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ORIGINAL RESEARCH

Effect of two Resin Cements and two Fiber Post Surface Treatments on Push-out Bond Strength between Fiber Post and Root Dentin

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ABSTRACT

Aim: To evaluate the effect of fiber post surface treatments on push-out bond strength between fiber post and root dentin.

Materials and methods: Sixty bovine mandibular teeth (N = 60) were sectioned (16 mm), prepared (12 mm), embedded with acrylic resin and then allocated into six groups (n = 10): Gr1- Silane coupling agent (Sil) + Conventional resin cement AllCem (AlC); Gr2- Sil + Conventional resin cement RelyX ARC (ARC); Gr3- tribochemical silica coating (TBS) + AlC; Gr4– TBS + ARC; Gr5- No treatment (NT) + AlC; Gr6– NT+ ARC. Specimens were sectioned in four slices (2 mm) and submitted to push-out test. Fracture analyses were executed at x200. The values of the push-out bond strength were submitted to two-way ANOVA and Tukey test ($\alpha = 0.05$).

Results: Resin cement did not affect the bond strength values ($p = 0.9674$), fiber post surface treatment affected the push-out bond strength ($p = 0.0353$), interaction between factors did not affected the values ($p = 0.338$). Tukey test did not show differences between the groups. Adhesive failure between cement and dentin was predominantly.

Conclusion: The fiber post surface treatment appears have no influence on bond strength between fiber post and root dentin.

Clinical significance: The tested fiber posts surface treatment appears do not influence the fiber post bond behavior.

Keywords: Adhesion, Fiber post surface conditioning, Intraradicular dentin, Push-out test.

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INTRODUCTION

Endodontic treatment may result in weakening of the tooth, leading to greater susceptibility to fracture and reduced coronary structure, complicating the use of an intracanal post to retain coronary restorations.[1] Prefabricated fiber reinforced posts are indicated in this context, as they generate lower stress concentrations in the remaining root as compared with metallic cast posts and cores.[2-4]

Clinical studies shown that the main causes of failure in teeth restored with fiber posts relate to post/restoration decementation, and can be attributed to defects in the interfaces between dentin and cement, or cement and the fiber post.[5-7] Many *in vitro* studies have investigated the factors that may affect the union at these interfaces, including evaluation of numerous adhesive systems, resin cements and post surface treatments.[8-12]

The main functions of resin cement are to confer retention and resistance to restorations, and to negate coronal microleakage.[13] Resin cements can be classified into three categories, based on the method of polymerization: chemical, photo activated and dual cure. Dual cure resin cements are the most commonly recommended, because they allow a control of the working time, have good flowable properties, providing a thin film of cement, are available in a variety of colors and opacities, and guarantee polymerization in zones of difficult access to light.[14-16] The use of a resin cement may have a considerable influence on post retention as compared with not-resinous cements.[14,17] Cohen et al (1997) and Rosin et al[17] found higher push-out bond values for groups cemented with resin cements in comparison with zinc phosphate cement.

If a conventional resin cement is chosen for fiber post cementation, an adhesive system should be used. Three-step etch-&-rinse adhesive systems combined with resin cements have yielded better bond strength values compared to self-etching adhesive systems and two-step total etching adhesive systems;[18-22] on the contrary, the technique is very sensitive. Furthermore, different fiber post surface treatments have been tested in order to increase the bond strength between fiber posts and resin cement.[8-10,19,23,24] These treatments include chemical (primers), micromechanical (etching, air-abrasion) and a combination of these methods.[11] Chemical treatment via the use of a silane-based primer, such as methacryloxipropyl-trimetoxi-silane, has proved very promising, and several authors have reported bond strengths similar to or greater than alternative surface conditioning techniques.[8,23,24] Silane coupling agents are able to increase the wettability and surface energy of the fiber post, and in addition, promote bonding between the organic components of the resin cement and the inorganic components of the fiber post (the silica from glass fibers).[11,23]

The tribochemical silica coating method involves a combination of micromechanical and chemical mechasnisms: it entails silica coating by air-abrasion with particles of aluminum oxide coated with silica on the post surface, and silane application. This generates a chemical/ mechanical network between the silica, the silane agent, and the resin cement.[20,25] Nergiz et al[26] have noted that as a consequence of this chemical/mechanical network, airborne particle abrasion increases the surface area available for adhesion, increasing retention between the fiber post and root canal. This conditioning method has yielded bond strength values superior to alternative surface treatments.[9,19] The tribochemical silica coating method is time-consuming compared to silane application however, and the air-particle abrasion aspect of it requires a specialized device (micro etcher).

Thus, this study aimed to evaluate the influence of different fiber post surface conditioning methods (untreated, chemical, and chemical-mechanical), using two conventional resin cements, on the bond strength between fiber post and root dentin. The hypotheses tested were: (1) different resin cements would not exhibit significant differences in terms of bond strength values; (2) different fiber post surface treatment methods would not be associated with significant differences in bond strength values.

MATERIALS AND METHODS

Specimen Preparation

Sixty single root bovine teeth (N = 60) had the coronal portion sectioned, and the length of the specimens was standardized at 16 mm. For tooth selection, the following inclusion criterion was applied: the mesiodistal and bucco-lingual dimensions of the coronal portion of the root canal should not be higher than 2.0 mm, corresponding to the coronal diameter of the fiber post system used in the study (White Post DC #3, FGM, Joinvile, SC, Brazil). These values were measured with a digital caliper (Starrett 727, Starrett, Itu, Brazil) and if the diameter exceeded 2.0 mm the specimen was eliminated from the study.

Root canals were prepared mechanically with NiTi instruments, followed by irrigation with 10 ml of Dakin solution. The root canals were prepared with the corresponding drill of the post system (White Post DC # 3, FGM), to a length of 12 mm. Subsequently the root apices were sealed with an adhesive system (Single Bond, 3M, ESPE, St Paul, MN, USA) and a resin composite (Oppalis, FGM) to avoid resin cement overflow.

The specimens were embedded in acrylic resin inside plastic cylinders (Dencrilay, Dencril, Caieiras, SP, Brazil). The embedding process was conducted as described in Bergoli et al.[18] To facilitate randomization, the specimens were numbered from 1 to 60 and six random sequences of ten teeth (n = 10) were generated by a computer program (Random Allocator, Department of Anesthesia, Isfahan, University of Medical Sciences, Isfahan, Iran), allocating the specimens to six different resin cement and the fiber post surface treatment groups (Table 1). The composition of the materials used in the study is listed in Table 2.

Fiber Post Surface Conditions

Three post surface conditions were evaluated as follows:

- In the silanization groups, the posts were first cleaned with 70% alcohol and air dried. The silane coupling agent was then applied with microbrushes (ProSil, FGM, Brazil) and allowed to react for 5 minutes.
- In the tribochemical silica coating groups, the post surfaces were air-abraded with 30 µm aluminum oxide particles modified with silica (Cojet Sand, 3M ESPE, St Paul, MN, USA) and the silane coupling agent (ProSil, FGM, Brazil) was applied as described in the previous group.
- In the control groups, the fiber post surfaces were only cleaned with 70% alcohol.

Cementation Procedures

The root dentin of all of the specimens was treated with a three step ‘etch & rinse’ adhesive system (Scotchbond Multi Purpose Plus, 3M ESPE, St Paul, MN, USA). The series of steps involved in adhesive application and resin cement insertion is described in Table 1.

The same luting procedures were used for both cements: they were applied into the root canal with acudose tips of the Centrix System (DFL, Rio de Janeiro, RJ, Brazil), the post was inserted with digital pressure, excess resin cement was removed with microbrushes (Endobrush, FGM, Brazil), and photoactivation (RadiiCal, SDI, Australia) was performed for 40 seconds from the coronal portion of the post.

After fiber post cementation, core reconstruction was conducted with a light cured composite resin (Opallis,

Table 1: Experimental design of the study

Adhesive system to dentin	*Post surface treatment*	*Resin cement*	*Group (N = 10)*
a,b,c,d	Silanization (Prosil*) (g,h)	AllCem* (e1,f)	Sil + AlC
		RelyX ARC** (e2,f)	Sil + ARC
	Tribochemical sílica coating (Cojet** (g,i,j)	AllCem* (e1,f)	TBS + AlC
		RelyX ARC** (e2,f)	TBS + ARC
	Control (Without treatment)	AllCem* (e1,f)	Untr + AlC
		RelyX ARC** (e2,f)	Untr + ARC

a: Etching the root dentin with phosphoric acid 37% for 20 seconds. The tip of syringe reached whole post space into root canal; b: Washing with com 10 ml of distilled water with a disposable syringe; c: Removing of the excess water/adhesive with #80 paper points; d: Application of multisteps ScotchBond Multi Purpose plus adhesive system (Activador, Primer, and Catalyst, 3M ESPE), using micro-brushes (Cavibrush, FGM); e1: Mixed the two pastes of cement AllCem and applied into the root canal with Centrix system (DFL, Rio de Janeiro, RJ, Brazil), using acudose points, and with the post; e2: Mixed the two pastes of cement RelyX ARC and applied into the root canal with Centrix system (DFL, Rio de Janeiro, RJ, Brazil), using acudose points, and with the post; f: Removed the cement excess and photo-activation for 40 seconds (Radii-cal, SDI, Austrália); g: Cleaning of the post with alcohol 70%; h: Silane application and drying waiting for 2 minutes; i: Blasting the surface of the post with particles of aluminum oxide 30µ; j: ESPE Sil application and drying waiting for 5 minutes; *(FGM, Joinvile, Brazil); **(3M ESPE, St Paul, USA)

Table 2: Composition of resin cements, coupling agent and tribochemical silica coating

Materials	*Manufacturer*	*Main composition*
RelyX ARC	3M ESPE, St Paul, USA	Etchant: 35% H_3PO_4 Adhesive: Bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, water, canphorquinone, photoinitiators, polyalkenoic acid copolymer, 5-nm silica particles Cement: Bis-GMA, TEGDMA polymer, zirconia/silica filler
Allcem	FGM, Joinville, Brazil	Bis-GMA, BIS-EMA, TEGDMA, photoinitiators (canphorquinone e dibenzoyl peroxide), barium-aluminum-silica glass particles, and SiO_2 nano-particles
Prosil	FGM, Joinville, Brazil	3- methacryloxypropyltrimethoxysilane (< 5%); ethanol (> 85%); water (< 10%)
Cojet	3M ESPE, St Paul, USA	(30 µm aluminum oxide particles coated by siliceous dioxides

FGM, Joenville, Brazil), aided by standardized plastic templates. Subsequently, the specimens were stored in a humid environment (± 37° C) for 7 days, prior to the push-out test.

Push-out Test

The specimens were attached to the LabCut1010 (Extec Corp, Enfield, CT, USA) cutting machine, and four slices (2 mm) were obtained per specimen, perpendicular to the long-axis of the root. The extrusion or 'push-out' was performed using a universal testing machine (DL 2000, Emic, São Jose dos Pinhais, Brazil) at a speed of 1 mm per min and the load was applied in the apical-coronal direction. Bond strengths (MPa) were derived using the formula R = C/A where C = breaking load (N) and A = interfacial area (mm^2). To calculate the adhered area, the formula of a circular straight cone with parallel bases was used: $A = \pi . g . (R_1 + R_2)$, where A = interfacial area, $\pi = 3.14$, g = slant height, R_1 = smaller base radius, and R_2 = larger base radius.[18] To determine the slant height the following calculation was used: $g^2 = h^2 + [R_2 - R_1]^2$, where h = height of the specimen. R_1 and R_2 were determined by measuring the internal diameters of the smaller and larger bases. The diameter and height of each specimen were determined using a digital caliper (Starret 727, Starret, Itu, SP, Brazil).

Fracture Analysis

Failure mode evaluations were performed for all the pushed-out specimens under an optical microscope (Olympus, BX60M, Japan) with 200x magnification. Failures were classified as: adhesive at interface dentin and cement (Adhes DC); adhesive at post and cement (Adhes PC); cohesive of post (Cohes P); cohesive of dentin (Cohes D); or cohesive of resin cement (Cohes C). Representative fractures were evaluated using scanning electron microscopy (JEOL-JSM-5400, Jeol Ltd, Tokyo, Japan). Cohesive fractures were not used for bond strength calculation, because they did not express the real values of bond strength.

Data Analysis

The means of the push-out bond of each tooth were calculated and were utilized for data analysis (n = 10). Push-out bond strength data were initially tested for normality and homoscedasticity, then two-way ANOVA and Tukey's tests ($\alpha = 0.05$) were performed.

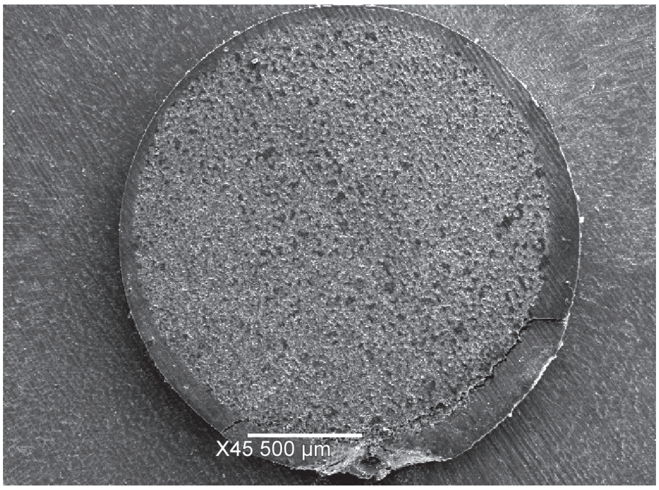

Fig. 1: Adhesive failure at resin cement-dentin interface (specimen from Gr1)

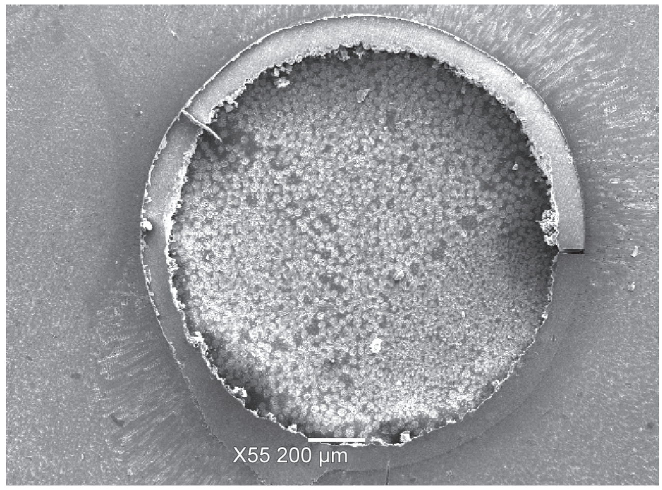

Fig. 2: Adhesive failure at fiber post-resin cement interface (specimen from Gr6)

RESULTS

Two-way ANOVA showed that post surface conditioning had a significant influence on bond strength values ($p = 0.0353$), but that the resin cement did not ($p = 0.967$) (Table 3). Conversely, Tukey's test showed no significant differences between the groups (Table 4). This could be related to the greater sensitivity of the ANOVA test.

Fracture analysis (Table 5) showed that failure mainly occurred at the resin cement/dentin interface (adhesive type) (Fig. 1), followed by failure of adhesive at the post/cement interface (Fig. 2).

DISCUSSION

Fiber post debonding from root canal is reportedly the main cause of failure of this system.[5-7] These failures can occur at the cement/ dentin interface or the post/cement interface, therefore it is important to promote higher bond strength at these interfaces. For fiber post cementation, the use of resin cements and surface conditioning agents from different manufacturers is common, thus it is important to analyze their relative performances.

After statistical analysis, the first hypothesis of the study was confirmed, as the different resin cements yielded similar bond strength values. This similarity suggested good interaction between materials from different manufacturers. The manufacturer of the adhesive system used in this study is not the same as the manufacturer of the resin cement tested, even though this interaction achieved higher bond strength values (9.5 ± 3.2 MPa).

Another factor that may be related to the similar bond strength values observed between the groups is the use of the same technique for resin cement insertion into the root canal, including the prevention of bubbles or defects that could influence interfacial resistance.[27-29] The cement insertion technique used in this study was reported by previous studies as capable of promoting a more uniform cement layer around the fiber post, avoiding

Table 3: Results from two-way analysis of variance (p = 0.05)

Source	*DF*	*SS*	*MS*	*F*	*P*
Cement	1	0.014	0.0144	0.00	0.9674
Surface Conditioning	2	60.710	30.3551	3.56	0.0353
Cement SurfCondit	2	18.882	9.4412	1.11	0.3380
Error	54	460.668	8.5309		
Total	59	540.275			

Table 4: Push-out bond strength means and standard deviation after Tukey's Test (MPa) (α = 0.05)

Cementation strategy	*Mean (SD)*
AllCem + Sil	9.5 (3.2) A
ARC + Sil	8.3 (2.6) A
AllCem + TBS	6.9 (3.6) A
ARC + TBS	6.6 (1.2) A
ARC + untreated	7.5 (3.3) A
AllCem + untreated	6.0 (2.7) A

Equal letters similar statistical results

Table 5: Number and percentage of fracture patterns analyzed

Failures	*Sil + AllC*	*Sil + ARC*	*TBS + AllC*	*TBS + ARC*	*Untr + AllC*	*Untr + ARC*	*Total (%)*
Adhes CD	33	36	39	39	32	34	219 (91.25)
Adhes PC	1	0	0	1	6	6	8 (3.33)
Cohes P	5	1	0	0	0	0	6 (2.5)
Cohes D	1	3	1	0	2	0	7 (2.91)

Adhes CD: adhesive cement/dentin; adhes PC: adhesive post/cement; cohes P: cohesive post; cohes D: cohesive dentin the formation of bubbles, and consequently, optimizing adherence to the substrates.[26-28]

Several techniques for fiber post surface conditioning have been tested in recent years.[8-10,19,23,24] Silica coating by air-abrasion with 30 µm silica-modified aluminum oxide particles has been used to increase the roughness of the post surface, facilitating micro-mechanical interlocking between post and cement, while the silica particles deposited on the surface react with silane, optimizing adhesion to the resin cement.[19,25,26] The application of silane coupling agent has also been reported as fundamental for good bonding between post and resin cement, since it increases the post's surface energy,[11,23] improving contact between the resin cement and the surface of the retainer. Silane coupling agent is capable of creating chemical couplings between the components of the post surface and matrix components of the resin cement.[10,11,23]

The two-way ANOVA suggested influence of the surface treatment on bond strength values, however this difference was statistically small, being not observed in data interaction. With regard to the second hypothesis of the study, Tukey's test showed no difference between the groups, emphasizing the lack of influence of the type of post surface treatment on the overall behavior of post/ cement/dentin interfaces (Table 4).

The insignificant differences in terms of bond strength observed among the 3 surface conditions (tribosilizatization, silanization, and no-treatment) can be explained by analysis of the mode of failures (Table 5); the most common type of failure was at the cement/ dentin interface (91.25%), consequently the bond strength test assessed that interface. This finding is in accordance with Monticelli et al.[11]

The predominance of adhesive failure at the cement/ dentin interface showed that the post surface treatments employed in this study promoted good adhesion at the cement/ post interface, since only 3.33% of failures occurred at this interface. Thus, the differences in bond strength values observed in this study are almost entirely related to differences at the adhesion cement/ dentin interface. Thus, studies with methodological designs that eliminate the dentin interface, such as that developed by Valandro et al,[19] could lead to the best assessment of the adhesion performance of resin cement to fiber posts.

Thus, based on the results of the present study and those reported by other authors,[5,7,11,20] the interface between dentin and cement appears to be the region more critical in teeth restored with fiber posts. Consequently, it is evidently more important to investigate approaches for improvement of adhesion to root dentin, than to the fiber post surface.

CONCLUSION

In teeth restored with fiber posts, the cement/ dentin interface appears to be the most susceptible to failure, while different post surface treatments appear to have little influence on bond behavior.

SIGNIFICANCE

The tested fiber posts surface treatment appears do not influence the fiber post bond behavior.

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