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Effect of Incorporating Two Different Woven Glass Fiber Reinforcent on the Flexural Strength of Acrylic Denture Base Materials

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ABSTRACT

Polymethyl methacrylate is widely used for denture base fabrication due to its pleasing aesthetics, ease of manipulation and cost-effectiveness compared to the metallic denture base materials and fixed prostheses. However, polymethyl methacrylate (PMMA) prostheses are prone to fracture due to their poor mechanical properties and degrade over time due to water sorption.

This study aimed at evaluating the effect of adding woven E-glass fibers on the flexural strength of Meadway heat cure Supercure denture base material. This was done by mutually comparing the flexural strength of unreinforced and StickNet™ (StickTech, Finland) and industrial (Iqbal Sons, Pakistan) glass fiber (GF) reinforced PMMA specimens respectively. Specimen preparation and testing was performed under the guidelines of ISO 20795-1 for DBM. Mechanical testing was performed using a Universal Testing Machine.

The results indicated that Glass fiber reinforcement resulted in significant enhancement in mechanical properties of PMMA. StickNETTM reinforced specimens showed highest flexural strength and fracture toughness, followed by the Industrial GF reinforced and unreinforced PMMA. This study clearly demonstrates positive improvement in strength and durability of PMMA after reinforcement with woven GFs. Future studies should focus on finding means to decrease the relatively higher solubility of glass fiber reinforced PMMA specimens in comparison to unreinforced PMMA.

INTRODUCTION

Regardless of their beneficial aspects, the PMMA based dentures are prone to fracture [1-4] under excessive masticatory forces or when dropped accidently [2,5,6]. In addition, they degrade with the passage of time owing to water sorption (WS) which causes subsequent hydrolysis [7-9] and leading to a resultant decrease in their physico-mechanical properties [8,10].

Various techniques have been devised to improve mechanical properties of the PMMA (DBM). One technique is to replace PMMA by another denture base material (e.g., Polyamides, Epoxy resins) [11], or the addition of a copolymer of rubber (e.g., butadiene styrene) within PMMA to increase its impact strength [3]. Other options include the use of metallic wires [1,11,12] or fiber reinforcement to strengthen the PMMA dentures.

Metallic wires substantially strengthen the acrylic dentures yet their incorporation into PMMA leads to poor aesthetics [13] and generation of areas of stress concentration and voids which can weaken instead of strengthening the denture base polymer [11]. Carbon/graphite fibers (CFs) [11,14] when incorporated into PMMA provide a substantial increase in the flexural and impact strengths [15] of acrylic resins. However, due to their grey color, they impart poor aesthetics to the dentures and are hence not commonly used for denture reinforcement [16]. Ultra-high molecular weight polyethylene (UHMWPE) [1,11,16,17] fibers are white in color and have shown an increase the impact strength and Young's modulus of the acrylic resins. However, these fibers require special pre-treatment (e.g. plasma treatment) in order to create a bond between the fibers and PMMA resin [15]. The aramid fibers (AFs) substantially enhance the tensile strength and toughness of PMMA based dentures [16] but they possess a yellow color and are also difficult to incorporate into PMMA. As a result, they have a limited use in clinical dentistry [16].

Recently, a great emphasis has been placed on the use of glass fibers (GFs) for denture base reinforcement [13,16,18-20,21-24]. Various studies have documented the beneficial effect of GFs on the mechanical properties of the denture resins [1,13,16,21,25,26]. GFs

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are highly aesthetic and possess excellent mechanical properties. As a result, they are a prime candidate for denture reinforcement [16]. In addition, GFs are highly amenable to bond formation with the PMMA resins when treated with a suitable silane coupling agent [1,27] such as Trimethoxysilylpropylmethacrylate silane (MPS).

Various forms and orientations of glass fibers have been experimented to reinforce acrylic resins. Mainly electrical (E-type) or Silica (S-type) glass fibers are used for this purpose [28]. Fibers that run in a single direction are called unidirectional fibers, while those incorporated in two or more directions are called multidirectional or woven fibers [22]. Increased impact [15,19,29] and fatigue strengths [30] have been reported with the use of woven GFs for the reinforcement of PMMA DBM, compared to the unidirectional fibers. However, a few studies have shown that higher mechanical strength is achieved by using unidirectional GFs compared to woven GFs [29,31,32]. However, they strengthen only in a single direction and their uniform incorporation distribution within PMMA is quiet difficult [33].

Apart from the obvious benefits of using fiber reinforced PMMA dentures, their high cost and difficulty of incorporation into PMMA limit their frequent clinical use, especially in the case of short and randomly oriented GFs which tend to "clump" [25,34] within the denture and can cause a reduction in the strength of the prosthesis. This has been overcome by the use of woven fibers that are available in the form of a mesh or network [35,36].

The problem of high cost of commercial dental GFs can be addressed by the use of industrial GFs that are not only cheaper but are also comparable in mechanical properties with dental grade GFs (StickNet™). Unfortunately, very few studies have focused on the use of industrial grade GFs [4,36-38]; for the reinforcement of dentures and most of these studies have only focused on the evaluation of mechanical properties of these reinforced DBM. Testing only some of the properties does not provide sufficient information about all the relevant properties of GF reinforced PMMA. Various other mechanical properties such as flexural strength (FS), fracture toughness (FT) and physical properties such as water sorption (WS) and solubility are essential to characterize PMMA dentures base materials that are reinforced with GFs.

The aim of this study was to investigate the effect of adding two different types of woven GFs on the flexural strength of PMMA denture base materials. This was done by mutually comparing the flexural strength of the GF reinforced PMMA as well as with the unreinforced PMMA.

MATERIALS AND METHODS

Materials

The components used for preparing PMMA specimens **(Table 1)**. Control specimens consisted only of PMMA, while the Exp specimens (Exp-I and Exp-II) contained PMMA that was reinforced with woven glass fibers.

Table 1. Components used in this study for preparing PMMA (control) specimens.

Material(s)		Brand Name	Manufacturer	Product code
Polymethyl methacrylate (PMMA)	Powder	Meadway Heat Cure Supercure	MR. Dental, UK	297
	Liquid	Meadway Universal monomer	MR. Dental, UK	20185
Type III Dental Stone	BEGO stone Plus		BEGO, Germany	54812
Sodium alginate separating Medium	ISO-8		BEGO, USA	52700

Additional components that were used for the preparation of glass fiber reinforced experimental PMMA (Exp-I and Exp-II) specimens, while the experimental component of this study are presented in **Table 2.**

Table 2. 3 PMMA (control and experimental) used in this study.

Control	Exp-I	Exp-II
PMMA	PMMA	PMMA
	StickNET	Industrial GFs
		Silane treatment

METHODS

Preparation and testing of the specimens in this study was carried out according to the guidelines of ISO 201795-1 for denture base materials **(Table 3)**.

Table 3. 2 Components used to prepare experimental materials (Exp-I and Exp-II) in this study of ISO 201795-1 for denture base materials.

No	Component Category	Type of Component / chemical nature	Brand name	Manufacturer
1	Glass fibers	Woven E-glass fibers	StickNet™	StickTech, Finland
			-	Iqbal Sons, Pakistan
2	Coupling Agent	98% (Trimethoxysilyl)propyl methacrylate silane (MPS)	Silane (A174)	Sigma Aldrich, USA

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Specimen Preparation for Measurement of Flexural Strength

A gypsum mold was prepared by investing a highly polished stainless steel (SS) plate measuring 65 mm (length) × 40 mm (width) × 3.5 mm (thickness) into a dental flask filled with freshly mixed type III dental gypsum, which was mixed according to the manufacturers recommendations. After the gypsum had set the SS plate was removed and a thin layer of an alginate separating medium was applied over the mold. Heat cured Meadway Supercure acrylic denture base powder and liquid components were mixed at a powder-liquid ratio of 2.35 g/mL and incorporated into the mold when the mixed resin had attained the doughy stage. Afterwards, both the halves of the flask were closed and the flask was kept under a pressure of 200 bars for 30 minutes by using a hydraulic press **(Figure 1)**.

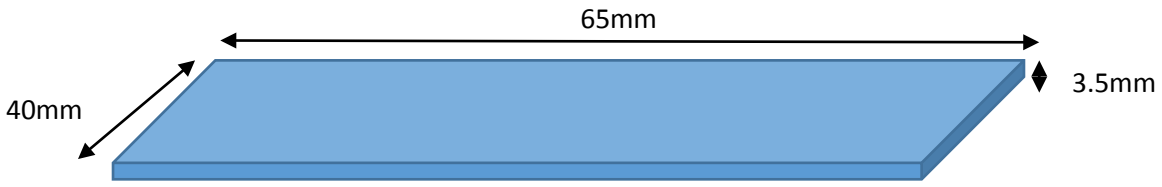

Figure 1. Schematic representation of stainless steel plate dimensions used to fabricate acrylic specimens for testing flexural strength in this study.

Subsequently, the flask was removed from the press and acrylic resin cured in a heat curing apparatus at 70°C for 2 hours followed by terminal boiling at 100°C for 1 hour. The cured acrylic plate was then retrieved from the flask and four specimens (65 × 10 × 3.3 mm) were obtained from each plate by cutting with a slow speed diamond saw (Isomet 4000 Linear) at an RPM of 600/min followed by grinding and polishing with a 600-grit metallographic paper under constant water cooling. Eight specimens (n=8) were prepared for each group.

In case of the control group, no fiber addition or silane treatment was performed while in case of Exp (Exp-I and Exp-II), woven E-glass fibers (i.e., StickNET and Industrial GFs respectively) were sandwiched within the acrylic resin dough.

The Volume of the industrial fibers was adjusted such that it was equal to that of StickNet™ GFs (Exp-I). Sheets of both the StickNET (Exp-I) and industrial fibers (Exp-II) were cut to dimensions of 60 mm length and 40 mm width. Thickness of the StickNET fibers was 0.06 mm while that of the industrial fibers was 0.02 mm. Hence, 3 layers of industrial fibers were used and one sheet of StickNET fibers was used to keep equal amount of volume for both the fibers.

Silane Treatment and Fiber Pre-Impregnation of Industrial Fibers

Unlike the StickNet glass fibers (GFs), which have already been treated with a silane coupling agent by the manufacturer, the industrial GFs require silane treatment prior to use. The Industrial GFs were washed in distilled water to remove surface impurities, followed by purification by immersing them in 1.6 M Hydrochloric acid for 60 seconds. Afterwards, the GFs were again washed with distilled water to remove any acidic remnants, and left in open air for 72 hours. Next, an 8% solution (v/v) of the silane coupling agent in 98% ethyl alcohol was prepared. A few drops of hydrochloric acid were also incorporated into the solution which acted as a catalyst for the reaction.

The GFs were then immersed into the solution for 24 hours so that all the ethanol evaporated out of the solution and a thin coating of the silane coupling agent remained on the fibers. The silane monomer coating on the GFs was cured by heating the fibers at 100°C for 24 hours. Afterwards, the fibers were washed with distilled water to remove any impurities, and now the fibers were ready for incorporation within the heat cured denture base PMMA.

Resin Impregnation of Glass Fibers

StickNET and Industrial glass fibers were immersed for 30 seconds in syrup of PMMA having a Powder/liquid ratio of 10/8. After removal from the resin syrup, they were immediately incorporated within the resin dough prepared for the fabrication of PMMA specimens.

Measurement of Flexural Strength

After the preparation of specimens (65 × 10 × 3.3 mm) for testing flexural strength, they were stored in a water bath at 37°C for 48 hours days prior to testing. When testing had to be performed, specimens were taken out of the water bath and placed onto the supports of the universal testing machine (AG20KNX Plus, Shimadzu, Japan). A three point bending test was then performed such that the distance between both the supports was 50 mm and load cell of 20 KN was used. A force applied at the crosshead was increased at a speed of 5 mm/min till the specimens fractured. The maximum force (F) required to fracture the specimens was measured in Newtons (N) from the Universal Testing Machine (UTM) and flexural strength, in Megapascal (MPa), of the specimens was calculated using the following formula, as directed by ISO 20795 1for DBM [39]:

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$$\sigma = \frac{3Fl}{2bh^2}$$

Where, σ=Flexural strength (Pa)

F=maximum load (Newtons) exerted on the sample

l=distance between supports of the UTM (mm)

b=width of the specimen (mm)

h=height of the specimen (mm)

Statistical Analysis

Statistical analysis was performed on Special Package for Social Sciences version 22, (SPSS 22). The means of flexural strength, fracture toughness, water sorption and solubility were analyzed by using a one way analysis of variance (ANOVA) test and by applying the post hoc Tukey test to compare the means of results between the groups.

RESULTS

Flexural Strength

Figure 2 shows the mean flexural strength (FS) for all the control and Exp (Exp-I and Exp-II) PMMA denture base materials. One way Analysis of Variance (ANOVA) revealed significant differences ($p<0.01$) between the FS of all the three groups of materials tested. The data was further analyzed by using a post hoc test (Tukey's Honest Significant Difference [HSD] test) to determine which means of the tested specimens were significantly different from each other. Among the tested specimens, StickNET reinforced PMMA specimens (Exp-I) showed the highest FS. The Industrial GF reinforced specimens (Exp-II) showed a FS which was significantly lower than Exp-I, but it was significantly greater than the unreinforced specimens (control). The maximum and minimum values for flexural strength for all the groups of tested specimens. Maximum FS value for control specimens was 85.3 ± 3.14 MPa, while the maximum FS for the Exp specimens (Exp-I and Exp-II) was found to be 135.48 ± 8.22 and 102.76 ± 6.81 MPa respectively.

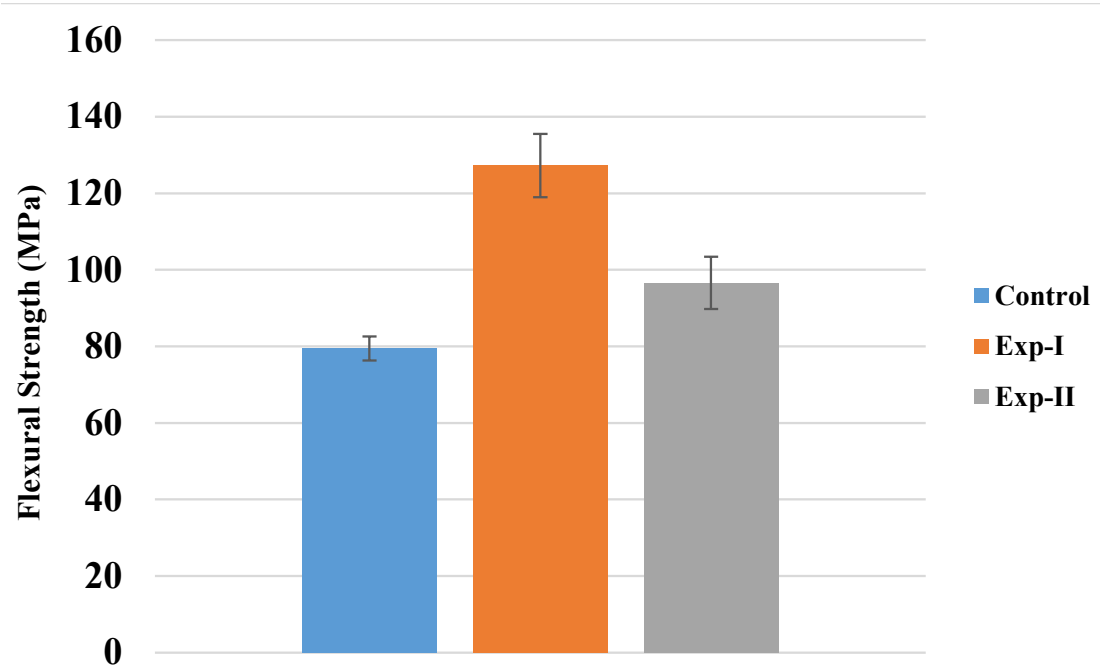

Figure 2. Mean values of flexural strength of unreinforced (control) and glass fiber reinforced (Exp-I and Exp-II) PMMA specimens.

Similarly, the minimum FS value for control PMMA specimens was 74.95 ± 3.14 MPa, while that of Exp PMMA specimens (Exp-I and Exp-II) was 116.37 ± 8.22 MPa and 83.27 ± 6.81 MPa respectively **(Figure 3)**.

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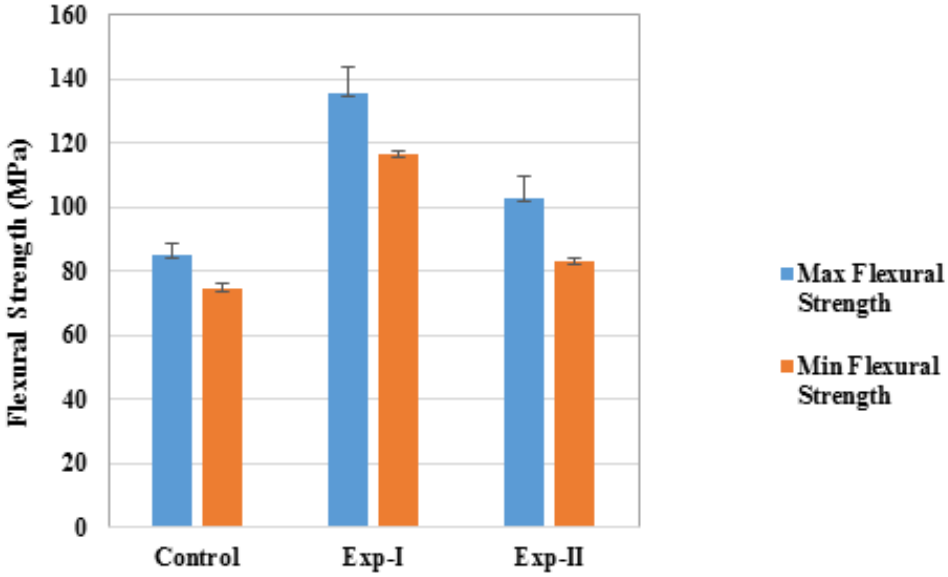

Figure 3. Minimum and maximum values of flexural strengths for the unreinforced and glass fiber reinforced PMMA specimens.

The SEM images of the transverse sections of fractured specimens at 17X showed that all types of specimens demonstrated a brittle type of fracture and a clear tensile and compressive fracture zones were visible **(Figures 3 and 4)**.

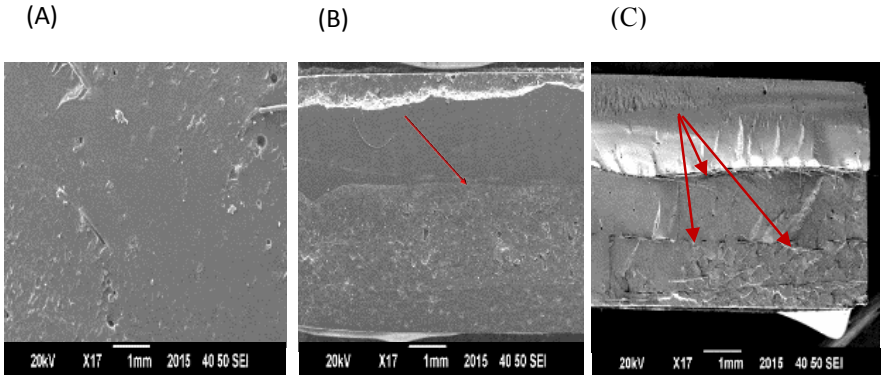

Figure 4. SEM image of fractured specimens (A) control, (B) Exp-I, (C) Exp-II, Images taken at 17X magnification.

DISCUSSION

Flexural strength (FS) of a material refers to its ability to resist fracture when a bending force is applied over it. Therefore, to ensure the success and longevity of prosthodontic appliances, they must possess high FS. In this study, the StickNET reinforced PMMA specimens (Exp-I) had an average FS of 127.22 ± 8.22 MPa, the industrial GF reinforced specimens (Exp-II) showed an average FS of 96.56 ± 6.81 MPa while the unreinforced PMMA control specimens had an average FS of 79.45 ± 3.14 MPa.

These results indicated that the incorporation of 1.7% (v/v) StickNET and Industrial GFs resulted in 57% and 20% increase in the FS of PMMA denture base resins respectively. These results are supported by previous studies [15,31,32,40,41] which concluded that there was an increase in the FS of PMMA when it was reinforced with woven GFs. In contrast, Koroglu et al. showed that there was a decrease in the FS of PMMA after the incorporation of StickNET GFs. However, the difference in their results might have been due to improper impregnation or placement of the fibers as the adhesion of the fibers to PMMA matrix was not assessed in the study.

In this study, a significant difference was observed between the FS of the Exp-I (122.34 ± 5.95 MPa) and Exp-II (92.94 ± 1.45 MPa) PMMA. Exp-II PMMA specimens demonstrated lower FS in comparison to Exp-I. However, FS of Exp-II was significantly higher than the control specimens, which indicates the beneficial effect of GF reinforcement on flexural strength of PMMA. The reason for the lower FS of Industrial fibers may be due to the imperfection in technique of the resin impregnation of the GFs which is also evident on the SEM images of the fractured specimens.

Since the concentration of the GFs was balanced by equalizing the volume of both types of GFs, it might be possible that the Industrial GFs may contain lesser concentration by mass of Industrial GFs in comparison to the StickNET fibers. In this study, imaging of the GFs under the scanning electron microscope (SEM) revealed that the mean thickness of a single sheet of the

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industrial GFs used in this study was 0.02 mm while that of the StickNET GFs was 0.06 mm. Similarly, the thickness of individual's fibers of the Industrial glass was found to be 5.2 µm while that of the StickNET GFs was 6.3 µm which implies that the industrial GFs were less dense than the StickNET fibers, which might have been the reason behind the inferior properties of Exp-II in comparison to Exp-I PMMA specimens.

Interestingly, it was also observed that there was a negligible effect of the number of layers of fiber sheets used for the reinforcement on the FS of PMMA [16]. The thickness of a single sheet of StickNET GFs is 0.06 mm, while that of the industrial GFs was 0.02 mm. As a result, 3 layers of the industrial GFs were used to equalize the volume of the StickNET GF sheets. Despite the use of multiple sheets in industrial GF reinforced PMMA specimens, the FS of these specimens was still lesser than the StickNET reinforced PMMA specimens that were reinforced with only a single fiber sheet. However, difference between the FSs of both the specimens might also have been due to the difference in the composition and mechanical properties of the industrial GFs in comparison to the StickNET GFs.

CONCLUSION

1. Incorporation of woven E-glass fibers within heat cured PMMA denture base materials results in a significant enhancement in the flexural strength and fracture toughness of this material.
2. Industrial GFs are a cheaper and convenient solution for enhancing the flexural strength of PMMA denture base materials, thereby reducing their fracture and need for replacement or repair.
3. Incorporation of woven GFs in the fracture prone zones of PMMA dentures can significantly reduce the fracture incidence of PMMA dentures.
4. Future studies should focus on the biocompatibility and repair flexural strength of PMMA after reinforcement with woven GFs.

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