

Effect of Surface Pretreatment and Thermal Activation of Silane Coupling Agent on Bond Strength of Fiber Posts to Resin Cement

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ABSTRACT

Introduction: For the restoration of grossly decayed root canal treated teeth, posts are used frequently to retain core for the definitive restoration. Therefore, it is necessary to create adequate adhesion at the post–root–cement interface for long-term performance of a post retained restoration.

Aim: To establish the outcome of surface pretreatments such as silanization, sandblasting, and silanization followed by thermal activation of fiber posts on bonding with a dual-cured adhesive resin-based cement.

Materials and methods: Eighty radiopaque, No #2 glass fiber-reinforced epoxy resin posts (Hi-Rem Posts, Overfibers, Switzerland), posts were wiped with alcohol (surgical spirit, 90% alcohol) for 5 seconds in a single stroke. A cylindrical plastic cap of diameter 10 mm and length of 15 mm, which is closed on one side, was selected. This was duplicated to form molds and fiber posts embedded perpendicularly in the polyester resin, and samples were made. Samples were randomly distributed into four groups based on the pretreatment done and each group contains 20 samples, group I—(control), group II—(silanization), group III—(sandblasting), and group IV—(thermal treatment of silane). After surface treatment, exposed post surface was uniformly coated with dual-cured resin cement. Cement was cured for 40 seconds with a halogen lamp. The samples are subjected to load in a universal testing machine (UTM) at a crosshead speed of 1.0 mm/minutes to evaluate the bonding failure at the interface.

Results: Group IV resulted in the highest bond strength values followed by group III. Group II showed a comparatively higher value than group I but less than groups III and IV.

Conclusion: Surface treatment procedure, on fiber post by silanization and sandblasting significantly improved adhesion between post and luting cement interface.

Clinical significance: The surface pretreatment, such as sandblasting, silanization, thermal activation of silane coupling agents significantly improved, the retention of the post within in the root canal system.

Keywords: Fiber post, Sandblasting, Shear bond strength, Silanization, Thermal activation.

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INTRODUCTION

For the restoration of grossly decayed root canal treated teeth, posts are used frequently to retain coronal restoration.¹ The fiber-reinforced posts are introduced by Duret et al.² Fiber post is frequently used for restoring root canal treated teeth. Fiber posts, when compared to the time, tested metal counterparts provide aesthetics, absence of corrosive reactions, comparative ease of removal in retreatment, optimal stress distribution, and adhesion.^{3,4} In spite of these advantages, 4.4% failures of fiber post occur at the post-restoration junction, of which 55% failures take place at the post-adhesive junction.³ Failures occur due to lack of functional alkoxy groups on the surface of the fiber post, results in insufficient and ineffective bonding of post/dual-cure adhesive resin interface.

Surface treatments of post surfaces improve adhesion between post and resin luting cement. Surface treatments can be chemical treatment, surface roughening, and a combination⁴ surface treatment with silane-coupling agents improves the bond strength by 20%.⁵

It mediates adhesion between inorganic and organic matrices by the formation of siloxane bonds, which makes the surface less polar and hence more compatible to bonding with adhesive resin.

Surface roughening treatment with sandblasting procedure improves the bond strength by 19%.⁵ Sandblasting roughens the surface of the fiber post, thereby promoting the larger surface area for bonding, which creates a mechanical interlock with the

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resin cement. Combination—surface treatment with sandblasting followed by silanization improved the bond strength by 50%.⁵

Hence, the aim of the present study is to investigate the outcome of surface treatment such as silanization, sandblasting, and silanization followed by thermal activation of fiber-reinforced posts on the adhesion with a dual-cured adhesive resin-based luting agent.

MATERIALS AND METHODS

Eighty radiopaque, translucent glass fiber reinforced epoxy resin posts (Hi-Rem Posts, Overfibers, Switzerland) with dimensions (diameter 2.2 mm and length 15 mm) were used in the study (Table 1). Posts were wiped with alcohol (surgical spirit, 90% alcohol) for 5 seconds. The post surface was conditioned for 1-minute with Eco-Etch Etchant that contains 37% phosphoric acid (Ivoclar-Vivadent, Schaan, Liechtenstein, USA. Lot No. U28587) and rinsed with deionized water (5 mL) for 20 seconds. The post surface is gently air-dried with a three-way syringe. A cylindrical plastic cap of diameter 10 mm and length of 15 mm, which is closed on one side, was selected for preparation of resin block. This is duplicated, and molds were made with addition silicone impression material. Polyester resin was carried into the mold. Fiber posts held with a needle holder were embedded perpendicularly in the polyester resin. The setting time of each sample was 15 minutes samples were randomly distributed into four groups based on the pretreatment done, and each group contains 20 samples.

Group I (control)—Surface of fiber post was neither treated with silanization nor sandblasting.

Group II (silanization)—In this group, post surfaces were applied with a silane-coupling agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein, USA. Lot No. E26882). Silane was applied onto the post surface with a disposable applicator tip and leftover on post surface for 60 seconds; later the post surface is gently air-dried, with a three-way syringe.

Group III (sandblasting)—In this group, the post surface was treated by sandblasting with 50 μ aluminum oxide particles using a sandblasting device (S-U Austral, Schuler Dental Germany) at 0.28 MPa air pressure for 10 seconds using 110 μ alumina particles. Surface of the post was held parallel to the direction of the incoming particle stream at a distance of 10 mm. The sandblasted post was then gently cleaned with alcohol-soaked cotton to remove any loose alumina particles.

Group IV (silanization followed by thermal activation)—Silane-coupling agent applied similar to group II followed by thermal activation post surface coated with silane was done at constant temp (80°C) for 10 minutes using preheated burnout oven (Temp Master M; Jelrus, Hicksville, NY, USA).

After surface treatment, post surfaces were coated with dual-cured resin cement (Multilink Speed, Ivoclar-Vivadent, Schaan,

Liechtenstein, USA, Lot No. 642977AN). Customized stainless steel metal ring of inner diameter 2.2 mm and depth of 3 mm was used for standardizing the bonding area of cement to the surface of the post. Stainless steel ring positioned onto the post surface and first increment of resin cement were applied to the post surface with a plastic instrument and cured with LED light (Bluephase C8, Ivoclar-Vivadent, Schaan, Liechtenstein, USA), with intensity of 800 mW/cm² at a distance of 2 mm for 40 seconds. For optimal curing, resin cement was cured for an additional 40 seconds using curing light.

The shear bond strength was measured at 1.0 mm/minute crosshead speed using Instron universal testing machine (High Wycombe, United Kingdom; Model No: 14115280010). Shear bond strength measured by using the formula:

Shear bond strength (MPa)

$$= \text{Load at failure (Newton's)} / \text{Bonding surface area (Sq mm)}$$

Statistical analysis was performed by transferring the tabulated observations to the software IBM SPSS version 20.0 (IBM, Armonk, NY, USA). To evaluate statistical differences among test groups, one-way ANOVA was performed. A Tukey *post hoc* test was performed to evaluate for multiple comparisons among groups.

RESULTS

The highest shear bond strength observed for silanization and thermal activation followed by sandblasting, silanization, and the lowest values are recorded in the control group. Thermal activation had achieved higher bond strengths than other surface treatments with minimum standard deviation compared to group II and group III. Tukey *post hoc* test shows that the highest mean difference of 26.86 MPa was seen between group IV and group I and the lowest mean difference of 6.85 MPa was seen between groups III and IV (Fig. 1). *Post hoc* test shows that all surface treatment method used in this study improved shear bond strength ($p \leq 0.05$). Of all surface treatments done, silanization showed the least improvement in the bond strength (Table 2). Significant difference ($p < 0.05$) in shear bond strength values were noted among all surface treatment groups (Table 3).

DISCUSSION

Increasing concern of patients towards more biocompatible and tooth-colored restoration leads to the development of esthetic post and core systems.⁶ Prefabricated posts became popular as they save number clinical visits by reducing laboratory procedure, removal of post system made easy and were economical.⁷ Reduced catastrophic fractures were seen with fiber posts when they are bonded to dentin using resin cement due to formation of a

Table 1: Material used in the study

Material	Composition	Manufacturer
Hi-Rem posts	Glass fibers (61.5 wt%) inserted in a polymer matrix of tri-methylene-glycol-di-meth-acrylate (16 wt%) and urethane di-meth-acrylate with highly dispersed silicon-dioxide filler (10 wt%).	Overfibers, Switzer Overfibers Mordano (BO), Italy
Silane coupling agent (Monobond-S)	1 wt% 3 methacryloxypropyltrimethoxysilane (3-MPS), ethanol/water-based solvent	Ivoclar-Vivadent, Schaan, Liechtenstein
Multilink speed	Base: dimethacrylate and HEMA (30.5 wt%), barium glass and silicon dioxide filler (45.5 wt%), ytterbium trifluoride (23 wt%), catalyst and stabilizers (1 wt%), pigments	Ivoclar-Vivadent, Schaan, Liechtenstein
Eco-etch etchant contains 37% phosphoric acid	37% phosphoric acid	Ivoclar-Vivadent, Schaan, Liechtenstein

homogeneous structure with root dentin.⁸ Fiber-reinforced posts composed of unidirectional glass fibers (61.5 wt%) are dispersed in a polymer matrix of trimethylene-glycol-dimethacrylate (16 wt%) and urethane dimethacrylate (12 wt%) and silicon-dioxide filler (10 in wt%).

Fiber posts improve retention and mechanical performance of the restored teeth. The elastic modulus of fiber post was close to dentin (18GB) that allows the flexure of post and root together, resulting in reduced fracture of root. Another advantage is that during endodontic retreatment, retrieval of fiber post system was more easy and predictable with less fracture. Most commonly, the failure of fiber post system was noticed at resin cement–fiber post interface of due to poor bonding.⁹

A major drawback with prefabricated fiber posts is that the polymer matrix between the post material fibers is highly cross-linked and therefore less reactive. This makes it difficult for these posts to bond to the resin luting agents and tooth structure.^{10,11} It has been estimated that 60% of the fiber post failures occurred between the fiber post and resin cement.¹²

The success of post and core restoration depends on the adhesion between post, resin cement, and dentin interface. The surface treatment procedures often result in improved adhesion at the post–cement interface by removing the matrix of epoxy

resin from superficial part and exposes the internal glass fibers. This helps in improved bonding of fiber post to bis-GMA based cement using coupling agents. Sahafi et al.¹⁶ had categorized various treatment procedures of post surface into three categories that include (1) surface roughening, (2) chemical adhesive optimization, and (3) combination of surface roughening and chemical adhesive optimization.¹³ Rough surface produced mechanically by sandblasting or chemically using chemical agents such as HF (20% w/v) or H₂O₂ (6% w/v) allows better penetration of the resin cement onto the surface of post, thus increasing micromechanical retention. Treating the surface of post with silane and followed by application of resin cement increases bond at the post–cement interface.

Post surfaces treated with alcoholic solutions before cementation removes oils and residues thereby increases the bond strength by 12%.^{5,14,15} Phosphoric acid treatment on fiber posts, etches post surface, and improves bonding.¹⁵ In this study, also posts were wiped with alcohol (surgical spirit, 90% alcohol) for 5 seconds followed by 37% phosphoric acid application to improve bonding efficiency.

Although surface treatment with hydrofluoric acid enhances bond strength by 15%, it causes structural microscopic damage to the post. Hydrogen peroxide (6% w/v) treatment improves bond strength by 12%, but there is no standard protocol for application time and concentration of the solution.

Pretreatment with silane-coupling agent was used frequently for ceramic prosthesis repair, all-ceramic crown cementation, bonding of ceramic brackets to tooth surfaces, and luting of fiber post to root dentin. Previous studies reported that significant improvements in bond strength after pretreatment with silane-coupling agents.^{16–18} However, Choi et al. reported that the application of silane to post surface results in an only minimal increase in the bonding capacity.¹

Moraes et al. reported that significant improvement in the retention of post surface seen only when the surface of post is treated appropriately before silane application. Surface pretreatment of post surface removes overlying epoxy resin and exposes glass fibers, which allow the formation of siloxane bonds between silane and glass.¹⁹

However, silane-coupling agents applications used in this study allow the formation of a bond between glass fibers and polymer groups. Organosilanes (R'-Si-(OR)₃) have an organic functional

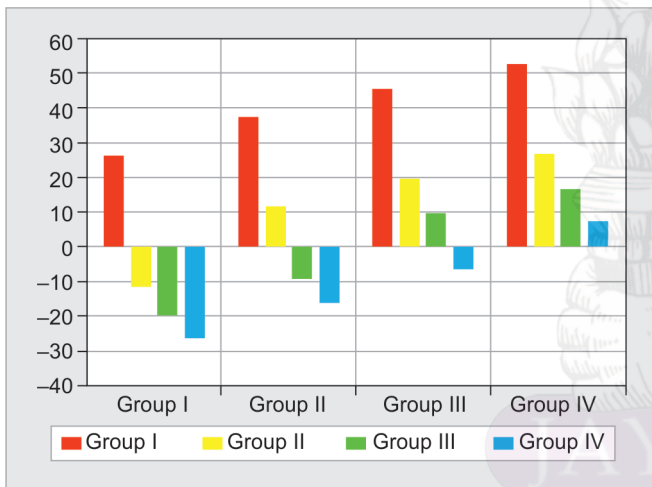


Fig. 1: Intergroup mean comparison of shear bond strengths

Table 2: Shear bond strength values of group I, group II, group III, group IV

Variable	Mean	Std. deviation	Std. error	95% confidence interval for mean			
				Lower bound	Upper bound	Minimum	Maximum
No treatment group I	26.07	0.51	0.132	25.783	26.349	25.31	27.16
Silanization group II	36.90	0.72	0.186	36.500	37.298	36.21	38.32
Sandblasting group III	46.07	0.69	0.178	45.692	46.457	45.22	47.30
Silanization and thermal activation group IV	52.93	0.52	0.135	52.638	53.219	52.16	53.62

Table 3: Mean comparison among groups

	ANOVA				
	Sum of squares	Df	Mean square	F value	p value
Between groups	6102.652	3	2034.217	5309.193	<0.001
Within groups	21.456	56	0.383		
Total	6124.109	59			

group and three alkoxy groups (R). Alkoxide groups (R) undergo hydrolysis reaction to form silanols (SiOH). Silanols undergo condensation to form siloxane bonds.^{12,20}

This siloxane bonds between silane-coupling agent and multilink link speed resin cement used in this study resulted in improved bond strength of group II. Smooth surface of fiber posts in control group minimizes the mechanical interlocking. Air abrasion technique used in this study creates rough surface on the post that improves micromechanical retention with luting cement. The increased roughness results improved bond strength in group III.

Group IV showed significantly greater values than groups I, II, III. Highest bond strength in group IV attributed to heat activation of the silane agent. Solvent present in the silane evaporates with heat application that result in increased reactivity of silane. Elimination of water, alcohol and volatile by-products during completion of the silane-silica condensation and the resultant facilitation of covalent bond formation are responsible for this effect.²¹ Monticelli et al. also reported that air drying at 38°C increases reactivity of silane agent, which results in increased bond strength of resin cement-post interface surface.²² Silva et al. reported that heat activated siloxane application to post surface resulted in increased bond strength to resin-based materials.²³

Design of this study has limitation that *in vitro* shear bond strength values may not exactly correlate with *in vivo* conditions. Further clinical studies are warranted.

CONCLUSION

Within the limitations of this study, surface pretreatment of fiber post significantly improved shear bond strength. The present study concludes that the surface treatment by sandblasting and silanization showed higher bond strength values than control. Shear bond strength at post-resin cement interface increased by 24% for silanization followed by thermal activation.

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