

The evaluation of flexural strength and impact strength of heat-polymerized polymethyl methacrylate denture base resin reinforced with glass and nylon fibers: An *in vitro* study

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With the advent of newer denture base materials, fiber reinforcement in polymethyl methacrylate (PMMA) acrylic resin by various fibers to improve the strength properties of PMMA denture base materials is common nowadays. So it has become imperative to evaluate which fiber suits best to improve both flexural and impact strengths of the denture base resin and to know up to what extent the fiber-reinforced PMMA denture base resin fulfills the strength requirement of an ideal denture base material.

This study compared the resistance-to-fracture properties of a commercially available heat-polymerizing PMMA denture base resin with those of the same material reinforced by glass and nylon fibers. The fibers were randomly oriented and used in concentration of 2% by weight. The 20 test specimens of similar dimensions were prepared for each of the 4 experimental groups, viz., conventional PMMA denture base resin; and the same resin reinforced with monomer-treated glass fibers, silane-treated glass fibers, and monomer-treated nylon fibers.

A total of 10 test specimens from each study group were subjected to three-point bend test on a Universal Instron testing machine, and the remaining 10 test specimens were tested for impact strength by Charpy's pendulum impact strength tester.

From the literature, it was found that the flexural and impact strengths of heat-polymerized PMMA denture base resin reinforced with fibers are significantly more than those of the conventional heat-polymerized PMMA denture base resin.

Key words: Charpy's pendulum impact strength tester, fixture, Instron testing machine, rollers, stress-applying rod

INTRODUCTION

Over the centuries, a variety of materials have been used for denture construction. The historic development of these materials, from the early dentures carved from stone, ivory, bone, and wood to the latest polymer, has been studied. Today the acrylic resin, namely, polymethyl methacrylate (PMMA), occupies a prominent place in the spectrum of denture base materials.^[1]

Fracture of the denture base is a major problem. In recent times, many approaches have been used to strengthen the PMMA denture base resin; among these, one approach is reinforcement with different types of fibers. A major difficulty in using reinforcing fibers is improper bonding of fibers with the resin.

Bearing these factors in mind, the present study was conducted to evaluate the flexural and impact strengths of heat-polymerized PMMA denture base resin reinforced with glass and nylon fibers.

MATERIALS AND METHODS

For convenience and clarity, the description of this study has been subdivided into the following heads:

i) Preparation of gypsum molds to obtain the specimen

The master die measuring 62 mm in length, 10 mm in width, and 3 mm in thickness was fabricated in stainless steel metal. The master die had one threaded hole on each end to facilitate easy removal from the investing material. After the verification of dimensions, 3 dies were selected for preparation of gypsum molds [Figure 1]; each threaded hole of master die was filled up with carding wax. A thin layer of petroleum jelly was applied over the die, and it was invested with die stone in the dental flask. Ensuring metal-to-metal contact between the base and its counterpart, the flask was closed under constant pressure on bench clamp.

In one flask, 3 master dies were invested at a time.

After the die stone set, the flask was opened and the carding wax within the holes was removed. The dies were carefully teased out from the investing material. The molds were then evaluated for any porosities and roughness. After that the prepared molds were immersed in hot water to remove any trace of impurities and to facilitate the application of separating medium (Stellon cold mold seal, DPI). The mold cavities obtained were then used for the preparation of acrylic resin test specimens [Figure 2].

ii) Preparation of polymethyl methacrylate resin specimens

Group A: Control group

In the pilot study it was found that for 3 mold cavities, 5 mL of monomer and 12 g of polymer in the ratio of 1:2.4, volume by weight, were required to make the specimens.

The control group test specimens were made with conventional heat-polymerized PMMA resin (DPI, heat cure). The required amounts of monomer and polymer in the ratio of 1:2.4 (v/w) for the 3 molds were mixed and allowed to reach dough stage. The dough was then kneaded and packed in the molds. The trial closure was performed with a hydropress at 2 kg/cm² and excess was removed.

The flask was then clamped; and after the bench curing polymerization, cycle was started at room temperature. Then the temperature was slowly raised up to 75°C and maintained for 90 minutes, and then up to 100°C and maintained for 40 minutes. After completion of polymerization cycle, the flask was allowed to cool in water bath to room temperature, and the acrylic resin specimens were retrieved after deflasking.

Group B: Reinforced with monomer-impregnated glass fibers

Glass fibers (Voltas Ltd., Pune, India) 2% by weight and 5 mm in length were soaked in monomer for 10 minutes in a Petri dish for better bonding of these fibers with the PMMA resin matrix.^[2] The fibers were removed from the monomer and excess liquid was allowed to dry. Then, the polymer- and monomer-treated fibers were mixed thoroughly to disperse the fibers. After that, the monomer and this polymer containing monomer-treated glass fibers were mixed in the ratio of 1:2.4 (v/w) and allowed to reach dough stage. The dough was then kneaded and packed into the prepared molds. The specimens were polymerized and retrieved in the same manner as described for the control group.

Group C: Reinforced with silane-impregnated glass fibers

Glass fibers 2% by weight and 5 mm in length were soaked in silane for 5 minutes in a Petri dish

for better bonding of these fibers with the PMMA resin matrix.^[3] The fibers were removed from silane and allowed to air-dry completely. The polymer- and the silane-treated fibers were mixed thoroughly to disperse the fibers. After that, the specimens were polymerized and retrieved in the same manner as described for group B.

Group D: Reinforced with monomer-impregnated nylon fibers

Nylon fibers (MRF Ltd., Chennai, India) 2% by weight and 5 mm in length were soaked in monomer for 10 minutes in a Petri dish for better bonding of these fibers with the PMMA resin matrix.^[2] The fibers were removed from the monomer, and excess liquid was allowed to dry. The polymer- and monomer-treated nylon fibers were mixed thoroughly to disperse the fibers. The specimens were polymerized and retrieved in the same manner as described for the other three groups.

Twenty specimens in each group were prepared, and the exposed fibers of the specimens at the peripheral border were trimmed with diamond bur at slow speed. Each specimen was then finished and polished. The dimensions of every specimen were verified with digital vernier caliper.

iii) Storage of specimens

To simulate the oral environment, all specimens were saturated by storing in normal saline at 37°C to maintain 100% humidity, for 1 week in an incubator.

iv) Testing

a) Flexural strength testing: Ten specimens from each study group (groups A, B, C, and D) were tested for flexural strength by three-point bend test on Universal Instron testing machine [Instron 4467, England] at a crosshead speed of 2 mm/min. To conduct the three-point bend test, a fixture was fabricated with the following dimensions: length, 80 mm; width, 30 mm; and thickness, 30 mm. On the top of the fixture, 2 grooves were made at a distance of 25 mm from the center on either side. A roller with diameter of 4.25 mm was placed in each groove, and a customized T-shaped stress-applying rod with the dimensions of 60 mm x 10 mm was fabricated [Figure 3], by which stress could be applied in the center of the specimen. The specimen was placed on the rollers in such a way that the center of the specimen coincided with the center of the distance between the 2 rollers. This whole unit was then mounted on the lower jaw, and the stress-applying rod was fixed in the upper jaw of the Universal Instron testing machine [Figure 4]. A load was applied with T-shaped rod in the center of the specimen until fracture



Figure 1: Metal dies used for mold fabrication



Figure 4: Flexural strength testing on Universal Instron testing machine



Figure 2: Die stone mold

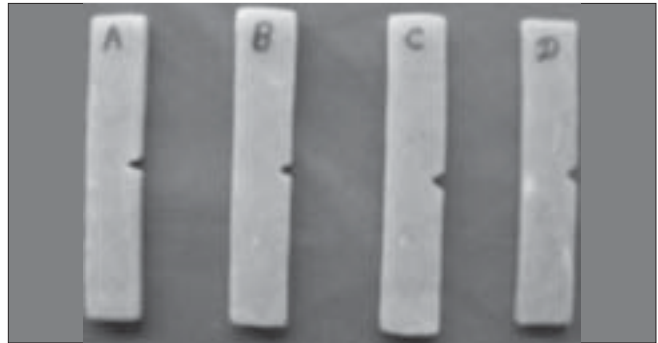


Figure 5: Specimens with V-shaped notch for impact strength testing

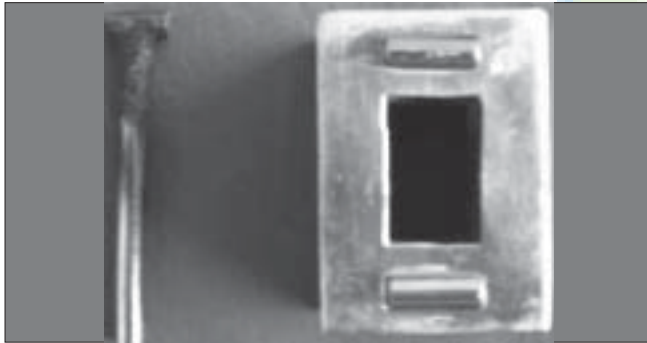


Figure 3: Fixture used in the study



Figure 6: Impact strength testing on Charpy's pendulum impact strength tester

occurred. In this manner, three-point bend test was done for each specimen.

b) Impact strength testing: Ten specimens from each study group (groups A, B, C, and D) were tested for impact strength. As per the requirement of Charpy's pendulum impact strength tester (Instron, England), on each specimen a 2-mm deep V-shaped notch was made in the center, on a lateral margin across the long axis of the specimen [Figure 5]. The specimen was fixed on testing platform in such a way that the V-shaped notch of the specimen faced the testing pendulum. Then, the specimens were subjected to impact strength test with Charpy's pendulum impact strength tester [Figure 6].

RESULTS

Flexural strength

The mean value for flexural strength with respect to fracture load and the corresponding flexural strength of the 4 study groups are presented in Table 1.

The flexural strength was calculated using the following formula:

$$FS = \frac{3 p l}{2 b d^2}$$

where

FS is the flexural strength,
p is the peak load applied,

l is the span length,
b is the specimen width, and
d is the specimen thickness.

The mean flexural strength in group A was 459.79 MPa; in group B, 605.81 MPa; in group C, 656.52 MPa; and in group D, 535.99 MPa [Table 1]. This shows that group C specimens presented the highest flexural strength, followed by group B, group D, and group A.

Comparative statistics on flexural strength in the four study groups are presented in Tables 2 and 3.

An analysis of difference in flexural strength of different groups was carried out using one-way analysis of variance (ANOVA) test, which is shown in Table 2. The 'F' value was 63.54, whereas the required 'F' value at 0.05 level of confidence was 2.87. This analysis reveals that the results are statistically significant. Then, the Scheffe's post hoc test of significance for flexural strength differences among the means of different groups was carried out, which is shown in Table 3. The critical difference, i.e., minimum significant range, was found to be 44.48. This reveals that all groups significantly differed from each other. Group C showed significantly higher flexural strength

compared to group B, group D, and group A, in that order. Group B showed significantly higher flexural strength compared to group A and group D. Group D showed significantly higher flexural strength compared to group A.

Impact strength

The mean value with respect to energy absorbed (in joules) to fracture and corresponding impact strength of the four study groups are presented in Table 4.

The impact strength was calculated using the following formula:

$$\text{Impact strength} = \frac{\text{Energy absorbed}}{\text{Width} \times \text{Thickness}}$$

The mean impact strength in group A was 1.26×10^{-03} J/mm²; in group B, 2.36×10^{-03} J/mm²; in group C, 4.42×10^{-03} J/mm²; and in group D, 1.56×10^{-03} J/mm² [Table 4]. This shows that group C specimens presented the greatest impact resistance, followed by group B, group D, and group A.

Comparative statistics of impact strength in the four groups are presented in Tables 5 and 6.

An analysis of differences in impact strength of different groups was done by using one-way analysis of variance (ANOVA) test, which is shown in Table 5. The 'F' value was 75.44, whereas the required 'F' value at 0.05 level of confidence was 2.87. This analysis reveals that the results are statistically significant. Then, the Scheffe's post hoc test of significance for impact strength differences among the means of different groups was carried out, which is shown in Table 6. The critical difference, i.e., minimum significant range, was found to be 0.67. This reveals that there was significant difference between each pair of groups; except between group A and group

Table 1: Mean values of flexural strength in the four study groups

Study groups	Load at break In kg [p]	Mean flexural strength (MPa)
Group A (Control)	10.8	459.79
Group B (Monomer impregnated glass fiber reinforced)	14.26	605.81
Group C (Silane impregnated glass fiber reinforced)	15.44	656.52
Group D (Monomer impregnated nylon fiber reinforced)	12.59	535.99

Table 2: One-way analysis of variance of flexural strength of different groups

Source of variance	Degree of freedom	Sum of square	Mean of square	"F" ratio	Required "F" ratio
Between group	(4-1) = 3	219528.92	73176.31	63.54*	2.87
Within group	(40-4) = 36	41460.54	1151.68		

*Significant at 0.05 level of confidence

Table 3: Scheffe's post hoc test of significance for flexural strength differences among the means of different groups

Group-A	Group-B	Group-C	Group-D	Mean difference	Critical difference
459.79	605.81			146.02*	
459.79		656.53		196.71*	
459.79			535.99	76.20*	
	605.81	656.53		50.72*	44.48
	605.81		535.99	69.82*	
		656.53	535.99	120.54*	

*Significant at 0.05 level of confidence

D, where the difference was insignificant. Group C showed significantly higher impact strength compared to group B, group D, and group A. Group B showed significantly higher impact strength compared to group A and group D. However, the difference between group D and group A was insignificant though group D showed higher impact strength compared to group A. Hence group C can be considered to be the most superior group among the four study groups.

DISCUSSION

The present study was conducted to compare the strength properties of conventional PMMA resin with the same resin reinforced with 5-mm chopped glass and nylon fibers in loose form. The fibers used in this study are those that are more economical and easily and readily available in the fiber industry.

According to the study by Gutteridge,^[4] the fiber incorporation beyond 3% by weight produces dry friable dough and provides no further beneficial effect on strength. So in this study, both glass and nylon fibers were used in the concentration of 2%

by weight.

Vallittu and Lassila,^[5] in 1992, again renewed interest in the subject and found that some of the features that result in reduced effect of the reinforcement could be associated with ineffective coupling between the acrylic matrix and fiber, poor wetting of the fibers, inclusion of voids, dry friable dough, nonuniform fiber distribution, or fiber breakage. Glass fibers on their own are hydrophobic in nature. They contain no polar groups, so their compatibility with PMMA resin is very poor. Untreated glass fibers act as inclusion bodies in the acrylic resin mixture; and instead of strengthening, actually they weaken the resin. In order to improve the adhesion between resin matrix and the glass fibers, Braden,^[6] in 1988, stated that surface modification has to be done, to improve chemical bonding between fibers and resin matrix. Some of the techniques followed are (i) silane treatment, (ii) monomer treatment, (iii) microwave treatment, and (iv) plasma treatment.

So in the present study to compare the bonding efficiency of silane and PMMA monomer treatment, glass fibers were monomer treated in group B and silane treated in group C. Silane coupling agents chemically bond glass fibers to the resin matrix more strongly than do the monomer-treated glass fibers.

From the literature, it appears that fibers placed perpendicular to the direction of the applied forces offer the greatest potential for improvement of the flexural fatigue and bending properties of denture base resins. However, the technical difficulties of maintaining the fibers centrally in the thickness of the denture base may outweigh any potential advantage. The using of randomly oriented fibers provide the best balance between improved properties and simplicity

Table 4: Mean values of impact strength in the four study groups

Study groups	Energy absorbed to break specimen (j)	Mean impact strength (X10-03 J/mm ²)
Group A (Control)	0.038	1.26
Group B (Monomer impregnated glass fiber reinforced)	0.071	2.36
Group C (Silane impregnated glass fiber reinforced)	0.13	4.42
Group D (Monomer impregnated nylon fiber reinforced)	0.047	1.56

Table 5: One-way analysis of variance of impact strength of different groups

Source of variance	Degree of freedom	Sum of square	Mean of square	"F" ratio	Required "F" ratio
Between group	(4-1) = 3	61.12	20.27	75.44*	2.87
Within group	(40-4) = 36	9.64	0.27		

*Significant at 0.05 level of confidence

Table 6: Scheffe's post hoc test of significance for impact strength differences among the means of different groups

Group-A	Group-B	Group-C	Group-D	Mean difference	Critical difference
1.26	2.36			1.1*	
1.26		4.43		3.17*	
1.26			1.56	0.30*	
	2.36	4.43		2.07*	0.67
	2.36		1.56	0.80*	
		4.43	1.56	2.87*	

*Significant at 0.05 level of confidence

of technique.^[4]

In the present study, the mean flexural strength was highest in group C (656.52 MPa), followed by group B (605.81 MPa), group D (535.99 MPa), and group A (459.79 MPa), as shown in Graph A. These observations are similar to those of John *et al.*, who found that all reinforced specimens showed better flexural strength than the conventional acrylic resin, and specimens reinforced with glass fibers showed the highest flexural strength, followed by nylon. The observations from this study reaffirm those made by Vallittu PK,^[7] that the modulus of elasticity of glass being very high, most of the stresses are received by the glass fibers without any deformation or transverse bending.

The statistical analysis of differences in significance [Table 3] carried out revealed that group C showed significantly higher flexural strength as compared to all other groups. Group B showed significantly higher flexural strength than group D. Group D showed significantly higher flexural strength than group A. Thus glass-reinforced specimens exhibited better flexural resistance as compared to other groups.

The mean impact strength was highest in group C (4.42×10^{-03} J/mm²), followed by group B (2.36×10^{-03} J/mm²), group D (1.56×10^{-03} J/mm²), and group A (1.26×10^{-03} J/mm²), as shown in Graph B. These observations are consistent with those made by Vallittu PK,^[8] who stated that the impact strength of PMMA reinforced with glass fibers seems to be higher than the impact strength of PMMA reinforced with other fibers or unreinforced PMMA.

The statistical analysis of differences in significance [Table 6] carried out revealed that group C showed significantly higher impact strength compared to all other groups. This was followed by group B, which showed significantly higher impact strength compared to group D and group A. However, the difference

between group D and group A was insignificant though group D showed higher impact strength compared to group A.

The results of this study matched consistently with those found in other studies with a similar design.

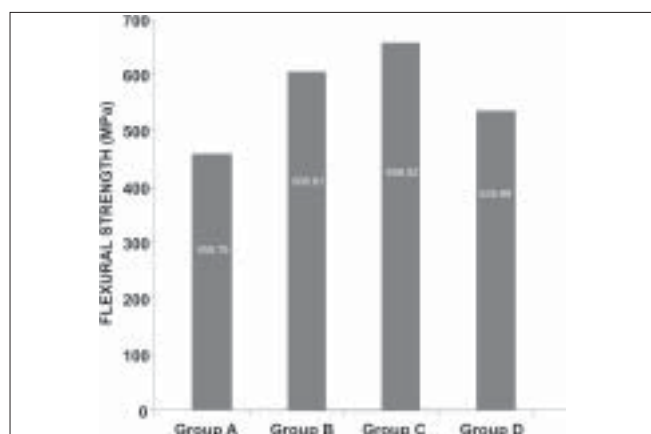
From this study, it is observed that reinforcement of denture base resin with either glass or nylon fiber improves the mechanical properties. Glass-reinforced or nylon fiber-reinforced dentures are much stronger and more resilient under flexural fatigue or impact stress conditions than conventional PMMA dentures, and hence the chances of fracture of the denture can be substantially eliminated. This improvement requires only a minor increase in the cost of material and technician's time.

CONCLUSION

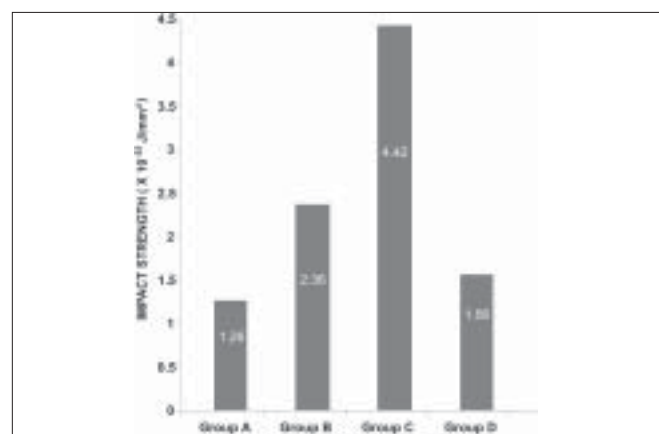
The search for still higher strength polymer continues, not only because it would be very useful to have 'unbreakable' dentures but also because it would then become possible to construct skeletally designed polymer-removable dentures. The cost implication and design potential of this are obvious.

The results of this study lead to the following conclusions:

- 1) On comparing the flexural and impact strength properties between conventional and fiber-reinforced heat cure PMMA denture base material, it was found that fiber-reinforced specimens were more resistant to impact and flexural fatigue than conventional PMMA specimens.
- 2) When flexural strength and impact strength of glass-reinforced and nylon fiber-reinforced heat cure PMMA denture base material were compared, it was found that glass fiber reinforcement considerably improves both impact and flexural strengths of denture base resin when compared with nylon



Graph A: Bar graph showing the mean values of flexural strength in the four study groups



Graph B: Bar graph showing the mean values of impact strength in the four study groups

fiber reinforcement.

3) Silane-impregnated glass fiber reinforcement suits best to increase the flexural and impact strengths of heat-polymerized PMMA denture base resin.

Reinforcement using 2% by weight of glass and nylon fibers substantially increased the fracture resistance of the specimens.

However, further research is required to ascertain the mode of arrangement of fibers; but from this study, it has been observed that randomly oriented fibers too provide improved strength. Moreover, it is technically easier to follow this procedure in the dental laboratory.

Further work is clearly needed to investigate the effect of long-term immersion in water on the fiber/resin interface and on mechanical properties. Other factors to be considered are the effect of the fibers on oral mucosa, whether or not they project from the resin following wear, and how various cleaning and polishing procedures affect the surface.

In order to establish the use of fiber-reinforced resin, it is mandatory also to research different techniques possible to enhance the bond between fibers and resin matrix and to make the process less technique sensitive.

REFERENCES

1. Peyton FA. History of resins in dentistry. *Dent Clin North Am* 1975;19:211-22.
2. Vallittu PK. Flexural properties of acrylic resin polymers reinforced with unidirectional and woven glass fibres. *J Prosthet Dent* 1999;81:318-26.
3. Solnit GS. The effect of methyl methacrylate reinforcement with silane treated and untreated glass fibres. *J Prosthet Dent* 1991;66:310-4.
4. Gutteridge DL. The effect of including ultra-high modulus polyethylene fibre on the impact strength of acrylic resin. *Br Dent J* 1988;164:177-80.
5. Vallittu PK, Lassila VP. Reinforcement of acrylic resin denture base material with metal or fibre strengtheners. *J Oral Rehab* 1992;19:225-30.
6. Braden M, Davy KW, Parker S, Ladizesky NH, Ward IM. Denture base poly(methyl methacrylate) reinforced with ultra-high modulus polyethylene fibres. *Br Dent J* 1988;164:109-13.
7. Vallittu PK, Lassila VP, Lappalainen R. Transverse strength and fatigue of denture acrylic glass fibre composite. *Dent Mater* 1994;10:116-21.
8. Vallittu PK, Vojtkova H, Lassila VP. Impact strength of denture polymethyl methacrylate reinforced with continuous glass fibres or metal wire. *Acta Odontol Scand* 1995;53:392-6.

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