Effects of post surface conditioning before silanization on bond strength between fiber post and resin cement

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PURPOSE. Post surface conditioning is necessary to expose the glass fibers to enable bonding between fiber post and resin cement. The purpose of the present study was to evaluate the effect of different surface conditioning on tensile bond strength (TBS) of a glass fiber reinforced post to resin cement. **MATERIALS AND METHODS.** In this *in vitro* study, 40 extracted single canal central incisors were endodontically treated and post spaces were prepared. The teeth were divided into four groups according to the methods of post surface treatment (n=10): 1) Silanization after etching with 20% H_2O_2, 2) Silanization after airborne-particle abrasion, 3) Silanization, and 4) No conditioning (Control). Adhesive resin cement (Panavia F 2.0) was used for cementation of the fiber posts to the root canal dentin. Three slices of 3 mm thick were obtained from each root. A universal testing machine was used with a cross-head speed of 1 mm/minute for performing the push-out tests. Two-way ANOVA and Tukey post hoc tests were used for analyzing data (α=0.05). **RESULTS.** It is revealed that different surface treatments and root dentin regions had significant effects on TBS, but the interaction between surface treatments and root canal regions had no significant effect on TBS. There was significant difference among H_2O_2 + Silane Group and other three groups. **CONCLUSION.** There were significant differences among the mean TBS values of different surface treatments. Application of hydrogen peroxide before silanization increased the bond strength between resin cements and fiber posts. The mean TBS mean values was significantly greater in the coronal region of root canal than the middle and apical thirds. ***[J Adv Prosthodont 2013;5:126-32]***

KEY WORDS: Resin cements; Dental bonding; Post and core technique; Composite resins; Fiber reinforced; Bond strength

INTRODUCTION

Retaining coronal restoration in endodontically treated teeth usually requires the use of intra-canal posts.[1-6] Although high success rates have been reported for fiber-reinforced composite (FRC) post restorations,[7,8] root fracture and loss of retention are still the most common reasons for failing these restorations.[9,10] Due to the similar elastic modulus of fiber posts and root dentin, distribution of loads in the root canal has an uniform pattern.[11,12] So, less occurrence of root fracture has been reported for these restorations in comparison to metallic post retained restorations.[7,8] The fiber post retention in root canal depends on the bond strength between different parts of the combined 'sandwich' post-cement-dentin assembly.[13-16] It has estimated that 60% of the fiber post failures occurred between the fiber post and resin cement.[17,18] So, achieving effective bond strength between the FRC post and resin cement is important.[4,19]

Many post surface treatment protocols (chemical and micro-mechanical modifications) have been reported to improve surface energy characteristics of dental fiber reinforced posts.[6,16,20-24] The bond strengths between post and resin cement can be improved chemically by post surface silanization.[9,22,25,26] However, there is no definitive agree-

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ment on this subject.[24,27] On the other hand, some studies concluded that roughening the post surface either with micro-mechanical methods like sandblasting[20,24,28-34] or application of hydrogen peroxide[35-37] can improve the retention of FRC posts that are cemented with resin cements. However, the effectiveness of these roughening techniques is not completely confirmed.[6,27,28]

Since chemical surface treatments alone may not create a strong bond between fiber posts and resin cements,[21] some micro-mechanical treatment methods have also been performed to remove a surface layer of fiber posts and exposes the quartz fibers for silanization.[38] This type of surface treatments that combine both a chemical and micromechanical method is called as alternative etching techniques.[21] Although, sandblasting and hydrofluoric acid etching are used to improve the bonding of fiber posts to resin cement,[18,24,27] these techniques can damage the glass fibers and affect the post integrity.[18] Hydrogen peroxide (H_2O_2) is one of the materials that can selectively dissolve the epoxy matrix without interfering with the glass fibers and can expose the fibers to be silanated.[6,20,39,40]

The purpose of this study was to investigate the effects of some post surface conditioning methods before silanization on the bond strength between fiber post and resin cement. The null hypothesis to be investigated was that the bond strength between fiber post and resin cement with the various surface conditioning methods are not significantly different.

MATERIALS AND METHODS

In this *in vitro* study, 40 human maxillary incisor single-rooted teeth free of caries, cracks, fractures, resorption, with fully developed roots and without previous root canal treatments or posts were selected. The teeth were cleaned of residues of soft tissue or calculus and disinfected by placing them in 2.5% NaOCl (Golrang, Golrang co., Tehran, Iran) for two hours and then stored in 0.1% NaN_3 solution.

Diamond discs (Ref.070, D&Z, Berlin, Germany) were mounted in a dental lathe machine (KaVo Polishing Unit. EWL 80, Germany) and used for removing coronal structures of teeth. This job was done at low speed under water irrigation to achieve a 15 mm root length. The pulp tissues were removed with barbed broach (Dentsply/Maillefer, Ballaigues, Switzerland). Canal working lengths were 1-mm short of the apical foramen. Canal instrumentation was done by a same operator using the step-back technique. Themaster apical file was the number 35 K file (Dentsply/Maillefer, Switzerland). Canals were irrigated with 2 mL of 5.25% solution of Sodium hypochlorite during the canal preparation stages (Golrang, Iran). Finally, paper points (Aria dent, Asia ChemiTeb Co, Tehran, Iran) were used for drying the root canals. The obturation was performed with vertical condensation method, using AH26 (Dentsply Caulk, Milford, Germany) and gutta-percha (Aria dent, Iran). A provisional restorative material (GC Caviton; GC Dental Products Corp., Tokyo, Japan) was used for filling the coronal root canal orifices, and then to ensuring the setting of the used sealer, the teeth were stored for one week in 100% humidity at 37℃.

After one week, the gutta-percha in the coronal aspect of each root was removed with a Gates Glidden drill #3 (Dentsply/Maillefer, Switzerland) so that, 4 mm gutta-percha was preserved in the apex to maintain the apical seal. The all post spaces were prepared to a depth of 10 mm, measured from top of the sectioned root surface, with dedicated drills provided by a post manufacturer (Fibio, Anthogyr, Sallanches, France). To standardize the final shape of the post space, a No. 3 drill of this system was used as the last drill. Finally, the canals were irrigated with distilled water and dried with paper points (Aria dent, Iran).

A glass reinforced fiber post (Hetco fiber post, Hakim Toos, Mashhad, Iran) (Table 1) size #3 was tried in the post spaces and then all posts were cut at a distance of 10 mm from the apical end with diamond discs (Ref. 070, D&Z) mounted in a dental lathe machine (KaVo Polishing Unit., KaVo EWL) under water irrigation. This procedure was done for post length standardization and creation diameter similarity among the used posts.[19] In all groups, the shortened Hetcoposts (Hakim Toos, Iran) were cleaned with 70% ethanol for 60 seconds, rinsed with distilled water and air dried.

The posts were randomly divided into four groups of 10 specimens each, according to surface treatments used:

H_2O_2 + Silane Group. At the first, the 30% H_2O_2 were diluted to 20% H_2O_2 and then the posts in this group were immersed in diluted H_2O_2 for 20 minutes at room temperature. After 2 minutes rinsing with running water, they were air dried. Then the posts surfaces were painted with a single layer of a silane coupling agent (Ultradent® Porcelain Etch and Silane, Ultradent Products Inc., UT, USA) for 60 seconds and dried for 60 seconds with gentle air stream.

Sandblast + Silane Group. The posts in this group were sandblasted with 50 µm aluminum oxide particles using an oral Microblaster (Dento-prep, Ronviga, Daugaord, Denmark) for 10 seconds at 0.25 MPa pressure from a 10 mm distance. After rinsing posts with 96% ethanol, they were air dried. The posts surfaces were painted with a single layer of thesilane coupling agent (Ultradent® Porcelain

Table1. Chemical composition of used Glass fiber in this study (as presented by manufacturer)

Chemical ingredients	%
Silicon dioxide	55
Calcium oxide	20
Baron oxide	12
Aluminum oxide	14
Sodium oxide	1
Potassium oxide	1
Magnesium oxide	4

Etch and Silane) and were air dried for 60 seconds with gentle air stream.

Silane Group. Without any surface pre-treatment, only a single layer of thesilane coupling agent (Ultradent® Porcelain Etch and Silane) was applied on the posts surfaces for 60 seconds and then air dried for 60 seconds with gentle air stream.

Control Group. This was a control group without any surface treatment.

In all groups, the root canal dentin walls were conditioned with an autopolymerizing primer (ED primer, Kuraray Medical Inc., Tokyo, Japan) before cementing the posts. For this purpose, one drop of each liquid (A and B) (ED Primer) were mixed and applied to the root canal walls with a microbrush (Atlas microbrush; Atlas Dandan Co., Tehran, Iran) for 60 seconds. Then, the post space was air dried and the excess primer was removed with paper points (Aria dent, Iran). For fiber post cementation, equal amounts of base and catalyst pastes of an adhesive composite resin cement (Panavia F2.0, Kuraray Medical Inc., Japan) were mixed and applied on the posts surface and into the root canals with a lentulo spiral instrument (Dentsply/Maillefer, Switzerland). Using gentle finger pressure, the fiber posts were inserted into the prepared post spaces to full depth. The excess cement was removed with a disposable brush. Finally, the remaining cement around the post was protected with oxygen inhibiting gel (Oxyguard II, Kuraray Medical Inc.).

After the initial chemical polymerization, the resin cement was light cured for 60 seconds in such a way that the tip of the light unit (Coltolux50, Coltene, Altstatten, Switzweland) was in close contact with the coronal end of the fiber post. Accurate light intensity of the light output was monitored before each light exposure by Coltolux light meter (Coltene, Altstatten, Switzerland).

After the cementation procedures, all dental root specimens were placed in a light-proof box, containing 37℃ sterile saline for one week. All the specimens were subjected to thermocycling treatment for 5,000 cycles at temperatures alternating between 5 and 55℃ for 30 seconds each, with an intermediate pause of 15 seconds.

The coronal part of each dental root was sectioned perpendicular toits long axis using a diamond disc (Ref.070, D&Z, Germany) mounted in a cutting machine (TL-3000, VafaeiIndustrial, Tehran, Iran) at low speed under constant distilled water irrigation to create 3 mm thick slices. In this manner,threepost/dentinsections (coronal, middle and apical) were obtained from each root. Due to usingtaperedfiber posts, the post diameters were measuredusingadigital caliper (Mitutoyodigital caliper 500-714-10, Mitutoyo Co., Tokyo, Japan) with 0.01 mm accuracy.

A universal testing machine (Walt + Bai AG Testing Machines Industriestrass 4, Löhningen, Switzerland) was used for applying tensile force at acrosshead speed of 1 mm/min. The push-out pin was placed on the center of the apical end of the post surface and in an apico-coronal direction without inserting extra forces on the surrounding root canal walls. Therefore, three push-out pins were constructed in three diameters (0.7, 0.9 and 1.0 mm) that were used for each three root sections respectively (apical, middle and coronal parts). The peak force (N) required to extrude the fiber post from each root slice was recorded for all specimens. The bond strength in MPa was calculated by dividing the load at failure (N) by the total area of the cemented post. This calculation was done with the following formula:

$$A = \pi(r_1 + r_2)\sqrt{(r_1 - r_2)^2 + h^2}$$

In this formula, π is the constant 3.14, r_1 is the coronal post radius, r_2 is the apical post radius and h is the slice thickness in mm.

The collected data were analyzed (SPSS/PC16.0; SPSSInc., Chicago, IL, USA) using two-way ANOVA and Post Hoc Tukeytest at $P<.05$ level of significance.

RESULTS

Table 2 shows the mean tensile bond strength (TBS) and standard deviation values for all experimental groups in different root canal regions. The two-way ANOVA showed that different surface treatments ($P=.004$) and root canal dentin regions ($P<.001$) had significant effects on TBS, but interaction between surface treatments and root canal regions ($P=.068$) had no significant effects on TBS (Table 3).

The H_2O_2 + Silane Group had the highest TBS mean value especially in the coronal region. The lowest TBS mean value was seen in the control group and in the apical region.

Post Hoc Tukeytest revealed that there was significant differences between H_2O_2 + Silane Group and other three groups ($P<.05$) (Table 4).

DISCUSSION

Based on the results of this investigation, the null hypothesis that the various surface treatments do not have any effects on the TBS between fiber post and resin cement was rejected.

Fiber posts are comprised of a matrix resin that surrounds different types of fibers. In the glass fiber posts, the fibers are made of glass and the matrix is made of epoxy resin.[5] The epoxy polymers cannot chemically bond with composite resin cements because of their highly cross-linked structure.[41] Therefore, some authors propose using silane coupling agent on the fiber post surface to increase the TBS between the fiber post and resin cement. However, it has been said that silane cannot increase the bond strength of the fiber posts to resin cements or composite core materials.[17,32,42-45] In fact this is due to this fact that the chemical bond is achieved mainly by creating covalent bonds between the silane coupling agent and the composite resin; and between silane and the exposed glass fibers or filler particles of the post.[46,47] In the present study, likes some other investigations, it was found that silane alone cannot increase the TBS of the fiber posts to resin cements (Table 4).

Table 2. Descriptive statistics and mean tensile bond strengths (MPa) and standard deviations for different surface treatments (n=10)

Treatment	Region	Mean	SD
H_2O_2 + Silane	coronal	21.5365	8.28316
	middle	19.0880	6.36184
	apical	9.1230	1.19001
Sandblast + Silane	coronal	18.4550	6.46585
	middle	10.1700	5.66025
	apical	6.5450	3.10299
Silane	coronal	20.5310	6.88138
	middle	14.5660	8.17108
	apical	6.3020	1.81350
Control	coronal	9.7650	4.26970
	middle	9.0770	4.69274
	apical	5.5850	2.15893

Table 3. Two-way ANOVA results on the effects of surface treatments, root canal regions and their interaction on the tensile bond strength

	Type III sum of squares	df	Mean square	F	Sig.
Corrected Model	3492.492[a]	11	317.499	9.656	.000
Intercept	20913.148	1	20913.148	636.050	.000
Treatment	472.255	3	157.418	4.788	.004
Region	2620.725	2	1310.363	39.853	.000
Treatment * Region	399.512	6	66.585	2.025	.068
Error	3551.008	108	32.880		
Total	27956.648	120			
Corrected Total	7043.500	119			

a. R Squared = .496 (Adjusted R Squared = .444)

Table 4. Post hoc test (Tukey's HSD) for comparing all test groups

Treatment (I)	Treatment (J)	Mean difference (I-J)	Std. error	Sig.	95% confidence interval	
					Lower bound	Upper bound
H_2O_2+ Silane	Sandblast + Silane	4.8592*	1.48053	.007	.9957	8.7226
	Silane	4.7292*	1.48053	.010	.8657	8.5926
	Control	3.9362*	1.48053	.044	.0727	7.7996
Sandblast + Silane	H_2O_2 + Silane	-4.8592*	1.48053	.007	-8.7226	-.9957
	Silane	-.1300	1.48053	1.000	-3.9934	3.7334
	Control	-.9230	1.48053	.924	-4.7864	2.9404
Silane	H_2O_2 + Silane	-4.7292*	1.48053	.010	-8.5926	-.8657
	Sandblast + Silane	.1300	1.48053	1.000	-3.7334	3.9934
	Control	-.7930	1.48053	.950	-4.6564	3.0704
Control	H_2O_2 + Silane	-3.9362*	1.48053	.044	-7.7996	-.0727
	Sandblast + Silane	.9230	1.48053	.924	-2.9404	4.7864
	Silane	.7930	1.48053	.950	-3.0704	4.6564

Based on observed means.
The error term is Mean Square (Error) = 32.880.
*. Denotes the mean difference is significant at the 0.05 level.

Roughening the fiber post surface with micro-mechanical procedures can bring the glass fibers in better contact with silane coupling agent.[6,20,40] Although, sandblasting is used to roughen the fiber posts surface for better bonding,[18,24,27] also it can damage the glass fibers.[18] Instead, hydrogen peroxide (H_2O_2) can selectively dissolve the epoxy matrix without damaging the glass fibers.[6,20,39,40] The present study showed that using H_2O_2 before silanization can significantly improve the TBS in comparison to Sandblast + Silane, Silane and Control groups ($P<.05$) (Fig. 1), which is in accordance with some other studies.[18,24,27]

Yet, Radovic *et al.*[48] showed that even if sandblasting can improve the bond strength between fiber post and resin cement but water aging can reduce the bond strength of sandblasted specimens.

It is said that by using H_2O_2 as a pretreatment for using silane coupling agent, a better chemical bonding between silane and fiber post was achieved.[32,36,40,43,47] In the studies that bonding agent was applied immediately after using H_2O_2 (without silane coupling agent as a mediator), the TBS mean value was reduced.[1] However, some authors claimed that pre-treatments are not necessary before silane application.[49]

For root canal regions, our results are in accordance with other studies that reported higher bond strength for coronal region of the root than other tworegions.[9,13,19] The tubular density and diameter of the coronal third of the root canal dentin are more than other regions. Furthermore, the dentinal hybridization is not uniform and lateral branches of resin tags are not seen in the apical regions of root canal dentin.[50] Furthermore, more access to the coronal portion of the canal makes etching and applying the adhesive agents[17,50] and light transmission to canal walls[50] become more effective in this area. However, Aksornmuang *et al.*[9] found no significant differences in the bond strengths among different regions of the root canal walls.

The present study had some limitations. For example, the specimens had no coronal tooth structure, only one type of fiber post and adhesive were evaluated and the influences of fatigue loading on the push out TBS of specimens were not investigated.

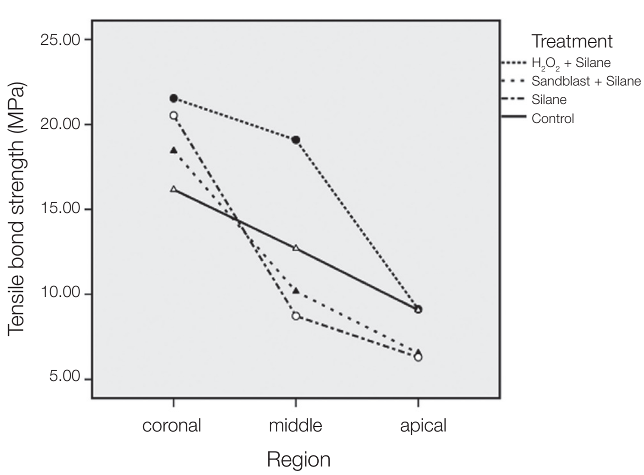

Fig. 1. Results for tensile bond strength in four experimental groups.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions were drawn:

There were significant differences between the mean tensile bond strength values of different surface treatments and root canal regions. Application of hydrogen peroxide before silanization increased the bond strength of resin cement to the fiber posts. The coronal region of the root canal showed significantly higher mean bond strength values than the other regions.

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