Comparison of the effect of shear bond strength with silane and other three chemical presurface treatments of a glass fiber-reinforced post on adhesion with a resin-based luting agent: An *in vitro* study

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Abstract

Background: Loss of retention has been cited to be the most common cause of the failure of postretained restoration with irreversible consequences when materials with different compositions are in intimate contact at the post/adhesive interface. With this background, a study was conducted to improve the adhesion at the resin phase of fiber posts using silane and other chemical pretreatments. **Materials and Methods:** Hundred glass fiber-reinforced posts were tested with 4 different protocols (n = 25) using silane as a control (Group A) and other three experimental groups, namely, Group B-20% potassium permanganate, Group C-4% hydrofluoric acid, and Group D-10% hydrogen peroxide were pretreated on the postsurface followed by silanization. These specimens were bonded with dual-polymerizing resin-based luting agent, which were then loaded at the crosshead speed of 1 mm/min to record the shear bond strength at the post/adhesive interface. The data were analyzed using one-way ANOVA test for multiple group comparisons and the *post hoc* Bonferroni test for pairwise comparisons ($P < 0.05$). **Results:** Group B showed more influence on the shear bond strength when compared to other protocols, respectively ($P < 0.001$). **Conclusion:** Alone silanization as a surface treatment did not improve the bond strength. Combination of chemical presurface treatments followed by silanization significantly enhanced the bond strength at the post/adhesive interface.

Keywords: Dual polymerizing resin cement, glass fiber-reinforced post, shear bond strength, silanization

Introduction

Restoration of a root-filled tooth will require the placement of a post to ensure adequate retention to the core when there is insufficient coronal tooth structure.[1] Among the different types of posts available, glass fiber-reinforced posts (GFPs) have gained popularity because they can be adhesively bonded to the root canal contributing to the formation of a homogenous dentin/post adhesive system, which in this case is known as a tertiary monoblock.[2,3] Nevertheless, because of their highly cross-linked epoxy resin-based structure, GFPs need to be superficially treated to improve its chemical interaction with resin cement.[4]

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Consequently, studies diverge about the real benefit of silanization in improving postretention;[5-10] therefore, other substances have currently been investigated such as acidic solutions that achieved positive results[11-13] although there is a lack of information about the negative effects on the poststructure that may occur.

Few have suggested, use of silane coupling agent in coating application on the postsurface before using the resin cement to promote adhesion between the postsurface and polymeric molecules of the resin material.[14,15] Silanes have reported to enhance the wettability and the chemical union between the resin-based materials and glass fibers.[9,16] Still in many cases, interfacial failure has been attributed to chemical incompatibility between the post and resin cement.[2,17-19] To improve the bond strength at the post/adhesive interface, surface treatments in the form of chemicals were aimed to produce roughness on the

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surface of the post, enhancing mechanical interlocking between the post and the resin cement.[14,15]

Hence, the aim of this *in vitro* study was to compare the effect of four chemical surface treatments, namely, silanization, potassium permanganate ($KMnO_4$ 20% Vol.), hydrofluoric acid (4% HF), and hydrogen peroxide (10% H_2O_2) of a GFP on adhesion with a resin-based luting agent. Two hypotheses were tested: (1) Application of silane would improve the bond strength at the post/adhesive interface and (2) application of silane plus other chemical pretreatments on the postsurface would enhance its adhesion to resin cement.

Materials and Methods

Preparation of specimens

Hundred polyvinylchloride cylinders packed with autopolymerizing acrylic resin (Methyl methacrylate resin, DPI Dental products of India Ltd, Mumbai.) were used for the preparation of the specimens. A casted metal post having dimensions same as the DT post was wrapped with a tin foil (Shandong Loften Aluminium foil Co. Ltd., China.) and inserted in a dough stage to obtain a standardized insertion depth and central position of the post.

After polymerization, the confirmation of the post space was done in such a way that 3 mm of the fiber post should protrude out for the attachment of an acrylic glass plate that encircled it. The acrylic glass plate with one side flattened of 3 mm thickness was fabricated (Bisco, Inc, Schaumburg, IL, USA) and mounted perpendicular to the cylinder and the whole assembly was confirmed on the Universal testing machine (Instron 4467; Instron Corp, Norwood, Mass) [Figure 1a].

Grouping of samples

Hundred radio-opaque translucent glass fiber-reinforced-posts (D. T. Light-Post, size no. 3; Bisco, Inc., Schaumburg, IL, USA), with a length of 20 mm and a maximum diameter of 2.2 mm, were randomly picked from the boxes and divided into four groups of 25 each, depending on the postsurface pretreatment to be performed.

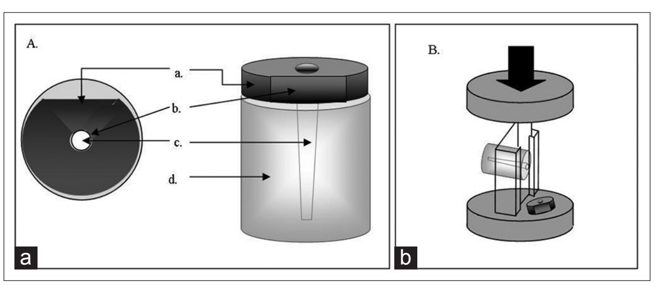

Figure 1: (a) Schematic representation of the specimen preparation for shear bond strength test. a: Acrylic glass plate, b: Bonding surface area of the resin luting agent and post, c: Glass fiber-reinforced post, d: Methyl methacrylate cylinder. (b) Schematic representation of the specimen loaded until failure

Surface treatments for the glass fiber-reinforced posts

The post spaces were cleaned with 95% alcohol and dried with an air stream and then the postsurface was subjected to four different surface treatments which were as follows:

- Group A: Silanization of the postsurface for 60s without any treatment of the resin phase. (Ivoclar, Vivadent AG, Schaan, Liechtenstein)
- Group B: Treatment with potassium permanganate (20% Vol.) followed by silanization
- Group C: Treatment with 4% hydrofluoric acid for 60s followed by silanization
- Group D: Treatment with 10% hydrogen peroxide for 20 min followed by silanization.

The etching procedure of Group B consists of subsequent application of three chemical solutions that firstly led to swelling of epoxy resin which altered the chemical structure, then etching which removed the epoxy resin matrix previously degraded by the solvent followed by neutralizing the excess permanganate that initiated the cleaning on the surface of the GFP. Each surface-treated post was rinsed with deionized water for 3 min, followed by air-drying.

The etching procedure for Group C includes fiber-reinforced posts that were immersed in 4% HF for 60s at room temperature and then rinsed with deionized water. The posts in the Group D were immersed in 10% hydrogen peroxide for 20 min at room temperature and then rinsed with deionized water.

A single layer of silane coupling agent (Ivoclar, Vivadent AG, Schaan, Liechtenstein.) was then applied with micro-tip brush to the postsurface of each of the four groups, i.e., Group A, Group B, Group C, and Group D and gently air-dried after 60s, according to manufacturer's recommendations.

Procedures for cementation

A single coat of Prime and Bond NT Dual cure bonding agent was applied to the etched post space. An air syringe was used to dry thoroughly for 5s to remove the excess. The bonding agent was then light cured for 10s.

Equal quantities of Calibra light shade base and regular viscosity catalyst (Dentsply, Caulk, Milford, U.S.A) were mixed for 20–30s till the paste obtained a uniform color. The luting agent was then applied on the postsurface which was then seated in the created post space, and firm pressure was maintained for 10s until the post is stable. Then, curing was done with a halogen polymerizing unit (600 mW/cm^2 output) (VIP; Bisco, Inc.) for about 20s with a tip to specimen distance held constant at 2 mm. An additional 40s of light polymerization was performed to ensure optimal polymerization of the luting agent.

All specimens were prepared by the same investigator on the same day to ensure the standardization. Before the shear bond strength test, the specimens were stored in deionized water for 24 h at 37°C.

Testing of samples

The specimens were loaded at an angle of 90° to the tip of the plunger. For this purpose, a customized mounting fixture of aluminum block was attached to the lower jaw of the universal testing machine (Instron 4467; Instron Corp., Norwood, Mass).

The load was applied to the specimen at a crosshead speed of 1 mm/min till the acrylic glass plate separated from the fiber-reinforced posts [Figure 1b], and the digital readings were recorded. The shear bond strength of the luting agent to the post was expressed in megapascals (MPa) which was then calculated by dividing the load (N) at failure by the bonding surface (mm^2).

Statistical methods

The data analysis was performed using SPSS (version 10.0 computer software for windows, Stanford, California) using one-way ANOVA test and also pairwise comparative analysis was done using *post hoc* Bonferroni test. The mean difference is statistically significant at the 0.05 level.

Results

The control group values obtained for Group A, i.e., 16.421 MPa were low which showed a less influence of silane as a surface treatment. The other experimental groups whose values obtained for Group B, Group C, and Group D were 27.233, 21.781, and 19.037 MPa, respectively. This showed that silanization improved the bond strength at the post/adhesive interface using chemical pretreatments [Table 1]. According to Table 2, comparative evaluation of the both control and experimental groups yielded a $P < 0.001$ indicating a highly significant difference between the tested groups at an alpha level of 0.05. Figure 2, graphically represented differences at significant levels in the mean and standard deviation values for tested groups.

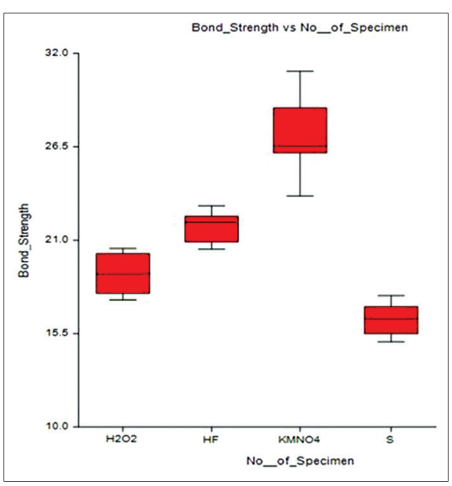

Figure 2: Box plot showing the mean bond strength values of four groups of chemical surface treatments plotted at significant levels

Discussion

The first hypothesis of the present study was that the application of silane would not improve the bond strength at the post/adhesive interface, and according to results displayed in Table 1, this hypothesis could be fully accepted because the application of silane promoted similar bond strength when compared with the values obtained for nonsilanized posts.[4,6]

In an attempt to maximize the resin bonding to the fiber-reinforced posts, silane application was introduced as it represented the most investigated surface treatment in the current literature.[16] The working mechanism of silane is based on increasing the postsurface wettability and consequent chemical bridge formation with the monomers of resin cement/composites.[20] The obtained values for Group A showed less influence on the bond strength between the fiber-reinforced post and resin-based luting agent ($P < 0.001$) [Table 2] probably because of the limited area on the postsurface exhibiting exposed glass fibers because they are superficially protected by epoxy resin;[20-22] in fact, the GFR post used in this study is constituted of 60 and 40 wt% of glass fibers and epoxy resin, respectively (manufacturer's information). Second, the application of presurface treatments produced between resin cement and silanized posts was

Table 1: Mean and standard deviations of the shear bond strength results obtained in the study

	Mean	SD	SE	95% CI for mean	
				Lower bound	Upper bound
Group A	16.421	0.89118	0.28182	15.7835	17.0585
Group B	27.233	2.21948	0.70186	25.6453	28.8207
Group C	21.781	0.81415	0.25746	21.1986	22.3634
Group D	19.037	1.06549	0.33694	18.2748	19.7992
Total	21.118	4.00402	0.70782	18.1272	20.7506

SD: Standard deviation; SE: Standard error; CI: Confidence interval

Table 2: Statistically significant differences among the control and experimental groups

Group (I)	Group (J)	Mean difference (I-J)	*P*	Significant/ nonsignificant
Group A	Group B	−10.81	<0.001	Highly significant
	Group C	−5.36		
	Group D	−2.61		
Group B	Group C	5.45	<0.001	Highly significant
	Group D	8.19		
Group C	Group D	2.74	<0.001	Highly significant

probably affected due to a more reactive surface of the quartz fibers that provided additional frictional resistance and sites for silanization that further enhanced the bonding ability, which was the second hypothesis of the study.[15,17]

The bond strength values for Group B obtained were high compared to other groups ($P < 0.001$) [Table 2]. This may be due to the partial removal of the epoxy resin matrix of the fiber posts that created "retention spaces" among the fibers for infiltration of the resin cement as confirmed microscopically.[14,23] A mild treatment protocol was chosen to develop superficial etching during the conditioning process that enhanced the adaptation of resin composite to the fiber posts. Furthermore, no cracking was seen on the underlining untreated epoxy resin of the fiber posts leaving the exposed quartz fibers undamaged.[14,15]

Other authors also demonstrated that the epoxy resin covering the fibers can be selectively dissolved by the action of oxidizing solutions, namely hydrogen peroxide and hydrofluoric acid.[14,15] These findings were confirmed microscopically when 4% hydrofluoric acid was used for conditioning methacrylate-based fiber posts where selective dissolution of the glass component of the fiber post produced an irregular pattern of microspaces that facilitated the penetration of the composite.[23,24] Some authors are also mentioning the use of procedures for treatment with hydrofluoric acid to be aggressive. However, these techniques can affect the integrity of the fiber post, and microscopic analysis has revealed an uneven removal of the epoxy resin matrix.[17] Despite fewer disadvantages still in the current study, good bond strength was achieved by Group C when compared to Group D, respectively [Table 2].

Hydrogen peroxide, on the contrary, is a considerably milder technique because the exposed quartz fibers remain smooth and leave the underlying epoxy resin matrix intact after the etching procedures.[15,16,24] Hence, the results obtained for Group D showed lowest bond strength values among the other groups ($P < 0.001$) [Table 2]. Other microscopic investigations also revealed the removal of the superficial layer of epoxy resin that exposed a less surface area for quartz fibers.[14,25]

Conclusion

The present *in vitro* study demonstrated enhanced bond strength at the post/adhesive interface using 20% Vol. $KMnO_4$ followed by silanization when compared with other experimental groups, respectively. Although this outcome was positive, the study demonstrated some limitations such as the absence of scanning electron microscope to characterize the failure patterns of the bonding surface and the lack of specimen aging. In addition, assessment of long-term durability and for improved simulation of the *in vivo* environment could have been applied. It would also be of interest to analyze other types of fiber posts and to compare their performances.

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Conflicts of interest

There are no conflicts of interest.

References

1. Gutmann JL. The dentin-root complex: Anatomic and biologic considerations in restoring endodontically treated teeth. J Prosthet Dent 1992;67:458-67.
2. Aksornmuang J, Foxton RM, Nakajima M, Tagami J. Microtensile bond strength of a dual-cure resin core material to glass and quartz fibre posts. J Dent 2004;32:443-50.
3. Tay FR, Pashley DH. Monoblocks in root canals: A hypothetical or a tangible goal. J Endod 2007;33:391-8.
4. Machado FW, Bossardi M, Ramos Tdos S, Valente LL, Münchow EA, Piva E. Application of resin adhesive on the surface of a silanized glass fiber-reinforced post and its effect on the retention to root dentin. J Endod 2015;41:106-10.
5. Bitter K, Meyer-Lückel H, Priehn K, Martus P, Kielbassa AM. Bond strengths of resin cements to fiber-reinforced composite posts. Am J Dent 2006;19:138-42.
6. Choi Y, Pae A, Park EJ, Wright RF. The effect of surface treatment of fiber-reinforced posts on adhesion of a resin-based luting agent. J Prosthet Dent 2010;103:362-8.
7. Kim YK, Son JS, Kim KH, Kwon TY. A simple 2-step silane treatment for improved bonding durability of resin cement to quartz fiber post. J Endod 2013;39:1287-90.
8. Liu C, Liu H, Qian YT, Zhu S, Zhao SQ. The influence of four dual-cure resin cements and surface treatment selection to bond strength of fiber post. Int J Oral Sci 2014;6:56-60.
9. Perdigão J, Gomes G, Lee IK. The effect of silane on the bond strengths of fiber posts. Dent Mater 2006;22:752-8.
10. Tian Y, Mu Y, Setzer FC, Lu H, Qu T, Yu Q. Failure of fiber posts after cementation with different adhesives with or without silanization investigated by pullout tests and scanning electron microscopy. J Endod 2012;38:1279-82.
11. Mohsen CA. Evaluation of push-out bond strength of surface treatments of two esthetic posts. Indian J Dent Res 2012;23:596-602.
12. Sipahi C, Piskin B, Akin GE, Bektas OO, Akin H. Adhesion between glass fiber posts and resin cement: Evaluation of bond strength after various pre-treatments. Acta Odontol Scand 2014;72:509-15.
13. Samimi P, Mortazavi V, Salamat F. Effects of heat treating silane and different etching techniques on glass fiber post push-out bond strength. Oper Dent 2014;39:E217-24.
14. Monticelli F, Toledano M, Tay FR, Cury AH, Goracci C, Ferrari M. Post-surface conditioning improves interfacial adhesion in post/core restorations. Dent Mater 2006;22:602-9.
15. Monticelli F, Osorio R, Sadek FT, Radovic I, Toledano M, Ferrari M. Surface treatments for improving bond strength to prefabricated fiber posts: A literature review. Oper Dent 2008;33:346-55.
16. Sumitha M, Kothandaraman R, Sekar M. Evaluation of post-surface conditioning to improve interfacial adhesion in post-core restorations. J Conserv Dent 2011;14:28-31.
17. Vano M, Goracci C, Monticelli F, Tognini F, Gabriele M, Tay FR, *et al.* The adhesion between fibre posts and composite resin cores: The evaluation of microtensile bond strength following various surface chemical treatments to posts. Int Endod J 2006;39:31-9.
18. Monticelli F, Goracci C, Grandini S, García-Godoy F, Ferrari M. Scanning electron microscopic evaluation of fiber post-resin core units built up with different resin composites. Am J Dent

2005;18:61-5.
19. Kalkan M, Usumez A, Ozturk AN, Belli S, Eskitascioglu G. Bond strength between root dentin and three glass-fiber post systems. J Prosthet Dent 2006;96:41-6.
20. Zicari F, De Munck J, Scotti R, Naert I, Van Meerbeek B. Factors affecting the cement-post interface. Dent Mater 2012;28:287-97.
21. Rathke A, Haj-Omer D, Muche R, Haller B. Effectiveness of bonding fiber posts to root canals and composite core build-ups. Eur J Oral Sci 2009;117:604-10.
22. Kim HD, Lee JH, Ahn KM, Kim HS, Cha HS. Effect of silane activation on shear bond strength of fiber-reinforced composite post to resin cement. J Adv Prosthodont 2013;5:104-9.
23. Mazzitelli C, Ferrari M, Toledano M, Osorio E, Monticelli F, Osorio R. Surface roughness analysis of fiber post conditioning processes. J Dent Res 2008;87:186-90.
24. Naves LZ, Santana FR, Castro CG, Valdivia AD, Da Mota AS, Estrela C, *et al.* Surface treatment of glass fiber and carbon fiber posts: SEM characterization. Microsc Res Tech 2011;74:1088-92.
25. Yenisey M, Kulunk S. Effects of chemical surface treatments of quartz and glass fiber posts on the retention of a composite resin. J Prosthet Dent 2008;99:38-45.