

# Flexural properties and impact strength of denture base resins reinforced with micronized glass flakes

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## Abstract

**Objectives:** To investigate the effect of the addition of glass flakes on physical properties of conventional heat cure denture base resins and to compare it with a high impact strength denture base resin.

**Materials and Methods:** Test specimens were divided into Group 1 - poly (methyl methacrylate) (PMMA) (Trevalon), Group 2 - Trevalon HI, Group 3 -5% glass flake +95% PMMA (Trevalon), Group 4 -10% glass flake +90% PMMA (Trevalon), and Group 5 -20% glass flake +80% PMMA (Trevalon). For glass flake modified groups, part of PMMA (powder) was substituted with the same weight of glass flake (GF003 m) as required, to bring it to 100% powder. Flexural strength was tested using three-point bending test and impact strength with Izod type impact tester.

**Results:** Plain PMMA (Trevalon), showed the highest value of flexural strength followed by Trevalon HI. Trevalon HI (highly cross-linked PMMA) group showed the highest value of impact strength.

**Conclusion:** Flexural strength of unmodified PMMA denture base resin decreases with increase in the concentration of glass flakes. Impact strength does not show any significant change at 5% concentration of glass flakes and impact strength significantly reduces with the addition of glass flakes in 10% and 20%.

**Key Words:** Flexural strength, glass flakes, high impact denture base resin, impact strength, poly (methyl methacrylate)

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## INTRODUCTION

The introduction of a more satisfactory plastic denture base material occurred in 1937 when Dr. Walter Wright described the results of his clinical evaluation of methyl methacrylate resin.<sup>[1]</sup> Poly (methyl methacrylate) (PMMA) has many advantages, particularly its appearance and ease of manipulation, but it has certain poor mechanical properties. Fractures may occur in use because of its unsatisfactory

transverse strength, impact strength or fatigue resistance. Attempts have been made to improve the mechanical properties of acrylic resin by giving maximum bulk to the material in the regions most heavily stressed, by copolymerization and cross-linking, reinforcement with carbon fibers.<sup>[2]</sup> The fracture of acrylic resin dentures is an unresolved problem in removable prosthodontics despite numerous attempts to determine its causes.<sup>[3]</sup>

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Vallittu and Lassila<sup>[4]</sup> studied the effect of different metal and fiber strengtheners on the fracture resistance of PMMA. Different types of commonly used metal wire and glass fiber, as well as carbon and aramid fibers, were used as strengtheners in test specimens. Each metal strengthener had a beneficial effect on the fracture resistance of the PMMA ( $P < 0.001 - 0.01$ ). Some fibers, which were silanized for better adhesion, also had strengthening properties.

A similar study<sup>[5]</sup> tested the effect on the fracture resistance of acrylic resin test specimens with different amounts of glass, carbon, and aramid fibers. The results indicated that an increase in the amount of fibers enhanced the fracture resistance of the test specimens ( $P < 0.001$ ). The scanning electronic microscope micrographs of transverse sections of test polymerized specimens revealed void spaces of different sizes inside the fiber roving. 1% glass fiber<sup>[6,7]</sup> concentration was found to give the best fracture strength and deformation results. Significantly higher glass fiber percentage was found to weaken the resin. Highly cross-linked reline resins<sup>[8]</sup> and the one that contained mainly PMMA and methyl methacrylate had a higher transverse bend strength and modulus of elasticity than the other reline resins.

Another study<sup>[9]</sup> compared the physical properties of a reinforced denture base polymer and concluded that polyethylene and glass reinforced acrylic resin specimens were significantly more resistant to impact. Fiber reinforcement had no significant effect on the transverse strength. Polyethylene reinforcement raised the deflection value. Carbon, thick Kevlar, and polyethylene reinforced specimens showed significantly higher elasticity modulus values.

Franklin *et al.*<sup>[10]</sup> conducted a study to evaluate the effect of a new material - glass flake reinforcement on PMMA denture base resin. Results showed that the addition of glass flake gave up to a 69% increase in fracture toughness compared to plain Trevalon material. The addition of 5% glass flake leads to an improvement in fracture toughness that was statistically significant compared to both plain Trevalon and the 10% and 20% groups.

Hence, a study of glass flakes was conducted to further evaluate its role in modification of physical properties such as flexural and impact strength. A comparison was also made between highly cross-linked denture base resin and glass flakes reinforced conventional heat cure denture base resin.

## MATERIALS AND METHODS

Glass flakes were procured on a sample basis for the study from Glassflakes Ltd., Leeds, Yorkshire, UK.

## Distribution of the specimens in groups

Five experimental groups were considered for the study, as follows:

- Group 1: PMMA (Trevalon)
- Group 2: Trevalon HI
- Group 3: 5% glass flake + 95% PMMA (Trevalon)
- Group 4: 10% glass flake + 90% PMMA (Trevalon)
- Group 5: 20% glass flake + 80% PMMA (Trevalon).

## Preparation of the specimens

### Flexural strength test specimens

Fifty specimens from two denture base resin groups (10 specimens for each of above mentioned five experimental groups) were processed to get specimens having dimensions of 64 mm × 10 mm × 3.3 mm according to ISO 1567:1999.<sup>[11]</sup> A custom-made three-piece stainless steel metal mold having five rectangular cavities of dimensions 64 mm × 10 mm × 3.3 mm in the middle part [Figure 1] was used. This resulted in five specimens at a time.

### Impact strength test specimens

Fifty specimens from two denture base resin groups (10 specimens for each of above mentioned five experimental groups) were processed to get specimens having dimensions of 50 mm × 6 mm × 4 mm as per ISO 1567:1999.<sup>[11]</sup> A custom-made three-piece stainless steel metal mold having five rectangular cavities of dimensions 50 mm × 6 mm × 4 mm in the middle part was used [Figure 2]. In the middle part, at the center of the mold along the thickness 1.2 mm V-shaped projection was incorporated into each cavity to get a V-shaped notch in the specimen. This resulted in five notched specimens at a time.

### Specimen processing

Each metal mold was coated with a thin layer of white petroleum jelly for easy retrieval of the heat polymerized specimens. All samples were prepared using the conventional compression molding technique and processed according to the manufacturer's recommendations as followed:

- Trevalon heat cure acrylic resin powder and liquid was mixed in a proportion as recommended by the manufacturer (24 g–10 ml)



**Figure 1:** Three-piece stainless steel metal mold having five rectangular cavities of dimensions 64 mm × 10 mm × 3.3 mm in the middle part

- Trevalon HI acrylic resin powder and liquid was mixed in a proportion as recommended by the manufacturer (25 g–11 ml).

**Control group (Group 1) processing**

For all groups polymer powder was measured in a digital weighing machine (Afcoset, Model ER182A, Bombay Burmah Trading Corp., Ltd., Mumbai, Maharashtra, India). Monomer Liquid was measured using a pipette. Powder and liquid is mixed in a prescribed ratio in a ceramic jar. When the dough stage was reached, it was kneaded properly and packed into the mold space of the customized mold. Trial closure was done at 1500 Psi, flash removed and final closure was done at 3500 Psi<sup>[12]</sup> under hydraulic bench press (Carlo De Giorgi S.R.L., Italy). The custom made mold was left under the hydraulic bench press for bench curing for 30 min.<sup>[12]</sup> After that screws were tightened at the four corners of the mold to maintain the pressure and then the mold was removed from the hydraulic press. The custom made mold assembly was kept in the water bath and heat cured in a digital acrylic curing unit (Model C-73, Confident Dental Equipment Ltd., India) as recommended by the manufacturer [Table 1]. Slow bench cooling was done, and the specimens were retrieved carefully, wet polished with silicon carbide papers 10 specimens for flexural strength and 10 specimens for impact strength were obtained. These were then stored in distilled water at room temperature for 24 h prior to testing.

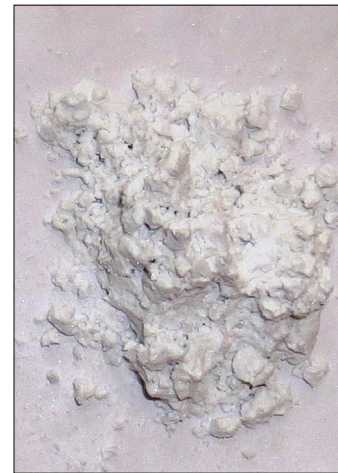
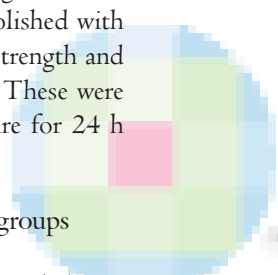
**Glass flake modified poly (methyl methacrylate) groups processing**

For modified groups, part of methyl PMMA (powder) was substituted with the same weight of glass flake (GF003 m) [Figures 3 and 4] as required, to bring it to 100% powder. For example, in 5% glass flake modified PMMA group, 5% w/w (5 g) glass flakes were added to 95% (95 g) PMMA

polymer to bring polymer powder to 100% (100 g) and then mixed with liquid as per manufacturer’s recommendation. Same was done for 10% and 20% glass flake modified PMMA group respectively and processed according to manufacturer’s instruction. Glass flakes were thoroughly mixed with PMMA powder using mixing spatula in a ceramic jar.



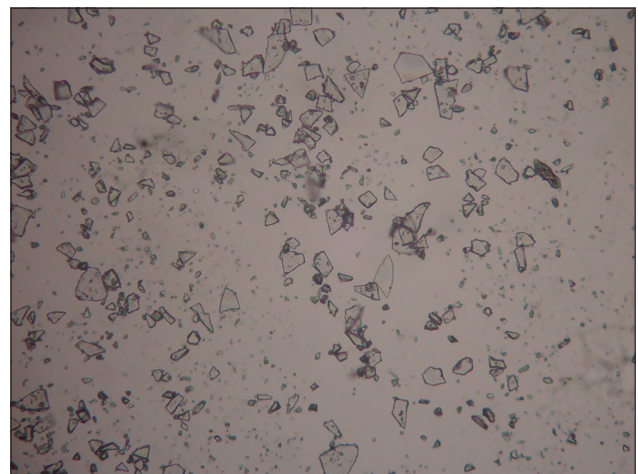
**Figure 2:** Three-piece stainless steel metal mold for impact strength specimen five rectangular cavities (50 mm × 6 mm × 4 mm) in the middle part



**Figure 3:** Glass Flakes-GF003 m

**Table 1: Manufacturer’s recommendation for processing**

Materials	Recommended powder-liquid ratio by manufacturer	Recommended curing methods by the manufacturer
Trevalon	24 g-10 ml	Boil sufficient water to cover the flask into the water. Add 200 ml of cold water for every 2 L of water and leave for 60 min. Apply low heat to maintain the temperature of water at about 68°C for 30 min, bring to boil in not <10 min and boil for a further 20 min. Total curing time 2 h
Trevalon HI	25 g-11 ml	Boil sufficient water to cover the flask into the water. Add 200 ml of cold water for every 2 L of water used and left for 60 min. Apply low heat to maintain the temperature of water at about 68°C for 30 min, bring to boil in not <10 min and boil for a further 20 min. Total curing time 2 h



**Figure 4:** Glass Flakes-GF003 m (40X microscopic view-Olympus CX41RF)

## Testing the specimens

### Flexural strength testing

Flexural strength test was performed according to ISO standard 1567:1999.<sup>[11]</sup> Prior to flexural strength testing, length, width and thickness of each specimen were measured with a digital Vernier caliper (Mitutoyo, Kawasaki, Japan) with a measuring accuracy of  $\pm 0.1$  mm. Ten specimens from each group were subjected to flexural strength testing under three-point loading [Figure 5] with a crosshead speed of 5 mm/min in a universal testing machine (model EZ 20, Lloyd Instruments Ltd., Fareham, UK). The flexural testing device consisted of a central loading plunger and two polished cylindrical supports, 3.2 mm in diameter and 10.5 mm long. The distance between the centers of the supports was 50 mm. This dimension represents the space between the maxillary molars in a complete denture. The load was applied perpendicular to the center of specimen strips until the deviation of the load-deflection curve and fracture of specimen occurred. Flexural strength was calculated by computer system associated with the machine (Nexygen, Lloyd instruments, Fareham, UK) using formula  $FS = 3 FL / (2bd^2)$ , where FS is flexural strength (MPa), F is the load or force at break (N), L is span of specimen between the supports (50 mm), b the width (10 mm), d the thickness (3.3 mm).

### Impact strength testing

Impact strength test was performed according to ISO standard 1567:1999/Amd. 1:2003(E).<sup>[11]</sup> Prior to impact strength testing, length width and thickness of each specimen were measured with a digital Vernier caliper (Mitutoyo, Kawasaki, Japan) with a measuring accuracy of  $\pm 0.1$  mm. Ten specimens from each group were subjected to impact strength testing using digital Izod type impact testing machine (Instron, Ceast 9050 Impact Tester, Italy). For this, a specimen of dimension 50 mm  $\times$  6 mm  $\times$  4 mm having a notch of 1.2 mm depth was kept on the jig in such a way that notch was facing towards the pendulum hammer [Figure 6]. A 5.5 J pendulum hammer was used to impart the energy at the

center of the specimen from the notched side. After deducting the attrition value (0.04 J), the net energy absorbed was obtained for each specimen and impact strength was calculated from the following formula:

$$IS = (\text{Energy absorbed} / [\text{effective width} \times \text{thickness}]) \times 1000$$

IS is impact strength (kJ/m<sup>2</sup>), energy absorbed is net energy absorbed in Joule, effective width is total width minus notch depth (6–1.2 mm = 4.8 mm), the thickness is 4 mm.

### Statistical analysis

Continuous data were presented as mean, standard deviation, confidence interval, median, and quartiles. Significant difference among the groups was obtained by parametric one-way ANOVA test and nonparametric test used was Kruskal–Wallis test. The further *post-hoc* analysis was carried out with Bonferroni test after one-way ANOVA.  $P \leq 0.05$  was considered for statistical significance. Statistical Package for Social Sciences (SPSS 17) and MS-Excel softwares were used to analyze the data.

## RESULTS

Table 2 shows the mean flexural strength (MPa) of specimens among five study groups.

Table 3 shows post-hoc analysis with Bonferroni test.

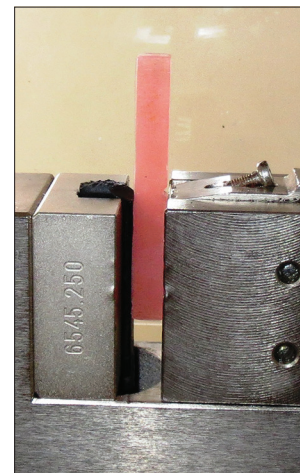
Table 4 shows the mean impact strength of specimens among five study groups.

Table 5 shows post-hoc analysis with Bonferroni test of unmodified Trevalon when compared to Trevalon HI.

Graph 1 represents comparison of mean flexural strength of specimens among five study groups.



**Figure 5:** Specimen subjected to flexural strength testing under three-point loading

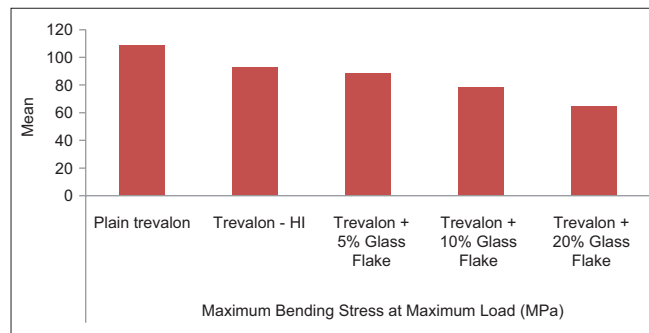


**Figure 6:** Specimen for impact strength testing kept on support arm of the jig for testing

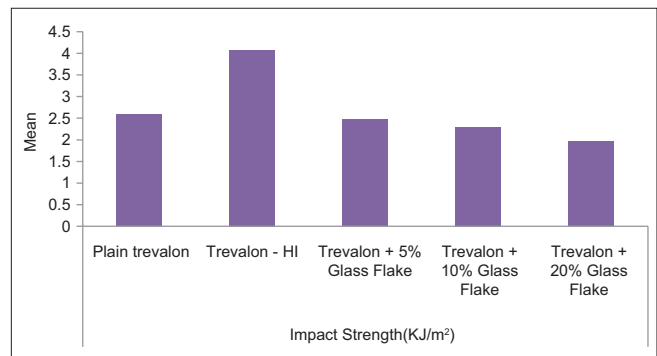
**Table 2: Mean flexural strength of specimens in five study groups (MPa)**

	n	Mean	SD	95% CI		ANOVA value	P
				Lower bound	Upper bound		
Plain Trevalon	10	108.6862	13.1065	99.3104	118.0620	28.820	0.000
Trevalon HI	10	92.8688	5.9257	88.6298	97.1078		<0.001, HS
Trevalon + 5% glass flake	10	88.5784	9.7112	81.6315	95.5254		
Trevalon + 10% glass flake	10	78.4884	10.7974	70.7643	86.2124		
Trevalon + 20% glass flake	10	64.8136	6.8495	59.9138	69.7134		

SD: Standard deviation, CI: Confidence interval, HS: Highly significant



**Graph 1:** Comparison of mean flexural strength of specimens among five study groups



**Graph 2:** Comparison of mean impact strength of specimens among five study groups

Graph 2 represents comparison of mean impact strength of specimens among five study groups.

**DISCUSSION**

Fractures in dentures result from two different types of forces—impact and flexure fatigue. While the impact may fracture dentures when they are dropped, repeated flexing from chewing ultimately fatigues many dentures in the mouth. Impact strength may be defined as the energy required to fracture a material under an impact force.<sup>[13]</sup> It is explained as a materials ability to withstand shock loading as measured by fracturing a specimen. Impact testing measures the energy required to break a specimen by dynamically applying a load.

Flexural strength, (transverse strength/modulus of rupture) is essentially a strength test of a bar supported at each end, or a thin disk supported along a lower support circle, under a static load.<sup>[13]</sup> The flexural strength of a material is a measure of stiffness and resistance to fracture.<sup>[14]</sup> Flexural strength tests were undertaken as these were considered relevant to the loading characteristics of a denture base in a clinical situation. The strength of a material in bending, expressed as the stress on the outermost fibers of a bent test specimen, at the instant of failure.

A study was conducted to evaluate the effect of glass flake reinforcement on PMMA denture base resin. Glass flake was added in 5%, 10% or 20% w/w to Trevalon denture base powder. The material was mixed, flaked, packed, and processed in a manner typical for a denture base material.

Fracture toughness was determined using a double torsion test technique. Results showed that the addition of glass flake gave up to a 69% increase in fracture toughness compared to plain Trevalon material. The addition of 5% glass flake leads to an improvement in fracture toughness that was statistically significant compared to both plain Trevalon and the 10% and 20% groups.

Glass flake (Glassflake Ltd., Leeds, Yorkshire, UK) is a high-aspect-ratio reinforcing additive with many commercial applications. The flake is a modified “C” glass composition and is supplied in a range of three thicknesses: 3.5–5.5, 1.9–2.5, and 1.4–1.9 mm. There are also three particle size distributions to choose from: Unmilled milled and micronized. As yet no literature exists regarding its ability to reinforce acrylic, though the manufacturers claim that its addition to some thermoplastics has resulted in significantly improved flexural modulus and planar reinforcement. They also claim the effect of adding to polytetrafluoroethylene (PTFE) outperformed glass fiber reinforced PTFE in terms of tensile strength, compressive modulus, dimensional stability, wear resistance and creep.

The addition of glass flakes in 10% and 20% w/w makes the consistency of mix thicker and stirring was difficult. The mixture was also found to be tackier to the touch with a higher concentration of glass flakes.<sup>[10]</sup>

Flakes were arranged randomly in the resin matrix as there is no chemical bonding between flakes and the resin. Glass flakes

**Table 3: Comparison of mean flexural strength of specimens among five study groups**

	Mean difference	SE	P
Plain Trevalon			
Trevalon-HI	15.8174	4.3114	0.006 HS
Trevalon + 5% glass flake	20.1078	4.3114	0.000 HS
Trevalon + 10% glass flake	30.1979	4.3114	0.000 HS
Trevalon + 20% glass flake	43.8726	4.3114	0.000 HS
Trevalon-HI			
Trevalon + 5% glass flake	4.2904	4.3114	1.000
Trevalon + 10% glass flake	14.3804	4.3114	0.017 Significant
Trevalon + 20% glass flake	28.0552	4.3114	0.000 HS
Trevalon + 5% glass flake			
Trevalon + 10% glass flake	10.0901	4.3114	0.238
Trevalon + 20% glass flake	23.7648	4.3114	0.000 HS
Trevalon + 10% glass flake			
Trevalon + 20% glass flake	13.6747	4.3114	0.027 Significant

Post-hoc analysis by Bonferroni test. Maximum bending stress at maximum load (MPa). HS: Highly significant, SE: Standard error

**Table 4: Mean impact strength of specimens in five study groups**

	n	Mean	SD	95% CI		ANOVA value	P
				Lower bound	Upper bound		
Plain Trevalon	10	2.582	0.472	2.244	2.920	43.752	0.000 <0.001, HS
Trevalon HI	10	4.077	0.571	3.669	4.485		HS
Trevalon + 5% glass flake	10	2.468	0.350	2.218	2.718		
Trevalon + 10% glass flake	10	2.298	0.174	2.173	2.423		
Trevalon + 20% glass flake	10	1.961	0.246	1.785	2.137		

Impact strength (kJ/m<sup>2</sup>). SD: Standard deviation, CI: Confidence interval, HS: Highly significant

**Table 5: Comparison of mean impact strength of specimens among five study groups**

	Mean difference	SE	P
Plain Trevalon			
Trevalon-HI	-1.49500	0.17465	0.000 HS
Trevalon + 5% glass flake	0.11400	0.17465	1.000 NS
Trevalon + 10% glass flake	0.28400	0.17465	1.000 NS
Trevalon + 20% glass flake	0.62100	0.17465	0.009 HS
Trevalon-HI			
Trevalon + 5% glass flake	1.60900	0.17465	0.000 HS
Trevalon + 10% glass flake	1.77900	0.17465	0.000 HS
Trevalon + 20% glass flake	2.11600	0.17465	0.000 HS
Trevalon + 5% glass flake			
Trevalon + 10% glass flake	0.17000	0.17465	1.000 NS
Trevalon + 20% glass flake	0.50700	0.17465	0.057 NS
Trevalon + 10% glass flake			
Trevalon + 20% glass flake	13.6747	4.3114	0.600 NS

Post-hoc analysis by Bonferroni test. Impact strength (kJ/m<sup>2</sup>). SE: Standard error, HS: Highly significant, NS: Not significant

can be seen on the fractured surface and also areas in the acrylic where flakes have been lost. The flexural strength of a material is a combination of compressive, tensile, and shear strengths. As the tensile and the compressive strength increases, the force required to fracture the material also increases.

Table 2 shows the mean flexural strength (MPa) of specimens among five study groups and plain Trevalon shows highly significant difference as compared to other groups. Table 3 shows post-hoc analysis with Bonferroni test of unmodified Trevalon when compared to Trevalon HI, Trevalon + 5% glass flake, Trevalon + 10% glass flake, Trevalon + 20% glass flake groups shows a mean difference of 15.81, 20.10, 30.19, 43.87 MPa, respectively.  $P < 0.001$  and hence plain Trevalon has highest flexural strength among all five specimen groups. Trevalon + 20% glass flake groups show least flexural strength.

Trevalon HI, when compared to Trevalon + 5% glass flake, shows a mean difference of 4.29 MPa.  $P = 1.000$  and hence its flexural strength is not significantly high as compared to Trevalon + 5% glass flake. When compared with Trevalon + 10% glass flake shows a mean difference of 14.38 MPa.  $P = 0.017$  and hence its flexural strength is significantly high. When compared to Trevalon + 20% glass flake group shows a mean difference of 28.05 MPa.  $P < 0.001$  and hence it has very high flexural strength.

Trevalon + 5% glass flake group, when compared to Trevalon + 10% glass flake, shows a mean difference of 10.09 MPa.  $P = 0.23$  and hence its flexural strength is not significantly high. But on comparison with Trevalon + 20% glass flake group shows mean difference of 23.76 MPa.  $P < 0.001$  and hence it has very high flexural strength.

Trevalon + 10% glass flake group, when compared with Trevalon + 20% glass flake group, shows mean difference of 13.67 MPa.  $P = 0.027$  and hence its flexural strength is significantly high.

Among glass flake modified groups, flexural strength decreases with increase in concentrations of glass flakes.

Table 4 shows the mean impact strength (kJ/m<sup>2</sup>) of specimens among five study groups. Table 5 shows post-hoc analysis with Bonferroni test of unmodified Trevalon when compared to Trevalon HI shows a mean difference of -1.495 kJ/m<sup>2</sup>.  $P < 0.001$  and hence it has high statistical significance. Hence Trevalon HI has very high impact strength as compared to plain Trevalon. Plain Trevalon group, when compared to Trevalon + 5% glass flake, Trevalon + 10% glass flake shows a mean difference of 0.114 and 0.228 kJ/m<sup>2</sup> respectively.  $P = 1.000$  and hence impact strength of plain Trevalon group is not significantly high as compared to Trevalon + 5% glass flake and Trevalon + 10% glass flake groups. Plain Trevalon, when compared to Trevalon + 20% glass flake shows mean

difference of 0.62 kJ/m<sup>2</sup>.  $P=0.009$  hence impact strength of plain Trevalon group is significantly high.

Trevalon HI when compared to Trevalon + 5% glass flake, Trevalon + 10% glass flake, Trevalon + 20% glass flake, shows mean difference of 1.609, 1.779, 2.116 kJ/m<sup>2</sup> respectively.  $P<0.001$  and hence it has highest impact strength as compared to all other groups.

Trevalon + 5% glass flake group when compared to Trevalon + 10% glass flake, Trevalon + 20% glass flake shows mean difference of 0.17 and 0.507 kJ/m<sup>2</sup> respectively.  $P>0.001$  and hence impact strength of Trevalon + 5% glass flake is not significantly high as compared to Trevalon + 10% glass flake and Trevalon + 20% glass flake.

Trevalon + 10% glass flake group, when compared with Trevalon + 20% glass flake group, shows a mean difference of 0.337 kJ/m<sup>2</sup>.  $P=0.600$  and hence impact strength of Trevalon + 10% glass flake group is not significantly high as compared to Trevalon + 20% glass flake group. Hence, Trevalon HI shows the highest impact strength while Trevalon + 20% glass flake group shows least impact strength among all five specimen groups.

Among glass flake modified groups, impact strength decreases with increase in concentrations of glass flakes.

The addition of rubber to PMMA produces a matrix of PMMA within which is dispersed an interpenetrating network of rubber and PMMA. If a crack develops in a rubber reinforced acrylic resin then it will propagate through the PMMA but will decelerate at the rubber interface. The rubber reinforced acrylic resins are believed to absorb greater amounts of energy at higher strain rate before fracture than the conventional acrylic resins and, therefore, offer an improved impact strength. A popular concept is that the rubber particles cause dispersion or deflection of the cracks.<sup>[14]</sup>

## CONCLUSION

From this study, it can be concluded that flexural strength of unmodified PMMA denture base resin decreases with increase in the concentration of glass flakes while the impact strength does not show any significant change at 5% concentration of glass flakes and impact strength significantly reduced with the

addition of glass flakes in 10% and 20%. Twenty percentage glass flake modified PMMA group showed the least value of flexural and impact strength. Trevalon HI has highest impact strength as compared to all other groups.

Hence, addition of glass flakes to improve physical properties<sup>[10]</sup> is contradicted by this study and adds to the drawback of using this material in dentistry.

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Nil.

## Conflicts of interest

There are no conflicts of interest.

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