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Influence of Endodontic Sealer Composition and Time of Fiber Post Cementation on Sealer Adhesiveness to Bovine Root Dentin

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This study aimed to assess the influence of the type of endodontic sealer (salicylate resin-based sealer vs. two endodontic sealers) and the time of fiber post cementation after root filling on the post adhesion to bovine root dentin. Sixty bovine roots were assigned to six groups (n=10), considering an experimental design with two factors (factorial 3x2): endodontic sealer factor in three levels [epoxy resin-based sealer (AH Plus), eugenol-based sealer (Endofill), and salicylate resin-based sealer plus mineral trioxide aggregate - MTA (MTA Fillapex)] and time for post cementation factor in two levels (immediate post cementation or 15 days after root canal filling). After post cementation, 2-mm-thick slices were produced and submitted to push-out test. The failure modes were analyzed under a 40× stereomicroscope and scored as: adhesive at cement/dentin interface; adhesive at cement/post interface; cement cohesive; post cohesive; dentin cohesive; or mixed. Data were analyzed using two-way ANOVA and Tukey's post-hoc tests (α=0.05). When the fiber posts were cemented immediately after the root canal filling, the bond strengths were similar, independent of the endodontic sealer type. However, after 15 days, the epoxy resin-based sealer presented higher bond strength than the other sealers ($p<0.05$). Comparison between each sealer in different experimental times did not reveal any differences. The main failure type was adhesive at dentin/cement interface (89.4%). The time elapsed between the root canal filling and post cementation has no influence on post/root dentin adhesion. On the contrary, the type of endodontic sealer can influence the adhesion between fiber posts and root dentin.

Key Words: bond strength, endodontic sealer, fiber post, root canal filling, root dentin.

Introduction

Restoration of endodontically treated teeth becomes more complex when the tooth crown is completely or partially lost. Root canal treatment is necessary to retain the restorative materials (i.e., posts, composite cores and crown) (1). Thus, fiber-reinforced composite posts (FRC) are alternatives to cast metal posts because their elastic moduli are similar to that of dentin, producing a favorable stress distribution and providing more aesthetic outcomes for anterior teeth, especially when compounded by glass fibers. Moreover, the cementation of FRC is less time-consuming compared with the indirect approach (cast post and core). Adhesive resin cements have been recommended to improve the retention of posts. The adhesive cementation of FRC for restoring endodontically treated teeth has demonstrated positive longitudinal results (2). Some authors have attributed this success to the similarity between the mechanical properties of the various materials (post-cement-dentin), which results in a homogeneous system (3).

However, the endodontic sealer may negatively affect the bond strength of fiber posts to root dentin. Some authors observed a loss of retention when eugenol-based sealers were used before post cementation with resin cements (4), while other studies found no significant difference when comparing eugenol- and non–eugenol-containing root canal sealers in terms of post retention using a resin cement (1,5). Currently, eugenol-based sealers are still widely employed in Endodontics due their long history of clinical success; however, epoxy resin-based sealers are preferred because of their satisfactory physical properties and adequate biological performance. In addition, the epoxy resin-based sealers are still closer to the ideal filling due to a significantly smaller number of gap-containing interface areas (6).

Recently, a new salicylate resin-based sealer has been launched to the market. It is a paste-paste sealer that contains mineral trioxide aggregate (MTA). It is biocompatible, radiopaque, and stimulates mineralization (7) and calcium ion release (8). According to the manufacturer, it provides easy handling and has the following physical properties: working time, 35 min; flow capacity, 27.66 mm; setting time, 130 min; optical density, 77%; and solubility, 0.1 (7). Some investigations of this compound, such as antibacterial activity (9), cytotoxicity

(10), solubility (11) and bond strength to root dentin (12) have been undertaken. However, there is no study that assessed the influence of salicylate resin-based sealers on the bond strength of glass fiber posts to root dentin and on the time elapsed between root canal filling and post luting.

The aim of this study was to assess the influence of the endodontic sealer type and the time elapsed between root canal filling and post cementation on the bond strength of fiber posts and bovine root dentin. The tested hypotheses were as follows: (1) when FRCs are cemented immediately after root canal filling, the salicylate resin-based sealer presents a bond strength similar to that of epoxy resins and higher than that of eugenol-based sealers; (2) 15 days later, the bond strength of FRCs to root dentin is similar, irrespective of the endodontic sealer; (3) post cementation 15 days after root canal filling increases the bond strength values observed in the eugenol-based sealer group.

Material and Methods

Initial Procedures

After approval by the institutional ethics committee (Process #23081.015314/2010-30), single-rooted bovine teeth with similar dimensions and no root defects were cleaned from surface-adhered debris using periodontal curettes and were stored in distilled water. The teeth were decoronated at the cementoenamel junction to standardize a remaining root length of 16 mm and those with coronal root canal diameter not greater than 2 mm, as measured with a digital caliper (Starrett 727; Starrett, Itu, SP, Brazil), were selected for the study. A sample of 60 roots was obtained.

The roots were embedded in acrylic resin (Dencrilay; Dencril, Caieiras, SP, Brazil) cylinders and the coronal portion of the canals was prepared with Gattes-Gliden drills (Dentsply Maillefer, Ballaigues, Switzerland), sizes 2, 3 and 4 at a depth of 10 mm. Next, a size 15 K-file (Dentsply Maillefer) was inserted to a depth of 15 mm, and the canals were prepared according to a stepback technique up to a size 60 K-file. The canals were irrigated with 1 mL of 1.0% NaOCl at each change of file during chemomechanical preparation, then filled with 2 mL of 17% EDTA for 3 min, flushed with 2 mL of 1% NaOCl, and dried with absorbent paper points (Dentsply Maillefer).

Test Groups

The roots were randomly allocated into 3 groups (n=20), according to the type of endodontic sealer: E - zinc oxide eugenol-based sealer (Endofill, Dentsply Maillefer); MTAF - salicylate resin-based sealer plus MTA (MTA Fillapex; Angelus, Londrina, PR, Brazil); AH - epoxy resin-based sealer (AH Plus, Dentsply Maillefer). Each group was divided into two subgroups (n=10), according to the time elapsed between root canal filling and fiber post cementation: immediate fiber post cementation (0 days) or 15 days after root canal filling (15 days). Thus, 6 test groups were formed (n=10): E_0, $MTAF_0$, AH_0, E_{15}, $MTAF_{15}$ and AH_{15}.

Specimens were filled with a size 60 master gutta-percha cone and accessory gutta-percha cones (Dentsply Maillefer), which were adapted to the tested endodontic sealers using lateral compaction. Root canal sealers were handled according to the manufacturers' instructions.

In groups E_{15}, $MTAF_{15}$, and AH_{15}, the specimens were sealed with a glass ionomer sealer (Vidrion R; SS White, Rio de JaneiroRJ, Brazil) after root canal filling and stored in distilled water at 37°C. In the groups in which post cementation was performed immediately after root canal filling (E_0, $MTAF_0$, and AH_0), the filling material was partially removed with a heated instrument and then a size 4 Largo Drill (Dentsply Maillefer) completed the removal to a depth of 10 mm. Post space was prepared with a size 3 drill from the Whitepost DC kit (FGM, Joinvile, SC, Brazil) to a depth of 10 mm.

Fiber Post Cementation

Before post cementation, the fiber posts were cleaned with ethyl alcohol 70% and coated with an MPS-based primer (Prosil; FGM). Root dentin was rinsed with distilled water and dried with paper points, followed by 37% phosphoric acid conditioning for 60 s, after which the dentin was rinsed with distilled water and dried. Using a microbrush, a multi-step etch-and-rinse adhesive system (ScotchBond Multipurpose; 3M ESPE, USA) was applied onto the dentin surface as recommended by the manufacturer (dentin etching using 37% phosphoric acid, followed by the application of the Activator®, Primer® and Catalyst® agents).

The dual-cure resin cement (AllCem, FGM) was prepared, taken into the root canal with a lentulo spiral, and the fiber post was inserted in a single movement. The cement was light-cured for 40 s using an LED light-curing unit (Radii Cal; SDI, Melbourne, Australia), maintaining the light guide tip of the light-curing unit placed perpendicular to the post (13). A single operator performed all procedures to avoid bias.

Specimen Production and Push-Out Test

Each specimen was fixed on the metallic base of a sectioning machine (LabCut 1010; Extec Corp, Enfield, CT, USA). The first cervical slice (approximately 1 mm thick) was discarded due the excess of cement in that region, which could influence adhesive resistance. Three other slices *per* specimen (thickness: 2 ± 0.3 mm) were obtained (n=30/group).

Each slice was positioned on a metallic device with a central opening (Ø=3 mm) larger than the canal diameter. The most coronal portion of the specimen was placed downward. For push-out testing, a metallic cylinder (Ø extremity = 0.8 mm) induced a load on the post in an apical to coronal direction without applying any pressure to the cement and/or dentin. Considering that the specimens were embedded into the epoxy resin parallel to the root axis and that the specimens were sectioned perpendicular to that axis, the post was submitted to parallel pressure to the greatest extent in relation to the root axis.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, São José dos Pinhais, PR, Brazil) at a speed of 1 mm/min. The bond strength (σ) in MPa was obtained with the formula σ=F/A, where F = load for specimen rupture (N) and A = bonded area (mm^2). To determine the bonded interface area, the formula to calculate the lateral area of a circular straight cone with parallel bases was used. The formula is: $A=2\pi g(R1 + R2)$, where π=3.14, g= slant height, R1= smaller base radius, R2= larger base radius. To determine the slant height, the following calculation was used: $g^2=(h^2 + [R2- R1]^2)$, where h= section height. R1 and R2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, which corresponded to the internal diameter between the root canal walls (3). These diameters and h were measured using the digital caliper.

Data Analysis

Disk specimens with cohesive fracture of the fiber post or dentin were excluded from the study to avoid misinterpretation of the results, as these types of specimens would not yield the real push-out bond strength.

After the push-out testing, the mean bond strength values of the six groups (n=10) were calculated and used for statistical analysis. The data were analyzed by two-way ANOVA (time for post cementation in two levels and endodontic sealer in three levels) and Tukey's test. The significance level was set at 5%.

Results

No specimen was lost during sectioning. After testing, some post cohesive failures and dentin fractures were observed. These specimens were excluded from the push-out bond strength calculations.

The two-way ANOVA revealed that the two variables, sealer composition and time for post cementation, affected the push-out bond strength values (p=0.0001 and p=0.3030, respectively). The interaction between groups is shown in Table 1. No significant difference was observed (p>0.05) when posts were cemented immediately after root canal filling. However, Tukey's post-hoc test demonstrated that the epoxy resin-based group promoted higher bond strength than that of the other groups after 15 days (p<0.05). Comparing the different times for the same endodontic sealer, there were no differences for any of the three tested sealers (p>0.05).

Failure mode distribution is illustrated in Table 2. Adhesive failure between the cement and root dentin (Ac/d) was predominant (89.4%), followed by mixed failures (7.2%). Adhesive failure between the cement and post occurred in 1.7% of the specimens, while post and dentin cohesive failures represented 1.1% and 0.6%, respectively. Figure 1 represents the failure modes.

Table 1. Bond strength mean values for the sealers according to time

Time	Means and standard deviations
Immediate	
Endofill	1.7 ± 1.1 a
MTA Fillapex	1.7 ± 0.6 a
AH Plus	2.8 ± 1.4 ab
15 days	
Endofill	1.6 ± 1.0 a
MTA Fillapex	2.5 ± 2.2 a
AH Plus	4.3 ± 1.4 b

Different letters indicate statistically significant difference at 5%.

Table 2. Failure mode distribution after the push-out test for the sealers according to time

Time	Ac/d	Ac/p	CC	DC	PC	M
Immediate						
Endofill	26	3	-	-	-	1
MTA Fillapex	27	-	-	1	1	1
AH Plus	25	-	-	-	-	5
15 days						
Endofill	28	-	-	-	-	2
MTA Fillapex	27	-	-	-	1	2
AH Plus	28	-	-	-	-	2
Total	161(89.4%)	3 (1.7%)	- (0%)	1 (0.6%)	2 (1.1%)	13 (7.2%)

*Ac/d = Adhesive at cement/dentin interface; Ac/p = Adhesive at cement/post interface; CC = Cement cohesive failure; DC = Dentin cohesive failure; PC = Post cohesive failure; M = Mixed failure.

Discussion

Debonding has been reported to be the primary failure of fiber posts (14). Additionally, the clinical long-term success of the adhesively cemented FRCs depends on chemical, mechanical and patient-related factors. Accordingly, the influence of endodontic sealers on FRC bond strength to root dentin was assessed. This study aimed to assess the influence of the type of endodontic sealer and the time of fiber post cementation after root filling on FRC adhesion to bovine root dentin. Because it is difficult to obtain single-rooted human teeth, bovine teeth are a good alternative for *in vitro* studies. Previous reports indicate few microscopic differences between bovine and human dentin (15). Moreover, the volume and morphology of bovine teeth are similar to those of human canines (15).

The FRCs presented similar bond strength to root dentin, irrespective of the type of endodontic sealer, when FRC cementation was performed immediately after root canal filling ($p>0.05$). However, the epoxy resin-based sealer promoted higher bond strengths when FRCs were cemented 15 days after root canal filling ($p<0.05$). Thus, both the first and second hypotheses were rejected.

According to Paschal et al. (16), post retention is lower in filled canals when compared with non-filled canals, regardless of the used cement type. This fact highlights the importance of using root canal sealer when the aim is to evaluate the bond strength of fiber posts to root dentin. Thus, it seems unnecessary to include a group without root canal fillings as a control, as the bond strength values cannot be compared. Some previous reports agree with these findings (1,5). Davis et al. (5) assessed the effects of two endodontic sealers (Sealapex, a non-eugenol-containing sealer and Tubli-Seal, a eugenol-containing sealer) on the adhesion of fiber posts to root dentin, and they did not observe any changes in bond strength when the push-out test was performed 1 week after the root canal filling. The main factors responsible for removing the free eugenol were: the time elapsed between the application of root canal sealer and post cementation, the action of the drill on root dentin for post space preparation and irrigation after post preparation (5).

Other, however, studies revealed that eugenol-based sealers decrease the bond strengths of both the composite resin core build-up and the FRCs to dentin (4). Menezes et al. (4) evaluated the bond strength of fiber posts to root dentin when post cementation was performed immediately or after 7 days. Using eugenol-based sealer for root canal filling, the bond strength values were lower when post cementation was performed immediately relative to the bond strength when post cementation was performed after seven days. These results may be explained by the similar diameters of the root canal at the moment of the root canal filling (1.1 mm diameter) and after post preparation (1.5 mm diameter). In this case, only 0.4 mm of possible sealer-impregnated dentin was removed; however, in the present study, 0.9 mm of dentin was excised. These factors can explain the similar results observed in the E_0 and E_{15} groups.

The polymerization reaction of resin cements for post luting and dentin adhesive agents is inhibited by the hydroxyl group in the eugenol-based sealers, which tends to block the reactivity of the radicals responsible for polymerization (17). Unlike previous reports, which found a negative influence of eugenol-based sealers on the retention of fiber posts (4) and indirect restorations (18), the present study found similar values at 0 and 15

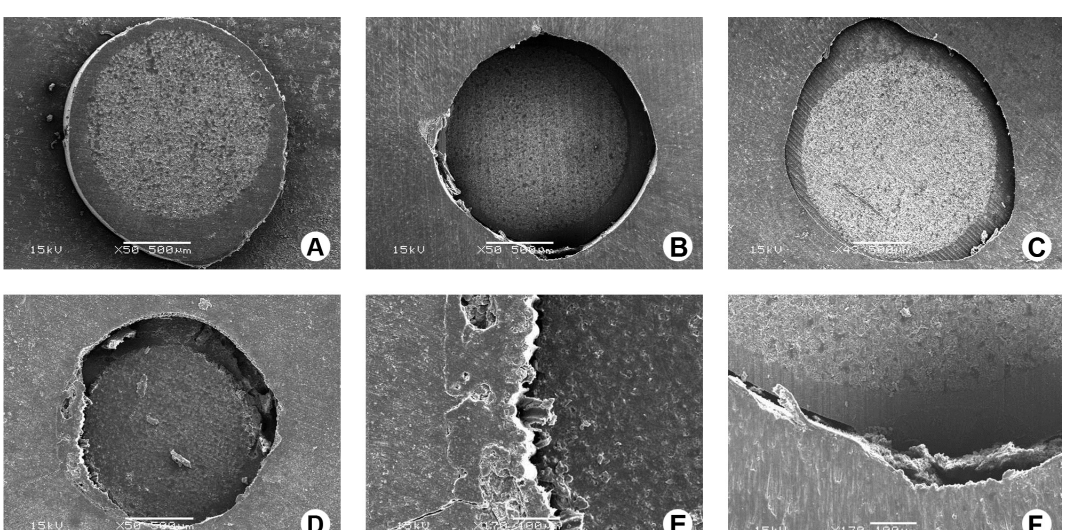

Figure 1. Failure modes. A-C: Adhesive at cement/dentin interface. D: Mixed. E: A high magnification (170×) of image 'D': adhesive at cement/post interface. F: A high magnification (170×) of image 'D': adhesive at cement/dentin interface.

days for eugenol-based sealers. Accordingly, the third hypothesis was also rejected. This fact may be explained by the mechanical removal of the sealer-impregnated dentin from the canal walls during post space preparation.

The removal of the sealer-impregnated dentin from root canals consists of a critical step in achieving optimum post retention when resin cement is used (1). Post preparation performed after root canal filling increases the push-out bond strengths of FRCs to root dentin relative to that of roots prepared before obturation (1). A large double-tapered post (2.0 mm diameter at the cervical portion) was used to simulate clinical practice by removing the major part of the sealer-penetrated dentin during post preparation as well as to ensure an intimate fit at the post-dentin interface. Therefore, the action of the drill for canal preparation may be able of removing the eugenol-impregnated dentin, which could impair the polymerization of the adhesive and resin cement (4). Thus, as reported by Davis et al. (5), the action of the drills and the irrigant can explain the similar results observed in E_0 and E_{15}.

When sealers are introduced into the canal and obturation forces are applied, it is possible that sealer constituents penetrate into the dentinal tubules. Currently, there is a lack of information about the depth of penetration of MTA Fillapex, and further investigations must be performed. Nonetheless, the reported penetration of eugenol-based sealer does not exceed 100 µm (19). However, epoxy resin-based sealers must achieve tubule penetration of up to 1,337 µm (20). Due to the major tubule penetration of the epoxy resin-based sealer relative to that of eugenol-based sealer, the procedures for post cementation were not efficient in removing the sealer-impregnated dentin. Therefore, some uncured epoxy resin-based sealer (AH_0) could have remained. This process may have an important role in adhesiveness when a two-step adhesive system is used (21). Thin resin layers of these adhesives may generate great amounts of uncured acidic monomers, which results in a resin that is not completely cured by the presence of oxygen (22). It is well known that these monomers can adversely react with the amine catalysts present in dual cure cements, preventing/retarding the cement polymerization (21). It can be speculated that one of the three amine groups present in non-cured epoxy resin-based sealers (AH_0) could also react with these monomers, impairing the adhesion of fiber posts to root dentin when cemented immediately, such as the procedure followed for dual cure cements (21). Furthermore, after 15 days, AH Plus is completely set. This circumstance can explain the more favorable results observed with this sealer relative to its performance in immediate testing conditions, although no statistically significant differences were observed.

According to the manufacturer, MTA Fillapex is composed of resins (salicylate, diluting, natural), bismuth oxide, nanoparticulated silica, mineral trioxide aggregate and pigments. To date, little is known regarding its adhesive properties. Previous works (12,23) reported higher adhesiveness to root dentin in AH Plus than in MTA Fillapex; however, no study has evaluated its influence on the adhesion between FRC and root dentin. The time for post cementation did not influence the bond strength of FRC to root dentin when root canals were filled with salicylate resin-based sealer ($p>0.05$). Thus, the composition of salicylate resin-based sealer did not impair the adhesiveness of FRC, as the obtained bond strength values were similar to those of eugenol-based sealers in both tested times for post cementation, and they were also similar to those of epoxy resin-based sealers in immediate testing conditions.

The predominance of adhesive failures (89.4%, Fig. 1) between dentin and cement agree with the literature, which stated that the bond strength on this interface is expected to be lower than that between fiber posts and cement (24). Most adhesive failures at the dentin/cement interface could be explained by the difficulty of controlling moisture inside the root canal, the high C-factor of the cavity and the decreased intensity of light transmission through the root. Additionally, the clinical affinity between post and cement plays an important role in bond strength (25). The low occurrence of cohesive failures (post cohesive, dentin cohesive or cement cohesive) in all experimental groups is due to the cohesive strength of the post, dentin or cement was most likely higher than the cement/dentin bond strength.

Under the experimental conditions of this study, the following conclusions can be drawn: 1. The time elapsed between root canal filling and fiber post cementation has no influence on post/root dentin adhesion; 2. The type of endodontic sealer may influence the adhesion. After 15 days, the epoxy resin-based sealer promoted higher bond strength values between the FRC and root dentin than did salicylate resin- and eugenol-based sealers.

Resumo

Este estudo objetivou avaliar a influência do tipo de cimento endodôntico (um cimento à base de resina de salicilato e dois cimentos endodônticos) e do tempo decorrido entre a obturação do conduto e a cimentação do pino de fibra na adesão de pinos de fibra à dentina radicular bovina. Sessenta dentes bovinos foram divididos em seis grupos (n=10), considerando um desenho experimental de dois fatores (3x2): cimento endodôntico em três níveis [à base de resina epóxica (AH Plus), eugenol (Endofill) e resina de salicilato e MTA (MTA Fillapex)] e o tempo para cimentação em dois níveis (cimentação imediata e 15 dias pós a obturação). Após cimentação do pino de fibra, fatias com 2 mm de espessura foram obtidas e submetidas ao teste de *push-out*. Os padrões de falha foram analisados em estereomicroscópio (40×) e classificados em: adesiva na interface cimento/dentina, adesiva cimento/pino, coesiva do cimento, coesiva do pino, coesiva da dentina e mista. Os dados foram analisados através dos

testes de ANOVA a dois fatores e post hoc de Tukey (α=0,05). Quando os pinos de fibra foram cimentados imediatamente após a obturação dos condutos, a resistência adesiva foi similar, independentemente do tipo de cimento endodôntico. Entretanto, após 15 dias, os dentes obturados com cimento resinoso à base de resina epóxica apresentaram os maiores valores de resistência adesiva (p<0,05). Os valores de resistência adesiva do mesmo cimento nos diferentes tempos experimentais não foram alterados. O principal tipo de falha foi adesiva na interface cimento/dentina (89,4%). O tempo decorrido entre a obturação dos condutos e a cimentação do pino não influenciou a adesão do pino de fibra à dentina radicular. Por outro lado, o tipo de cimento endodôntico influencia a adesão entre dentina radicular e pinos de fibra.

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