Original Article

Effect of Intracanal Dentin Conditioning with Xylene, Phosphoric Acid and Chlorhexidine on Bond Strength to Glass Fiber Post with Self-Adhesive Cement

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Abstract

Background and Aim: Debonding is the most commonly encountered failure in teeth restored with fiber posts that mainly occurs at the weakest interface (dentin-cement interface). Thus, reinforcement of this interface is mandatory. The aim of this study was to evaluate the effect of different root dentin surface treatments after post space preparation on bond strength of fiber posts cemented with a self-adhesive resin cement.

Materials and Methods: Forty extracted sound single-rooted teeth underwent root canal therapy. After post space preparation, the teeth were assigned to four group of 10 teeth according to the type of dentin surface treatment: Group 1: 2% chlorhexidine rinse, group 2: 37% phosphoric acid etching and then irrigating with saline, group 3: rinse with xylene and then irrigation with saline, group 4: rinse with saline (control group). Then, fiber posts were cemented with Total cem cement and the teeth were sectioned horizontally. Specimens were observed under a stereomicroscope at X10 magnification and failure modes of each group were determined. Results were analyzed by one-way ANOVA.

Results: There was no significant difference between the mean bond strength of the four groups (P=0.174). The highest mean bond strength value was found in phosphoric acid group (8.18±3.19 MPa) and the lowest belonged to the control group (6.21±1.81 MPa).

Conclusion: Our results showed that dentin surface treatment with phosphoric acid before cementation of fiber posts with a self-adhesive cement improves the bond strength.

Key Words: Root Dentin, Conditioned Dentin, Fiber Post

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Introduction

Restoration of endodontically treated teeth is challenging since they have lost a large amount of tooth structure due to caries, fracture or defective restorations [1]. In most cases, endodontically treated teeth do not have adequate coronal tooth structure and require post-space preparation in the root canal for coronal build-up. Fiber posts are increasingly used due to having mechanical properties similar to those of dentin, biocompatibility, resistance to corrosion and light transfer [2]. To increase intra-canal post retention

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and the strength of endodontically treated teeth, fiber posts are cemented with resin cements [2].

Bond to root canal is weak since it is affected by several factors such as a thick smear layer covering the root surface, presence of sealer residues, high C factor and inability to completely clean the canal [3-7]. The cement-dentin interface is the weakest point of attachment of fiber post to tooth structure [2].

At present, self-etch cements are increasingly used due to their simplicity and lower technical sensitivity compared to three-step adhesive systems. The manufacturers of these cements claim that adhesive-cement complex eliminates the need for tooth or restoration preparation [2]. However, some studies have reported limited etching ability and limited reaction of superficial dentin in use of these types of cements and higher nano-leakage [8].

During application of resin cements for intracanal post cementation, dentinal walls must be cleaned from gutta-percha debris and smear layer since the mechanism of bonding of resin cements is based on penetration of acidic monomers of the cement into dentin and dentin demineralization, which result in micromechanical interlocking of cement [2,7]. Previous studies attempted to clean the canal wall as much as possible [9-11].

Xylene is an organic solvent used for elimination of gutta-percha in endodontic re-treatment and post space preparation. It plays an effective role in increasing the bond strength of fiber post to root dentin [12].

Considering the recent approach to increase resin penetration into dentin and increasing the bond strength, this study aimed to assess the effect of root canal dentin preparation with xylene, phosphoric acid and chlorohexidine on bond strength of fiber post to root dentin using self-adhesive system.

Materials and Methods

In this in vitro, experimental study, 40 sound single canal human premolars extracted due to orthodontic or periodontal reasons were collected (ethical approval code:21151). The teeth were evaluated in terms of crack and wear and those with cracks and wear were excluded. The teeth were cleaned with gauze and periodontal tissue remnants and calculus were removed by hand instruments. The teeth were immersed in saline at room temperature until the experiment. One week prior to testing, they were transferred to 0.5% chloramine T solution for infection control. Tooth crown was cut 1mm above the cement enamel junction by a low speed diamond disc under water spray. Canal diameter was measured by a digital caliper (Mitutoyo, Kanagawa, Japan) with 0.001 mm accuracy and teeth with canal diameter over 1mm were excluded. Canals were prepared using ProTaper NiTi files (SX, S1, S2, F1 and F2) (Dentsply, Maillefer, Ballaigues, Switzerland) according to the manufacturer's instructions. F2 was the final file used in all teeth. Next, 0.9% saline was used in all canals for irrigation and root canals were filled with gutta-percha (Iran Iron, Tehran, Iran) and AH26 resin sealer (Dentsply DeTrey GMBH, Konstanz, Germany). The teeth were then stored in 100% humidity in black film boxes for one week. Size 1 radiopaque glass fiber posts (AngeluesExacto, PR, Brazil) with 1.1 mm diameter were used. For post-space preparation, first a #1 peesoreamer was used to remove root filling material and then post space was prepared using size 1 drill with 10 mm length (provided by the intracanal post manufacturer) such that root canal was prepared to 9 mm apical to the cementoenamel junction, and 4-5 mm of gutta-percha remained after post-space preparation at the apex. Next, 40 teeth were randomly divided into four groups of 10. A separate solution was used for post-space preparation in each group.

Group 1. Canals were rinsed with 5mL of 2% chlorhexidine (Clorhexidina S, FGM, Joinville, Brazil) for two minutes.

Group 2. Canals were rinsed with 32% phosphoric acid gel (Uni-etch; Bisco, Schaumburg, IL, USA) for 15 seconds and rinsed again with 5 mL of saline.

Group 3. Canals were rinsed with xylene for 60 seconds and were then irrigated with 5 mL of saline.

Group 4 (control). Canals were rinsed with 5 mL of 0.9% saline. After preparation, root canal was dried with paper point (Iran Iron, Tehran, Iran).

For post cementation, dual cure resin cement (TotalCem, ITENA, Paris, France) was used according to the manufacturer's instructions.

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Cement was mixed by a syringe provided by the manufacturer and delivered into the canal by an applicator. Some cement was also applied on the post. The post was introduced into the canal and guided by gentle finger pressure. Light curing was performed using a LED light curing unit (Woodpecker, Guangxi, China) for 40 seconds. The intensity of light was checked before each curing by a radiometer (Demetron, Kerr, Orange, CA, USA). Power of light curing unit was 1000 mW/cm^2.

Spongy blocks were placed in prefabricated metal molds. After fixing the teeth in vertical position in the sponge, the mold was filled with resin and allowed to set. Only the apical 4-5 mm of teeth was inside the sponge and the remaining parts were embedded in resin. Molds were placed in a cutting machine and the teeth were sectioned by low speed diamond disc (Isomet, Buehler, Dusseldorf, Germany) perpendicular to the longitudinal axis of the root under water coolant.

In each tooth, a section was made at the coronal part of the root with 1±0.1 mm thickness. Thus, each group contained 20 specimens. The coronal side of the specimens was marked by a water-resistant marker. Thickness of each slice was measured by a digital caliper with 0.001 mm accuracy (Mitutoyo Crop., Kakogawa, Japan). Intracanal post diameter was measured in the coronal and apical parts of each specimen using a graded microscope.

For bond strength testing, the specimens were transferred to a universal testing machine (Zwick Roell, Ulm, Germany). An acrylic block with 10 mm thickness was used. A cylinder was cut out of the acrylic to create a cylindrical hole measuring 3 mm in diameter and 5 mm in height. The specimens were placed on the block such that the cross-section of the post was placed directly over the hole. Block was positioned in the machine under adequate light and magnification such that plunger applied load with 1 mm diameter only to the post and had no contact with the cementum or dentin. Since posts were conical-shaped, they were positioned such that the apical part was in contact with plunger so that the post dislodged from the larger part. Load was applied at a crosshead speed of 0.5 mm/minute until post dislodgement. To calculate shear strength, load at failure was divided by the bonding surface area of post and tooth structure. Since in each specimen post was in the form of an incomplete cone, surface area of the periphery of this cone was calculated using the equation below:

$$A = \pi\left(R_1 + R_2\right)\sqrt{\left(R_1 - R_2\right)^2 + h^2}$$

Where R1 is the radius of the post in the coronal part of the specimen, R2 is the radius of the post in the apical part of the specimen and h is the thickness of specimen.

Load at failure was divided by the surface area.

$$\pi = \frac{F}{A} = \frac{F}{\pi\left(R_1 + R_2\right)\sqrt{\left(R_1 - R_2\right)^2 + h^2}}$$

After push-out bond strength test, separated post segments were evaluated under a stereomicroscope at x10 magnification to determine the mode of failure as follows:

1.Adhesive failure between post and resin cement (no cement on the post).

2.Mixed failure such that 0% to 50% of the post surface was coated with cement.

3.Mixed failure such that 50% to 100% of the post surface was coated with cement.

4.Adhesive failure between dentin and resin cement (post completely coated with cement).

5.Cohesive failure of the post

6.Cohesive failure of dentin

One-way ANOVA was applied to assess the effect of dentin surface preparation on bond strength. All statistical analyses were performed using SPSS version 20 at $P<0.05$ level of significance. Normal distribution of data was confirmed using one-sample Kolmogorov-Smirnov test (P=0.874).

Results

As seen in Table 1, the highest bond strength was noted in 37% phosphoric acid and the lowest was noted in saline (control) group. One-way ANOVA compared the mean bond strength of the groups and showed no significant difference (P=0.174). Table 2 shows the frequency percentage of modes of failure in each group. The results showed that in phosphoric acid, chlorhexidine and xylene groups, most failures were adhesive and occurred at the cement-post interface while in the control group most failures were adhesive at the dentin-cement

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Table 1. Mean and standard deviation of bond strength of post to root dentin in the four groups (n=20)

Group	Minimum	Maximum	Mean	Standard deviation
Phosphoric acid	1.72	14.79	8.18	3.19
Xylene	1.18	11.76	7.16	3.23
Chlorhexidine	2.72	11.43	7.06	2.66
Control	2.40	10.03	6.20	1.81

Table 2. Mode of failure after push out bond strength test in the four groups

Groups	Adhesive (post-cement) (1)	Mixed (2)	Mixed (3)	Adhesive (dentin-cement) (4)	Cohesive (dentin)	Cohesive (post)
Control	15%	15%	5%	60%	5%	0
Phosphoric acid	60%	0	15%	15%	10%	0
Xylene	35%	10%	25%	30%	0	0
Chlorhexidine	40%	5%	15%	35%	5%	0

interface. Statistical power for detection of three-unit difference in bond strength of the groups was calculated to be 0.87.

Discussion

This study assessed the effect of different dentin surface preparation methods on bond strength of fiber post tested by push out test. The results showed no significant difference in the mean bond strength of fiber post among the four groups of saline, xylene, phosphoric acid and chlorhexidine (P=0.174). The highest bond strength was noted in 37% phosphoric acid group (8.18±3.19 MPa) and the lowest was noted in the control group (6.20±1.81 MPa).

After tooth preparation, dentin is covered with smear layer, which is composed of cut mineralized collagen fibrils. No continuity exists between the smear layer and underlying dentin, However, smear layer covers the dentin surface and occludes dentinal tubules. As the result, smear plug is formed [13].

In endodontically treated teeth, smear layer also contains crushed odontoblastic residues, necrotic materials and microorganisms. During post-space preparation with rotary instruments, sealer and gutta-percha are also added to the composition of smear layer and a new smear layer is formed [9]. Resin bonds to dentin by penetration into dentinal tubules and the space between collagen fibrils [13]. Bonding mechanism in use of adhesive resin cements for cementation of fiber posts is micromechanical and based on the formation of hybrid layer and resin tags in demineralized surface. In total-etch resin cements, elimination of smear layer and debris from the dentin surface is necessary for the formation of hybrid layer [14]. Bond to root dentin depends on resin penetration into demineralized inter-tubular dentin and formation of resin tags as well as possible chemical interactions on resin-dentin surface [15]. In total-etch adhesive system, etching with phosphoric acid and rinsing with water is the most commonly practiced method to obtain a suitable bond to root dentin [16,17]. Self-etch cement was used in our study. Some researchers believe that dentin preparation with phosphoric acid may compromise the bond strength of self-etch systems [18]. They believe that dentin preparation with phosphoric acid causes deep demineralization of dentin and inhibits proper resin penetration and thus, results in formation of a destructive area under the hybrid layer [17]. In our study, root dentin etching with 37% phosphoric acid improved the bond strength of self-etch adhesive system. Hybrid smear layer in self-etch adhesive systems is a weak area of bond to dentin and since this layer contains modified collagen fibrils, its bond strength gradually decreases and degrades [11]. On the other hand, mineral parts of the smear layer are strong buffers that increase the pH of acidic monomers and decrease their demineralizing ability [2]. Thick smear layer formed on canal walls during endodontic treatment and post space

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preparation prevents effective demineralization and also prevents the penetration of self-etch adhesive and consequently compromises the bond strength.

Root dentin preparation with 37% phosphoric acid eliminates the smear layer formed during endodontic treatment or post-space preparation [18] and opens dentinal tubules, yielding a clean surface. It also increases surface roughness and causes greater wetting of root dentin [19,20]. Thus, bond strength of self-etch resin cement to root dentin increases. Our results were in line with those of Pisaniet al, Scotti et al, Demiryureket al, and Zhang et al [14,17,18,21]. Assessment of the frequency of mode of failure showed that the frequency distribution of adhesive failure between dentin and cement in phosphoric acid group was lower than that in the control group. Thus, it may be stated that post-space preparation with 37% phosphoric acid improves the quality of self-etch bond to root dentin. This result was in line with that of Zhang et al [17].

Based on the current results, it appears that xylene dissolves gutta-percha and sealer and eliminates the secondary smear layer formed during post space preparation and therefore, improves the bond strength of self-etch cement to root dentin. This result was in line with that of Da Silva et al, [12]. In xylene group, the frequency of adhesive failure between cement and dentin was lower than that in the control group, which indicates the positive efficacy of xylene in improving the quality of bond of self-etch systems.

Mubashir Mushtaqet al, [22] in their study on solubility of root canal sealers showed that xylene can dissolve AH Plus and Apexit Plus sealers. Chloroform and xylene are the most commonly used gutta-percha solvents [23,24]. Due to existing concerns regarding the carcinogenicity of chloroform, researchers are in search for other solvents [24]. Da Silva et al, [12] in their study on the effect of root dentin preparation on push out bond strength of fiber posts showed that bond strength in xylene group was higher than that in saline group and it was in line with our findings. Our study showed no significant difference between chlorhexidine and control group although chlorhexidine group showed higher bond strength.

Frequency of mode of failure showed that adhesive failure had a lower frequency in chlorhexidine group compared to the control group, which points to its positive effect on bond quality. This finding was in agreement with that of Zhou et al [2].

Complete cleaning and disinfection of pulp chamber and root canal is necessary for long-term success of endodontic treatment. Chlorhexidine has a wide antimicrobial spectrum, low toxicity and high substantivity and is commonly used during root canal cleaning and shaping and also in endodontic retreatment [25]. Evidence shows that application of chlorhexidine prior to acid etching has no negative effect on composite bond to coronal dentin, pulp chamber dentin, enamel or bond to resin reinforced glass ionomer cement [25]. Sontoset al. [26] stated that 2% chlorhexidine solubilized in water or in gel form does not interfere with the bond of self-etch adhesive systems to pulp dentin [26]. Lindblad et al. reported that chlorhexidine had no significant effect on push out bond strength of fiber posts and final rinse of post space with 2% chlorhexidine after etching (in use of etch and rinse adhesive systems) and also prior to the use of self-etch resin cements is recommended for post cementation [27].

Da Silva et al. [2] reported that the bond strength in the group using chlorhexidine was higher than that in saline group, which was in line with our results [12]. Zhou et al. stated that use of chlorhexidine had no negative effect on primary bond strength of fiber post to root dentin, which was in agreement with our findings. Leitune et al, [28] reported that chlorhexidine did not interfere with primary bond strength but could not prevent reduction in bond strength after six months. However, bond strength at six months in chlorhexidine group was still higher than that in the control group [28]. It appears that positive effect of chlorhexidine on bond strength is due to its high substantivity and strong electrostatic bond. When chlorhexidine salts are disintegrated, positively charged ions are released. These free cations can form strong electrostatic bonds to phosphate groups of hydroxyapatite. Strong electrostatic bonds formed by anionic molecules play a role in bond of chlorhexidine to anionic functional groups of other molecules such as phosphate in lipopolysaccharides and carboxylic groups in proteins. It appears that penetration of

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chlorhexidine into dentin enhances resin penetration into dentinal tubules. However, future studies are required to confirm it [2].

Evidence shows that chlorhexidine can increase the durability of hybrid layer and bond strength [29,30]. This ability is probably due to the inhibitory effect of chlorhexidine on matrix metalloproteinases [25]. Matrix metalloproteinases are members of an enzymatic family present in sound root dentin and coronal dentin and play an important role in destruction of collagen network in bonded restorations. Collagen degradation and structural changes in collagen fibrils are believed to be responsible for reduction in bond strength [25]; all these are believed to be caused by matrix metalloproteinases present in dentin [31].

Bacteria present in post space can play a role in matrix degradation. Chlorhexidine eliminates these bacteria and further contributes to bond strength especially when rubber dam is not used during post space preparation [12,30]. In bond to root dentin, several factors such as variations in dentin structure, difficult bonding procedure in root canal and anatomical variations in canal shape can result in controversy in reported bond strength values, which has also been discussed in some other studies [2,9,17].

Conclusion

1.The highest push out bond strength was noted in phosphoric acid group and the lowest in control (saline) group. The difference among groups was not significant in this regard (P=0.174).

2.Assessment of the modes of failure showed that in phosphoric acid, chlorhexidine and xylene groups, most failures were adhesive and occurred at the cement-post interface. In the control group, most failures were adhesive and occurred at the dentin-cement interface.

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