 1 consisted of Heat-cure PMMA; Group 2 consisted of Heat-cure PMMA with aluminium oxide reinforcement; Group 3 consisted of Polyamide; Group 4 consisted of Polyamide with nano-additives. Sub-group A and B in each group are for flexural test and impact test, respectively. Ten specimens were prepared for flexural strength testing and ten for impact strength for each material.<sup>10</sup> The parent materials have been shown in Table 1. The coding along with the tests are described in Table 2.

Aluminium oxide particles (3  $\mu\text{m}$ ) and aluminium oxide nanoparticles (50 nm) were selected as reinforcement and nano-additive, respectively, based on their proven ability to enhance the mechanical properties of denture base resins.<sup>10,11</sup> The distinction in particle sizes is important, as micron-sized fillers primarily contribute to improvements in bulk strength and stiffness, whereas nanoparticles, due to their extremely high surface area-to-volume ratio, act at the molecular level to improve interfacial bonding, toughness, and resistance to crack propagation.<sup>12,13</sup>

The high-precision HL-stainless steel dies were fabricated at Achiever Tooling Solutions, Delhi, India, using a Computer Numerical Control (CNC) machine. The dies, as shown in Figure 1, were standardized according to ADA specification No. 12 (65 mm  $\times$  10 mm  $\times$  3 mm) for flexural strength testing and ASTM specification D-256 (80 mm  $\times$  12.7 mm  $\times$  3.17 mm) for impact strength testing.<sup>14,15</sup>

For the experimental design, the specimens were divided into groups:

- **Control groups:** Unreinforced PMMA and unreinforced Polyamide.(Groups 1A,2A,3A,4A)
- **Reinforced group:** PMMA + aluminium oxide microparticles (3  $\mu\text{m}$ ) (Groups 1B,2B)

- **Nano-additive group:** Polyamide + aluminum oxide nanoparticles (50 nm). (Groups 3B,4B)

The aluminium oxide microparticles (3  $\mu\text{m}$ ; Sigma-Aldrich, St. Louis, USA) and aluminium oxide nanoparticles (50 nm; Nanostructured & Amorphous Materials Inc., Houston, USA) were incorporated at predetermined concentrations. The term nano-additive here refers to the submicron reinforcement strategy, in which nanoscale particles are dispersed within the polymer matrix to improve physical and mechanical properties without adversely affecting handling or aesthetics.<sup>16</sup>

Group 1A–2A and 1B–2B specimens were compression moulded in No. 7 and No. 9 Varsity flasks, respectively, as shown in Figure 2. The metal dies were thinly coated with petroleum jelly and placed on the unset vacuum-mixed Kaldent (type II dental plaster, Kalabhai) to expose only one die surface. Wax rolls of 3 mm diameter and 6 mm length were also quickly aligned towards one side of the dies in the unset plaster for easy retrieval of the dies after processing. After separating the media, another plaster mix was prepared and poured into the scope of the Varsity flask. The flask was clamped, and the plaster was left to set for 1 hour. Then, the Varsity flask was opened from the middle, and the wax was removed. This helped in engaging instruments for the removal of the metal dies. The space created by the wax was filled with alginate, and the excess was carefully incised with a BP blade.

For Groups 3A–3B and 4A–4B, unique flasks for injection moulding were used, as shown in Figure 3. Gypsum was vacuum-mixed and poured into the lower half of the flask. Wax sheets were rolled into cylindrical pieces to prepare channels. The over-extended edges of Groups 1A and 1B were finished with 300-grit sandpaper. Polishing was performed with

pumice slurry. Ten samples each for Groups 2A and 2B were fabricated in the same manner as described, except for the reinforcement of 50  $\mu\text{m}$  aluminium oxide powder particles, which were obtained from Nanolabs, India.

The cylinder was prepared for Group 3A–3B, and the furnace was set to 288 °C. The heating cylinder was kept in the slot until the preset temperature was achieved. In the meantime, the flasks were opened and heated to 65 °C. The heating cylinder was then removed from the furnace, and the Valplast cartridge was inserted. This was kept in the furnace for 16 minutes to allow the granules to melt. The material was then injected into the flask at a pressure of 5 bars for 5 minutes and released. Bench cooling of the flask was carried out until it attained room temperature. The samples were retrieved, grossly trimmed with diamond discs and acrylic burs, and finished with smooth blue rubber wheels after the specimens were separated from the sprue with diamond discs. Ten samples were prepared in this way for Groups 3A and 3B.

For Groups 4A and 4B, the mean of five Valplast cartridges was weighed separately, and empty cartridges were also weighed. 0.5% of the difference in the means of the weights was calculated as the amount of aluminium oxide nano-additive required in each cartridge. The cover of the Valplast cartridge was removed and placed on the vibrator. Nano-particle powders were gradually added, and the cover of the cartridge was closed by tapping. The cartridge was shaken manually for a minute to ensure uniform distribution of nanoparticles in the Valplast granules. The processing of Valplast was then performed as described for Groups 3A and 3B.

All the finished samples (Figure 4) were immersed in distilled water in labelled jars and incubated at 37 °C for 7 days to simulate intra-oral conditions, as shown in Figure 5.

### Testing for Flexural Strength

**Group 1A and 2A:** The sample was loaded onto the holder of the Instron Universal Testing machine 3382 (Figure 6) at a cross-head speed of 1.28mm/ minute and a span length of 48 mm. The calculations were done according to the Annual Book of ASTM Standards 2005, which describes the specifications for plastics D256 to D3159.<sup>13</sup> The machine was connected to a computer that gave us flexural strength and modulus readings.

**Group 3A and 4A:** The samples were loaded onto the Instron Machine. Nylon samples, being flexible materials, deflected beyond the machine's capacity and did not fracture. Therefore, the maximum load (F) was noted once the load-deflection curve became constant. For materials that deformed significantly but did not break, flexural yield strength is the stress at which material strain changes from elastic deformation to plastic deformation, causing it to deform permanently.<sup>10</sup>

### Testing for Impact Strength

The samples were mounted on the Notch Vis (Ceast) Machine to a depth of 2.5mm to create a V-shaped notch.<sup>10</sup> The width of the sample after notching becomes 10.2 mm (Figure 7, left). The Izod impact test component of the Resil Impactor Junior Impactometer, CEAST, Italy, with 0.01 J accuracy, was used. The arrow in the diagram shows the point and direction of the impact that is to be applied with the help of the pendulum (Figure 7, right). Notches represent stress concentration areas, as seen in the frenal areas of denture bases.<sup>10</sup> After the notches were made, the samples were immersed in distilled water at room temperature for 24 hours to release the stresses created within the sample during the preparation of the notches. The samples were vertically mounted, and a pendulum of 2J was released from them. ASTM impact energy is expressed in J/m or ft-lb/in. The impact strength was calculated by

dividing the impact energy(E) in J or ft-lb by the thickness of the specimen. The thickness of the specimen after notching was 10.2mm. The calculation for impact strength was E/10.2. The impact strength of the samples was noted for further comparison. The statistical analysis was conducted using IBM SPSS 24.0 Statistics, South Asia Private Limited.

## RESULTS

In the present case, four groups' means were to be compared, and multiple pair-wise comparisons were also required; therefore, a one-way analysis of variance was done, followed by post hoc multiple pair-wise comparisons. The cut-off 'p' value is taken as < 0.05 for the significance difference test. The comparison of flexural strength (in MPa) for 4 groups of denture-base material is presented in Graph 1. The mean flexural strength of PMMA was 91.69 MPa  $\pm$  13.66, which is the highest. The Analysis of Variance (ANOVA) revealed a significant difference in the mean values of flexural strengths of 4 groups of materials ( $p=0.000$ ).

Graph 2 compares the entire distribution of flexural modulus across the four groups of denture-base materials. This revealed the superiority of PMMA over the remaining three groups of denture-based materials. Even though polyamide has the lowest mean value, the difference between polyamide and polyamide with nano-additive is not very distinct when we look at their range of distribution, median and inter-quartile ranges in the box plot.

Graph 3 depicts the median with interquartile ranges and the range of the entire distribution with the box plot. This revealed the superiority of Polyamide over the remaining 3 groups of denture-base materials. Even though the polyamide with reinforcement has a lower mean value, the difference between polyamide and polyamide with nano-additive is not

very distinct when we look at their range of distribution, median and inter-quartile ranges in the box plot. Similarly, the difference between

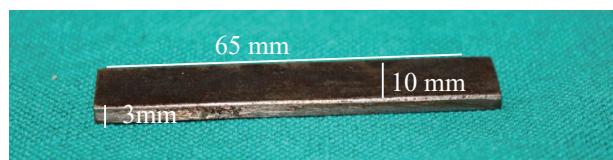
PMMA and PMMA reinforcement could be more distinguishable.

**Table 1:** Parent Materials for the Study

Composition	Trade name	Manufacturer	
Heat-cure Poly Methyl Methacrylate	Coltene Heat-cure Acrylic	Coltene Whaledent, Mumbai, India	Batch no: 170201
Polyamide (Nylon 6-6)	Valplast	Valplast International Corporation, USA	CAT: 21102

**TABLE 2** Group of materials and their codes

MATERIAL	TEST	Group
Heat-cure PMMA	Flexural strength	1A
Heat-cure PMMA	Impact strength	1B
Heat-cure PMMA with reinforcement	Flexural strength	2A
Heat-cure PMMA with reinforcement	Impact strength	2B
Polyamide	Flexural Strength	3A
Polyamide	Impact Strength	3B
Polyamide with nano-additive	Flexural strength	4A
Polyamide with nano-additive	Impact strength	4B



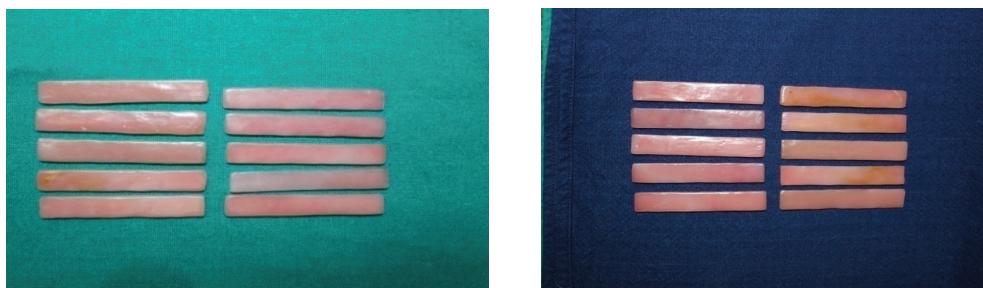
*Figure 1:* HL-13 stainless steel dies



*Figure 2:* Flask with metal dies embedded in plaster for Group 1A, 1B, 2A, 2B



*Figure 3:* Metal dies embedded in plaster with wax sprues for Group 3A, 3B, 4A, 4B



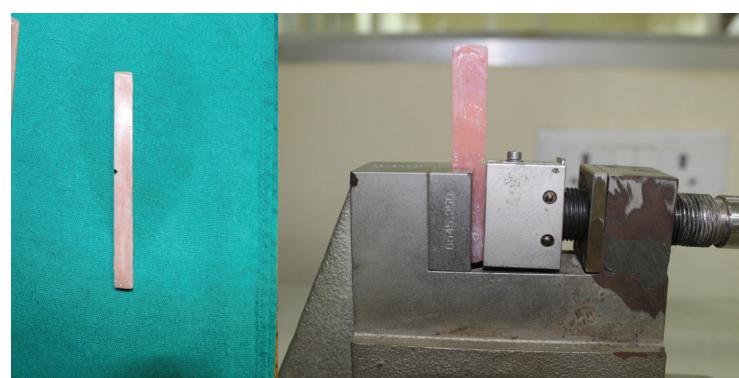
**Figure 4:** Samples After Finishing



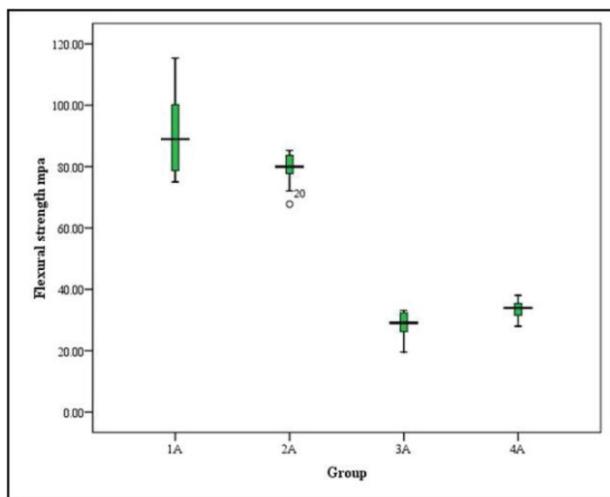
**Figure 5:** Incubation of samples at 37° Celsius



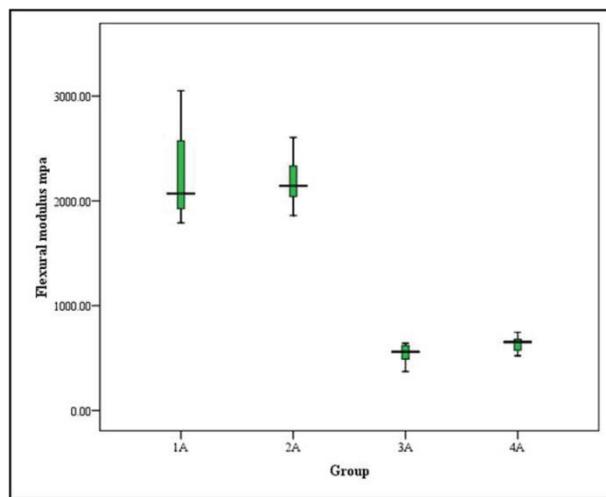
**Figure 6:** A prepared sample under flexural load



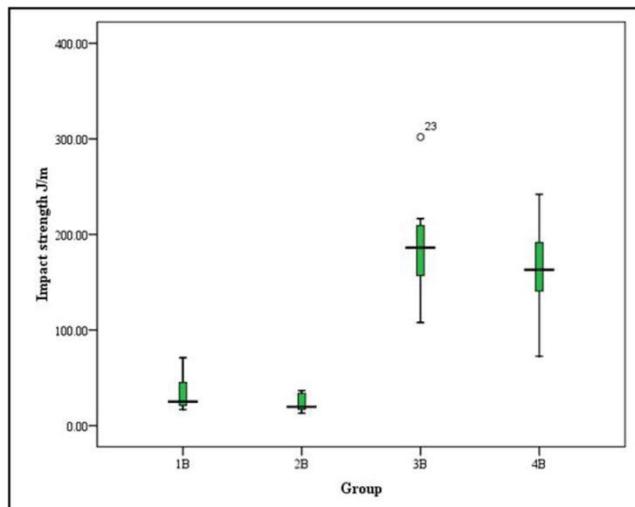
**Figure 7:** Sample mounted for testing with direction of impact



**Graph 1:** Box plot of Flexural strength (MPa) for different groups of denture-base materials



**Graph 2:** Box plot of Flexural modulus (MPa) for different groups of denture-base materials



**Graph 3:** Box plot of Impact strength (J/m) for different groups of denture-base materials

## DISCUSSION

This experiment compared the flexural and impact properties of four denture base materials: conventional heat-cure PMMA (Groups 1A–1B), PMMA reinforced with 2.5% aluminium oxide microparticles (Groups 2A–2B), polyamide (Groups 3A–3B), and polyamide with 0.5% aluminium oxide nanoparticles (Groups 4A–4B). Specimens with processing errors were excluded to ensure accuracy. Mechanical requirements for denture polymers are defined by ANSI/ADA Specification No. 12 (ISO 1567), with a minimum flexural strength of 65 MPa, flexural modulus of 2.0 GPa, and impact strength of 15 J/m.<sup>14,15</sup>

### Flexural Strength:

Group 1A (conventional PMMA) demonstrated the highest mean flexural strength ( $91.69 \pm 13.66$  MPa), exceeding the ISO minimum and confirming its suitability in situations requiring resistance to bending stresses.<sup>11,16</sup> Incorporation of 2.5% aluminium oxide microparticles in PMMA (Group 2A) further enhanced flexural strength compared to unmodified PMMA, consistent with Vojdani et al.<sup>16</sup> and Anne G et al.<sup>11</sup>, who reported improved flexural properties at specific filler concentrations. In contrast, polyamide groups (3A and 4A) exhibited lower flexural strength, with values falling below ISO minimum standards, reflecting the flexible

nature of nylon resins.<sup>7,10</sup> Addition of 0.5% aluminium oxide nanoparticles to polyamide (Group 4A) did not produce significant improvements in flexural strength, consistent with findings by Al-Noori et al.<sup>7</sup>, who observed minimal mechanical changes with low-concentration nanoparticle additions.

### Impact Strength:

Polyamide (Group 3B) showed the highest impact strength among all groups, demonstrating its ability to absorb sudden forces without fracturing.<sup>10</sup> Addition of aluminium oxide nanoparticles (Group 4B) maintained impact resistance comparable to unmodified polyamide, suggesting that the nanoparticle concentration did not compromise flexibility.<sup>7</sup> Conventional PMMA (Group 1B) exhibited the lowest impact strength, reflecting its brittle nature.<sup>11</sup> PMMA reinforced with microparticles (Group 2B) showed modest improvement in impact resistance, indicating that particulate reinforcement can slightly enhance fracture toughness but cannot match the inherent impact resilience of polyamide.<sup>16,17</sup>

### Comparison and Reinforcement Effect:

The study confirms the trade-off between flexural and impact properties. PMMA provides superior rigidity, whereas polyamide offers higher impact resistance and flexibility.<sup>10,11</sup> Reinforcement strategies enhanced specific properties: microparticles improved PMMA's flexural strength, and nanoparticles preserved polyamide's impact performance without detrimental effects.<sup>7,16,17</sup> The concentrations used in this study—2.5% microparticles in PMMA and 0.5% nanoparticles in polyamide—were chosen based on prior studies demonstrating mechanical improvements without compromising biocompatibility.<sup>7,17</sup> Pentapati et al.<sup>17</sup> reported further improvements in flexural strength and hardness with higher concentrations (15%) of aluminium oxide

in PMMA, indicating potential for future optimization.

Further research should evaluate the influence of different concentrations and types of reinforcements on both PMMA and polyamide. Long-term *in vivo* and *in vitro* studies simulating oral aging factors such as thermal cycling, fatigue loading, and enzymatic degradation are necessary. Comparative studies on additional mechanical properties like hardness, wear resistance, and dimensional stability will help refine material selection. Patient-centered clinical trials could provide stronger evidence for the functional and esthetic performance of reinforced denture base materials in daily use.

### LIMITATIONS

This study has certain limitations. Being an *in vitro* investigation, it may not fully replicate the complex intraoral environment. Factors such as thermal cycling, masticatory fatigue, enzymatic degradation, and long-term water sorption were not simulated. The evaluation was limited to a single concentration of aluminium oxide microparticles (2.5%) and nanoparticles (0.5%), which may not represent the full range of potential effects. Additionally, properties such as surface hardness, wear resistance, and dimensional stability were not assessed, and the influence of long-term aging on reinforced PMMA and polyamide remains unexplored. Only flexural and impact strength were tested, whereas other mechanical properties critical for clinical performance were not considered. Furthermore, manual mixing and specimen fabrication may have introduced minor variability, potentially affecting the consistency of results.

### CONCLUSION

Within the above limitations, following is the conclusion of the study :

- Heat-cure PMMA exhibited the highest

flexural strength, while polyamide demonstrated superior impact strength.

- b) Reinforcement of PMMA with aluminium oxide microparticles enhanced its flexural strength compared to unmodified PMMA.
- c) The incorporation of aluminium oxide nanoparticles into polyamide did not significantly improve flexural strength but maintained impact resistance similar to unmodified polyamide.
- d) These outcomes suggest that reinforced PMMA is suitable where high flexural resistance is required, while polyamide is advantageous when impact resistance and flexibility are of greater clinical importance.
- e) Reinforced PMMA may be recommended for patients prone to midline fractures of complete dentures or with high occlusal loads. Polyamide, with or without nano-additives, may be chosen where flexibility, shock absorption, and esthetics (such as tooth- or gingiva-colored clasps) are critical.

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