

Evaluation of Impact Strength of Dental Acrylic Resins by Incorporation of TiO₂ Nanoparticles Using Two Different Processing Techniques

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ABSTRACT

Aim: The present study aims to evaluate the impact strength of PMMA incorporated with TiO₂ nanoparticles by two different processing techniques i.e. water bath and microwave processing techniques.

Materials and methods: A total of 80 samples made of PMMA were divided into four groups. Each group includes 20 samples with group I and II comprising of samples made of normal acrylic resin and acrylic resin reinforced with 1 wt% TiO₂ nanoparticles processed with conventional water bath technique respectively, groups III and IV include normal acrylic resin and acrylic resin reinforced with 1 wt% TiO₂ processed using microwave technique respectively. The specimens were tested for impact strength using IZOD pendulum impact tester. The impact energy values obtained were analyzed statistically.

Results: The results through one-way ANOVA showed a high mean impact strength with group IV samples (23.13) and lowest with respect to group I (19.42) with highest statistical significance ($p < 0.001$). A *post hoc* Tukey test intergroup analysis showed a statistically significant difference between group I and other groups, whereas there was no statistical significance associated with other intergroup comparisons.

Conclusion: PMMA with its current drawbacks in the physical and mechanical properties requires modifications to make it an ideal denture base material. The current investigation evaluates that a high mean impact strength with samples made of PMMA incorporated with TiO₂ nanoparticles processed by microwave technique was obtained when compared with normal acrylic resins processed by water bath technique.

Clinical significance: The methodology of current study can be used while processing of denture bases for patients to evaluate the effect of oral environment on inclusion of TiO₂ nanoparticles with microwave processing in a clinical setup. This could help in reducing the amount of fractures associated with heavy load masticatory stresses and improving the mechanical properties in denture bases.

Keywords: Acrylic denture base, Impact strength, Microwave, TiO₂ nanofillers.

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INTRODUCTION

A breakthrough in the field of prosthetics was the invention of poly(methyl methacrylate) (PMMA) and is considered as an ideal denture base material compared to many. PMMA is a routinely used material in the fabrication of removable partial and complete dentures as it possesses a wide range of beneficial properties like ease of handling, inexpensive fabrication, and stability in oral environment, lack of toxicity, color matching ability, biocompatibility and accurate fit. Despite the favorable properties, masticatory forces have a deforming effect on PMMA during function and it lacks particularly in terms of transverse and impact strength. So, any factor that increases the distortion of the denture base and alters the stress distribution may lead to denture fracture.^{1,2} Even though PMMA is an excellent denture base material, it has few disadvantages like low in strength resulting in denture fractures. Fracture may occur because of unsatisfactory impact strength, transverse strength, or fatigue resistance.³

However, due to its poorer physical and mechanical properties, PMMA structure is further modified by rubber copolymerization or by reinforcement with different types of fillers like metallic wires, fibers (glass, aramid, nylon, polyurethane and carbon)^{4,5} and use of metallic oxides to reduce the drawbacks and to improve the fracture resistance of PMMA denture based resins.^{6,7} Metal oxides such as aluminum oxide, zinc oxide, zirconium oxide and titanium

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dioxide (TiO₂) are used along with PMMA, among which TiO₂ is routinely used as they could improve the flexural strength, fracture toughness, hardness, thermal conductivity, antimicrobial property, impact strength and a substantial decrease in water sorption and solubility.⁸

Nanotechnology is a growing field which determines the future of science and technology. It is based on the idea of creating functional structures by controlling molecules on a structural basis and manipulating the matter at billionth of meters or nanometers.⁹ According to the definition of the National Nanotechnology Initiative, nanotechnology is the direct manipulation of materials at the nanoscale.¹⁰ This term defines a technology that enables almost complete control of the structure of matter at nanoscale dimensions. It will give us the ability to arrange atoms as we desire and subsequently to achieve effective, complete control of the structure of matter. These advances in nanotechnology provided suitable changes in dentistry from diagnosing the disease to varying treatment options.¹¹

The approaches in which nanotechnology can be used in dentistry can be on therapeutic basis, regeneration of dental tissues or by incorporating them in dental materials.¹² As mentioned earlier, the usage of these nanoparticles in PMMA denture base is by alteration and addition of TiO₂ filler size to improve the performance of the material. The nanomaterials present with variety of morphologies such as particles, flowers, cubes, rods, tubes etc.⁸

A number of processing techniques like compression molding, injection molding or fluid resin techniques are proposed in the fabrication of denture bases using acrylic resins. Variable dimensional changes tend to occur in PMMA, which is attributed to the type of processing technique.¹³ Recently, various studies were conducted using microwave processing, the principle of which depends on the effect of microwave energy on the monomer components promoting an uniform and immediate heating of the polymer mass, that activates the decomposition of benzoyl peroxide, and quickly yields free radicals for the polymerization process, which decreases in the same proportion as polymerization increases.¹⁴

Studies conducted on TiO₂ nanoparticles incorporation into denture base resins showed improved physical and mechanical properties. The other studies conducted on comparison of various processing techniques have shown improved mechanical properties of denture bases with microwave curing technique by decreasing the residual monomer content.¹⁵ Hence, considering the above said facts, the present study was conducted to evaluate the effect of TiO₂ nanoparticles on impact strength of dental acrylic resins using microwave processing and conventional water bath methods.

Table 1: Study groups

Group	Method	Sample size
Group I	Normal acrylic resin processed with conventional water bath technique	20
Group II	Acrylic resin reinforced with 1 wt% TiO ₂ nanoparticles processed with conventional water bath technique	20
Group III	Normal acrylic resin processed using microwave technique	20
Group IV	Acrylic resin reinforced with 1 wt% TiO ₂ processed using microwave technique	20

MATERIALS AND METHODS

In the present study, the concentrations of TiO₂ incorporated into PMMA was restricted to 1 wt%. A total of 80 samples divided into four groups with 20 samples each with dimensions of 60 mm × 12 mm × 3 mm were prepared as per ASTM (American Standards for Testing and Material). The sample size and methodology in each group are summarized in Table 1.

A total of 40 metal sample analogs with the above-mentioned dimensions were invested into flasks through two pour technique using dental plaster as the investing medium in conventional water bath technique (groups I and II). After the investing medium has reached its final set, the two portions of the flask were gently separated. The metal sample analogs were also lifted from investing medium along the beveled edges, thereby creating mold space into which acrylic resin was packed (Fig. 1A). For microwave oven processing (groups III and IV), separate microwavable flasks were used. The above mentioned procedure was repeated with microwavable flasks (Fig. 1B).

To pack acrylic resin into the mold space, a selected acrylic material was mixed according to manufacturer's recommended polymer: monomer ratio and packing was done. Then curing of samples in water bath technique was done in an acrylizer at 74°C for 8 hours with no terminal boiling. Following which, the flasks were allowed to cool to room temperature and removed from the water bath and bench, cooled for 30 minutes. Subsequently the flask was immersed under cold tap water for 15 minutes. The same procedure was repeated for microwavable flasks and cured at 540 watts for 10 minutes in microwave oven (Fig. 2).



Figs 1A and B: (A) Metal analogs invested in brass varsity flask; (B) Nonmetallic (microwavable) flasks



Fig. 2: Microwave oven



Fig. 3: Amalgamator

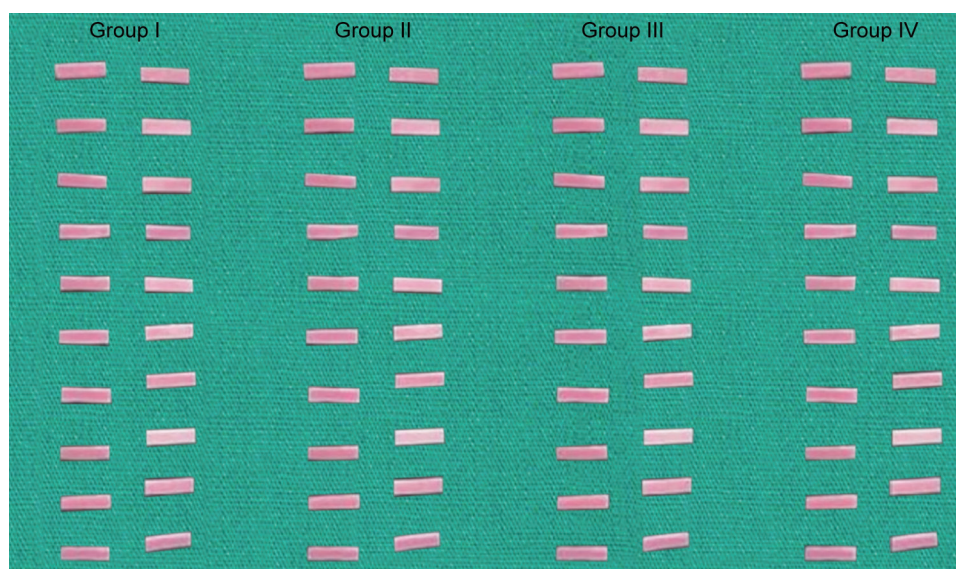


Fig. 4: Test samples from various groups

TiO₂ nanoparticles were incorporated into the polymer with a concentration of 1 wt% i.e., 1 g of nanoparticles to 99 g of polymer and mixed using an amalgamator (Fig. 3) to achieve a homogeneous mixture. Polymer and monomer were mixed according to manufacturer's recommendations (3:1 by volume).

After completing the process, the cured samples were retrieved from the flask by deflasking. The samples were trimmed of the excess resin flash using tungsten carbide burs and fine abrasive of grit size 120/240 microns without altering the specimen surfaces and dimensions. Samples from all the groups are depicted in Figure 4.

Specimens from all the four groups were tested for impact strength using a pendulum impact tester—IZOD (Tinius Olsen IZOD/Charpy Impact Tester Model IT504, USA) (Fig. 5). It conforms to ASTM standards D256 and D6110; ISO standards 179 and 180. It has a pendulum, a digital display and an anvil for supporting the specimen Model IT 504.

The fabricated samples were notched in the middle, with a notch cutter (Fig. 6) as in ISO 179:1993 to a depth of 0.2 mm leaving a residual depth of 2.8 mm beneath the notch. In this test, the sample piece was used as a cantilever i.e., the specimen was

clamped upright in the anvil with a v-notch at the level of clamp. The dimensions of the sample were entered in the display box. The pendulum was released after entering the data. The test piece was hit by pendulum which was aerodynamically controlled. It was allowed to fall freely from fixed height, to give a blow of 120 ft lb. energy. After fracturing the test piece, the height to which the pendulum rises was recorded by a friction pointer from which absorbed energy was read. The impact energy of the test specimen was displayed in J/m. The results obtained for each test specimen were analyzed statistically using SPSS version 22. Descriptive analysis for scale data, one-way ANOVA and independent *t* test for intergroup comparison are the statistical analysis done with a *p* value of <0.05 was considered significant and <0.01 as highly significant.

RESULTS

The present study was conducted to evaluate the impact strength of PMMA incorporated with TiO₂ nanoparticles by two different processing techniques. The test samples of different groups



Fig. 5: Izod impact tester



Fig. 6: Notch cutter

Table 2: Mean and SD of impact strength in various groups

Group	N	Minimum	Maximum	Mean	Std. deviation
Group I	20	17.000	27.398	19.42	2.47
Group II	20	19.200	27.398	22.53	2.06
Group III	20	19.300	27.000	21.73	1.92
Group IV	20	19.333	32.400	23.13	2.96

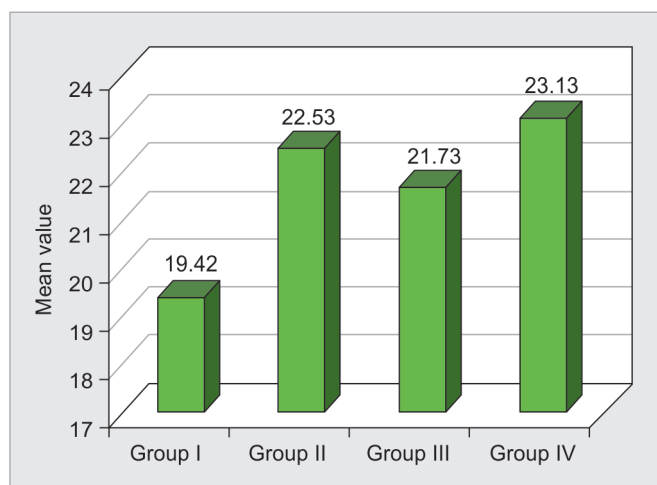


Fig. 7: Mean impact strengths between the four groups

mentioned in Table 1 were subjected to pendulum impact tester. The mean values and standard deviation obtained in various groups were summarized in Table 2 and Figure 7. The mean impact strength was highest in group IV with mean value of 23.13 and lowest in group I with 19.42. The results were analyzed statistically through

one-way ANOVA (Table 3), which showed a highly significant difference between the groups with a "*p*" value of 0.001.

The comparisons between the individual groups was statistically using *post hoc* Tukey test (Table 4). *Post hoc* Tukey analysis showed statistically significant differences in the mean impact strength values between group I with all other groups individually ($p < 0.05$). The analysis of mean impact strength between group II with group III ($p = 0.716$), group II with group IV ($p = 0.856$), group III and group IV ($p = 0.256$) showed no statistical significance. A negative value (–) in the mean difference indicates that the second variable has higher mean value than first variable in that comparison.

DISCUSSION

PMMA is widely used to fabricate denture bases due to various desirable properties. Despite having these, it lacks physical and mechanical strength that could support the masticatory stresses.¹⁶ A study conducted by Johnston et al. on flexure fatigue of 10 commonly used denture base resins concluded that 68% of dentures break within their first year of clinical service.¹⁷ Few other studies also showed varied reports on fractures of denture, with fatigue and impact resulting in maxillary dentures to break in the midline and with impact as a cause in 80% of mandibular denture fractures.¹⁸ Due to the above said facts, there are various

Table 3: Overall comparison of mean impact strength in various groups by one way ANOVA test

Comparison	Sum of squares	df	Mean square	F	Sig.
Between groups	158.423	3	52.808	9.276	<0.001*
Within groups	432.658	76	5.693		
Total	591.081	79			

*Highly significant

Table 4: Post hoc Tukey test for individual intergroup comparisons

Comparison	Mean difference	p value
Group I–Group II	–3.106650*	0.001**
Group I–Group III	–2.307950*	0.016*
Group I–Group IV	–3.707800*	<0.001**
Group II–Group III	0.798700	0.716 NS
Group II–Group IV	–0.601150	0.856 NS
Group III–Group IV	–1.399850	0.256 NS

*Significant; **Highly significant
NS, nonsignificant

new interventions put forth into PMMA by adding nanofillers and also introduced newer processing techniques alternative to the conventional methods. TiO₂ nanofillers compared to others are proved to have good mechanical and antimicrobial properties.¹⁹

In the present study, the evaluation of impact strength of PMMA material incorporated with TiO₂ nanofillers was done and also comparison between acrylic resins with/without TiO₂ nanofillers using two different processing techniques (microwave processing and conventional water bath methods) was done. The test samples were divided into four groups (Table 1) and all the specimens were tested for impact strength. The obtained results showed that impact strength was increased by adding TiO₂ nanoparticles. Highest impact strength was observed for group IV (23.13) followed by group II (22.53), group III (21.73), group I (19.42). i.e. PMMA reinforced with 1 wt% TiO₂ using microwave technique showed higher impact strength compared to others because in this technique microwaves are used to generate heat inside the resin.

The concentration of TiO₂ in the present study was restricted to 1 wt%, because in a study conducted by Shirkavand et al. on the effect of various concentrations of (0.5, 1 and 2 wt%) TiO₂ nanoparticles on tensile strength of dental acrylic resins, incorporation of 1 wt% of TiO₂ to PMMA showed improved mechanical properties.²⁰ Increased concentrations of TiO₂ (5%) will lead to worsening in impact strength of the resin material. On increasing the filler content above the saturation point, the resin cannot incorporate more filler particles leading to saturation of the material and further leads to deterioration. Further attempts to add filler particles above saturation point leads to disruption of resin matrix causing decreased strength of specimens.²¹ The other theory is that, the nanoparticles incorporated into PMMA will take up the applied load and the resin matrix will distribute the load and provides structural integrity.²⁰ The present study showed that the impact strength was comparatively higher in the test specimens belonging to group II and IV (the acrylic resins incorporated with 1 wt% TiO₂) than group I and III (the normal acrylic resins). These results are in concordance with the studies conducted by Shirkavand et al.,²⁰ Ahmed et al.,²¹ Ghahremani et al.²² and Acosta-Torres et al.²³ The study of Acosta-Torres et al. on the effect of nanotechnology on the improvement of conventional prosthetic acrylic resins proved that TiO₂ nanofillers improved the mechanical properties of the resin materials.

The impact strength of a material also depends on the processing technique used. Conventional water bath method has been in use for a very long time because of its easy and cost effective procedure; however, due to exothermic polymerisation reaction, there is evidence of porosities and low impact strength and it takes longer processing times to prevent the formation of voids within the denture.²⁴ Microwave processing of denture based resins is based on the generation of electromagnetic field inside the microwave oven which allows the acrylic molecules to orient

themselves. Nishii et al. in 1968 first proposed the use of microwave energy as an alternative PMMA processing method.²⁵ Kimura et al. in their studies concluded that a similar behavior is exhibited by acrylic resins processed through both conventional water bath method and using microwave technique, but with early processing time using microwave processing.¹⁵ Jadhav et al. investigated the impact strength of denture base resins polymerized by various processing techniques. Their results showed denture bases polymerized by microwave processing technique had highest impact strength compared to water bath technique.²⁴ In microwave processing, the polymer and monomer mixer is heated directly before the flask is heated, so the heat generated gets dissipated to the investment and flask. In water bath method, water gets heated first followed by the heating of the flask, investment polymer and monomer mass. This delay in the heating of polymer and monomer mass results in longer processing times.²⁵ The limitations of the present study includes: even though the dimensions of the specimens were standardized, the strength of the reinforced PMMA orally in the presence of the saliva cannot be anticipated and the simulation with the clinical conditions is not done. Another limitation was that surface of acrylic samples were flat when compared to any removable prostheses which has curved surfaces.

CONCLUSION

Specimens of heat cured acrylic resin materials fabricated with incorporation of TiO₂ nanoparticles showed high impact strength compared to the normal acrylic resins. Among the groups tested, the acrylic resins processed with microwave technique with incorporation of TiO₂ particles showed highest impact strength. Further tests can be done to evaluate the effect of oral environment on inclusion of TiO₂ nanoparticles with microwave processing of denture bases in a clinical setup.

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