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Original article

Evaluation of flexural strength of resin interim restorations impregnated with various types of silane treated and untreated glass fibres

Sqn Ldr K.S. Naveen^{a,*}, Maj Gen J.P. Singh^b, Col M. Viswambaran^c,
Col R.K. Dhiman^d

^a Resident, Division of Prosthodontics, Department of Dental Surgery, Armed Forces Medical College, Pune 411040, India

^b Commandant, Command Dental Adviser, CH (EC), Kolkata, India

^c Commanding Officer, Military Dental Centre, Jabalpur 482001, (MP), India

^d Associate Professor, Division of Prosthodontics, Department of Dental Surgery, Armed Forces Medical College, Pune 411040, India

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ABSTRACT

Background: Flexural strength is an important mechanical property that determines the long-term prognosis of interim restorations. Studies are lacking regarding the effect of silanation of the various types of glass fibre impregnation on the flexural strength of resin interim restorations.

Methods: A customized metal die was milled to simulate the prepared abutments of a three-unit fixed dental prosthesis. A total of seventy five samples of interim fixed dental prostheses were prepared using autopolymerizing tooth colour acrylic resin. Unidirectional and woven forms of glass fibres (Stick and Stick Net), which were silane treated and untreated were used to reinforce the resin matrix. Fifteen samples were prepared for each group along with unreinforced group serving as control. The flexural strength was evaluated with universal testing machine.

Results: The means and standard deviations of flexural strength for different groups were 13.90 ± 2.96 (control), 61.58 ± 5.26 (unidirectional fibres), 30.89 ± 3.60 (woven fibres), 112.05 ± 5.51 (silane treated unidirectional fibres) and 73.85 ± 4.10 (silane treated woven fibres) respectively. The mean flexural strength of silane treated unidirectional fibres (112.05 MPa) was highest and statistically highly significant ($P < 0.0001$) compared to all other groups.

Conclusions: Within the limitations of the current study, flexural strength of the reinforced PMMA interim fixed dental prosthesis was significantly higher ($P < 0.0001$) when compared to the unreinforced PMMA interim fixed dental prosthesis. The use of silane treated unidirectional glass fibres is an effective method of reinforcing interim fixed restorations made of PMMA resins.

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* Corresponding author. Tel.: +91 9689095740 (mobile).

E-mail address: dmaveenks@gmail.com (K.S. Naveen).

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Introduction

Interim or provisional restorations are integral part of fixed prosthodontic practice. Generally, there is a waiting period before the delivery of the definitive prosthesis due to the extensive laboratory procedures involved. An interim restoration made up of either acrylic or composite is provided to the patient during this interim phase. The interim restorations should fulfil biological, mechanical, and aesthetic requirements to be considered successful.¹ Achieving these requirements depends on important properties of resins, including polymerization shrinkage, wear resistance, colour stability, compressive strength, tensile strength and flexural strength. Flexural strength is defined as force per unit area at the point of fracture of a test specimen subjected to flexural loading². The flexural strength of interim prostheses is a critical property, particularly in long-span interim prostheses with short height pontics and connectors and also when the patient exhibits parafunctional habits such as bruxism or clenching. Flexural strength is also important, when these restorations are worn over a long period of time to assess the results of periodontal, endodontic, temporomandibular joint dysfunction therapies and during the restorative phase of implant reconstructive procedures. The maintenance of these interim prostheses can present considerable difficulty for both the patient and the dentist. Not only can repair procedures be time consuming, but also breakage of these restorations can lead to tooth movement and functional and aesthetic problems.^{3,4}

The most commonly used material for fabricating these interim restorations is polymethyl methacrylate. The lack of sufficient strength of this material has led to various methods of reinforcement of the interim restorations fabricated from polymethyl methacrylate. The concept of using fibres to reinforce an interim restoration appears to have an acceptable success rate. Investigations have shown that carbon fibres produce a significant increase in the flexural strength of polymers; however, their black colour limits their use for provisional restoration.⁵ Surface treatment of the fibres used for reinforcement has shown conflicting results. The transverse strength did not improve significantly when polyethylene fibres without the surface treatment were used. However, the use of plasma-treated polyethylene fibres showed a significant increase in strength.⁶ Glass fibres have been used in either continuous or woven form as a strengthening material.⁷ Use of silanised glass fibres as a means of reinforcement is promising due to their good adhesion to the polymer matrix, high aesthetic quality, and increased strength of the resulting composite.⁸ Other researches have shown that the position, quantity, and direction of the fibres and the degree of adhesion between the fibres and the polymer affect the degree of reinforcement.^{9–16}

There has been a lack of concrete information regarding the quantitative analysis of the effect of various types of silane treated and untreated glass fibre impregnation on the flexural strength of resin matrix. The purpose of this study was to analyze the change in flexural strength of the provisional restorations by impregnation with various types of silane treated and untreated glass fibres.

Materials and method

A metal die of brass (Fig. 1) was made to simulate the prepared abutments of a three-unit fixed dental prosthesis (FDP). A custom impression tray was fabricated to fit the metal die. A wax pattern was fabricated using modelling wax (S-U-Modelling wax, Schuler-Dental, Germany) for a three-unit fixed dental prosthesis on the metal master die. The wax pattern had a connector dimension of 5 mm height $\times$ 5 mm width with a sanitary pontic having a clearly demarcated central fossa (to be later used for placing the steel ball for testing the flexural strength). The three-unit metal FDP casting was done with Co–Cr alloy (Wironium plus, Bego, Germany) using an induction casting machine (Fig. 2). The three-unit metal fixed dental prosthesis was placed on the master die. Block-out of all the dead space below the three-unit fixed dental prosthesis on the master die was carried out with putty rubber base impression material to prevent locking of the main impression material and to ensure standardization of the under surface of the pontic and the connector dimensions. Impression was made with polyvinyl siloxane impression material (Betasil®, Mueller-Omicron Dental, Germany) using the custom metal tray to yield a master index to be later used for fabrication of interim prosthesis samples in resin. Sluiceways were provided in the index for the escape of excess resin material.

The tooth coloured polymethyl methacrylate (PMMA) acrylic resin (DPI–Dental Products of India Ltd, Mumbai, India) was used to make the interim prosthesis samples. The samples were fabricated with the polymer to monomer ratio as per manufacturers recommended ratio of 3:1 by volume. A total of seventy five samples were made and divided into five groups of 15 samples each, depending on the method of reinforcement, i.e. Group 1: unreinforced group; Group 2: reinforced with Untreated unidirectional glass fibres; Group 3: reinforced with Untreated woven glass fibres; Group 4: reinforced with Silane treated unidirectional glass fibres; and Group 5: reinforced with Silane treated woven glass fibres.

For Group-1 the resin mixture was poured into the master index which was then placed on the metal die under pressure using a metal clamp for 15 min. The sample was retrieved from the master index and the excess was trimmed and

Fig. 1 – Master die.

Fig. 2 – Three-unit metal FPD.

polished. For Group-2, the unidirectional glass fibres (Stick® E glass fibres, Stick Tech Ltd., Turku, Finland) were precut with scissors to the size of 20 mm length, 2 mm of width and 1 mm in thickness. The resin mixture was first poured up to one third of the master index. A single piece of glass fibre of precut dimensions was carefully placed with the tweezers in the middle of the index at the connector region extending from one abutment to another. The remaining two thirds of the index was filled with PMMA resin mixture. The tray with the die was then pressed with a clamp to ensure that the tray remained completely seated throughout the polymerization procedure under firm uniform pressure for 15 min. The sample was retrieved from the index and final finishing and polishing was done. For Group-3, the woven glass fibres (StickNet® E glass fibres, Stick Tech Ltd., Turku, Finland) were precut with scissors to the size of 20 mm length, 2 mm wide and 1 mm thickness. Steps similar to Group-2 were followed to fabricate the samples.

For Group-4 and Group-5, the pre-cut fibres of the same dimensions as in Group-2 & 3 were dipped in silane coupling agent (Silano®, Angelus, Brazil) for 5 min (Fig. 3). After air drying, the fibres were immediately placed at the site of reinforcement. The samples were fabricated using similar procedure as described for Group-2. All the samples (Fig. 4) were stored in water for 24 h for complete elimination of the residual monomer prior to testing.

Fig. 3 – Treating the fibres with silane coupling agent.

Fig. 4 – Samples of all groups.

The samples were tested for flexural strength using three-point bending test with the help of a Universal Testing Machine (Instron Corp. 4204). A three-point bending test was done to evaluate the flexural strength. Each interim FDP sample was firmly seated with hand pressure on the brass master die and held on the Universal Testing Machine (Instron Corp. 4204). It was ensured that each sample fitted snugly on the die with no rocking of the sample. The test samples were loaded with a 6.36 mm diameter steel ball placed in the specifically demarcated region of the sample, i.e. the central fossa of the pontic with a crosshead speed of 5 mm/min till the fracture occurred (Fig. 5). The load resulting in fracture of the sample was recorded. Flexural strength was automatically calculated by the equipment software (the reading displayed on the system) using the formula: $FS = \frac{3WL}{2bd^2}$, where FS = flexural strength (MPa or MN/m²), W = maximum load before fracture (N), L = distance between the supports (mm), b = width of the samples (mm) and d = thickness of the samples (mm).

All the data collected was subjected to statistical analysis. The mean and standard deviation for each group were determined. The data was analyzed for differences using one way ANOVA test to determine statistically significant (P value ≤ 0.0001) differences between the means. The inter group comparisons were made using Tukey's pairwise comparison test.

Results

The mean and standard deviations of flexural strength for different groups are presented in Table 1. The comparisons between the mean flexural strength among all the groups are depicted in Graph 1. The mean and standard deviation values of flexural strength were subjected to one way ANOVA test to find the statistical significance. Table 2 shows the one way ANOVA test values to check the statistical significance between the mean flexural strength of each group. On analyzing the results of the ANOVA test, it was seen that the flexural strength differed significantly between the groups ($F = 1112.76$, $P = 0.0001$ very very highly significant).

Fig. 5 – Testing the sample.

Since ANOVA test reflected significant differences in the mean performance levels of the five groups, the Tukey's pairwise comparison test was used for pairwise comparison of difference amongst means of each group. The Tukey's significance differences (TSD) were checked at 4.56 and 5.52, which is equivalent to P value 0.05 and 0.01 respectively. The Tukey's pairwise comparisons for difference between means of each group are presented in Table 3. The comparison of the difference means between Group-1 and the all other Groups were highly significant with TSD value ≥ 0.01 . The comparison between Group-2–Group-4 and Group-3–Group-5 was highly significant (TSD ≥ 0.01). The results of Tukey's pairwise test showed statistically highly significant differences with respect to mean flexural strength (MPa) within each group.

The mean flexural strength of Group-2 (61.58 MPa) improved very significantly compared to Group-4 (112.05 MPa), when the unidirectional glass fibres were treated with silane coupling agent. The flexural strength of Group-3 (30.89 MPa) increased significantly compared to Group 5 (73.85 MPa), when the woven form glass fibres were treated with silane coupling agent.

Discussion

Interim fixed dental prostheses are subjected to flexure under function and the fracture can occur under occlusal load resulting in failure of the restoration. Therefore, the interim

Table 1 – Comparison of flexural strength in MPa among groups.

Method of reinforcement	Flexural strength
	Mean $\pm$ SD ($n = 15$)
Group 1: unreinforced (control)	13.90 $\pm$ 2.96
Group 2: unidirectional glass fibres	61.58 $\pm$ 5.26
Group 3: woven glass fibres	30.89 $\pm$ 3.60
Group 4: silane treated unidirectional glass fibres	112.05 $\pm$ 5.51
Group 5: silane treated woven glass fibres	73.85 $\pm$ 4.10

Graph 1 – Box plot comparison of the mean flexural strength (MPa).

restorations should have adequate flexural strength to withstand the masticatory forces. Although, restorations should be properly designed to avoid it, they may nevertheless fail leading to discomfort and time loss. The flexural strength of interim restoration materials is one of the important factors and should be considered prior to selecting a provisional restorative material for the clinical success.³

With the recent introduction of improved fibre reinforcing materials, the concept of using glass fibres to reinforce interim restorative materials has gained popularity. Glass fibres were tested as reinforcement for PMMA denture base. The various glass fibres routinely used for reinforcements are unidirectional, woven and free form.^{7,10,15,17} Many studies have investigated the transverse strength of the glass fibre-PMMA composite.¹⁶ It has been observed that the glass fibre incorporated into PMMA considerably increased the transverse strength of PMMA, especially if the concentration of fibres in the PMMA matrix is sufficiently high.

The adhesion between fibres and polymer matrix plays an important role in transferring the stress from the matrix to the fibres. The chemical bond between the polymer and the fibres should ideally be of a covalent nature. Silane coupling agents have been used successfully to improve the adhesion between polymers and glass fibres.⁸ The function of the silane coupling agent is to form a siloxane bridge by a condensation reaction of silanol groups and the silica surface of the glass fibre. Silanes can also be used for improving the adhesion between fibres other than glass fibre and polymer. Here, the silanes on the surface of the fibres improve the physical adsorption of polymer to the micro irregularities of the surface by improving the surface wettability.¹⁸

Table 2 – One way ANOVA: flexural strength versus groups.

Source	DF	Sum of square	Mean square	F	P
Group	4	87,960.5	21,990.1	1112.76	0.0001
Error	70	1383.3	19.8		
Total	74	89,343.8			

Table 3 – Tukey's pairwise comparisons.

GROUPS	Group-1	Group-2	Group-3	Group-4
Group-2	47.68**	—	—	—
Group-3	16.79**	30.69**	—	—
Group-4	98.15**	50.47**	81.16**	—
Group-5	59.95**	12.27**	42.96**	38.20**
Family error rate (P values) = 0.0500, 0.0100. Tukey's significant difference: 4.56, 5.52. Difference between means < TSD (0.05) – not significant (NS) ≥TSD (0.05) – significant (*) ≥TSD (0.01) – highly significant (**).				

There is evidence from dynamic in-vitro tests that glass fibre reinforcement increases fatigue resistance of dental appliances up to 100 times compared with fatigue resistance of an unreinforced restoration.¹⁵ Few other researches have also shown that unidirectional and woven types of glass fibres increase the flexural strength of PMMA.^{19–22} But there has been lack of research on the effect of silanisation on the unidirectional and woven types of fibres on the flexural strength of the interim restorations. Various authors have recommended different directions for placement of glass fibres. In the present study, fibres were placed in a direction longitudinal to the loading forces. It has been proven by various researches that this kind of placement provided the most effective reinforcement.^{11,22}

The glass fibres used in this study were also silanated to improve the adhesion of the fibres to the polymer matrix. It has been proven by various authors that the silanation provided good adhesion to the polymer matrix, high aesthetic quality and increased strength.^{8,9,13} The fibres were dipped in the monomer prior to incorporating in the resin matrix for proper wetting of the fibres with polymer. This procedure improved the adhesion. An in-vitro study by Vallittu⁹ has shown that wetting with monomer before application increases the adhesion of fibres to the polymer matrix. This was a pre-requisite for bonding of fibres to the polymer matrix. An effective impregnation process allows the resin to come into contact with surface of every fibre.

The site of placement of fibres is also crucial for strength of the restoration. As per literature the various sites of reinforcement include occlusal, middle and cervical third.^{21,23} In this study the reinforcement materials were placed at the middle of the master index in the occlusal one third, because interim restoration resins, like most of brittle materials, have a greater compressive than tensile strength. Therefore, fracture is usually initiated in the tension side of the restoration, which will be in the middle to cervical third of the pontic and the connector areas. It has been proven that by placing the fibres at middle third, there is considerable improvement in flexural strength.

The results of this study have shown that, the unidirectional fibres had better flexural strength than the woven form of fibres. This may be due to the fact that, reinforcement with unidirectional fibres resulted in anisotropic pattern, whereas, the woven fibres had isotropic pattern. Similar findings were observed by other studies.^{11,19} The mode of failure of the fibre-reinforced resin samples in this study showed a partial fracture pattern, where the joints remained intact and a small

portion of the pontics was separated as a result of cohesive failure of resin materials. In fact, a crack would occur on the tension side but would not propagate through to the compression point. The embedded fibre could not be stretched enough for the crack in the resin to continue. The fibre appeared to hold two pieces together. The fracture failure is usually related to the initiation of the crack and its subsequent propagation until displacement. A complete failure with the embedded fibre may not occur. In clinical situations, this is perhaps the most favourable mode of fracture of the interim restoration because the restoration remains intact and the treatment is unlikely to be compromised by partial separation of the pontics.

The effect of the luting agent on flexural strength of interim FDP was not investigated in this study. It is likely that cementing the FDP to the abutments increases the fracture resistance of the FDP by transferring stresses more evenly to the FDP abutment system. Another limitation of the present study was the difficulty in securing the fibre in the exact location; however, the results may provide a rational clinical protocol for the fabrication of fibre-reinforced interim FDP.

Conclusions

Within the limitations of the current study, the following conclusions can be drawn:

1. The flexural strength of the reinforced PMMA interim fixed dental prosthesis was significantly higher ($P < 0.0001$), when compared to the unreinforced PMMA interim fixed dental prosthesis.
2. The flexural strength of PMMA interim fixed dental prosthesis reinforced with unidirectional fibres was higher than with the woven type fibres.
3. The silane treatment with silane coupling agent of both the types of fibres significantly improved the adhesion of the glass fibres with the polymer, thereby, increasing the flexural strength.
4. The overall results of the in-vitro study carried out showed that the use of silane treated unidirectional glass fibres was an effective method of reinforcing interim fixed restorations made of PMMA resins.

Conflicts of interest

All authors have none to declare.

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