Effect of different adhesion strategies on fiber post cementation: Push-out test and scanning electron microscopy analysis

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Abstract

Aim: The aim of this study was to investigate the effect of phosphoric acid etching and the dentin pre-treatment with sodium hypochlorite (NaOCl) on the push-out bond strength between fiber post and root canal dentin. **Materials and Methods:** Root canals of 48 human incisors were selected, post spaces were prepared and assigned to four groups: G1-37% phosphoric acid (15 s); G2-5.25% NaOCl (2 min) +37% phosphoric acid (15 s); G3-37% phosphoric acid (60 s); and G4-5.25% NaOCl (2 min) +37% phosphoric acid (60 s). Fiber post cementation was performed with two-step etch-and-rinse adhesive system/dual-cured resin cement according to the manufacturer's recommendation. After 24 h, each root was sectioned transversally into three slices (cervical, middle and apical) and the bond strength of each section was determined using a push-out bond strength test. Morphology analysis of the bonded interface was evaluated using a scanning electron microscopy. Push-out strength data (MPa) were analyzed by Analysis of Variance and Tukey-Kramer (= 0.05). **Results:** Considering the NaOCl pre-treatment, no statistically significant differences were observed among groups; however, when the phosphoric acid was applied during 60 s in the apical portion without NaOCl pre-treatment, the bond strength was statistically significant increased. **Conclusion:** The NaOCl pre-treatment did not improve the bond strength of adhesive luting cement to root canal dentin. The findings suggest that the use of 37% phosphoric acid for 60 s may have a beneficial effect on bond strength in the apical root third.

Keywords: Dentin, fiber post, phosphoric acid, sodium hypochlorite

Introduction

Fiber posts have been widely used in endodontically treated teeth in the last few years. One of the advantages of fiber posts is the adhesive bonding technique, which requires minimal intervention on the dentin surface. In addition, fiber posts produce an elastic modulus similar to dentin, resulting in homogeneous stress distribution along the root canal system. As a result, fiber post minimizes the risk of root fractures or lead to more favorable fracture mode than metallic posts.[1-4]

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The clinical success of restorative treatment using fiber post relies on the bond strength between fiber posts and canal dentin surface.[5] One of the most common causes of glass-fiber post failure occurs in the bonding interface between adhesive luting material and dentin. It may be due to different factors such as poor polymerization of adhesive materials along the canal,[6] adverse effects of endodontic sealers,[7] high polymerization shrinkage,[8] morphological changes of dentin tissue[9] and the presence of residual tissues.[10]

Concerning the etch-and-rinse adhesive systems, a preliminary etching step with phosphoric acid can remove the smear layer, demineralize the dentin walls, open the dentin tubules and expose the collagen fibrils, allowing resin infiltration into the collagen matrix.[11-13] On the other hand, sodium hypochlorite (NaOCl), a non-specific proteolytic agent, acts as an organic solvent and leads to greater smear layer removal.[14,15] In addition, the dentin pretreatment using 5.25% NaOCl seems to positively affect the bond strength.[16,17]

Based on these considerations, the aim of this study was to evaluate the effect of phosphoric acid etching duration (15 or 60 s) and additional pretreatment using 5.25% NaOCl (2 min) on the push-out bond strength between fiber posts at the cervical, middle and apical thirds of the root canal dentin. Morphology of the bonded interface was analyzed using a scanning electron microscopy (SEM). The null hypothesis tested was that the extended etching time (60 s) and additional pre-treatment with NaOCl could not affect the bond strength between fiber post and root canal dentin regardless of the root third.

Materials and Methods

Specimen preparation

This study was conducted after the approval of the local Ethical Committee for Research. A total of 48 human permanent anterior maxillary extracted teeth were selected. None of them had received restorative or endodontic treatment and the roots presented a length between 14 and 16 mm. Teeth were stored in 0.1% of thymol solution for no more than 6 months.

Teeth were sectioned below the cement-enamel junction, perpendicularly to the long axis. Standard endodontic preparation was carried out using a crown-down technique. The root canal was manually enlarged and flushed with 2% NaOCl. After chemical-mechanical preparation, distilled water was used as a final rinse. Then, the root canal was blot dried with paper points and filled with gutta-percha cones (Dentsply Ind, Petrópolis, RJ, Brazil). To eliminate the effect of endodontic sealers, gutta-percha cones were used without endodontic cement.[18] Afterwards, provisional filling material (Coltosol, Vigodent S/A Ind., Rio de Janeiro, RJ, Brazil) was used to seal the access cavity. Specimens were kept at 37°C for 3 days in 100% humidity. All the procedures were performed by the same operator.

Post-space preparation was initially created with heated instruments and the residual gutta-percha was removed with a Gates-Glidden bur (No. 3, Dentsply Maillefer) up to a final depth of 11-12 mm, maintaining at least 3 mm of apical seal. Root canal walls were enlarged with calibrated bur (No. 3, FRC® Postec Plus, Ivoclar-Vivadent, Schann, Liechtenstein, Germany). The canals were rinsed with distilled water and dried with paper points. Then, a total of 40 specimens were used to push-out bond strength test ($n = 10$) and 8 specimens were prepared for SEM analysis ($n = 2$). The roots were randomly assigned to groups according to the method used and four adhesion strategies (dentin pretreatment and 37% phosphoric acid etching duration): G1-37% phosphoric acid (15 s); G2-5.25% NaOCl + 37% phosphoric acid (15 s); G3-37% phosphoric acid (60 s); and G4-5.25% NaOCl + 37% phosphoric acid (60 s). After acid etch step, the root canal was washed with distilled water and dried using paper points. The NaOCl pre-treatment was performed for 2 min and the excess was removed with a gentle air stream and paper points.

The fiber posts (No 3, FRC Postec® Plus, Ivoclar/Vivadent) were placed in the root canal to test the fit. Then, posts were sectioned 2 mm above the root margin using double-faced diamond disc (#7020, KG Sorensen, Baueri, SP, Brazil). Before cementation, each fiber post was cleaned with 70% alcohol, dried and a silane agent was applied according to manufacturer's instructions (Monobond-S, Ivoclar/Vivadent). The two-step etch-and-rinse adhesive system (Excite Dual-self-cure [DSC], Ivoclar/Vivadent) and dual-cured resin cement (Variolink II, Ivoclar/Vivadent) were used according to the manufacturer's recommendation. The composition of these materials are described in Table 1. The adhesive system was applied in dentin canal surface with an extra-fine microbrush and the excess was removed. Afterwards, the resin cement was applied into the root canal space with a lentulo drill and the post was immediately seated. Excess cement was removed and the photopolymerization was performed for 60 s. The light output of the halogen-curing unit (Optilight 600, Gnatus, Ribeirão Preto, SP, Brazil) was monitored (600 mW/cm^2) by a radiometer (Curing Radiometer, model 100, Kerr Corporation, Orange, CA, USA). Then, all specimens were stored in distilled water at 37°C.

Push-out bond strength

Initially, each root was sectioned perpendicularly to the long axis into three slices thickness of 2.3 mm ±0.1 mm using a low-speed saw (Labcut 1010 Extec Corp.®, Enfield, CT, USA) with a diamond disc under water cooling. The thickness of each slice was measured using a digital electronic caliper (Mitutoyo Sul Americana Ltda., São Paulo, SP, Brazil), rounded to the nearest 0.001 mm. Then, each specimen was attached with cyanoacrylate-based adhesive (Super Bonder Gel-Loctite Brazil Ltda., Itapevi, SP, Brazil) to an adapted device, which was carried out on a universal testing machine (EMIC, Curitiba, SC, Brazil). A compressive load was applied using diameter cylindrical plunger (0.8 mm) at a constant speed of 0.5 mm/min in an apical-coronal direction until the post was dislodged. The plunger was positioned in the center of each specimen, directly in contact with the post fiber.

Push-out bond strengths (MPa) were calculated for each specimen by the maximum force required to dislodge

Table 1: Compositions of the materials (adhesive system and resin cement) used in this study (information supplied by the manufacturer)

Materials (manufacturers)	Composition
Excite DSC (Ivoclar/Vivadent, Schaan, Liechtenstein, Germany)	2-Hydroxyethyl methacrylate, dimethacrylates, phosphonic acid acrylate, silica, initiator, stabilizer, ethanol
Variolink II (Ivoclar/Vivadent, Schaan, Liechtenstein, Germany)	Bisphenol-A-diglycidylmethacrylate, urethane dimethacrylate, triethylene glycol dimethacrylate, glass, ytterbium trifluoride, benzoylperoxide, initiator, stabilizer

DSC: Dual-self-cure

the post (N) by the area (A) of the bonded interface. The area of the bonded interface was calculated using Maple 5.1 software (Maple, Waterloo Inc., Waterloo, Ontario, Canada).[7] The statistical analysis was performed using the Analysis of Variance (ANOVA) in a split plot design. The dentin treatments (NaOCl pre-treatment and acid etch time) represented the plot factor while the three-thirds for the root canal (cervical, middle and apical) were described as a sub-plot factor. The multiple comparisons were performed using the Tukey-Kramer test at a significance level of 5%.

SEM

The ultrastructural interface analysis was performed through a scanning electron microscope (JSM 2900, JEOL, Peabody, MA, USA). After post cementation, teeth were sectioned parallel to the long axis using a low-speed saw under water-cooling. The resin cement/dentin interfaces were wet polished (APL-4, Arotec, Cotia, SP, Brazil) with 600-, 1000- and 1200-grit SiC paper. Specimens were polished with soft cloths and diamonds pastes of decreasing abrasiveness (6, 3, 1 and 0.25 μm). Then, specimens were ultrasonically cleaned, air dried, mounted on a metallic stub and sputter coated with gold (Balzers-SCD 050 Sputter Coater, Balzers, Liechtenstein, Germany). Representative areas were photographed at ×500 and ×1500 magnification.

Results

The means and standard deviations of push-out bond strength means are described in Table 2. The dentin pre-treatment (5.25% NaOCl for 2 min) did not improve the bond strength regardless of the acid etch duration and root third ($P = 0.420$). However, bond strength produced by 60 s etching time was higher than 15 s in the apical root third. Regarding the extended etching time, no statistically significant differences were observed for cervical and middle-thirds ($P > 0.05$).

SEM findings are illustrated in Figure 1 (×500 and ×1500). Bonding interface examination showed morphological variations among root dentin regions (cervical, middle and apical thirds). Regarding the cervical third, the resin-dentin interdiffusion zone showed long and dense tags [Figure 1a]. Photomicrography analysis of middle root third demonstrated shorter resin tags when compared to cervical third [Figure 1b]. On the other hand, the hybrid layer was less apparent and discontinuous gap was demonstrated in the apical third [Figure 1c].

Discussion

The hypothesis was partly supported by the results; the additional pre-treatment with 5.25% NaOCl during 2 min did not affect the bond strength. However, extended etching time (60 s) provided statistically significant increase in the bond strength values in the apical third of the root. The coronal and middle thirds were not influenced by dentin treatments tested in this study.

NaOCl has been considered as an excellent non-specific proteolytic agent and leads to greater dissolution of organic compounds during root canal therapy.[14,15] According to Gu *et al.*[19] 5.25% NaOCl can promote the smear layer removal on coronal and middle thirds; however, the smear layer still remained in the apical third of the root. Interestingly, no resin tags formation were observed between adhesive layer and root canal dentin when the smear layer were not removed.[12] Concerning the 5.25% NaOCl dentin pre-treatment, this current study did not show statistically significant differences on the bond strength between fiber post and root dentin. Therefore, the application of phosphoric acid alone was enough to promote the smear layer removal and hybrid layer formation.

The acid etching of dentin removes the smear layer and demineralizes the subsurface.[11,13] SEM analysis exhibited morphological differences among the root portions. The cervical third demonstrated an authentic hybrid layer with long and dense resin tags. On the other hand, no distinguishable hybrid layer and less apparent resin tags were revealed in the apical third. Since the apical portion may gather a greater amount of remnants after post space preparation, it may impair the complete resin infiltration. On the other hand, the extended etching time to 60 s, without any NaOCl pre-treatment, seems to increase the bond strength significantly in apical third. It also may be related to the fact that apical dentin contains a lower number of tubules,[9] and hence 37% of phosphoric acid applied for 60 s may contribute to more efficiently smear layer removal when compared to 15 s. In addition, the closer contact between fiber post and apical root canal wall can improve the bond strength means. Thus, the frictional retention seems to play a key role on the total retention of the fiber posts on the root canal preparation.[7,20]

Table 2: Mean (SD) of bond strength (MPa) among tested groups

Acid etching time	Without NaOCl pre-treatment			With NaOCl pre-treatment		
	Cervical	Middle	Apical	Cervical	Middle	Apical
15 s	6.3 (2.9)a	7.6 (3.7)a	6.9 (2.5)a	8.3 (2.4)a	8.9 (4.0)a	9.2 (3.2)a
60 s	6.8 (2.7)a	7.8 (2.2)a	10.1 (3.1)b	7.4 (2.8)a	7.2 (4.1)a	8.2 (3.5)a

Values followed by distinct letters in the same pre-treatment (without or with NaOCl) and third of the root (cervical, middle and apical) are significantly different (ANOVA and Tukey-Kramer/$P \leq 0.05$). ANOVA: Analysis of variance; SD: Standard deviation; NaOCl: Sodium hypochlorite; MPa: megapascal

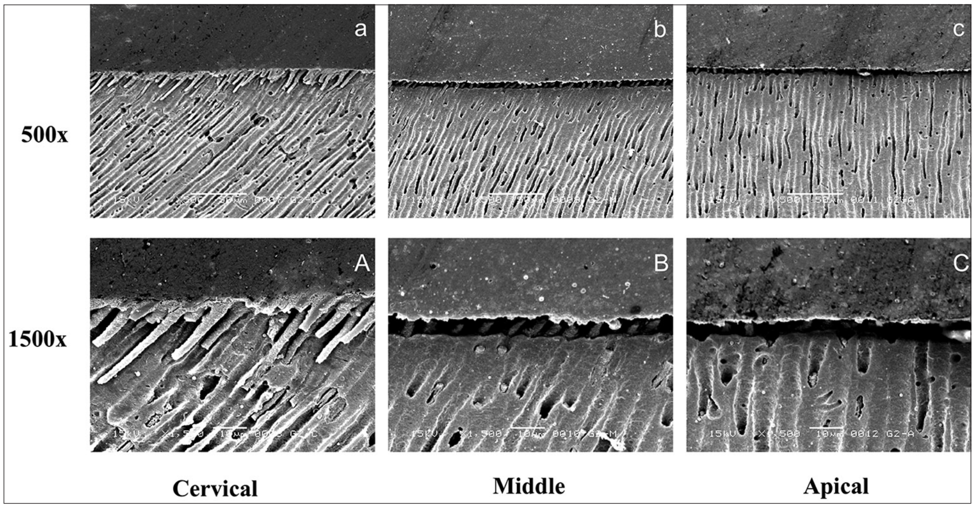

Figure 1: Photomicrographs of bonding interface for group 2 (×500 and ×1500). (a, A) Resin-dentin interdiffusion depicted long and dense tags in the cervical third. (b, B) For the middle third, shorter resin tags were observed and (c, C) less apparent and discontinuous gap were demonstrated in the apical third

Bond strength of post retention could be affected by several factors. In this study, the adhesive luting materials were used from the same manufacturer to avoid possible chemical incompatibility between materials. The dual-polymerized resin cement was combined with Excite DSC adhesive system. According to Ferrari *et al.*,[21] Excite DSC adhesive system consists of catalyst particles that might allow polymerization in the absence of light. In addition, dual-polymerized resin cements when light-cured showed a higher degree of conversion.[22,23] In this investigation, it was assumed that light-transmission through the post space did not affect the results. In accordance with Boff *et al.*,[6] no statistically significant differences on the bond strength were observed in the middle and apical third. In addition, Foxton *et al.*[24] reported similar behavior among root regions.

The previous reports showed that eugenol-based endodontic sealer may also affect resin cement polymerization during post cementation, decreasing bond strength between fiber post and root canal dentin.[7,25] In this study, standardized endodontic treatment was carried out to simulate clinical procedures and endodontic sealer cement was not used to prevent any influence of this agent on adhesive cementation procedures, enabling more accurate assessment of the variables studied (5.25% NaOCl pre-treatment and acid etching).

Another relevant issue to determine the post retention was the methodology used. To assess the bond strength of fiber post to root canal, push-out test allows evaluating the post retention and the regional configuration differences of the root canal dentin. Thus, this *in vitro* design appears to be more closely to clinical scenarios than others mechanical tests (i.e., microtensile bond test), which evaluate only the adhesive bond strength.[7,20] In addition, it is important to emphasize that, despite extended etching time (60 s) improve bond strength means in the apical third, this procedure may affect the dentin permeability and hence increase the hydrolytic degradation of resin-dentin interface. Therefore, long-term studies are needed to evaluate the beneficial effects of extended the pre-etching with phosphoric acid in the apical third of root canal.

Conclusions

Dentin pre-treatment with 5.25% NaOCl had no effect on the bond strength between fiber posts and root canal walls. However, the use of 37% of phosphoric acid for 60 s increased the bond strength in the apical third of the root dentin.

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