

Differential hydrolytic degradation of dentin bonds when luting carbon fiber posts to the root canal

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Abstract

Objective: The purpose of the study was to evaluate the effect of water storage, adhesive system and root canal region on the hydrolytic degradation of dentin bonds to carbon fiber posts.

Study design: Fiber posts were bonded to the root canals using different adhesive systems and composites: SB1 XT group (Scotchbond 1 XT/RelyX ARC), OBF group (One Up Bond F Plus/Ionotite F), and AB2 group (All Bond 2/C&B) After water storage (10 days), all roots were sectioned into 1 mm beams and divided into coronal or apical group. The specimens were stored in water at 22-26 °C temperature for 15 or 60 days and tested for microtensile bond strength at a crosshead speed of 0.9 mm/min after the calculation of the bonding area. Statistical analysis was performed with ANOVA followed by Tukey test to detect differences between groups ($\alpha=0.05$). SEM investigation was performed to determine the mode of fracture.

Results: Bond strength at coronal and apical half showed significant differences between experimental groups both after 15 and 60 days of water storage. Premature failures were observed in 16-20% of specimens before storage in water.

Conclusions: For the bonding systems tested, clinicians should consider that bond strength inside the root canal at apical half is lower than at coronal half irrespective of the adhesive system. The adhesion within the root canal is possible for SB1 XT and OBF groups unlike the AB2 group where adhesion to root canal dentine is not reliable. Nevertheless, apical half represents the worst scenario in which is possible to obtain a durable adhesion.

Key words: *Fiber post, adhesion, water degradation, storage.*

Introduction

Fiber posts may be a suitable alternative to cast or pre-fabricated metal posts for restoring endodontically treated teeth (1). Fiber-reinforced, epoxy resin-based posts are commonly used in clinical practice with methacrylate-based adhesives, dentine bonding agents (DBAs) and composite resin cements (2). Several authors have evaluated adhesion to root canal dentine both by microtensile bond strength (μ TBS) tests and push-out tests (3,4). Microtensile bond strength can produce uniform stress distribution (5) and attain adhesion at different levels of the root canal (3). The force is applied perpendicularly to the surface without external interferences (5), unlike the push-out test that has a considerable friction component (6).

Authors have reported the regional differences in bond strength among various levels of the root (3,7), and showed that the coronal third created the best adhesion probably because of easier access (8). Adhesion in the root canal is complicated because it is difficult to control the humidity of the substrate required for application of some etch-and-rinse adhesives (9). Moreover, the degree of conversion of light-polymerized adhesives, presence of debris or sclerotic dentine, the effects of irrigants on the dentine surface (3,4), and the unfavorable C-factor, which is the ratio of the surface areas in contact and out of contact with the tooth, are also very important (10,11).

Bouillaguet et al. (3) reported that C-factor inside the root canal could exceed 200, compared to values between 1 and 5 for intracoronal restorations. In addition, Sauro et al. (9) recently reported that root dentine is a wet substrate and current dentine bonding agents (DBAs) pose several problems related to the formation of voids and blister-like structures that may induce deterioration over time (12). Water plasticizes polymer chains and lowers the mechanical properties of hydrophilic resins promoting hydrolysis of components (13-15). The purpose of this study was to compare regional bond strength of carbon posts luted with different bonding systems/composite resins in different levels of root canals (from apical to coronal) after 2 different periods of water storage. The second objective was to evaluate the fracture morphology of tested specimens. The null hypothesis tested was that bond strength would not be affected by adhesive systems applied or by hydrolytic degradation.

Material and Methods

Forty-four human incisors extracted for periodontal reasons were obtained from patients with age between 35 and 60 years old and stored in saline solution at 4°C no more than 2 months. The teeth were horizontally sectioned 0.5 mm below the cement-enamel junction. The root canal preparation was performed with NiTi rotary

instruments (Mtwo; Sweden & Martina, Padova, Italy) operated at 350 rpm under constant irrigation. Irrigation with 5 mL 5% NaOCl (Niocl 5% Dental, Ogna, Italy) and 5 mL 10% EDTA (ethylenediaminetetraacetic acid) (Tubuliclean, Ogna, Italy) was performed at each step. The final preparation had a .06 taper and a diameter of 0.3 mm at the apex (ISO size 30). The canal was then rinsed with water and dried with paper points (Mynol, Jersey City, NJ). Each canal was obturated using System B device (EIE/Analytic, Technology, Orange, Calif) and AH plus (Dentsply DeTrey, Konstanz, Germany). All specimens were stored in water 26°C for 3 days and then the post space was prepared with Gates-Glidden #2, #3 (Dentsply Maillefer, Ballaigues, Switzerland) and with post-hole drills (#10) (Isasan, Rovello Porro, Italy). All teeth received the same type of carbon fiber post #10 (Tech 2000 Xop Isasan, Rovello Porro, Italy). Specimens were divided into 3 experimental groups (n=11) and a control group (n =11) based on the type of adhesive/resin cement used.

In the SB1 XT group, the root canal dentine was etched for 15 s with a 35% phosphoric acid gel (Scotchbond 1 XT; 3M-ESPE, St. Paul, Minn) and rinsed for 30 s with water. The canal was dried with paper points (Mynol, Jersey City, NJ) avoiding the exsiccation of the dentine. One coat of the etch and rinse adhesive (Scotchbond 1 XT; 3M-ESPE, St. Paul, Minn) was applied for 15 s inside the canal and to the post with a microbrush, gently air dried and polymerized for 20 s using a halogen light-curing unit (XL-2500; 3M ESPE, St. Paul, Minn, USA) with an output power intensity of 600mW/cm², at a standardized distance of 5 mm. Equal amounts of RelyX ARC pastes A and B were mixed for 10 seconds, placed inside the canal with a lentulo spiral #40 (PD SA, Vevey, Switzerland) and light cured for 60 seconds after fiber post placement in the canal.

In the OBF group, one coat of the all-in-one self-etch adhesive One Up Bond F Plus (Tokuyama Dental Co, Tokyo, Japan) was applied onto the root canal walls and to the post, gently air dried and polymerized for 20 s. Resin cement (Ionotite F) was mixed at a ratio of 1:1 and introduced into the canal with #40 lentulo spiral. The resin cement was chemically polymerized after post placement.

In the AB2 group, the fiber post was placed with the etch and rinse All Bond 2 adhesive and C&B resin cement (Bisco Inc, Schaumburg, Ill). Root canal dentine was etched as previously described and rinsed for 30 s with water. The canal was dried with paper points without dehydrating the dentine. Five to six coats of mixed primer (Primer A & B, All Bond 2 Kit, Bisco) were applied with a microbrush in the post-space and to the post. Dentine/enamel bonding resin (D/E Resin, All Bond 2 Kit, Bisco) was mixed with Pre-Bond (All Bond 2 Kit, Bisco) to make the adhesive resin dual-

polymerizable. Resin cement (C&B resin cements A and B) were mixed for 10 s and placed inside the canal with a #40 lentulo spiral. The post was covered with luting cement, inserted into the canal and finally polymerized for 60 s.

The teeth were stored in water at 22-26°C temperature for 10 days after post insertion. Teeth were sectioned perpendicularly to the long axis (1 mm thick) with a water cooled low speed saw (Remet, Casalecchio di Reno, Bologna, Italy). Obtaining three to five beams from each resin-bonded root dentine. The thickness of each specimen was measured with a digital caliper (Mitutoyo, Sakado, Japan) (Accuracy: ± 0.02 mm) (~ 0.9 mm). Specimen preparation was completed by trimming each of them to an hourglass profile using a fixing device and a flexible polishing disc (Sof-Lex; 3M-ESPE) under continuous water spray (trimming-modified-technique). Each specimen was clamped between the rubber coated surfaces of the fixing device and trimmed using a flexible polishing disc mounted in a low-speed handpiece at 20.000 rpm (Bien Air; Chemin des Grillons, Bienne, Switzerland) to reduce the amount of vibration. Some specimens failed during the trimming phase, in particular in the control group where no etching and bonding were used. The specimens were then stored in water at 22-26°C temperature for 2 different periods: 15 days or 60 days in order to evaluate the effect of hydrolytic degradation on bond strength.

Cyanoacrylate adhesive (Zapit, Corona, Calif) was used to glue the specimens to the jig of the microtensile tester (Bisco Inc., Schaumburg, Ill). Subsequently, the specimens were tested to failure at a crosshead speed of 0.9 mm/min. Bond strength was calculated at coronal and apical halves and expressed as the force at failure divided by the bonded cross-sectional surface area with a formula used previously by Bouillaguet (3). Since the bonding interface was curved, the exact length of the interface was calculated by measuring the cord (L) and then calculating the length of the arc (L'), where θ is the angle formed between the cord and the center of the post. $L' = r \times 2 \sin \theta^{-1} \times (L/2r)$

After μ TBS testing, the morphology of the fractured specimens was classified on the basis of mode of failure at dentine-cement interface or cement-post interface as observed visually and using SEM.

Bond strength values were expressed in MPa and analyzed by ANOVA to ascertain the effect of the different adhesives, root dentin zone and water storage. Interactions were included in the analysis. Post hoc multiple comparisons were performed using the Tukey test ($\alpha = 0.05$).

Pre-test failures, that are failures during specimens preparation, were also recorded.

Three apical and 3 coronal half specimens tested were selected for each group and analyzed by SEM

(JSM-5200; JEOL, Tokyo, Japan). The digitized SEM images were subjected to image analysis using a digital slow-scan image recording system (SemAfore; JEOL, Sollentuna, Sweden). The thickness of the marginal hybrid layer was evaluated every 100-150 μ m. All the values were averaged to calculate the mean marginal hybrid layer thickness for the inspected specimens.

Results

The table 1 shows the μ TBS results obtained at 15 and 60 days of storage in water. SB1 XT, OBF and AB2 groups showed significant differences at coronal half both after 15 ($P=0.005$) and 60 days ($P=0.037$) (Table 2). SB1 XT, OBF and AB2 groups did not show significant differences at apical half both after 15 ($P=0.098$) and 60 days ($P=0.14$). SB1 XT and OBF groups did not show statistically different bond strength at coronal half both after 15 and 60 days ($P>0.05$). SB1 XT and AB2 groups showed statistically different bond strength at coronal half both after 15 and after 60 days ($P<0.05$), while OBF and AB2 groups only after 15 days ($P<0.05$).

Table 1 lists the percentage of failure on the basis of the morphology of the fractured interface. No differences in the type and the percentage of fracture were found between specimens stored for 15 or 60 days. In the SB1 XT and OBF groups the most frequent type of failure was adhesive between post and composite cement.

In the SB1 XT group resin tags and lateral branches were frequently observed both in apical (Fig. 1a) and coronal half (Fig. 1b). SEM observations showed voids in the composite cement thickness. At apical half the diameter of the voids was greater (about 70 μ m) than at coronal half (about 40 μ m).

In the OBF group, apical specimen showed rare porosities. Resin tags were rare at apical half (Fig. 2a) but clearly visible at coronal half (Fig. 2b).

The AB2 group showed more frequent fractures at the resin cement-dentine interface both for apical and for coronal half specimens. Residual resin cement was present on the post surface after microtensile test (Fig. 3a) therefore, the fractures occurred between DBA and the dentine interface. Resin tags detached from the dentinal surface (Fig. 3b) were visible on the post surface. At the coronal half no voids were visible inside the thickness of the resin cement. In the control group, all the fractures occurred at the resin cement-dentine interface (Table 1).

The AB2 group showed a higher percentage of pre-test failures (20.2%) than SB1 XT (16.6%) and OBF groups (14.3%). The control group presented the highest percentage of failures (81.3 %). The length of resin tags and the thickness of the hybrid layer are reported in (Table 1).

Table 1. Mean (SD) microtensile strength values, percentage of change in bond strengths after 15 days and 60 days of storage in water (MPa), the fracture morphology the length of resin tags and thickness of hybrid layer (μm).

Material	Coronal half (Mpa)mean \pm SD		Apical half (Mpa)mean \pm SD		% change from 15 to 60 days of storage		% dentine-cement fracture		% cement-post fracture		Coronal half		Apical half	
	15 days	60 days	15 days	60 days	Coronal half	Apical half	Coronal	Apical	Coronal	Apical	Thickness of ibrid layer	Length of resin tags	Thickness of ibrid layer	Length of resin tags
SB1 XT/RelyX ARC n=11; s= 34	9.3 (3.0) ^A	6.4 (2.9) ^B	6.0 (3.6) ^E	4.0 (2.0) ^E	31.2	33.3	13.6	30.0	86.4	70.0	40	25-30	15	25-30
OBF/Ionote F n=11; s= 36	8.1 (4.1) ^A	6.2 (3.6) ^B	5.3 (3.1) ^E	5.5 (2.2) ^E	23.5	+3.8	7.1	18.2	92.9	81.8	4	25-30	3	20-25
AB2/C&B n=11; s= 29	4.1 (3.7) ^C	2.9 (2.8) ^D	2.6 (2.4) ^E	3.5 (0.8) ^E	29.3	+34.6	90.9	71.4	9.1	28.6	10-12	-	20	8-10 on post surface
Control Group (RelyX ARC) n=11; s= 3	1.4 (1.0)				-	-	100.0	-	0.0	-	-	-	-	-

SB1 XT = Scotchbond XT; OBF = One Up Bond F Plus; AB2 =All Bond 2

SD = standard deviation

n = number of teeth per group

s = number of sticks obtained after specimens preparation

Groups with different superscripted letters are significantly different (P<.05).

Table 2. One-way analysis of variance.

Dimension	Source	Sum of squares	df	Mean square	F	P
μTBS 15 days coronal	Between groups	159.9	2	76.4	6.2	0.005
	Error	430.3	35	12.3		
	Total	583.1	37			
μTBS 15 days apical	Between groups	51.5	2	25.8	2.5	0.098
	Error	263.9	26	10.2		
	Total	315.5	28			
μTBS 60 days coronal	Between groups	71.5	2	35.7	3.7	0.037
	Error	269.3	28	9.6		
	Total	340.7	30			
μTBS 60 days apical	Between groups	15.5	2	7.8	2.2	0.14
	Error	70.5	20	3.5		
	Total	86	22			

μ TBS = microtensile bond strength; df = degrees of freedom.

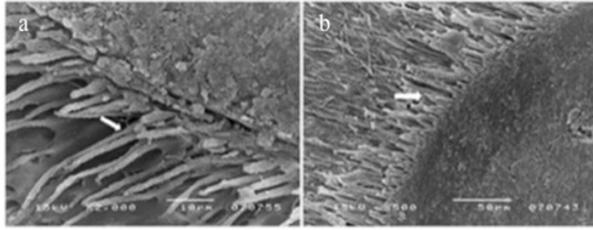


Fig. 1. SEM micrograph of hybrid layer of SB1 XT. Several resin tags and lateral branches (white arrow) are displayed on apical (a) and coronal (b) half specimen.

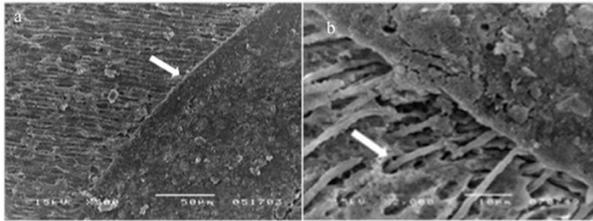


Fig. 2. SEM micrograph of hybrid layer of OBF group. No voids are visible inside the cement and no gaps are present between cement and dentine (white arrow) on the apical half specimen (a). Some resin tags and lateral branches are displayed on coronal half specimen (b).

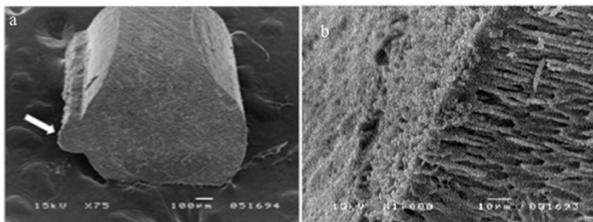


Fig. 3. SEM micrograph of AB2 group. Residuals of resin cement are present on post surface (white arrow) of the apical half specimen (dentine-cement fracture after μ TBS) (a). No hybrid layer is evident on coronal half specimen (B).

Discussion

The most relevant and novel objective of the present study is the challenging of the specimens for hydrolytic degradation. The results showed that the exposure of the specimens to water was responsible for reducing of the bond strength due probably to the hydrolytic degradation (12-14). Therefore, the null hypothesis that bond strength would not be affected by adhesive systems applied or by hydrolytic degradation must be rejected. A relatively longer storage time produced a significant decrease of bond strength for SB1 XT group and a lower decrease of bond strength for OBF group.

Etch and rinse adhesives, such as SB1 XT, are more sensitive to hydrolytic degradation because the exposure of collagen matrix of dentin by acid etching may activate matrix metalloproteinase (MMPs), which

are known to cause collagenolysis in presence of water. Nanoleakage due to incomplete resin penetration in collagen network is considered as pathways for degradation of the adhesive interfacial region through water permeation into hybrid layer (16). The degradation has also been shown to result in significant decrease in bond strength over time after water storage as showed in the present study. OBF showed a lower decrease of bond strength over time also because, using self-etch DBA, collagen fibrils could be completely covered by the resin phase and are inaccessible to water needed to effect collagenolysis (17). SEM analysis disclosed that high values of adhesion to the root dentine surface in the SB1 XT and OBF groups were associated with formation of a homogeneous hybrid layer, in accordance with the results of a recently published investigation (18). By contrast, the AB2 group showed lower values of adhesion and a thinner hybrid layer confirmed by the higher percentage of pretest failure (20.2%) than SB1 XT and OBF groups. SEM micrographs disclosed that AB2 group specimens were fractured at the cement-dentine interface (Fig. 3a) and the hybrid layer was not detected in apical half (Fig. 3b). Interestingly, SEM analysis of the post surface revealed that resin tags were stretched out of the dentine tubules during the microtensile bond strength test.

Voids and porosities were found both in the coronal and apical halves in the SB1 XT group that may be responsible for reduced bond strength at the apical level of root. The OBF group did not demonstrate any voids in the thickness of composite cement either at the coronal or the apical half.

The C-factor increases with the increase of the length of the root canal (10). Therefore, relief of the shrinkage stresses is hampered by the high cavity-configuration factors encountered in these long narrow channels, the non-fitting nature of adapting circular posts to irregular post spaces, and the polymerization that is initiated from the top of the post space (11). As a result, bond strength inside the root canal appears to be lower than results obtained in coronal restorations and may explain the fairly wide standard deviation values of the microtensile bond strength principally in the AB2 and Control groups. Materials with low viscosity, such as RelyX ARC and Ionotite F, could be responsible for an increase in flow during cement polymerization reducing high shrinkage stresses especially inside the root canal. The only surface where cement flow could have occurred is the free surface at coronal third of the canal. This could explain the reduction of microtensile bond strength from coronal to apical level.

Several studies have addressed bond strength at different levels of the root canal using the microtensile test, and concluded that adhesion strength progressively decreases from the coronal to the apical level (3,4). The

present study confirmed this trend reporting adhesion values similar to those reported by Bouillaguet et al. (3) (Table 1). In addition a control group was added without etching and bonding with posts luted only with RelyX ARC to rule out any friction and establish whether adhesion occurs within the canal despite a C-factor value exceeding 200 (3,10). The present study showed a high number of pre-test fractures in the control group (81.3%), where the formation of a hybrid layer is not possible, and post-microtensile fractures occurred between the cement and the dentine. This high number of pre-test fractures was responsible for the low number of beams obtained from the control group after specimens preparation. Indeed 11 teeth were used in this group and only 3 beams were obtained. However, the SBI XT and OBF groups showed fewer pre-test fractures than the control group (16.6% and 14.3 %). Moreover, post-microtensile fractures in the SBI XT and OBF groups occurred principally between the cement and the post surface. These results show that adhesion within the root canal is possible for the SBI XT and OBF groups unlike AB2 group where adhesion to root canal dentine is not reliable. Recently published investigation showed that the predominantly occurring failure mode was adhesive between dentin and resin cement (19). The different failure mode may be partially explained by the fact that in the present investigation it was not used a surface treatment of the post with silane, hydrofluoric acid or hydrogen peroxide that appear to increase post-cement adhesion (20). In fact microtensile bond strength results were lower than those showed in other similar studies (19).

Microtensile bond strength was used because of its uniform stress distribution (5), the possibility to obtain multiple specimens, evaluate bond strength at different levels of the root canal (cervical, middle, apical) (3) and the lack of friction between post and cement unlike push-out (4,6). Considering the higher number of premature failures reported by Bouillaguet et al. (3) using SBI XT/RelyX ARC, the present study shows that the trimming-modified technique (TMT) we used was responsible for the lower number of premature failures: 16.6% as against 40.7% in the Bouillaguet et al study (3).

Conclusions

Within the limitations of this study, the DBAs tested applied to apical halves showed lower bond strength values than those measured for coronal halves. Evaluating the number of pre-test fractures and the mode of failure, it may be concluded that adhesion within the root canal is possible for SBI XT/RelyX ARC and OBF/Ionotite F groups, unlike AB2/C&B group where adhesion to root canal dentine is not reliable. Nevertheless, apical half represents the worst scenario in which is possible to obtain a suitable adhesion. Storage in water affects

hybrid layer by hydrolytic degradation of collagen fibers resulting in significant decrease of bond strength over time. Etch and rinse bonding systems are more prone to hydrolytic degradation due to the exposure of collagen matrix of dentin by acid etching than results in activation of metalloproteinase.

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