

**To evaluate the efficacy of addition of 1% titanium dioxide nanoparticles on the flexural strength of double layered acrylic resin denture base: An in vitro study and clinical report.**

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**Type of Publication:** Case Report

**Conflicts of Interest:** Nil

**Abstract**

**Introduction:** The effect of incorporating a thin layer of 1% Titanium dioxide modified acrylic resin to conventional acrylic resin during packing on the flexural strength of the resulting hybrid material has been uncertain.

**Purpose:** The purpose of this in vitro study was to develop a new technique by addition of 1% Titanium dioxide nanoparticles to improve the flexural strength of the processed hybrid heat cure acrylic resin denture base material.

**Methodology:** Thirty specimen were divided into three groups.

Group 1- Unmodified heat cure polymethylmethacrylate resin specimen.

Group 2- 1% Titanium dioxide modified heat cure polymethylmethacrylate resin specimen which were packed conventionally in one step.

Group 3- Double layered specimen which were packed in two steps, first with unmodified polymethylmethacrylate resin and then by adding a thin layer of 1% Titanium dioxide modified polymethylmethacrylate before the final packing.

**Results:** Flexural strength of Group 3 was higher-  $172.092 \pm 33.512$  as compared to Group 2-  $171.708 \pm 16.752$  and Group 1-  $140.172 \pm 24.340$ . ANOVA test was applied to compare the flexural strength among the groups. ANOVA showed statistically significant difference among the groups ( $p=0.014$ ).

**Conclusion:** The inclusion of 1% Titanium dioxide nanoparticles in the double layer technique improved the flexural strength, hence this technique can be used for the fabrication of dentures having antimicrobial properties.

**Keywords:** Denture Stomatitis (DS), Flexural strength (FS), Polymethyl methacrylate (PMMA), Titanium Dioxide nanoparticles ( $\text{TiO}_2$  NPs).

## Introduction

Polymethyl methacrylate (PMMA) is extensively used in the prosthetic rehabilitation of completely and partially edentulous patients owing to its inherent favourable features such as biocompatibility, satisfactory aesthetics, cost effectiveness and ease of use. However, inadequate mechanical properties and poor antimicrobial properties constitute its major drawbacks.[1] Heat polymerized acrylic resins are most commonly used denture base material. Shakir et al mentioned several causes for PMMA denture fractures and found that accidental fractures accounted for 56% of the analysed cases, poor denture fit 35%, heavy masticatory load 13.8%, fracture due to single complete denture opposed by natural teeth 12% and acrylic resin breakage due to prolonged period of usage 5%. Hence, mechanical properties such as flexural strength, elastic modulus and surface hardness of the denture base resin are pivotal for durability of the prostheses.[2] The flexural strength of acrylic resin also called as the modulus of rupture is the property that manifests every time the prosthesis is subjected to cyclic deformation. It represents the highest stress experienced

in this material at the time of rupture. A low ability to withstand such deformation under load force is the most important factor for the clinical incidence of premature fracture of the prosthesis. The minimum flexural strength value of standard dental base polymers is prescribed as 65 MPa by the ISO 1567 International Standard.[1-3] The surface topography and wettability of denture base material can affect microbial adhesion and biofilm formation contributing to the occurrence of Denture Stomatitis. Denture stomatitis occurs in 15 to 70% of the denture wearers and the main pathogen responsible is *Candida albicans* (*C. albicans*). The use of acrylic resin that is resistant to candida adhesion appears to be an effective way to avoid the development of DS. Moreover, hygienic denture materials (containing antifungal agents) could help maintain oral and systemic health in patients using acrylic resin dentures.[4] Different techniques are used to improve the mechanical properties and clinical usage of PMMA which include incorporation of a rubber phase, metal wires, metal oxides and fibres. Developments in the field of nanotechnology has led to the use of nanoparticles, nanotubes and nanofibers to reinforce acrylic resins. Nanoparticles are characterized by their small size, large specific surface area and strong interfacial interaction with organic polymers. Nanomaterials may also provide an antibacterial effect related to their large active surface area compared with their size and chemical reactivity. Commonly used nanoparticles include silver (Ag), aluminium oxide ( $\text{Al}_2\text{O}_3$ ), silicon dioxide ( $\text{SiO}_2$ ), zirconium dioxide ( $\text{ZrO}_2$ ) and titanium dioxide ( $\text{TiO}_2$ ).[1-4] Titanium dioxide nanoparticles have a large spectrum of activity against microorganisms including Gram-negative, Gram positive bacteria and fungi. Incorporation of  $\text{TiO}_2$  nanoparticle in denture base resin significantly reduces microbial adhesion. The integration

of TiO<sub>2</sub> Nanoparticles to PMMA reduces the porosity of the denture bases which reduces the growth of candida hyphae that is accountable for denture stomatitis.[5]Hence, the purpose of this study is to evaluate the efficacy of addition of a thin layer of titanium dioxide modified acrylic resin to conventional acrylic resin during packing on the flexural strength and compare it with the flexural strength of fully modified denture base resin and the flexural strength of unmodified denture base resin. The null hypothesis being tested is that there is no difference in the flexural strength between modified 2- layer, modified 1-layer and unmodified denture base resin.

### Materials and Methodology

The sample size was established using the G Power software version 3.0.1(Franz Faul Universitat, Kiel, Germany). Considering the effect size to be measured(f) at 60%, power of the study at 80% and the margin of error at 5%, the total sample size needed was 30. Three groups of 10 samples each (10 samples× 3 groups = 30 samples).

Thirty wax samples of dimensions 65mm×10mm×25mm were prepared. These wax samples were invested in a flask using two pour technique, the first pour being of Plaster of Paris (Gypsum type II) and the second pour of dental stone (following manufacturer's instructions). Once the second pour was set, the flasks were immersed in thermally controlled hot water bath for dewaxing. Following dewaxing separating media was applied in the mold space and the flasks were left to reach room temperature



Figure 1: Preparation of wax specimens measuring 65mm×10mm×25mm.



Figure 2: Wax samples invested in Plaster of Paris



Figure 3: Mold space after dewaxing procedure

**Group 1:** The control group received a full mix of unmodified polymethylmethacrylate resin. Denture base resin powder was mixed with the liquid according to the manufacturer's instruction and was packed into the mold space.



Figure 4: Conventional one step packing of unmodified PMMA for Group 1 .

**Group 2:** 1% TiO<sub>2</sub> (99.9% purity, 30-50nm size) was added to the acrylic resin powder and homogenized. The prepared polymer and monomer were mixed in a proportion of 5gm:3.5ml and the mixture was packed into the mold at dough stage in one step.

**Group 3:** Two mixes were prepared at an interval of fifteen seconds. The first mix was of unmodified polymethylmethacrylate resin and the second one was of 1% titanium dioxide modified acrylic resin. The first layer was packed against the cameo surface and covered with two layers of aluminium foil cut to the same dimension of the samples. A wet cellophane sheet was used as a separator between the stone and the acrylic resin during trial closure. The flask was opened, cellophane sheet, silver foil was removed and any excess acrylic resin was trimmed. Now, a thin layer of titanium dioxide modified acrylic resin was applied at the intaglio surface of the flask before reassembling.



Figure 5: Double layer packing technique for Group 3.

The final closure for all the groups was carried out by using the pneumatic press for five minutes followed by thirty minutes storage in the flask frame before polymerizing. The flasks were submerged in a thermally controlled polymerization unit at 165 degrees F for 90 mins followed by 30 minutes at boil. After polymerization, the samples were retrieved and finished and polished similar to the conventional method of denture polishing. The specimens were stored in distilled water for one week before testing.

A three-point flexural test [International Organization for Standardization (ISO)] was used to test the flexural strength of freshly retrieved samples from the distilled water by using a universal testing machine (Mecmesin Multi Test 10-i). The maximum load at fracture was recorded and flexural strength of each sample was calculated using the formula:

$$FS(Mpa) = \frac{3 WL}{2 bd^2}$$

where FS is flexural strength(MPa), W is maximum load at fracture(N), L is distance between supports (50mm) and b and d are specimen width (10mm) and thickness (2.5mm) respectively.

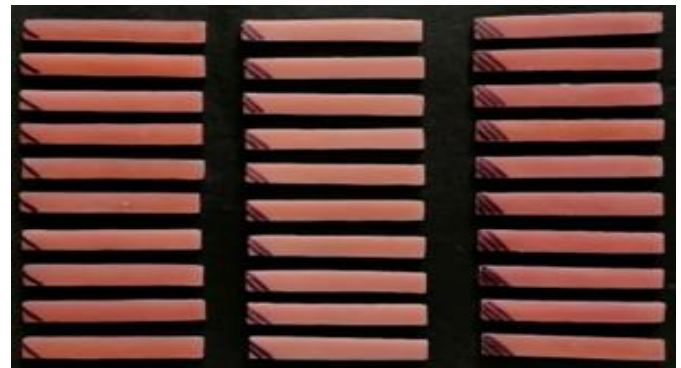


Figure 6: Acrylized samples

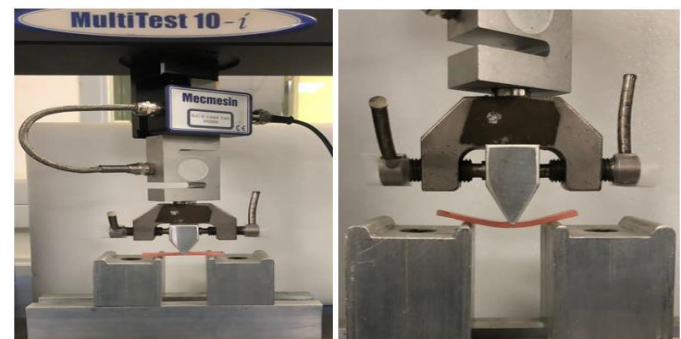


Figure 7: Three point flexural test using universal testing machine

## Results

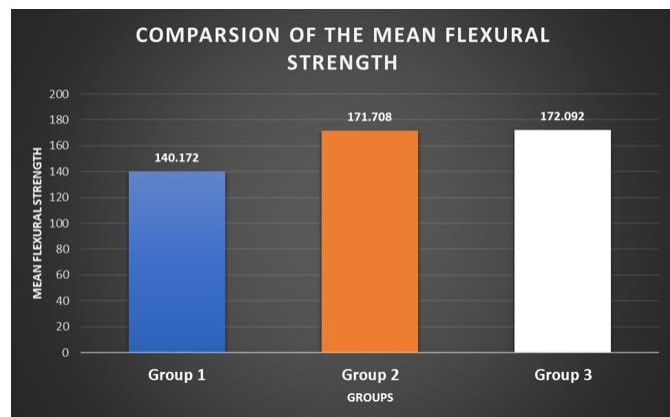
SPSS (Statistical Package For Social Sciences) version 20. (IBM SPASS statistics [IBM corp.released 2011]) was used to perform the statistical analysis. The values obtained were subjected to ANOVA test to compare the flexural strength among the groups with post hoc

Bonferroni test for inter group comparison and the level of significance is set at 5%. Flexural strength of Group 3 was higher-  $172.092 \pm 33.512$  as compared to Group 2-  $171.708 \pm 16.752$  and Group 1-  $140.172 \pm 24.340$ . ANOVA test was applied to compare the flexural strength among the groups. ANOVA showed statistically significant difference among the groups ( $p=0.014$ ).

| Group   | N  | Min    | Max    | Mean   | S.D   | F vaue | p value |
|---------|----|--------|--------|--------|-------|--------|---------|
| Group 1 | 10 | 93.00  | 177.0  | 140.17 | 24.34 | 5.044  | .014*   |
| Group 2 | 10 | 144.84 | 192.24 | 171.70 | 16.75 |        |         |
| Group 3 | 10 | 128.76 | 216.36 | 172.09 | 33.51 |        |         |

Table 1: Comparison of the mean flexural strength between the groups using independent sample t test.

Inter group comparison was done using post hoc Bonferroni test. Statistical significant difference with flexural strength was seen between Group 1 V/s Group 2 ( $p=0.033$ ) with mean difference of -31.53 and between Group 1 V/s group 3 ( $p=0.03$ ) with mean difference of -31.92 whereas there was no significant difference seen between Group 2 V/s Group 3 ( $p=1.00$ ).



Graph 1: Graph of mean flexural strength of all the groups.

|                     | Mean Difference | P value |
|---------------------|-----------------|---------|
| Group 1 V/s Group 2 | -31.53          | 0.033*  |
| Group 1 V/s Group 3 | -31.92          | 0.03*   |
| Group 2 V/s Group 3 | -0.384          | 1.00    |

Table 2: Inter group comparison of flexural strength using post hoc bonferroni test.

## Case Report

A 51 years old female patient reported to the Department of Prosthodontics, Vokkaligara Sangha Dental College and Hospital, Bengaluru with the chief complaint of missing teeth in her upper and lower arches since three months and wanted replacement for the same. The patient gave a history of undergoing extraction of her upper and lower teeth due to periodontal reasons.

The patient was diagnosed with completely edentulous, well formed, U shaped maxillary arch with anterior undercuts and completely edentulous, well formed, U shaped mandibular arch with lingual undercuts present.

In the treatment plan explained to the patient, the patient opted for conventional maxillary and mandibular complete dentures. Hence, for antimicrobial properties and enhanced flexural strength without compromising the aesthetics, dentures were fabricated incorporating 1%  $\text{TiO}_2$  using double layered technique.



Figure 8: Completely edentulous maxillary and mandibular arches

## Procedure

- Preliminary impressions were made in impression compound (Functional impression compound, Pinnacle) using non-perforated edentulous stock trays for both maxillary and mandibular arches.
- Primary casts were obtained (**Figure 9**). The undercuts were blocked with wax (Modelling wax, Hindustan) and wax spacer was adapted. Autopolymerizing acrylic resin (DPI RR Cold cure) was used to fabricate custom tray of uniform thickness on both maxillary and mandibular casts.

- The tray was adjusted in patient's mouth and low-fusing compound (DPT Pinnacle Tracing Stick) was used to perform border molding. Zinc oxide non eugenol cement (Prime Image Eugenol Free Impression Paste) was used for secondary impression for both the arches (**Figure 10**).
- The final impressions were poured in type III dental stone (**Figure 11**). Modelling wax (Modelling wax, Hindustan) was used to block undercuts. Autopolymerizing resin (DPI RR Cold cure) was used to make record bases and wax (Modelling wax, Hindustan) occlusal rims were made on the record bases. The maxillomandibular relationship was recorded and the mounted on the articulator (**Figure 12**).
- The maxillary and mandibular artificial teeth were arranged (**Figure 13**) and try in was carried out and the occlusion, aesthetic and phonetics were evaluated.
- Waxed and finished trial dentures were sealed to the cast. After flasking and dewaxing (**Figure 14,15**), separating media was applied. Two mixes were prepared at an interval of fifteen seconds. The first mix was of unmodified polymethylmethacrylate resin and the second one was of 1% titanium dioxide modified acrylic resin. The first layer was packed against the cameo surface (**Figure 16**) and covered with two layers of aluminium foil (**Figure 17**). A wet cellophane sheet was used as a separator between the stone and the acrylic resin during trial closure. The flask was opened, cellophane sheet, silver foil was removed and any excess acrylic resin was trimmed (**Figure 18**). Now, a thin layer of titanium dioxide modified acrylic resin was applied at the intaglio surface of the flask before reassembling (**Figure 19**),

- The final closure was carried out by using the pneumatic press for five minutes followed by thirty minutes storage in the flask frame before polymerizing. The flasks were submerged in a thermally controlled polymerization unit at 165 degrees F for 90 mins followed by 30 minutes at boil. After polymerization, the samples were retrieved and finished and polished similar to the conventional method of denture polishing (**Figure 20**).
- The finished prosthesis was inserted into the patient's mouth (**Figure 21**) and necessary adjustment was carried out. The patient was given training for placement of the prosthesis and post insertion instructions for maintenance were given.



Figure 9



Figure 10



Figure 11



Figure 12



Figure 13



Figure 14



Figure 15



Figure 16



Figure 17



Figure 18



Figure 19



Figure 20

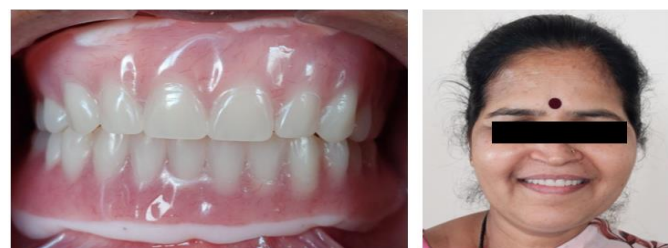


Figure 21

## Discussion

Based on the obtained data, the null hypothesis was rejected. The flexural strength of modified denture base resin was affected compared with the unmodified. A significant increase in flexural strength was noted in one and two layer TiO<sub>2</sub> modified denture base resin compared with the control. Flexural strength improvement of TiO<sub>2</sub> modified acrylic resin may be related to particle size, homogenous dispersion or improved bonding between TiO<sub>2</sub> nanoparticles and the resin matrix.

In the study by Shirkavand and Moslehifard <sup>[6]</sup>, TiO<sub>2</sub> nanoparticles in 0.5, 1 and 2wt% concentrations were added to polymethyl methacrylate acrylic resin. They showed that addition of 1wt% nanoparticles had the greatest efficacy for enhancement of tensile and impact strength. Thus, 1wt% TiO<sub>2</sub> nanoparticles was added to acrylic powder in the present study. Shirkavand and Moslehifard also demonstrated that addition of 1wt% TiO<sub>2</sub> nanoparticles reinforced the acrylic resin but adversely affected its colour. Sodagar et al<sup>[7]</sup> also showed that addition of TiO<sub>2</sub> nanoparticles improved the flexural strength of acrylic resin, which was in line with the findings of the present study.

Limitations of the present study include the in-vitro design that lacked complete oral simulation such as the absence of saliva, masticatory stress and irregular configuration of the actual denture base. Hence, based on the double layered technique using 1% TiO<sub>2</sub> a removable maxillary and mandibular complete denture was fabricated for a patient.

## Conclusion

Within the limitations of the study, it was concluded that:

1. The flexural strength was increased by the incorporation of 1 wt% Titanium dioxide nanoparticles.
2. Incorporation of Titanium dioxide nanoparticles in the one or two – layer denture bases increased the flexural strength and did not show statistical significant difference.
3. The two- layer denture bases containing antimicrobial agent such as the Titanium dioxide nanoparticles can prove to be a promising technique for the fabrication of dentures with antimicrobial properties.

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