Push-out bond strength of FRC posts using conventional and wet-ethanol bonding systems: an ex-vivo study

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Abstract

Purpose: The aim of this study was to compare the bond strength obtained on root canal walls when using two different adhesive systems to bond a fiber reinforced composite (FRC) post inside a root canal and to evaluate the type of failure at the resin-dentine interface. **Methodology**: Thirty-eight central incisors were root canal treated, divided in 2 groups, and restored using FRC posts luted using resin cement (Multilink). Excite DSC was used as bonding agent in group 1 and an experimental ethanol-wet bonding system was used in group 2. The specimens were cut into 1mm-thick sections and a push-out test was carried out after 24h. Fracture type was assessed using optical and scanning electron microscopy. **Results:** Mean and standard deviation for the excite DSC and ethanol-wet groups were respectively 7.6±5.5 and 7.4±5.6 MPa. The Mann-Whitney rank sum test showed no statistical difference between the 2 groups (P=0.708). With regards to failure type, adhesive failure prevailed in the 2 groups. SEM evaluation revealed that adhesive failures occurred mainly at the resin-dentin interface. **Conclusions:** The 2 bonding systems exhibited similar short-term behavior. A baseline is established to test potential improvement of long-term behavior when using ethanol-wet bonding systems.

Running title: Push-out bond strength of FRC posts
Key Words: adhesion, fiber-post, ethanol-wet bonding, interface, dentin, photodarkening

Introduction

In the actual wet-bonding concept, acid-etching of the dentin is required to generate a superficial collagen layer. This layer should remain moist and will receive a primer in order to allow the infiltration of hydrophobic monomers thus creating the hybrid layer [1]. Simplification of bonding procedures reduced 3-step techniques to 2 steps, and later to 1-step with the introduction of self-etching primer adhesives. This simplification was possible by increasing the amount of acidic monomers dissolved in primers.[2] However, some potential problems are associated with the simplification of bonding procedures [3,4] as these simplified adhesives behave as permeable membranes that allow the fluids to cross the adhesive layer after polymerization.[5] The water that migrates to the composite-adhesive interface is trapped as water blisters, which might act as stress concentration sites, which might result in deterioration of the resin-dentin bond.[6] Furthermore, long-term survival of bonded restorations is compromised due to the hydrophilic properties that allow for hybrid layer disintegration at the dentin resin interface.[7,8] This disruption of the hybrid layer was reportedly due to water sorption and also to the action of matrix metalloproteinases that were liberated from the dentinal tissues after the etching process.[9-11] As such processes are mainly water-based, recently, a

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different bonding process based on wetting collagen matrices with ethanol rather than water was suggested.[12] The authors postulated that among other advantages, using an "ethanol-wet" bonding system would improve long-term behavior of resin-dentin bonds. This preliminary study even demonstrated superior bond strengths to dentin for the ethanol-wet bonding system. However, we cannot extrapolate these findings to intra-radicular bonding processes, as the previous study was performed on coronal dentin that differs from root canal dentin.[13,14]

The aim of this study was to compare the bond strength obtained on root canal walls when using two different adhesive systems to bond a fiber reinforced composite (FRC) post inside a root canal and to evaluate the type of failure at the resin-dentine interface. The null hypotheses tested were that there are no differences (1) in the bond strength achieved at the resin-dentine interface between the adhesive systems tested and (2) in the failure modes between the different groups.

Material and Methods

Preparation of the specimens

38 central incisors free from caries or previous restorations were cleaned of external debris, examined by transillumination for cracks, and stored in an incubator at 37°C at 90% relative humidity before testing. Teeth were extracted for periodontal reasons and patients' consent was obtained. Crowns were cut perpendicularly to the long axis of the tooth 2mm above the cemento-enamel junction using a diamond disk under water-cooling. Root canals were instrumented using stainless steel K-files # 10, 15, 20 (Dentsply-Maillefer, Ballaigues, Switzerland) and Glyde (Dentsply-Maillefer) to the visual working length (1 mm from the apex). Cleaning and shaping was continued using ProTapers (Dentsply-Maillefer) according to the manufacturer's instructions and to F3 size. Root canals were irrigated between instrumentation with 2mL 5.25% NaOCl and all teeth were obturated using the Schilder technique, using calibrated gutta-percha points (F3, Dentsply-Maillefer) and AH Plus sealer (Dentsply-Maillefer).

Root canal filling was removed with a Largo drill No. 1 (Dentsply-Maillefer) to a depth of 9 mm and a post-space was prepared with #3 calibrating drills (Ivoclar-Vivadent, Schaan, Liechtenstein) and the specimens were randomly distributed into 2 groups (n=19).

38 FRC Postec Plus® #3 (Ivoclar-Vivadent) were used (Max Ø: 2mm, Min Ø: 1mm). Each post was cleaned with Total Etch® (Ivoclar-Vivadent) for 60s according to the manufacturer's instructions, rinsed with water and dried. The posts were then placed in 10% H2O2 for 20 minutes then rinsed with water and dried after which they were dipped in a 10% sodium ascorbate solution for 10 minutes and treated with a silane coupling agent for 60s (Monobond-S®, Ivoclar-Vivadent). For both groups, surfaces were carefully air dried after silane application and the two different adhesives were applied respectively onto the posts of each group in the following manner: a generous amount of adhesive was applied with a microbrush to the posts with gentle agitation to ensure complete coverage. Air-drying followed at a distance of 15-20 cm to evaporate the solvent, moving closer as the resin stopped moving. Light-curing followed for 20s.

The canal walls were etched (Total Etch®) for 15s, then rinsed with water and dried with paper points. Excite DSC® (Ivoclar-Vivadent) was applied to the canal walls and excess material was removed using paper points. The posts were then luted using Multilink Automix (Ivoclar-Vivadent) and light polymerized using a halogen light-curing unit (Blue Phase, Ivoclar-Vivadent) for 40s with the tip of the unit directly in contact with the post.

In Group 2, 100% ethanol was used to flush the water from the canal and left in place for 1 minute. Care was taken to keep the canal moist with ethanol at all times during the procedure unless otherwise specified. Three 1-minute iterations were performed; after which the two components of the autopolymerizing version of resin 2 were prepared (solvate 50% resin A in 50% ethanol, and 50% resin B in 50% ethanol) (1:1 in weight). They were mixed in a 1:2 ratio as a primer for a hydrophobic dentin adhesive and placed on the root canal walls using a microbrush (Microbrush X). Ethanol was allowed to slowly evaporate. The posts were then luted using Multilink Automix (Ivoclar-Vivadent) and light-curing will follow using a halogen light-curing unit (Astralis10, Ivoclar-Vivadent) for 40s with the tip of the unit directly in contact with the post.

Push-out test

After 24h of storage at room temperature, 1mm-thick horizontal sections from each specimen was obtained using a precision cutting device and a diamond coated disc (Buehler, Lake Bluff, IL) and push-out test was conducted using a Universal Testing Machine (Controls, Milan, Italy). A stainless steel pin (1mm diameter) was placed in contact with the post section and was loaded at a crosshead speed of 0.5 mm/min until failure occurred by dislodgment of the post section. The retentive strength of the post segment was expressed in mega

Table 1

Descriptive statistics of Excite DSC and wet-ethanol groups (PF: premature failures; Means are given in MPa).

Group	n	PF	Mean (SD)
Excite DSC	90	8	7.6 (29.1)
Wet-ethanol	98	5	7.4 (30.8)

Pascals (MPa), by dividing the load at failure in newtons by the interfacial area (A) of the post fragment. The latter, being the lateral surface of a truncated cone, was calculated by the formula: $A= \pi(R+r)[h^2 +(R-r)^2]^{0.5}$, where $\pi = 3.14$, R = coronal post radius, r = apical post radius and h = root slice thickness.

Fracture type

Specimens were examined using stereomicroscopy to determine the type of failure whether cohesive, adhesive, or mixed and randomly selected samples were examined using scanning electron microscopy (SEM).

The level of significance was set at $P<0.05$. Statistical analyses were performed using SPSS version 11.0 software (SPSS Inc., Chicago, IL).

Results

Premature failures were considered as zero-bond values and were included in the study. Descriptive statistics (Table 1) revealed that mean and standard deviation for the excite DSC and ethanol-wet groups were respectively 7.6±5.5 and 7.4±5.6. However as the data failed the Kolmogorov-Smirnov test, a Mann-Whitney rank sum test was performed and showed no statistical difference between the 2 groups ($P=0.708$).

With regards to failure type, adhesive failure prevailed in the 2 groups (Table 2). Although, there were differences between the groups, they were not statistically significant (Fisher exact test).

SEM evaluation revealed that, in some samples, adhesive failures occurred mainly at the resin-dentin interface (Figure 1). It also revealed that such failures were not purely adhesive but rather either mainly adhesive with minor cohesive areas or with minor occurrences on the resin/fiber-post interface (Figure 2). Although present, cohesive failures were less frequent and displayed residual cement on both fiber-post and dentin (Figure 3).

Discussion

The main advantage of using "ethanol-wet" bonding systems would reside in the improvement of the long-term behavior of resin-dentin bonds.[15] However, this improvement cannot be properly defined unless we have a baseline from which to start, namely a comparison of immediate bond-strength values between conventional and wet-ethanol bonding systems. Push-out test has several advantages when compared to other bond strength measurement techniques. It allows using thin sections from every specimen and thus several readings could be obtained from every single specimen. Additionally, it reduces the influence of mechanical retention associated when trying to pull an entire post from the root canal. However, even a single poorly prepared sample could translate into biased results. The premature failures were predominantly observed in the apical-most part of the posts. Coronal and apical data

Table 2

Distribution of adhesive vs. cohesive failures in the 2 groups.

Group	Adhesive	Cohesive
Excite DSC	75	15
Wet-ethanol	73	25

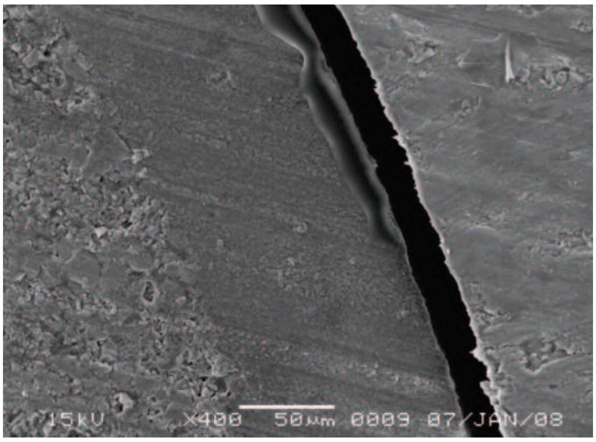

Figure 1: Scanning electron image of adhesive failure. The luting resin is still bonded to the fiber-post with the failure occurring on the resin/dentin interface.

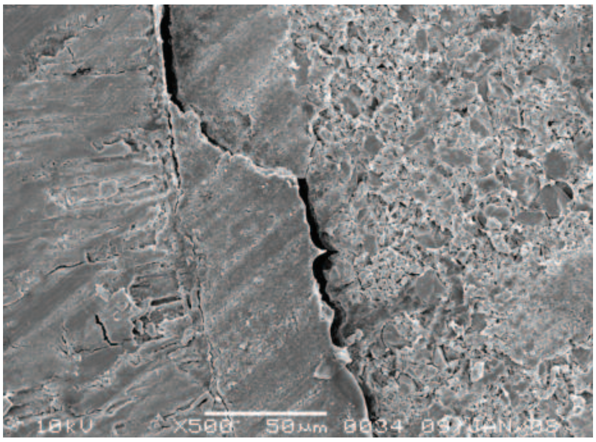

Figure 2: Scanning electron micrograph of a double adhesive failure with part of the failure occurring on the resin/dentin interface and the other part on the resin/fiber-post interface.

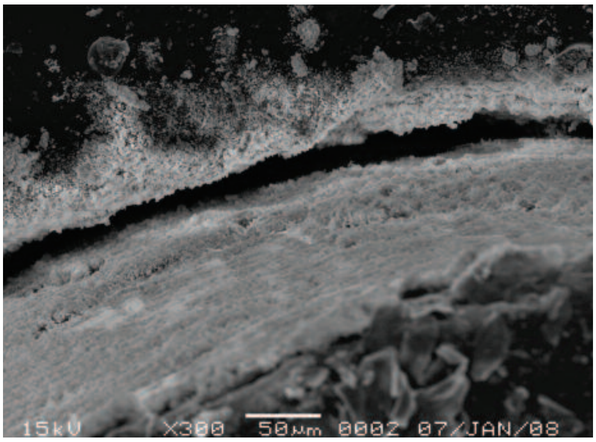

Figure 3: Scanning electron micrograph of a cohesive failure displaying residual luting cement on both interfaces.

were however pooled as the distribution appeared to be similar for the 2 groups and the aim of the study was primarily to compare the two bonding systems.

The presence of water inside the hybrid layer seems to be responsible for bond strength reduction since water droplets trapped inside would act as stress raisers.[16] Furthermore, water will allow for aging/degradation phenomenae as it provides a suitable environment for matrix metalloproteinases hydrolytic action on collagen.[7,17] Ethanol-wet bonding was developed theoretically in an attempt to reduce water content/uptake in hybrid layers by dehydrating the collagen mesh obtained after etching prior to infiltrating it with the hydrophobic resin.[17]

Following smear layer removal, dentinal tubules on the canal wall are rendered patent and possess physical properties similar to that of capillary tubes.[18] One advantage of solvating the bonding resin in ethanol would be to decrease the surface activity of the resin thus allowing for better wetting and penetration. The surface tension reducing ability of ethanol is well documented. Cunningham et al.[19] investigated the effect of ethanol on the spreading property of NaOCl as measured in a capillary tube. Ethanol reduced the surface tension of the NaOCl and significantly improved the ability of the irrigant to spread in vitro. Furthermore, as surface wetting can also be affected by altering the surface activity of the dentin,[18] wetting root canal walls with ethanol prior to applying the bonding agent should also improve resin penetration. Obviously, resin penetration depth into dentinal tubules could be correlated to leakage resistance. The data remains inconclusive as some studies showed correlations [20,21]and others did not.[18] Further research remains needed to assess relationship between sealer penetration and leakage resistance.

When tested on coronal dentin[15], the ethanol-wet bonding technique demonstrated improved bond strength after occluding dentinal tubules. According to these authors, the reduced bond strength in patent tubules could be attributed to water recontamination of the hybrid layer in deep dentin. They hypothesized that water derived from the pulp chamber could have contaminated the chemically dehydrated dentin surfaces hence resulting in poor wetting by hydrophobic monomers, which justified the use of tubular occlusion materials as a possible solution to the problem of water recontamination. While many studies assessed water content of endodontically treated teeth, few papers described water movement.[21,22] Hypothetically, this "weeping dentin phenomenon" should require positive pulpal pressure in order to extrude liquid through the dentinal tubules such as in vital teeth and would be unlikely in endodontically treated teeth. Hashimoto et al.[23] showed that without pulpal pressure, no fluid movement was detected and furthermore that with the notable exception of air blows, most other procedural steps produced an inward movement of fluid in the dentinal tubules. Furthermore, a recent study[24] showed that water affecting hybrid layers did not spontaneously permeate from root dentin of endodontically treated teeth but rather originated from an incomplete evaporation of primer solutions.

A possible explanation to the low push out strength observed in the apical sections could be related to the light transmission characteristics of the posts.[25] These authors showed a 60-68% reduction in light transmission between 4mm and 8mm from the canal entrance. Dual-cure resins include monomers that are polymerized by self and light activation. Light activation initiator systems are generally based on camphoroquinone, which absorbs visible light energy between 400 and 500nm[26] and associates with a tertiary amine, then dissociates into free radicals. Although these modes of activation are independent, light activation is required to increase the degree of conversion.[27] Another possibility could be the inclusion of rare earth elements in the composition of some fiber-posts. The post used in this study contains ytterbium for radiopacity. This rare earth element has associated with the photodarkening phenomenon, which is an optical effect described for amorphous light transmitting media.[28] It involves the interaction of radiation with amorphous glass thus creating absorbing color centers in the optical media due to the resonant interaction of light with the rare earth compounds. In particular, Ytterbium-doped optical fibers have exhibited severe transmission losses, which are strongest at short wavelengths (e.g., in the visible spectral range).[28] As such losses are reported to increase during operation (proportionally to the seventh power of the density of excited ytterbium ions), ytterbium-doped fiber posts could theoretically lose their light transmission characteristics, and the amount of light that reaches the resin cement beyond a certain depth would not be effective for setting off the light-induced initiation of the polymerization reaction. However, this remains speculative and further research is required to confirm or otherwise infirm this hypothesis.

The type of failure is adhesive and seemed to happen mostly on the resin/dentin interface. This is accordance with a study by Ounsi et al.[29] as push-out resistance between the post and the resin was found to be around 30MPa while push-out strengths observed were around 7.5MPa. It should be stressed that alternate bonding strategies are being studied. Bouillaget et al.[30] improved push-out bond strength of intracanal bonded resin cones by using an indirect technique. They hypothesized that bond strengths to radicular dentin could be improved by utilizing procedures that compensate for polymerization shrinkage. However, several studies admitted that there are no immediate applications for this technique before its optimization.[30,31] Based on these findings, the proposed hypothesis was accepted.

Since this study established identical baseline behavior for both bonding systems, long-term follow-up studies can now be conducted to investigate the influence of water and fatigue on bond strength performance.

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