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ORIGINAL ARTICLE

Study to evaluate the Effect of Silane Treatment and Three Different Woven Fiber Reinforcement on Mechanical Properties of a Denture Base Resin

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ABSTRACT

Background and objectives: Considering the variance obtained in literature, as far as mechanical properties of fiber-reinforced poly(methyl methacrylate) (PMMA) resin is concerned, an investigation was done to evaluate and compare the properties of a denture base resin reinforced with and without silane-treated glass, polyethylene, and carbon fibers in woven form.

Materials and methods: A total of 140 specimens of PMMA (Trevalon) of standard dimension (65 × 10 × 3 mm) as per American Dental Association (ADA) specification no. 12/International Standards Organization (ISO) specification no. 1567 for transverse deflection test and American Society for Testing Materials (ASTM) specification no. D6110/ISO specification no. 179 for impact strength test were prepared: 20 for each of the seven study groups. Reinforced PMMA specimens were prepared by incorporating single-layer woven (Twill 2 × 2) glass, polyethylene, and carbon fiber mats (55 × 6 mm) into their respective groups. Silanization of specimens of respective groups was carried out by dipping the fiber samples in silane compound A-174 (γ-methacryl oxypropyl trimethoxy silane). Specimens were subjected to Charpy's impact test and transverse strength using "Impact Testing Machine" and three-point bending "Universal Testing Machine" (which was calibrated to show deflection values) respectively. Modulus of elasticity values were obtained from deflection readings. The readings thus obtained were tabulated and were subjected to statistical analysis. Comparison of the mean values between control and other groups was done by unpaired t-test. For all the tests, a "p" value of 0.05 or less was set for statistical significance.

Results: Values obtained for mechanical properties tested of specimens in group IIIC (heat-cured denture base resin—Trevalon, with silane treated, woven polyethylene fiber reinforcements) far exceeded the values of all other groups. When comparing silanized specimens with nonsilanized specimens, it was seen that in general, use of a silane coupling agent statistically significantly improved the impact strength of the tested specimens (one way analysis of variance, F = 208.2; *post hoc* Tukey's, $p < 0.001$, highly significant).

Conclusions: Silanized woven polyethylene fiber reinforcement resulted in the greatest improvement in mechanical properties of PMMA resin specimens for any group tested.

Keywords: Denture base resin, Mechanical properties, Poly(methyl methacrylate), Reinforcements, Silanization, Woven fibers.

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INTRODUCTION

An inherent disadvantage of PMMA as an acrylic denture base resin is its susceptibility to break during service due to its poor strength characteristics, including low impact strength and low fatigue resistance.[1]

It has long been hypothesized that the addition of synthetic fibers to the monomer/polymer mixture may strengthen the resultant acrylic resin. Several different types of fibers have been used, with varying results.[1] Various types of fibers including carbon fiber, whisker fiber, aramid fiber, polyethylene fiber, and glass fiber have been used as reinforcements.[2-6] Carbon fibers have been shown to improve flexural and impact strength, prevent fatigue fracture, and strengthen the resin. However, carbon fibers have a dark color, which might pose an esthetic problem. For this reason, more appropriate strengtheners are needed.

Likewise, polyethylene fibers have also been observed to increase the impact strength, modulus of elasticity, and flexural strength. Unlike carbon and Kevlar fibers, polyethylene fibers are almost suitable in denture base acrylic resins.[4] The other factors that relate to the strength of the fiber composite are quantity of fibers in polymer matrix, orientation of fibers, and adhesion of fibers to polymer matrix. Untreated fibers act as inclusion bodies in the acrylic resin mixture and, instead of strengthening, actually weaken the resin. Silane coupling agents, which chemically bond fibers to the resin matrix, may make the mixtures more homogeneous, resulting in strong PMMA.[1]

Considering the variance obtained in literature, as far as mechanical properties of fiber-reinforced PMMA are concerned, an investigation was carried out to evaluate the effect of silane treatment and three different woven fiber reinforcement on mechanical properties of a denture base resin.

MATERIALS AND METHODS

Preparation of Working Mold Space for Fabrication of Test Specimens

Ten rectangular stainless steel blocks measuring 65 mm in length, 3 mm in thickness, and 10 mm in width were prepared (according to ADA specification no. 12/ISO specification no. 1567 for transverse deflection test and ASTM specification no. D6110/ISO specification no. 179 for impact strength test) to create uniform mold spaces in gypsum, for the fabrication of test specimens. Before the dental stone was set in the base flask, the rectangular steel blocks were invested side by side in two rows of five blocks each exposing their superior surface. After the first pour was set, a thin film of sodium alginate separating media was applied over the surface and the counter flask was invested with die stone and allowed to set for 1 hour. Later, the flask was opened; the steel blocks were carefully retrieved to obtain mold spaces for preparing test specimens.

Grouping of Test Specimens

For the purpose of the study, the test specimens were divided into the following groups as given in Table 1.

Preparing PMMA Resin Test Specimens

Test specimens were fabricated by the compression molding technique. Finishing and polishing of the retrieved specimens were carried out using tungsten carbide bur and wet 600 grit sandpaper. Later, all the test specimens were kept in water at room temperature for 48 hours before testing, so as to ensure minimal residual monomer content within the test specimens. The same processing sequence was applied for all the test specimens.

Preparing Fiber-reinforced PMMA Resin Test Specimens

Acrylic resin fiber specimens were prepared by cutting woven mats of fiber slightly short of the size of the slots, and placing them in the mold approximately in the middle of the acrylic resin dough. Single-layer, nonsilane-treated, and silane-treated glass, polyethylene, and carbon fiber mats measuring 55 × 6 mm were added to the specimens from their respective groups as shown in Table 1.

Preparing Silane-treated Fiber-reinforced PMMA Resin Test Specimens

Silanization was carried out by dipping the different woven fiber mats in silane compound γ-methacryl oxypropyl trimethoxy silane (Silquest A-174®, Batch No. D672051309, Momentive Performance Materials, India) and allowing to air dry for 20 minutes prior to their incorporation into the acrylic resin dough.

Testing of Mechanical Properties of Test Specimens

Impact Strength

Ten specimens of each from groups I, IIA, IIB, IIC, IIIA, IIIB, and IIIC were used for impact strength determination. The test specimens were placed on the platform of the Charpy type impact testing machine (Karl Frank, Germany, Model 580M) with their un-notched side facing away from the pendulum, which was then released (from an angle of 165° to the horizontal). The energy absorbed by the test specimen at the time of fracture was given by a calibrated scale. Impact strength was calculated using the following formula:

$$\delta = \frac{E}{A}$$

where E = energy absorbed by the specimen at breaking, A = area of cross-section (30 mm^2).

Table 1: Grouping of test specimens used during the study

Group I: Control group; heat-cured denture base resin (Trevalon) with no fiber reinforcements (n = 20)	Group II: Heat-cured denture base resin (Trevalon) with nonsilane-treated, woven fiber reinforcements (n = 60)	Group III: Heat-cured denture base resin (Trevalon) with silane-treated, woven fiber reinforcements (n = 60)
	Group IIA: Heat-cured denture base resin with nonsilane-treated, woven glass fiber reinforcements (n = 20)	Group IIIA: Heat-cured denture base resin with silane-treated, woven glass fiber reinforcements (n = 20)
	Group IIB: Heat-cured denture base resin with nonsilane-treated, woven carbon fiber reinforcements (n = 20)	Group IIIB: Heat-cured denture base resin with silane-treated, woven carbon fiber reinforcements (n = 20)
	Group IIC: Heat-cured denture base resin with nonsilane treated, woven polyethylene fiber reinforcements (n = 20)	Group IIIC: Heat-cured denture base resin with silane treated, woven polyethylene fiber reinforcements (n = 20)

Transverse Strength

A Universal Testing Machine (Tinius Olsen, USA, Model H5KS) was utilized for this study and a three-point loading system was used for the application of load. Ten specimens each from their respective groups were placed on a customized jig and load was applied at a cross-head speed of 5 mm/min. The load at which fracture occurred was noted and the transverse strength was calculated using the following formula:

$$\sigma = \frac{3LP}{2WT^2}$$

where P = fracture load, L = the distance between the supports (50 mm), W = specimen width (10 mm), and T = specimen thickness (3 mm).

Modulus of Elasticity

For determination of Young's modulus of elasticity (Y), the Universal Testing Machine was calibrated, so that deflection of the specimen could be determined. Modulus values were calculated using the formula:

$$Y = \frac{PL^3}{4DWT^3}$$

where D = deflection, rest, P = fracture load, L = the distance between the supports (50 mm), W = specimen width (10 mm), and T = specimen thickness (3 mm).

All the readings thus obtained were tabulated and were subjected to statistical analysis. Comparison of the mean values between control and other groups was done by unpaired t-test (Table 2). For all the tests, a p-value of 0.05 or less was set for statistical significance.

RESULTS

The aim of this study was to evaluate the effect of silane treatment and three different woven fiber reinforcement on mechanical properties of a heat cure denture base resin. From the statistical analysis, the following results have been obtained.

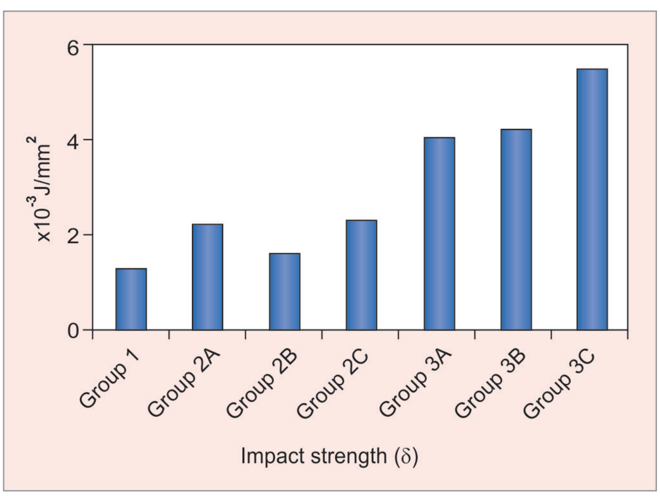
Graph 1 shows the mean impact strength of all study groups. Group IIIC showed highest mean impact strength value of 5.60 × 10^{-3} J/mm^2. The lowest mean impact strength value was of group I (1.36 × 10^{-3}J/mm^2).

Graph 2 shows the mean transverse strength of all study groups. Group IIIC showed highest mean transverse strength value of 130.89 MPa. The lowest mean transverse strength value was of group I (93.51 MPa).

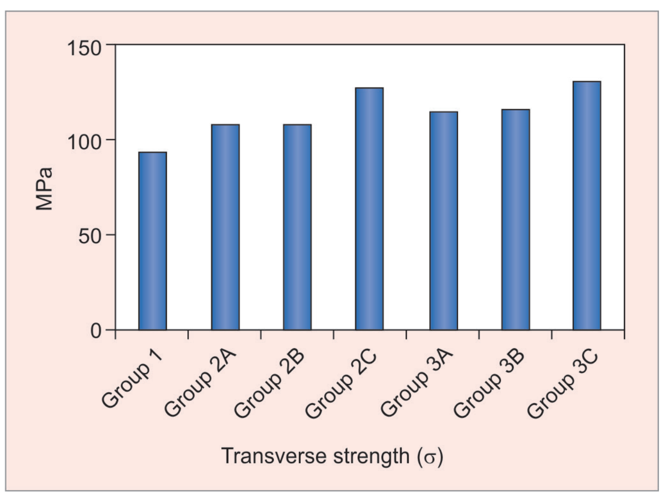
Table 2: Descriptive information on the various mechanical properties tested and their comparison with the control group

	Impact strength (δ) (×10^{-3}J/mm^2)		*Transverse strength (σ) (MPa)*		*Modulus of elasticity (γ) (MPa)*	
Groups	*Mean ± SD*	*vs control*	*Mean ± SD*	*vs control*	*Mean ± SD*	*vs control*
I	1.36 ± 0.24	–	93.52 ± 3.20	–	5021.9 ± 773.9	–
IIA	2.26 ± 0.26	p <0.001, S	107.44 ± 1.55	p<0.001, S	5439.7 ± 711.2	0.83, NS
IIB	1.63 ± 0.29	0.61, NS	106.99 ± 1.34	p<0.001, S	5288.7 ± 563.6	0.98, NS
IIC	2.36 ± 0.29	p<0.001, S	126.46 ± 2.86	p<0.001, S	7113.3 ± 762.4	p<0.001, S
IIIA	4.10 ± 0.50	p<0.001, S	114.43 ± 0.58	p<0.001, S	5467.0 ± 477.8	0.78, NS
IIIB	4.30 ± 0.46	p<0.001, S	115.36 ± 1.06	p<0.001, S	5259.2 ± 513.5	0.98, NS
IIIC	5.60 ± 0.31	p<0.001, S	130.59 ± 1.12	p<0.001, S	7600.4 ± 933.6	p<0.001, S

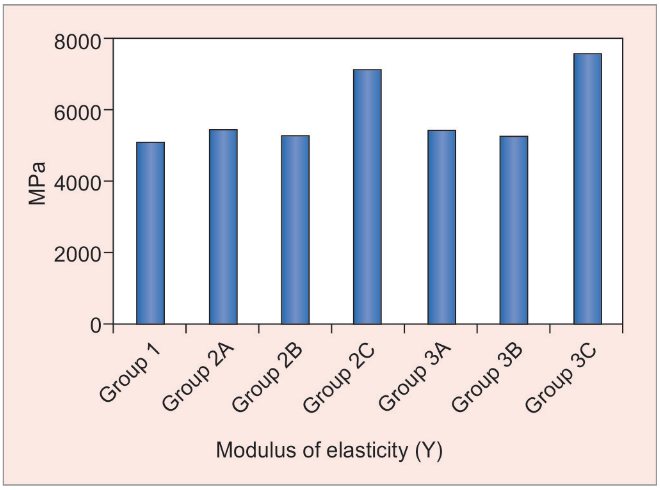
Unpaired t-test; SD: Standard Deviation; NS: Not significant; S: Significant



Graph 1: Comparison of mean impact strength of various groups tested



Graph 2: Comparison of mean transverse strength of various groups tested



Graph 3: Comparison of mean modulus of elasticity of various groups tested

Graph 3 shows the mean modulus values of all study groups. Group IIIC showed highest mean modulus value of 7600.42 MPa. The lowest mean modulus value was of group I (5021.99 MPa).

The results of this study thus show that, in general, test specimens of group III, i.e., silane-treated specimens, showed superior mechanical properties as compared with all other groups. For a particular mechanical property under consideration, the groups have been arranged in the descending order of their values as follows (as seen from Graph 1 to 3 respectively):

- *Impact strength*: Group IIIC>IIIB>IIIA>IIC>IIA>IIB>I
- *Transverse strength*: Group IIIC>IIC>IIIB>IIIA>IIA>IIB>I
- *Modulus of elasticity*: Group IIIC>IIC>IIIA>IIA>IIB>IIIB>I

DISCUSSION

Since the 1930s PMMA has been the most commonly used material in denture fabrication, and it adequately satisfies the esthetic demands. Color stability, ease of manipulation, and polishing make it a desirable material.[1,2]

Despite its wide usage as a main component of denture base polymer for many years, fractures or cracks of this material were observed in clinical use.[4] Most of the denture fractures occur inside the mouth during function, primarily because of resin fatigue. The denture base resin is subjected to various stresses during function; these include compressive, tensile, and shear stresses.[7] Some of the factors responsible for denture fracture include stress intensification, increased ridge resorption leading to an unsupported denture base, deep incisal notching at the labial frena, sharp changes at the contours of the denture base, deep scratches, and induced processing stresses.[8,9]

Various types of fibers including carbon fiber, aramid fiber, polyethylene fiber, and glass fiber have been used as reinforcements. Fibers can be used in three forms, namely, continuous parallel, chopped, and woven.[4,5,10,11] Reinforcement with fibers enhances the mechanical strength characteristics of denture bases, such as transverse strength, ultimate tensile strength, and impact strength. In addition, fiber reinforcement has advantages compared with other reinforcement methods, including improved esthetics, enhanced bonding to the resin matrix, and ease of repair.

Surface treatments, including plasma and silane, have been used to improve the bond strength and wettability between the fibers and PMMA.[1,12,13] Silanes are mainly used as adhesion promoters in ceramic restorations and their repairs with resin composites, fiber-reinforced polymer composites, glassy fillers in resin composite, and to form a durable bond between resin composite to silica-coated metal and metal alloys.

It can be seen from the results of the present study that the addition of fibers does in fact improve the mechanical properties of PMMA resin. Also evident is the fact that, surface treatments, such as silanization can further enhance these properties. In addition, it can also be stated that among all the specimens, silanized woven polyethylene fiber reinforcement of PMMA yielded the most superior results in terms of the mechanical properties tested.

CONCLUSION

Within the limitations of this *in vitro* study and from the results obtained, the following conclusions can be drawn:

- Regardless of the type of fiber used, addition of reinforcing woven fibers to the PMMA resin statistically significantly improved the mechanical properties tested, i.e., impact strength, transverse strength, and modulus of elasticity of the resin when compared with nonreinforced specimens.
- Further, silanization of fibers yielded better mechanical properties over specimens containing nonsilanized fibers.
- Thus, highest values for the above-mentioned mechanical properties were obtained for test specimens of group IIIC, i.e., heat cure denture base resin (Trevalon) with silane-treated, woven polyethylene fiber reinforcement, in comparison to any other group tested.

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