Effect of silane activation on shear bond strength of fiber-reinforced composite post to resin cement

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PURPOSE. Among the surface treatment methods suggested to enhance the adhesion of resin cement to fiber-reinforced composite posts, conflicting results have been obtained with silanization. In this study, the effects of silanization, heat activation after silanization, on the bond strength between fiber-reinforced composite post and resin cement were determined. **MATERIALS AND METHODS.** Six groups (n=7) were established to evaluate two types of fiber post (FRC Postec Plus, D.T. Light Post) and three surface treatments (no treatment; air drying; drying at 38℃). Every specimen were bonded with dual-curing resin cement (Variolink N) and stored in distilled water for 24 hours at 37℃. Shear-bond strength (MPa) between the fiber post and the resin cement were measured using universal testing device. The data were analyzed with 1-way ANOVA and by multiple comparisons according to Tukey's HSD (α=0.05). The effect of surface treatment, fiber post type, and the interactions between these two factors were analyzed using 2-way ANOVA and independent sample T-tests. **RESULTS.** Silanization of the FRC Postec Plus significantly increased bond strength compared with the respective non-treated control, whereas no effect was determined for the D.T. Light Post. Heat drying the silane coupling agent on to the fiber-reinforced post did not significantly improve bond strength compared to air-syringe drying. **CONCLUSION.** The bond strength between the fiber-reinforced post and the resin cement was significantly increased with silanization in regards to the FRC Postec Plus post. Bond strength was not significantly improved by heat activation of the silane coupling agent. ***[J Adv Prosthodont 2013;5:104-9]***

KEY WORDS: Post and core technique; Silane; Resin cements; Shear strength; Heat treatment

INTRODUCTION

Fiber-reinforced composite posts are widely used to restore endodontically treated teeth with all-ceramic crowns. The major advantage of these posts is the similarity of the elastic modulus to dentin, which reduces stress concentration as well as root fracture.[1,2] However, fiber post-restored teeth occasionally fail, most commonly as the result of debonding of the post at the post-cementation junction.[3]

To enhance the adhesion of the resin cement to the fiber-reinforced composite post, several surface treatment methods have been suggested.[4-8] In a strategy aimed at achieving micro-mechanical interlocking between the resin cement and the fiber-reinforced post, increasing the roughness of the post surface has been examined.[9] Airborne-

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particle abrasion (sandblasting) was shown to significantly increase the bond strength between the post and the resin cement.[6,7] Alternatively, roughening can be enhanced by etching the post surface using hydrogen peroxide. This is a clinically simple method to improve the bonding strength without the need for acid etching of the post.[10]

Chemical bonding between the resin cement and the fiber-reinforced composite post also has been tested. For example, silane coupling agents have an organic functional group such that covalent bonds are formed between the resin cement and the quartz fibers of the post. Silane has been shown to increase bond strength between ceramics and resin composite materials,[11,12] thus strengthening the case for using silane coupling agents in dentistry, in order to improve the surfaces of fiber-reinforced composite posts for resin bonding.[1,13]

Nonetheless, among the post surface treatment methods, studies examining the efficiency of silanization in enhancing bond strength have reported conflicting results. There were no significant difference in bond strength between silane-treated posts and untreated posts.[7,14,15] However, silanization improves the microtensile bond strength between resin cement and fiber-reinforced composite posts in other reports.[1,13,16]

In the glass industry, silane treated glass is routinely heat-treated to maximize bond strength.[11] In dentistry applications, Shen *et al.* reported that silane drying under a stream of warm air was effective in enhancing the tensile bond strength of the composite to ceramic.[12] A similar approach to fiber-reinforced composite posts has been proposed to activate silanization, based on the premise that silane dried under warm air (38℃) has a higher bond strength than when dried at room temperature (21℃).[17]

The objective of this study was to determine the effect of silanization, heat activation after silanization, and fiber-reinforced composite posts differing in composition on the bond strength between the post and the resin cement. The null hypothesis was that none of these parameters would improve bond strength.

MATERIALS AND METHODS

Two post types were tested in this study: FRC Postec Plus (Ivoclar Vivadent AG, Schaan, Liechtenstein), with a maximum diameter of 2.0 mm; and D.T. Light Post, Size 3 (BISCO Inc., Schaumburg, IL, USA), with maximum diameter of 2.2 mm. The FRC Postec Plus is composed of glass fiber (70%), ytterbium fluoride (9%), and dimethacrylate (21%), i.e., UDMA and TEGDMA. The D.T. Light Post is composed of quartz fibers (60%) and an epoxy resin matrix (40%).[18]

The posts were embedded perpendicularly with unsaturated polyester resin (Formica Polycoat; Aekyung Chemical Co., Seoul, Korea) in the mold of a metal ring with a diameter of 15 mm and a length of 23 mm. By incremental pouring of the unsaturated polyester resin, the top surface of fiber posts were positioned at an even level with the top surface of the resin blocks (Fig. 1). The exposed surface of the post was cleaned using phosphoric acid (Total Etch; Ivoclar Vivadent AG, Schaan, Liechtenstein), rinsed with distilled water, and dried as recommended by the respective manufacturers.[7]

Six experimental groups (n=7 per group) were established to evaluate the two different types of fiber post and the three surface treatments. Group 1 served as the untreated control for the FRC Postec Plus. A metal hexagon nut with a 2 mm inner diameter was placed on the exposed top of the post embedded in a polyester resin block. An Accudose syringe and needle tip (Centrix, Shelton, CT, USA) were used to add dual-polymerizing resin cement (Variolink II; Vivadent AG, Schaan, Liechtenstein) so as to completely fill the metal hexagon nut, thereby establishing a uniform bonding surface area. The specimen was light-polymerized with a polymerizing light-emitting diode (Mini L.E.D.; Satelec, Paris, France) at a distance of 2 mm for 40 seconds. In Group 2, prior to resin cement adhesion, the post surface was silanized using a two component silane coupling agent (Porcelain Liner M; Sun Medical, Moriyama City, Japan) applied with a brush to the upper surface of

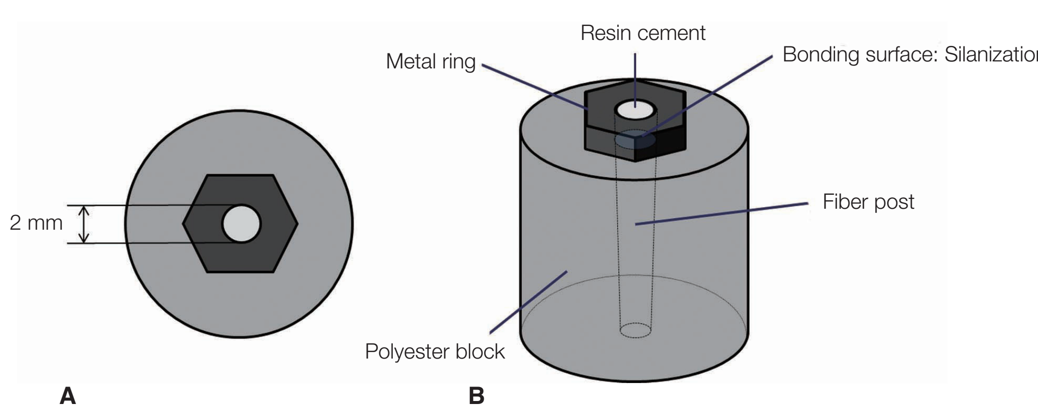

Fig. 1. Schematic drawing of specimen preparation for shear bond strength test. A: cross-sectional view for shear bond test, B: total view of mounted specimen.

the fiber post. The silanized post was then dried at room temperature (21℃) for 60 seconds from a distance of 30 cm using an air-syringe. In Group 3, the silane coupling agent was applied with a brush after which the post was dried under warm air mode (38℃) using a blow dryer (EDDY UN-1552, Unix electronics, Seoul, Korea) for 60 seconds at a distance of 30 cm. In Group 4-6, the procedures were the same but the post was a D.T. Light Post, likewise embedded in a resin block .

Before their analysis in a shear bond strength test, the 42 specimens prepared for the study were stored in distilled water for 24 hours at 37℃, since water storage has been shown to influence post retention similar to the oral environment.[5]

Each specimen was mounted in a universal testing device (Instron 3366, Instron Corp., Norwood, MA, USA) and loaded at a crosshead speed of 1.0 mm/min (Fig. 2A). When the metal hexagon nut separated from the resin block, the load at failure was measured (Fig. 2B).

Dividing the load (N) at failure by the bonding surface area (mm^2), defined the shear bond strength, expressed in mega-Pascals (MPa).

The data were statistically analyzed, beginning with a 1-way ANOVA to examine the effects of the different surface treatments: no treatment, silanization plus air drying at 21℃ (S21), and silanization plus air drying at 38℃ (S38). Tukey's HSD multiple comparison was then applied in a post-hoc analysis, followed by a 2-way ANOVA and independent sample t-tests to examine the effects of surface treatments, the fiber post composition, and the interactions between the two. The alpha value was set at 0.05. SPSS 18.0 software (SPSS, Inc, Chicago, USA) was used for the ANOVA, post-hoc analysis, and t-tests.

RESULTS

For the FRC Postec Plus post, the mean shear bond strength was 21.72 ± 2.35 (MPa) for the control group (no surface treatment), 30.72 ± 3.57 (MPa) for the S21 group, and 30.48 ± 4.75 (MPa) for the S38 group (Table 1). The differences between these groups were significant according to the 1-way ANOVA (Table 2, Fig. 3). Further analysis with Tukey's HSD test showed significant differences in the shear bond strengths of the control group and either the

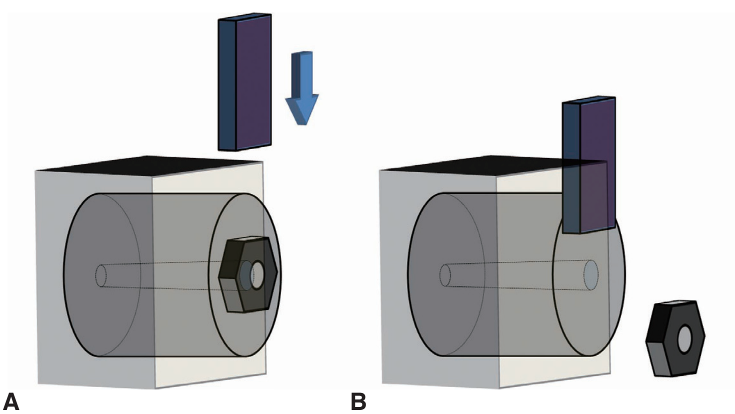

Fig. 2. Schematic presentation of shear bond test. A: the mounting specimen and loading, B: failure.

Table 1. Mean value and standard deviation (MPa) of shear bonding strength

Fiber post		Mean	SD	Std. error	95% confidence interval Lower	95% confidence interval Upper
FRC Postec Plus	Control (no treatment)	21.72	2.35	.88	19.54	23.89
	Silane + Drying (21℃)	30.72	3.57	1.34	27.41	34.02
	Silane + Drying (38℃)	30.48	4.75	1.79	26.08	34.88
	Total	27.64	5.53	1.20	25.12	30.16
D.T. Light Post	Control (no treatment)	21.27	1.93	.73	19.48	23.05
	Silane + Drying (21℃)	24.24	2.31	.87	22.10	26.39
	Silane + Drying (38℃)	24.17	4.50	1.70	20.00	28.34
	Total	23.23	3.29	.71	21.73	24.72

S21 group or the S38 group. However, the differences between the latter two groups were not significant.

For the D.T. Light Post, the mean shear bond strength was 21.27 ± 1.93 (MPa) for the control group, 24.24 ± 2.31 (MPa) for the S21 group, and 24.17 ± 4.50 (MPa) for the S38 group (Table 1). However, unlike the FRC Postec Plus post, none of the differences between the groups were significant (Table 2, Fig. 3).

As seen in Table 3, the 2-way ANOVA showed significant effects of the type of the fiber-reinforced composite post and the surface treatment on shear bond strength. There was also significant interaction between the type of the fiber-reinforced composite post and the surface treatment.

In the independent sample t-test, the shear bond strength values obtained for the FRC Postec Plus and D.T. Light Post were compared with respect to the surface treatment. In Levene's test for the equality of variances, the assumed equal variances of the three surface treatment methods were confirmed. For the two control groups, there were no significant differences between the FRC Postec Plus and D.T. Light Post. However, for the S21 and S38 silanization groups, the shear bond strength of the FRC Postec Plus was significantly higher than that of the D.T. Light Post (Table 4).

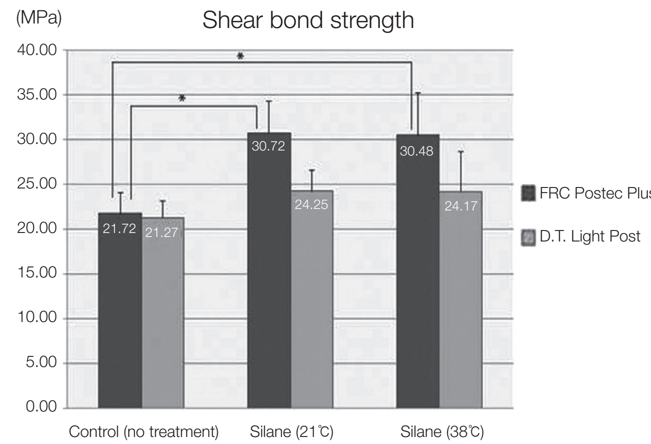

Fig. 3. Shear bond strength of the FRC Postec Plus and the D.T. Light Post as a function of the surface treatment. *: Mean values were significantly different by 1-way ANOVA (P<.05).

Table 2. One-way ANOVA results for the FRC Postec Plus and the D.T. Light Post

Fiber post		Sum of squares	df	Mean square	F	Sig.
FRC Postec Plus	Between groups	368.192	2	184.096	13.500	.000
	Within groups	245.453	18	13.636		
	Total	613.645	20			
D.T. Light Post	Between groups	40.288	2	20.144	2.055	.157
	Within groups	176.470	18	9.804		
	Total	216.758	20			

Table 3. Results of a 2-way ANOVA with fiber post and surface treatment as the independent variables and shear-bond strength as the dependent variable

Source	Sum of squares	df	Mean square	F	Sig.
Corrected model	612.859(a)	5	122.572	10.458	.000
Intercept	27174.652	1	27174.652	2318.642	.000
Fiber post	204.378	1	204.378	17.438	.000
Surface treatment	326.034	2	163.017	13.909	.000
Fiber post * Surface treatment	82.447	2	41.223	3.517	.040
Error	421.923	36	11.720		
Total	28209.433	42			
Corrected total	1034.781	41			

a R Squared = .548 (Adjusted R Squared = .486)

Table 4. Independent sample t-test to compare the shear bond strength of the FRC Postec Plus and the D.T. Light Post as a function of the surface treatment

Surface treatment		Levene's test for equality of variances		t-test for equality of means		
		F	Sig.	t	df	Sig. (2-tailed)
Control (no treatment)	Equal variances	.445	.518	.391	12	.702
	Not equal variances			.391	11.566	.703
Silane + Drying (21℃)	Equal variances	4.187	.063	4.024	12	.002
	Not equal variances			4.024	10.297	.002
Silane + Drying (38℃)	Equal variances	.092	.767	2.548	12	.026
	Not equal variances			2.548	11.965	.026

DISCUSSION

The results of this study partially support the null hypothesis, according to which neither silanization, heat activation nor fiber post composition would improved the strength of the bond between the fiber-reinforced composite post and the resin cement. Higher shear bond strengths between the fiber-reinforced composite post and the resin cement were achieved with silanization of either post. Between the two post types, silanization of the FRC Postec Plus resulted in higher shear bond strengths. However, heat activation did not significantly improve shear bond strength as there were no significant differences, in this respect, between heat-dried and room-temperature-dried posts.

Clinical failure of a tooth restored with fiber-reinforced post is usually the result of cementation failure. In this study, bonding at the junction between the resin cement and the fiber-reinforced post was evaluated in posts subjected to three types of surface treatment using testing method that was designed based on the results of a previous study by Choi *et al.*[7]. To ensure a bonding surface of constant area, a metal hexagon nut, as a mold, and posts with same diameter were used. In a pilot study, we noted that air bubbles became trapped during deposition of the resin cement, leading to irregularities in the bonding surface area. This problem was remedied by using an Accudose needle tip and syringe (Centrix, Shelton, CT, USA), which allowed the resin cement to be contained within the metal hexagon nut without the trapping of air bubbles. Consequently, we were able to reduce the standard variation of the experimental data reported herein compared to those of the pilot study.

In the present work, the bond strength with the resin cement was higher for fiber-reinforced posts of the silanization group than for control, untreated posts. Conflicting results regarding the effect of silanization on the bond strength between fiber-reinforced posts and resin cement have been reported. Our results are in accordance with those of previous studies showing a significant increase in bond strength after silanization of the fiber-reinforced post,[1,16] although in other studies the improvement obtained with silanization was not significant.[7,13,14]

The higher shear bond strength of the FRC Postec Plus may have been due to the composition of its matrix. While both the glass fibers of the FRC Postec Plus and the quartz fibers of the D.T. Light Post contain hydroxyl groups that can react with a silane coupling agent, the matrix of the D.T. Light Post is an epoxy resin, which lacks these functional groups. By contrast, the matrix of the FRC Postec Plus, which contains UDMA, TEGDMA, ytterbium trifluoride, and highly dispersed silicon dioxide, is similar to the composition of resin cement (Variolink II) such that better coupling is achieved between the two.

Blow dryer was chosen because it is easy to get and can be applied easily in clinical setting. It was examined for capability of maintaining heat temperature and proved maintaining the temperature at 38-40℃ with 30 cm distance in warm mode. But, in this study heat treatment after silanization of the post was not effective in enhancing bond strength. Silane coupling is a technique-sensitive step, given that incomplete evaporation of the solvent may compromise chemical reactions between silane and the glass or quartz fibers of the post.[19] Heat treatment after application of the silane coupling agent is thought to promote complete evaporation of the solvent. Although the optimal temperature has not been determined, in the glass industry, the silane coupling agent is heat-treated at a temperature close to 100℃.[20] Monticelli *et al.* reported that, in dental applications, heat treatment of the silane coupling agent at a temperature of 38℃ but not at 21℃ effectively enhances bond strength.[17] Based on that study, we examined the effects of heat treatment at 38℃ but failed to detect a significant difference compared to drying at room temperature.

Pre-activated silane coupling agents, allows forming siloxane bonds, but they have short shelf life. In this study two-component system (Porcelain liner M) was used in which hydrolysis of the functional group occurs only after the silane coupler (γ-MPTS) is mixed with the acidic monomer (4-META). Thus, in two-component system, complete solvent evaporation is more likely than a pre-activated system. In the previous study, silanization using a two-component silane coupling agent resulted in greater bonding strength when the posts were dried at a higher temperature

(38℃) than at room temperature (21℃).[17] Accordingly, in this study, we used a two-component silane coupling agent, expecting that it would clearly demonstrate the benefits of heat treatment after silanization. However, this was not the case as the two temperatures did not yield significant differences in bonding strength. Similar results were obtained in a previous study, in which the two-component silane produced higher bond strength when air drying was carried out at room temperature (23℃) rather than at a higher temperature (60℃).[21] In that report, it was speculated that while heating may improve solvent evaporation, the extent of the improvement is probably related to the volatile nature of the specific solvent, such that bonding strength might even decline after heating.[21]

The specimen design of this study offered a simple method to control the bonding surface area. In addition, the depth of the light transmission was relatively short, enabling a high rate of light-induced polymerization. A limitation of this study design was the above-described difficulty with the blow dryer not capable of discriminating the effect of evaporation of the solvent from heat activation of silane. The use of a higher controlled range of heat radiating device with small aperture may allow more precise evaluation of the effect of surface treatment, including combinations of silanization, heat treatment, in addition to the micro-mechanical surface processes, such as airborne-particle abrasion.

CONCLUSION

In this *in vitro* study of the bond strength between a fiber-reinforced post and resin cement, the results support the following conclusions:

Silanization of the FRC Postec Plus but not of the D.T. Light Post significantly increased the bond strength between the fiber-reinforced post and the resin cement. Heat treatment after silanization of the fiber-reinforced post did not significantly improve the bond strength between the fiber-reinforced post and the resin cement compared to the strength achieved with silanization followed by drying at room temperature.

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