

Effect of Surface Treating Fibre Posts on Shear Bond Strength of Composite Resin- An In vitro Study

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ABSTRACT

Introduction: Fibre-reinforced post is a common choice for restoring endodontically treated teeth which are badly broken. To improve the retention of post, surface treatments are performed to modify the surface structure and to enhance the adhesion of fibre post and resin cement.

Aim: To evaluate and compare the effect of three different surface treatment protocols on Fibre-Reinforced Composite (FRC) post to increase the bond strength between FRC post and dual cure adhesive resin luting agent using Universal Testing Machine (UTM).

Materials and Methods: Sixty four radiopaque fibre-reinforced posts were selected. The samples were divided into two groups of glass fiber (Group A) and carbon fiber (Group B) each (n=32) embedded into acrylic resin which were subdivided into four groups each [I-no surface treatment, II-ethyl alcohol and silanization, III-hydrogen peroxide and silanization, IV-sand blasting and silanization (n=8)]. A customised metal

ring was placed around the exposed fibre post surfaces after surface treatment. The dual cure adhesive resin cement was loaded in the ring and polymerised with halogen polymerising light. Observations were then statistically analysed using (One-way ANOVA) to evaluate the mean difference among four groups and Tukey's Post-Hoc test to determine inter group comparisons.

Results: Statistically significant difference was found among all groups with sub-group IV of A and B i.e., both carbon and glass fibre posts treated with sand blasting and silanization showing significantly higher value ($p < 0.05$) compared to all other groups and glass fibre posts showed significantly higher value than carbon fibre posts.

Conclusion: Sand blasting followed by silanization is the most effective surface treatment than other treatments done for improving the bond strength of resin cement to the fibre post surface.

Keywords: Dual cure adhesive resin cement, Silanization, Universal testing machine

INTRODUCTION

The use of fibre posts for the restoration of endodontically treated teeth is currently a common practice in dental offices due to good mechanical properties of the fiber posts when compared to traditional metal cast posts. Fibre posts provide modulus of elasticity and diametral tensile strength similar to the dentin; therefore, diminishing the risk of root fracture [1]. It has been mentioned that fibre posts can also increase the transmission of light within the root canal, thereby increasing the degree of conversion of monomers to polymers (resin cements) and also eliminating the problem of curing deeper areas of the root when using resin cements [2]. Carbon fibre posts have better mechanical properties as their modulus of elasticity is closer to that of dentin and they have elevated resistance to fatigue [3], with less microleakage. Failure of fiber post and core restorations occurs because of debonding between the fiber post and resin luting and/or between fiber post and root canal dentin due to its inadequate bond strength. As the polymer matrix in the post material fibres is highly cross-linked, and less reactive making it difficult for the posts to bond with resin luting agents and tooth structure.

Machado AC et al., had categorised several surface treatments available for fibre posts into three categories: rough surface promotion, chemical adhesive optimisation and association of these methods [4]. The various procedures to improve the bond strength of fibre post were by use of alcoholic and phosphoric acid solutions by removing oils and residues from the surface of fibre posts [5]. The use of alcoholic solutions for surface treatment of

fibre posts contributed in degreasing post surfaces by removing oils and residues [6]. The surface treatment of fiber posts with hydrofluoric acid etching works by forming micro spaces between the exposed fibers [7]. Other surface roughening approaches used were application of sodium ethoxide, and potassium permanganate which enhance bond strength by increasing the surface energy of fibre post [8,9].

The present study was conducted to evaluate the effect of surface treatments of carbon fibre posts and glass fibre posts on bond strength of composite resin and was followed in accordance with the study of Choi Y et al., [1]. The research hypothesis was that significantly different shear bond strength values will be found between the composite resin and the two different fibre posts, after different surface treatments.

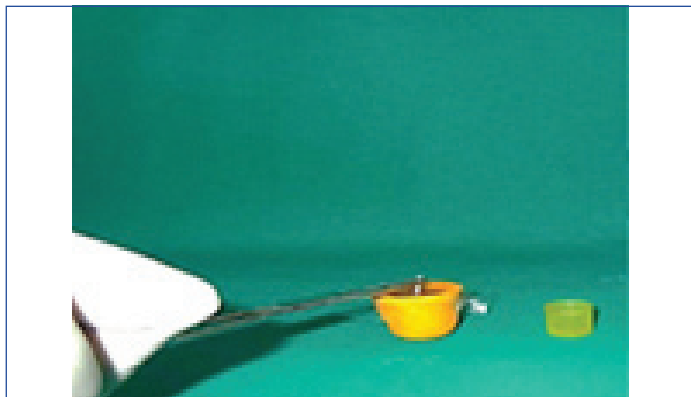
MATERIALS AND METHODS

The present in vitro study was conducted in the Department of Prosthodontics and Implantology, Government Dental College and Hospital, Hyderabad, Telangana, India, from August-October 2016. The ethical clearance for this study was obtained from the academic ethical clearance committee of the institute. The significance levels for the study was $p < 0.05$, the power of the study was 80% and confidence interval was 95%.

1. Sample selection: Thirty two radiopaque translucent fibre reinforced posts each of glass and carbon fibre type were taken with the specific dimensions of 15 mm in length and 1.1 mm diameter where the dimensions were verified with the help of a Vernier calliper (Deccan Dental Depot, Hyderabad,

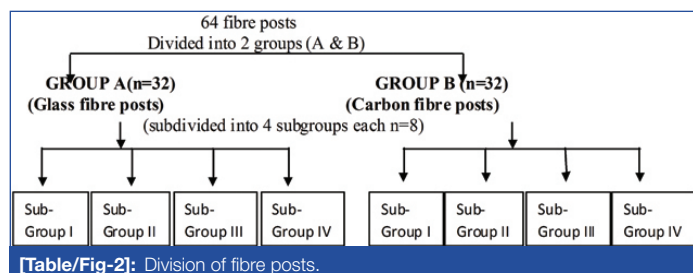
India). All the samples were wiped with alcohol (Surgical Spirit-Century Pharmaceuticals, Vijayawada, India) for 5 seconds for decontamination.

2. **Preparation of resin mould:** A cylindrical plastic cap of diameter 15 mm and length of 20 mm which was closed on one side was duplicated and moulds were made with addition silicone impression material (Aquasil, Dentsply, Germany). Fibre posts held with a needle holder were embedded perpendicularly into the acrylic resin [Table/Fig-1].



[Table/Fig-1]: Preparation of resin mould and placement of fibre post.

Sixty four samples of fibre posts were divided into thirty two samples each of glass fibre-reinforced epoxy resin posts (Reforpost, Angelus, Soul Dental solutions, Chandigarh, India) and Carbon fibre-reinforced epoxy resin posts (Reforpost, Angelus, Soul Dental solutions, Chandigarh, India) which were then randomly divided into four sub-groups each (n=8) [Table/Fig-2].



[Table/Fig-2]: Division of fibre posts.

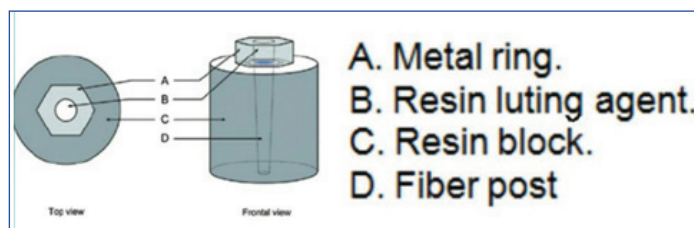
- a. **Sub-group I (Groups A and B)-Control:** In both the groups, the surface of fibre posts were not treated with any of the solution.
- b. **Sub-group II (Groups A and B)-Ethyl alcohol and silane:** In both the groups, the post surfaces were treated with 96% ethyl alcohol (Changshu Yangyuan Chemical Co. Ltd., China) for three minutes. Then the post surfaces were rinsed with distilled water for two minutes and air dried for 10 seconds. A single layer of a silane coupling agent (Calibra, Dentsply, Germany) was applied on the surface with a brush, after which they were left undisturbed for 60 seconds and later gently air dried.
- c. **Sub-group III (Groups A and B)-Hydrogen peroxide and silane:** In both the groups, the post surfaces were treated with 6% hydrogen peroxide (Swastik Pharmaceuticals, Vijayawada, India) for 20 minutes. Then the post surfaces were rinsed with distilled water for two minutes and air dried for 10 seconds. A single layer of a silane coupling agent (Bifix-SE, Calibra) was then applied on the surface of post with a brush, after which they were left undisturbed for 60 seconds and later gently air dried.
- d. **Sub-group IV (Groups A and B)-Alumina particle abrasion and silane:** In both the groups, the post surfaces were sandblasted with an extra oral sand blasting device (Anquistexim Inc, Mumbai, India) using 110 μ alumina particles (A. B. Enterprises, Mumbai, India) at 0.28 MPa for five seconds. The post surface was held parallel to the direction of incoming particle stream at a distance

of 10 mm. Then the surface was air blown with three-way syringe to remove the remaining alumina particles.

The sand blasted post was then gently cleaned with alcohol soaked cotton held with tweezer to remove any loose alumina particles. Then the blocks were allowed to dry for 10 minutes at room temperature. The surfaces were then coated with silane coupling agent (Bifix-SE, Calibra), after which they were left undisturbed for 60 seconds and later gently air dried.

3. Preparation for luting agent adhesion:

- a. The exposed post surfaces of all the samples were now luted using dual-polymerising resin-based luting material (Bifix-SE, Voco, Germany). A customised stainless steel metal ring [Table/Fig-3] of inner diameter 1.1 mm and depth of 3 mm was used for standardising the bonding area of cement to the post surface.



[Table/Fig-3]: Placement of metal ring for bonding of post with composite.

- b. After positioning the ring on the post surface, the first increment of luting agent was applied to the post surface with a plastic filling instrument and light polymerised for 40 seconds with a Halogen polymerising light (600 mW/cm² output-400 to 500 nm wave length), at a distance of 2 mm. An additional 40 seconds of light polymerisation was performed to ensure optimal polymerisation of the luting agent. The luting agent was carried in three increments of 1 mm each till the ring was completely filled. As the inner diameter of the metal ring and diameter of the fibre post was 1.1 mm, the inner surface of the metal ring was coated with composite and was bonded to the fibre post.

The sample from each group was placed horizontally between two movable jigs and held tight by locking the jigs to prevent movement of samples in a UTM (Dak systems Inc, Mumbai). The load was applied with a metal bar held between two movable jigs at the junction of metal ring and adhesive cement at a crosshead speed of 1.0 mm/minute until the metal ring was separated from the post surface, showing the failure at bonding interface.

The shear bond strength of the luting agent to the post was calculated by dividing the Load (N) at failure by the bonding surface area (mm²). It is expressed in Mega Pascal's (MPa).

$$\text{Shear Bond Strength (MPa)} = \frac{\text{Load at Failure (Newton)}}{\text{Bonding surface area (mm}^2\text{)}}$$

STATISTICAL ANALYSIS

The statistical analysis were done by mean comparisons among groups by ANOVA and Tukey's Post-Hoc test. The significance level was considered to be $p < 0.05$. The software used was IBM SPSS Version 20.0.

RESULTS

The [Table/Fig-4] represents the mean comparisons among groups of glass and carbon fibre posts. Among the group of glass fibre posts, bond strength was higher with Group IV A as compared to other sub-groups and among the group of carbon fibre posts, bond strength was higher with Group IV B i.e., sandblasting with silanization than other sub-groups.

Groups	N	Mean	Std. deviation	Std. error	95% Confidence interval for mean		Minimum	Maximum
					Lower bound	Upper bound		
I A	8	13.2402	0.74860	0.26467	12.6143	13.8660	12.13	14.10
II A	8	24.6148	0.74595	0.26373	23.9912	25.2384	23.15	25.50
III A	8	25.4789	0.42630	0.15072	25.1225	25.8353	24.84	25.98
IV A	8	33.7678	0.63341	0.22395	33.2383	34.2974	33.09	34.99
I B	8	13.4951	0.98034	0.34660	12.6755	14.3146	12.14	14.95
II B	8	21.8961	1.06579	0.37681	21.0050	22.7871	20.57	23.36
III B	8	22.5163	0.74284	0.26263	21.8953	23.1373	21.39	23.94
IV B	8	33.7266	0.34024	0.12029	33.4422	34.0110	33.27	34.21
Total	64	23.5920	7.37144	0.92143	21.7506	25.4333	12.13	34.99

[Table/Fig-4]: Mean and standard deviation values for both the groups.

Statistical analysis (one-way ANOVA) revealed that both the posts had a significant influence on bond strength ($p < 0.05$). There was significant difference ($p < 0.05$) among the groups of glass and carbon fibre posts with different surface treatments [Table/Fig-5].

	Sum of squares	df	Mean square	F	Sig. $p < 0.05$
Between groups	3392.050	7	484.579	868.351	0.001
Within groups	31.251	56	0.558		
Total	3423.301	63			

[Table/Fig-5]: Mean comparison of different groups.

df: Degree of freedom, F: ANOVA value

Statistical Analysis: ANOVA one way test. Statistically significant if $p < 0.05$

[Table/Fig-6] shows the results obtained by Tukey's Post-Hoc test. This test was done to evaluate the inter-group significant differences. The p-value was significant (< 0.05) between all groups, the inference shows that the bond strength improves with surface treatment than without any treatment. Of all surface treatments done, ethyl alcohol with silanization on carbon fibre posts showed least improvement in the bond strength. There was no statistical difference among the groups of glass and carbon fibre posts on all the surface treatments. When compared to the surface treatment by ethyl alcohol and silanization and hydrogen peroxide and silanization, there was no statistical difference among groups of glass and carbon fibre posts.

DISCUSSION

The success of intra radicular-retained restorations using fibre post depends on the bonding interface between post, resin cement and tooth. Thus, to improve this interaction, it is necessary to perform surface treatments of the fibreposts, seeking to remove the superficial epoxy resin matrix and expose the internal glass fibres. This is done to improve chemical bonding to Bis-GMA based materials through coupling agents.

The surface wetting theory by Pape PG and Plueddemann EP recognises a key role of the wetting capacity of the silane for improved adhesion. According to this theory, the silane with its low viscosity, would assist substrate wetting, and once an intimate contact between the interfacing materials is established, van der waals' forces would become effective, providing a physical adhesion, which contributes to the chemical reaction [10]. In the present study, though the glass and carbon fibre posts were not surface treated, positive shear bond strength was observed. This positive shear bond strength value was due to the mechanical and physical properties of the dual-polymerisation resin luting agent to maximise strength and rigidity [11].

In this study, ethyl alcohol with silanization group showed comparatively higher value than the control group but less than the hydrogen peroxide and sandblasting with silanization groups. The degreasing of the post surface, removes oils and residues which is then followed by the benefit of silane application for enhancing

S. No.	Sub-group	Sub-group	p-value
1	IA	IIA	0.0001**
		IIIA	0.0001**
		IVA	0.0001**
		IB	0.997
		IIB	0.0001**
		IIIB	0.0001**
2	IIA	IVB	0.0001**
		IIIA	0.305
		IVA	0.0001**
		IB	0.0001**
		IIB	0.0001**
		IIIB	0.0001**
3	IIIA	IVB	0.0001**
		IVA	0.0001**
		IB	0.0001**
		IIB	0.0001**
4	IVA	IVB	0.0001**
		IIIB	0.0001**
		IIB	0.0001**
		IB	0.0001**
5	IB	IVB	0.0001**
		IIIB	0.0001**
		IIB	0.0001**
6	IIB	IVB	0.0001**
		IIIB	0.712
7	IIIB	IVB	0.0001**

[Table/Fig-6]: Tukey's Post-Hoc test to compare each sub-group of both the groups i.e., glass and carbon fibre groups.

** indicate that there was statistically significant difference with pair wise comparison with $p < 0.05$

the micro tensile bond strength of a dual-cure resin core material to translucent fibre posts [12]. Prithviraj DR et al., conducted a study to evaluate the effect of surface treatment of glass fibre posts, carbon fibre posts and cast metal posts with ethyl alcohol, resin primer and air-borne alumina particle abrasion on retention. They concluded that treating the surface of the posts with resin primer and ethyl alcohol produced no statistical significant difference in the retentive strength [13].

In the present study hydrogen peroxide and silanization group showed significantly greater values than control group and ethyl alcohol and silanization group but lower than sandblasting and silanization group in both glass and carbon fibre post. Roberto R et al., stated that the bonding capability of conventional and self-adhesive resin cements to fibre posts were affected by different post surface pretreatments.

Post surfaces treated with 24% H₂O₂ or 96% ethyl alcohol showed significantly stronger bond interactions to resin cements than only silane application [14]. Naves LZ et al., investigated on morphology and etching pattern, surface modification and characterisation of two different fibre posts: glass fibre post and carbon fibre post, were surface treated with 4% hydrofluoric acid for 1 minute; 37% phosphoric acid for 30 seconds; 10% hydrogen peroxide for 20 min; 24% hydrogen peroxide for 10 minutes. Results have concluded that H₂O₂ followed by silane coupling agent, improved the bond strength between post and composite resin, facilitating the stress distribution and improving the post retention [15]. Mazitelli C et al., conducted a study on quartz fibre posts treated with 10% H₂O₂ for 20 minute, 30% H₂O₂ for 10 min, 21% sodium ethoxide for 20 minute, etching with potassium permanganate, etching with 4% hydrofluoric acid for one minute sand blasting and silicate-silane coating. The study concluded that etching with potassium permanganate or sodium ethoxide increases surface roughness through partial removal of the epoxy resin matrix and improves the surface area available for adhesion by creating micro-retentive spaces [16].

In the present study group with sand blasting followed by silanization showed significantly greater values than all groups for both glass and carbon fibre posts. By removing a surface layer of the epoxy resin, a greater surface area of exposed glass fibres is available for silanization. Sandblasting leads to roughening of post surface by removing the resin matrix between the silicon fibres making it more retentive in addition to the chemical reaction of silanes which relies on the formation of Si-O-Si siloxane bonds and conversion of the mineral surface into a less polar surface compatible with organic bonding agent, thus improving bond strength than other groups. Wang Y et al., reported that sand blasting and silanization improve bond strength between fibre post and resin cement and recommended fibre posts should be pre-treated with sandblasting or combined with silanization before clinical use [17]. According to Rodig T et al., the push out bond strength between fibre posts and different composite resin following different surface treatments of the posts-silanization, sand blasting plus silanization and tribochemical coating and concluded that post type, type of surface treatment and type of resin cement were significant factors for bond strength [18].

LIMITATION

In the present study, only one composite resin and three surface treatments of the posts were evaluated, using in vitro conditions. Different results might be obtained with different composite resins or with different surface treatment techniques. However, this in vitro study does not give an exact prediction whether the in vitro performance of the fiber posts is the same as the performance in vivo. In this in vitro study the post were pretreated and was immediately followed by the application of the resin composite for the core build-up. Further in vitro and in vivo studies are necessary to evaluate whether the positive effect on post-core bond strength is still retained by pre-treating the post surface well in advance of the clinical use. Evaluation of such a strategy will enable manufacturers to

supply pretreated fibre posts in presealed sachets, as well as saving clinicians valuable chair-time. More parameters like flexural strength, fracture resistance, microleakage are needed to be evaluated for a successful restoration. Further studies on these fibre post systems are required to validate the results of the present study.

CONCLUSION

Within the limitations of this study, the pretreatment of fibre post reduce the chances of bond failure. The present study concludes that the surface treatment by sandblasting and silanization showed higher bond strength values than with ethyl alcohol and hydrogen peroxide along with silanization.

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