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Comparative evaluation of impact strength of mechanically modified heat polymerized polymethyl methacrylate (PMMA) resin with addition of 0.5, 1, 2 wt% of silver nanoparticles (AgNPs): An in-vitro study

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Abstract

OBJECTIVE: Polymethyl methacrylate (PMMA) is one of the most widely used denture base material because of favorable esthetics and desirable characteristics such as easy handling. Acrylic resins, although, have some inherent disadvantages such as relatively poor physical and mechanical properties. The objective of the present study was to evaluate and compare the impact strength of unmodified and modified heat cure PMMA-based denture base material with addition of different concentrations of silver nanoparticles (AgNPs) (0.5, 1, 2 wt%).

METHODS: The present in-vitro study comprised of a total of 60 samples allocated to four groups with Group A samples consisting of heat cure acrylic resin without any modification (used as control); Group B samples consisting of heat cure acrylic resin modified with 0.5 wt% of AgNPs; and subsequently, Group C and Group D samples consisting of heat cure acrylic resin modified with 1 wt% and 2 wt% of AgNPs, respectively. The impact strength of the prepared acrylic samples was evaluated using Izod/Charpy Impact Tester, while the values obtained were tabulated and subjected to statistical analysis. Statistical analysis was done using Statistical Package for Social Sciences (SPSS) version 17.0 (SPSS Inc., Chicago, IL, USA), while one-way analysis of variance and Tukey's multiple post-hoc procedures were used for statistical analysis. $P < .05$ was considered statistically significant.

RESULTS: The highest impact strength was found in Group C samples consisting of heat cure acrylic resin modified with 1 wt% of AgNPs to be closely followed by Group B samples consisting of heat cure acrylic resin modified with 0.5 wt% of AgNPs. Group D samples consisting of heat cure acrylic resin modified with 2 wt% of AgNPs, although, revealed relatively lower impact strength compared to Group B and Group C samples.

CONCLUSIONS: Within the limitations of the present study, it was concluded that the impact strength of mechanically modified heat polymerized PMMA resin was significantly enhanced with addition of varying concentrations of AgNPs, although, it was observed that with an increase in the concentration of AgNPs, a subsequent decrease in the tensile strength of the final polymer material was observed.

Keywords:

Heat polymerized polymethyl methacrylate (PMMA) resin, impact strength, in-vitro study, silver nanoparticles (AgNPs)

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Introduction

Edentulism is a matter of great concern to majority of the population. The loss of teeth may lead patients to seek care for functional or esthetics reasons. Tooth loss and use of artificial substitutes for the prosthetic rehabilitation of patients is not a new idea. Missing teeth to be replaced with prosthesis have been described throughout history. In this context, polymethyl methacrylate (PMMA) is one of the most widely used materials in prosthodontics because of favorable esthetics and desirable characteristics such as easy handling.^[1] Acrylic resins have excellent esthetic properties, adequate strength, low water sorption, low solubility, and are least toxic. They can reproduce surface details accurately and can be easily repaired.^[2,3] Acrylic resins, although, have some inherent disadvantages such as relatively poor mechanical properties.^[4] Denture base acts as an intermediary between the teeth and the jaws and has to transmit all or part of the masticatory forces from the teeth to the underlying tissues including bone. One of the most common problems confronted in the provision of such prosthesis is whether the constraints of strength and design meet the functional requirements of the oral cavity. There are a few drawbacks of acrylic resins, which during processing lead to the ingress of dimensional inaccuracies and comparatively low modulus of elasticity which cause their rapid deformation at even low stresses due to properties such as low thermal conductivity and high coefficient of thermal expansion. Material ageing, as well, can drastically affect the physical and mechanical properties of acrylic resins.^[4] The flexural and impact strength of PMMA-based denture base material can be significantly improved by reinforcing it with carbon and/or polyethylene fibers. The physical and mechanical properties of acrylic resins including their thermal conductivity and polymerization shrinkage can, also, be improved by the addition of metal fillers such as silver, copper, and/or aluminum in the form of microparticles/nanoparticles (powder) to the resins.^[5] Several studies have shown that when silver nanoparticles (AgNPs) are added to acrylic resin, they exhibit antimicrobial behavior as well. Chladek *et al.*^[4] reported that the growth of *Candida albicans*, one of the most common pathogens in the oral cavity, can be restricted by adding AgNPs to acrylic resin. Monteiro *et al.*^[6] incorporated colloidal AgNPs in denture base resin in different concentrations and found that AgNPs can attain an effective antimicrobial property. The addition of AgNPs has, also, been proven to be useful in the form of additives to certain drugs, wound dressings, suture materials, surgical masks, venous and urinary catheters, endotracheal tubes, artificial tendons, and orthopedic metallic rods and grafting materials and orthodontic adhesives in the literature.^[7,8] Literature reports various studies related to AgNPs-based

composites with antimicrobial application in the medical field. However, there have been very few studies which have been conducted to check the efficacy of AgNPs on the impact strength of denture base resin because denture fracture is a problem commonly encountered by denture wearers. The aim of the present study was to evaluate and compare the impact strength of unmodified and modified heat cure PMMA-based denture base material with addition of different concentrations of AgNPs (0.5, 1, 2 wt%).

Materials and Methods

The present study was designed as an in-vitro study for the comparative evaluation of impact strength of mechanically modified heat polymerized PMMA resin with addition of 0.5, 1, 2 wt% of AgNPs. The experimental design for the present study was constituted by a total of 60 samples allocated to four groups with 15 samples in each group. Group A samples consisted of heat cure acrylic resin without any modification (used as control); Group B samples consisted of heat cure acrylic resin modified with 0.5 wt% of AgNPs; and subsequently, Group C and Group D samples consisting of heat cure acrylic resin modified with 1 wt% and 2 wt% of AgNPs, respectively. The objectives and need for the study were approved by the Institutional Ethics and Review Board via. Letter approval no. SDDC/IERB/01-41-2022 before the start of the study.

Standardized mould preparation: To standardize the acrylic samples, five metal bars [Figure 1] with dimensions of 50mm × 6mm × 4mm were milled for mould space preparation according to the specifications provided by the American Dental Association for testing impact strength.^[9] A thin layer of wax (Modelling Wax, Hindustan Dental Products Pvt. Ltd., Hyderabad,



Figure 1: Metal bars of dimension 50mm × 6mm × 4mm used for mould space preparation according to specifications provided by American Dental Association (ADA)

India) was applied to both the ends of metal bars and coated with a thin layer of petroleum jelly (Biopharm Labs, Bangalore, India) to all the surfaces of bars for easy removal from the investment material. The metal bars were, then, invested in the lower component of the denture flask, embedding half of the thickness of the bars into the dental stone (Asian Chemicals, Rajkot, India) used as investment material. This was allowed to set for half an hour. A single layer of sodium alginate solution (Cold mould seal, ProDent, Ratnagiri, India) was painted using a painting brush on to the dental stone. The counter part of the flask was, then, assembled and another mix of dental stone was poured to complete flasking. Care was taken not to incorporate any air bubbles in the dental stone. The lid was placed and the flask was clamped tightly until the final set of the dental stone after which the two halves of the flask were opened and the preformed metal bars were retrieved from the investment [Figure 2]. The two halves of the flask were separated and thoroughly flushed with hot water to remove any residual wax. The moulds were cleaned with soap water and dried in open air. While the moulds were still warm, a single layer of sodium alginate solution was painted using a painting brush on to the dental stone on both parts of the flask for the fabrication of the acrylic samples [Figure 3]. For this, five metal bars were invested in a single flask, while similarly, two other dental flasks were prepared, so, a total of three flasks were prepared for the fabrication of 15 samples for each group.



Figure 2: Standardized mould spaces prepared for fabrication of acrylic samples

Preparation of Group A samples consisting of heat cure acrylic resin without any modification (used as control): The 15 samples of Group A served as control group. For this, manufacturer's recommended amounts of powder and liquid components (Polymer:Monomer ratio of 3:1, i.e., 12g: 4mL) of PMMA-based denture base material [DPI Heat Cure- Dental Products of India (DPI- Mumbai, India)] was taken and mixed in a porcelain jar. The mixed resin was left in the mixing pot until it reached dough stage and then, the mix was kneaded thoroughly to make homogenous dough. The dough was packed into the mould space of the dental flask. Care was taken to avoid porosity due to entrapment of air bubbles. Trial closure was carried out, while the excess flash was removed and final closure was done under a hydraulic bench press [Figure 4]. The flask was, then, shifted to a clamp and left in the clamp for bench curing for 30 minutes at room temperature to allow for proper penetration of the monomer into the polymer. Similarly, the other two flasks were, also, packed with PMMA denture base material. The flasks were immersed in water at room temperature in an acrylizer [Figure 5]. Curing was done using a short curing cycle where the temperature was slowly raised from room temperature to 73°C and held for 90 minutes followed by boiling at 100°C for 30 minutes. After the curing was completed, the flasks were removed and left for bench cooling. Once the flasks were cooled, the samples were retrieved from the flask. A total of 15 samples were retrieved, trimmed using carbide burs, and finished with 120-grit sandpaper in a mandrel with slow speed. Minimal finishing and polishing was done. Care was taken not to generate heat during the finishing and polishing procedure. All the samples were measured for length, width, and height using a digital vernier caliper [Figure 6]. The prepared samples were stored at room temperature in water for 7 days before evaluation of impact strength in impact testing machine.

Preparation of Group B samples consisting of heat cure acrylic resin modified with 0.5 wt% of AgNPs: After preparing 15 mould spaces for Group B samples similar



Figure 3: Prepared acrylic samples for evaluation of impact strength in impact testing machine

to Group A samples, Group B samples were prepared by adding 0.5 wt% of AgNPs to PMMA denture base material by mixing 0.02g of AgNPs with 4mL of the liquid (monomer) for proper dispersion of the nanomaterial into the monomer component and then, quick mixing with 12g of the powder (polymer) to prevent agglomeration with the help of a magnetic stirrer for 20 minutes. When the mixture reached the dough stage, it was taken in to the mould space and packed. The prepared samples were, then, processed and finished similar to Group A samples.

Preparation of Group C (consisting of heat cure acrylic resin modified with 1 wt% of AgNPs) and Group D (consisting of heat cure acrylic resin modified with 2 wt% of AgNPs) samples: Similar to Group B samples, Group C and Group D samples were prepared with the measured amount of AgNPs, while after finishing of all 60 samples, a V-shaped notch of depth 1.2 ± 0.1 mm was cut in the middle of each sample leaving behind a residual depth of 4.8 ± 0.1 mm beneath the notch in the prepared samples using a diamond disk at the center of the finished acrylic samples.

Evaluation of impact strength: The impact strength of the prepared acrylic samples was evaluated using

Izod/Charpy Impact Tester (Pacorr Testing Instruments Pvt. Ltd., Ghaziabad, India) [Figure 7] with a pendulum of 0.5 J. The samples were clamped at one end with the samples being vertically positioned such that the notch of the prepared samples was facing toward the pendulum and the swinging pendulum was used to break the notched sample. All 60 samples were subjected to fracture load, while the load at which the samples fractured was noted in terms of the impact value (in J) and impact strength (in J/m). The values obtained were tabulated for statistical analysis.

Statistical analysis used

Statistical analysis was done using Statistical Package for Social Sciences (SPSS) version 17.0 (SPSS Inc., Chicago, IL, USA), while one-way analysis of variance and Tukey's multiple post-hoc procedures were used for statistical analysis. The mean values and standard deviations of the impact strength between the control group and mechanically modified groups were calculated using descriptive statistics. With $P < .05$, the null hypothesis

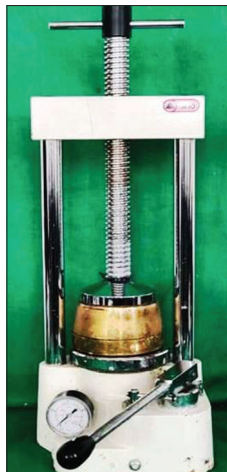


Figure 4: Hydraulic bench press used in preparation of acrylic samples

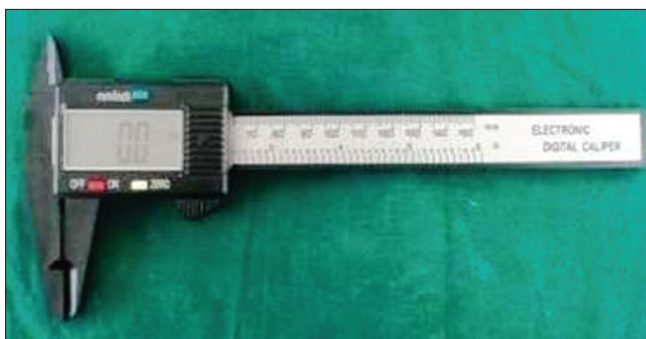


Figure 6: Digital vernier caliper used for conducting measurements of prepared acrylic samples



Figure 5: Acrylizer used in preparation of acrylic samples



Figure 7: Izod/Charpy Impact Tester used for evaluation of impact strength of prepared acrylic samples

was rejected, while the data were considered statistically significant.

Results

The aim of the present study was to evaluate and compare the impact strength of unmodified and modified heat cure PMMA-based denture base material with addition of different concentrations of AgNPs (0.5, 1, 2 wt%). For this, the values of peak load that were obtained during the fracture of the tested samples from the impact testing machine were tabulated and subjected to statistical analysis. Table 1 shows the descriptive statistics, mean values of impact strength (in J/m), and standard deviations observed for Group A to Group D wherein the mean value of impact strength (in J/m) for Group A samples was calculated as 7.1 J/m, while the mean values for Group B, Group C, and Group D samples were calculated as 19.1 J/m, 20.4 J/m, and 13.1 J/m, respectively. On analyzing the results, it was found that the highest impact strength values were found in Group C samples consisting of heat cure acrylic resin modified with 1 wt% of AgNPs to be closely followed by Group B samples consisting of heat cure acrylic resin modified with 0.5 wt% of AgNPs. Group D samples consisting of heat cure acrylic resin modified with 2 wt% of AgNPs, although, revealed relatively lower impact strength values compared to Group B and Group C samples. The lowest impact value (in J) for Group A samples in the present study was calculated at 0.02 J with the lowest impact strength (in J/m) being 3.3 J/m. Likewise, the highest impact value (in J) for Group A samples in the present study was calculated at 0.07 J with the highest impact strength (in J/m) being 11.6 J/m. Similarly, the lowest impact value (in J) for Group B samples in the present study was calculated at 0.07 J with the lowest impact strength (in J/m) being 11.6 J/m, while the highest impact value (in J) and impact strength (in J/m) for Group B samples were calculated as 0.15 J and 25.0 J/m, respectively. The lowest impact value (in J) for Group C samples in the present study was calculated at 0.07 J with the lowest impact strength (in J/m) being 11.6 J/m, while the highest impact value (in J) and impact strength (in J/m) for Group C samples were calculated as 0.15 J and 25.0 J/m, respectively. For Group D samples, the lowest impact value (in J) in the present study was calculated at 0.06 J with the lowest impact strength (in J/m) being 11.6 J/m, while the highest impact value (in J) and impact strength (in J/m) for Group D samples were calculated as 0.15 J and 25.0 J/m, respectively. Table 2 shows the intergroup and intragroup comparisons between Group A and Group D using one-way analysis of variance wherein a significant difference was observed between all four groups when intergroup and intragroup comparisons were made, while the results were found to be statistically highly significant ($P < .00001$).

Likewise, Table 3 shows the intergroup comparisons between Group A and Group D using Tukey's multiple post-hoc procedures wherein Tukey's multiple post-hoc procedures used for the subsequent multiple comparisons between different groups revealed the results showing a significant difference in impact strength between all treatment groups except on comparison between Group B and C which was found to be statistically insignificant ($P = .7780875$).

Discussion

PMMA is the most commonly used material for fabricating denture bases. The properties like low weight, less water absorption, low solubility, and less cost further enhanced its popularity. PMMA, also, possesses excellent esthetic properties and is easy to repair. The material, although, has certain inherent disadvantages such as low thermal conductivity, low mechanical strength, and low modulus of elasticity. It is brittle in nature and has a high coefficient of thermal expansion. The most common causes of denture fractures are the poor fit and lack of a balanced occlusion established in the final fabricated dentures. Fractures in the dentures usually result from different types of forces like flexural, fatigue, and impact forces.^[10-16] Beyli and von Fraunhofer^[12] in the year 1980

Table 1: Descriptive statistics, mean values of impact strength (in J/m), and standard deviations observed for Group A to Group D

Group	Mean (in J/m)	Std. deviation	Standard Error	95% Confidence Interval for Mean	
				Lower bound	Upper bound
A	7.1	3.1685	0.82	8.699	5.501
B	19.1	4.2402	1.08	16.975	21.225
C	20.4	4.9649	1.28	17.89	22.91
D	13.1	3.515	0.9	11.329	14.871

Table 2: Intergroup and intragroup comparisons between Group A and Group D using one-way ANOVA

One-way ANOVA	Sum of Squares	Df	Mean Square	F	P
Between groups	1685.3672	3	561.7891	34.55867	<0.00001**
Within groups	910.3413	56	16.2561		
Total	2595.7085	59			

** $P < 0.001$ - Highly significant

Table 3: Intergroup comparisons between Group A and Group D using Tukey's multiple post-hoc procedures

Group	Treatment Pair	Tukey's HSD Q-statistics	Tukey's HSD P
A	B	11.5271	0.0010053**
	C	12.8079	0.0010053**
	D	5.7571	0.0010053**
B	C	1.2808	0.7780875
	D	5.7699	0.0010053**
C	D	7.0507	0.0010053**

** $P < 0.001$ - Highly significant

conducted a study to analyze the causes of fracture of acrylic resin dentures and found that midline fractures in dentures are a flexural fatigue failure. In a similar previous study, Smith DC,^[10] also, concluded midline fractures as a type of fatigue fractures. The author, also, observed that the strength of a denture depends on the shape, residual stress, conditions of loading, and the mechanical properties of the material, while concluding that the incisal notch acts as a crack initiator similar to the findings of the study conducted by Beyli and von Fraunhofer.^[12] Impact failure extraorally and flexural fatigue failure intraorally are the two most important causes of fracture of denture bases. Materials with good impact strength, although, absorb the energy of impact through the elastic action of the material.^[10,17-19]

Vallittu PK^[20] stated in his study that the impact strength of PMMA-based denture base material is quite low. Numerous studies have been conducted in the field of denture reinforcement to overcome the disadvantages of PMMA-based denture base material and have suggested a variety of materials for the same. The aim of all these studies was to alter and improve the mechanical properties of denture base material.^[13,17,20-30] In similar context, Mahmood WS^[31] conducted a study in which he incorporated carbon nanotubes to high-impact denture base material and concluded that the addition of carbon nanotubes significantly improved the physical and mechanical properties of the material. Some researchers have, also, demonstrated that PMMA-based denture base material can show fatigue resistant behavior with an improved impact strength when it is reinforced by carbon fibers.^[13,29,32]

Few researchers have, also, concluded that the mechanical properties of PMMA-based denture base material can be improved by addition of glass fibers.^[13,20,23,30,33,34] Recently, more attention has been directed toward the incorporation of nanoparticles into PMMA-based denture base material to improve its properties.^[4,6,16,19,27,35-43] Oyar *et al.*^[16] conducted a study to compare the mechanical properties of heat-polymerized PMMA resin with AgNPs added at different concentrations and sizes, while concluding that the properties of the resultant material depend upon the type of the incorporated nanoparticles, their size, shape, and the concentration and interaction of the particles with the polymer matrix. It has, also, been observed that the material properties of polymer nanocomposites are superior to the pure polymer matrix or composites with larger sized inclusions.

AgNPs are one of the most commonly used nanoparticles because of their clinically proven antimicrobial properties against a variety of microorganisms including *Escherichia coli*, *Candida albicans*, *Streptococcus mutans*, and *Staphylococcus aureus*.^[19,6,38,39,42] The smaller

size of the nanoparticles has, however, been linked to an increased cytotoxicity, especially when the dimensions of AgNPs are less than 3nm.^[6,19,39,44] In the present study, size of the AgNPs used was 20nm-30nm. A notable finding in relation to AgNPs was, although, observed in a study conducted by Sodagar *et al.*^[19] wherein the authors found that adding AgNPs significantly improved the flexural strength of PMMA-based denture base material; however, addition of 0.05wt% of AgNPs was found to be more effective than a concentration of 0.2wt% of AgNPs. Likewise, similar observation was made in the study conducted by Ghaffari *et al.*^[40] who used 5wt% of AgNPs and concluded that addition of 5 wt% of AgNPs decreased the tensile strength of acrylic resin. Hamed-Rad *et al.*,^[41] also, concluded from the findings of their study that addition of 0.2 wt% and 2 wt% of AgNPs to acrylic resin significantly improved its tensile strength compared to the control group, although there was a decrease in tensile strength observed with an increase in the concentration of AgNPs which can be explained on the basis of the fact that AgNPs act as impurities and that the tensile strength would decrease as their concentration increases in the final polymer. This is further explained on the basis of the fact that the dispersion of AgNPs in the polymer matrix decreases the reaction of monomer with the polymer, thus increasing the amount of unreacted monomer in and lessening the physical properties of the final product.^[3,6,37,38] Also, higher concentrations of AgNPs in the polymer matrix produce agglomeration sites that adversely affect the physical properties of the final product.^[43,45] 0.5, 1, and 2 wt% of AgNPs were used in the present study, while the findings of the present study were found to be in accordance with the findings of the previous studies wherein lower concentrations of AgNPs were found to improve the mechanical properties of mechanically modified heat-polymerized PMMA resin.

The acrylic resin test samples in the present study were prepared according to the specifications provided by the American Dental Association (Specification No. 12 for heat cure denture base acrylic resin) to standardize the dimensions of the samples during the testing procedures (50mm × 6mm × 4mm).^[16-19] Also, the samples were fabricated using a custom-made metallic mould. There are different approaches for the preparation of samples. In a method followed by Hamed-Rad *et al.*,^[41] the samples were prepared by mixing AgNPs with the polymer. Few other researchers have, also, used the same method for incorporation of AgNPs.^[19,29,32] Adding nanoparticles to the monomer is another method of modifying PMMA resin to reduce agglomeration. In the present study, the AgNPs were dispersed in liquid (monomer) using a magnetic stirrer and then mixed with the powder (polymer) of the acrylic material similar to the method adopted in various other

studies for the proper dispersion of the nanomaterial into the monomer component before mixing with the powder (polymer) to prevent agglomeration.^[6,16,39]

Also, multiple methodologies have been described to evaluate impact strength of the polymers with notched and unnotched samples.^[1,6,9,14,17-19,30,32,44,46] For the present study, a V-shaped notch of depth 1.2 ± 0.1 mm was cut in the middle of each sample leaving behind a residual depth of 4.8 ± 0.1 mm beneath the notch in the prepared samples using a diamond disk at the center of the finished acrylic samples similar to the methodology followed in other studies.^[17-19,32] The highest mean impact strength of PMMA was found to be of the group which was modified with 1 wt% of AgNPs, followed by PMMA which was modified with 0.5 wt% and 2 wt% of AgNPs, while the least impact strength was found for the control group similar to the findings of previous studies where addition of lower concentrations of AgNPs was found to significantly improve the mechanical properties of acrylic resin.^[3,6,16,38,41]

Conclusions

Within the limitations of the present study, it was concluded that the impact strength of mechanically modified heat-polymerized PMMA resin was significantly enhanced with addition of varying concentrations of AgNPs. Also, it was observed in the present study that the highest impact strength was found in Group C samples consisting of heat cure acrylic resin modified with 1 wt% of AgNPs to be closely followed by Group B samples consisting of heat cure acrylic resin modified with 0.5 wt% of AgNPs. Group D samples consisting of heat cure acrylic resin modified with 2 wt% of AgNPs, although, revealed relatively lower impact strength compared to Group B and Group C samples confirming that with an increase in the concentration of AgNPs, a subsequent decrease in the tensile strength of the final polymer material was observed. Further studies may be required to validate the results obtained in the present study and to evaluate the effect of varying concentrations of AgNPs that can be used to enhance the physical properties of the final product obtained without compromising esthetic requirements.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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