

Effect of calcium hydroxide dressing on push-out bond strength of endodontic sealers to root canal dentin

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Abstract: The aim of the present study was to evaluate the effect of calcium hydroxide dressing on the bond strength of three commercially available endodontic sealers (MTA Fillapex, Sealapex, and AH Plus) to root canal dentin. Sixty slices of extracted human canines were obtained from cervical, middle, and apical root thirds. Root canals were standardized and specimens were filled and divided into six groups ($n = 10$): G1, MTA Fillapex; G2, Sealapex; and G3, AH Plus, with prior application of calcium hydroxide dressing; and G4, G5 and G6, without prior application of intracanal dressing. After 7 days, specimens were submitted to a push-out test. The data obtained were analyzed using the ANOVA and Tukey tests ($\alpha = 5\%$). Fracture modes were classified as adhesive, cohesive or mixed. The results of sealer bond strength to root canal dentin varied according to the sealer, root third and prior dressing application. Overall, calcium hydroxide dressing reduced bond strength in all root thirds, but the reduction was significant only for AH Plus, at the cervical (3.25 ± 1.69) and apical (4.43 ± 1.65) thirds ($p < 0.05$). AH Plus showed the highest bond strength for all root thirds ($p < 0.05$) compared to the other groups. G1, G2, G4 and G5 showed similar bond strength values for all root thirds ($p > 0.05$). In conclusion, the calcium hydroxide dressing only had a negative effect on the bond strength of AH Plus, at the cervical and apical thirds. On the other hand, the bond strength values for MTA Fillapex and Sealapex were lower than those for AH Plus and, whereas the mixed failure mode predominated for AH Plus, the adhesive failure mode predominated for MTA Fillapex and Sealapex.

Keywords: Endodontics; Calcium Hydroxide; Dentin.

Introduction

Chemomechanical preparation of root canals is followed by canal filling using a biocompatible sealer with adequate dimensional stability.^{1,2} Solid filling materials such as gutta-percha are normally used in conjunction with different endodontic sealers.³

AH Plus is an epoxy-based sealer used as a standard in several bond strength studies.⁴⁻⁶ On the other hand, other sealers have also been used in endodontic treatment. Sealapex, with a new composition, is a salicylate-based resinous sealer with low cytotoxicity, but with a lower bond strength compared to iRoot and AH Plus sealers.^{7,8} MTA Fillapex is a new salicylate-based resinous sealer, and has shown reasonable biologi-

cal properties and antimicrobial action.^{9,10} Its bond strength to root dentin, however, is lower than that of AH Plus, and can be affected by the presence of moisture in the dentin.^{11,12}

An intracanal calcium hydroxide dressing is recommended between treatment sessions in several clinical situations.¹³ Prior to filling the canal, the dressing should be carefully removed to provide adequate adhesion between the endodontic sealer and the root canal dentin.¹⁴ However, studies have shown that calcium hydroxide-based pastes cannot be completely removed from root canals.¹⁵⁻¹⁷ Calcium hydroxide residue can interact physicochemically with the filling material, increasing apical leakage and compromising the prognosis of endodontic treatment.¹⁸⁻²⁰ Despite recent technological advances in methacrylate-based resin materials, which provide adequate sealing of the root canal, it has been demonstrated that calcium hydroxide residue can have a negative effect on the bond strength of this type of sealer.^{14,21}

Even though it has been hypothesized that the prior use of calcium hydroxide dressing may adversely affect the bond strength of endodontic sealers to root canal dentin, to our knowledge there are no studies in the related literature that have tested this hypothesis in the case of MTA Fillapex and Sealapex sealers. The aim of the present study was thus to evaluate the push-out bond strength of three commercially available endodontic sealers (MTA Fillapex, Sealapex and AH Plus) with and without prior application of an intracanal calcium hydroxide dressing, in the cervical, middle and apical root thirds.

Methodology

The present study was approved by the Research Ethics Committee of the Araraquara School of Dentistry, *Universidade Estadual Paulista* (UNESP), under report no. 67/10. Sixty single-rooted extracted human canines were sectioned transversally at the cemento-enamel junction. The roots were adjusted to 16 mm in length, and the working length was established 1 mm short of the apex. The root canals were explored with a #15 K-file (Dentsply Maillefer, Ballaigues, Switzerland), enlarged to a #25 K-file (Dentsply Maillefer), and irrigated with 5 mL of a 2.5% sodium hypochlo-

rite solution (Asfer, São Caetano do Sul, Brazil), after using each instrument.

The root specimens were then centered in plastic rings (20 mm length × 16.7 internal diameter) and embedded in polyester resin (Maxi Rubber, Diadema, Brazil). All specimens remained intact for 24 h to allow resin polymerization. After the resin was cured, 2.0 mm-thick slices were obtained from the cervical, middle, and apical root thirds. The cervical sections were cut 1 mm apically to the cemento-enamel junction, the middle sections were cut 5 mm apically to the cemento-enamel junction, and the apical sections were cut 8 mm apically to the cemento-enamel junction, thus obtaining 60 slices of each root third, for a total of 180 slices.

The diameter of the root canal slices was standardized as follows. Each slice was enlarged using a low-speed handpiece and a #703 conical steel bur (Vortex Prod. Odontológicos, São Paulo, Brazil) attached to the arm of a surveyor. The arm of the device was lowered to a depth previously determined by a silicone stop to produce a standardized specimen with the following dimensions:

- largest diameter = 1.65 mm,
- smallest diameter = 1.40 mm.

During specimen preparation, the root canals were irrigated with distilled water. Next, the specimens were immersed in a 2.5% sodium hypochlorite solution (Asfer, São Caetano do Sul, Brazil) for 15 minutes, dried and then immersed in a 17% EDTA solution (Biodinâmica, Ibiporã, Brazil) for 3 minutes, and finally washed in distilled water to remove the smear layer.

The specimens were then randomly distributed into 6 groups. Three experimental groups (G1 through G3, $n = 10$) were previously filled with calcium hydroxide paste (Calen; SS White, São Paulo, Brazil) and kept at 37°C and 95% humidity for 21 days. The other three control groups (G4 through G6, $n = 10$) received the same treatment and were stored under identical conditions, but without prior application of a calcium hydroxide dressing. After this period, all specimens were individually immersed in a 2.5% sodium hypochlorite solution and placed on a shaker table (SP Labor, Presidente Prudente, Brazil), operating at level 4, for 3 minutes. After this step, all specimens were observed with a stereomicroscope

(S8APO; Leica Microsystems, Wetzlar, Germany), at 20× magnification, to assess their integrity and the quality of the preparation.

Specimens were then filled with one of the following materials:

- G1, MTA Fillapex (Angelus, Londrina, Brazil; $n = 10$ for each root third);
- G2, Sealapex (SybronEndo, Orange, USA; $n = 10$ for each root third), mixed at a 1:1 ratio (w:w); or
- G3, AH Plus (Dentsply Caulk, Milford, USA; $n = 10$ for each root third).

Groups G4, G5, and G6 were filled with the same materials, respectively.

All sealers were manipulated in accordance with manufacturer instructions. Table 1 shows the composition of the tested endodontic sealers.

Immediately after filling, the specimens were stored at 37°C and 95% humidity for seven days. After this period, the slices were washed, dried and fixed to a metallic apparatus, so that the side with the smaller diameter of the root canal faced upwards. The tip of the plunger used for load application in the push-out test had a diameter of 1.3 mm and was aligned perpendicularly to the upper face of the slice. The push-out test was performed using an electromechanical testing machine (EMIC DL; Emic, São José dos Pinhais, Brazil), calibrated at a constant speed of 0.5 mm/min. The filling was subjected to axial force until it was dislodged from the root canal section.

The force needed to dislodge the filling material (in kN) was transformed into tension (in MPa). The bond strength of each material (in MPa) was calculated according to the following equation:

$$MPa = F/AA$$

where MPa is the bond strength, F is the force, and AA is the bonded area.

AA was calculated according to the following equation:

$$AA = \pi (R + r).g$$

where AA is the bonded area, R is the radius of the canal at the cervical surface (in mm), r is the radius of the canal at the apical surface (in mm), and g is the relative height of the inverted cone (in mm).

The value of g was calculated according to the following equation:

$$g^2 = (R - r)^2 + (2.0)^2$$

The results obtained for each group and for their respective root thirds were subjected to the ANOVA and Tukey tests ($p = 0.05$), using Graph Pad Prism 5.01 statistical software (Graph Pad Software, San Diego, CA).

After performing the push-out test, each specimen was examined under a stereomicroscope (S8APO; Leica Microsystems, Wetzlar, Germany), at 20× magnification, to determine the failure mode. Failure was classified as:

- adhesive, when occurring along the sealer/dentin interface;
- cohesive, within the filling material; and
- mixed, when both types of failure were combined.

The failure frequencies within each group and root third were observed and tabulated.

Table 1. Composition of the endodontic sealers.

Sealer	Composition
MTA Fillapex	Salicylate resin, diluting resin, natural resin, bismuth trioxide, silica, MTA, pigments
Sealapex	Paste A: isobutyl salicylate resin, silicon dioxide, bismuth trioxide, titanium dioxide pigments; Paste B: N-ethyl toluene, sulfonamide resin, silicon dioxide, zinc oxide, calcium oxide
AH Plus	Paste A: epoxy resin, calcium tungstate, zirconium oxide, aerosil, dye; Paste B: 1-adamantane amine, N,N'-dibenzil-5-oxanonandiamine-1,9,TCD-diamine, calcium tungstate, zirconium oxide, aerosil and silicone oil

Results

Table 2 presents the mean push-out bond strength and standard deviation values for the study groups, at the cervical, middle, and apical root thirds, with or without prior application of calcium hydroxide dressing.

Prior application of a calcium hydroxide dressing had a negative effect on the bond strength of AH Plus to root canal dentin, at the cervical and apical root thirds ($p < 0.05$). AH Plus presented higher bond strength values compared to MTA Fillapex and Sea-

Table 2. Mean push-out bond strength values and standard deviation (in MPa) for tested sealers at different root thirds, with or without prior application of calcium hydroxide intracanal dressing.

Root third	Dressing	MTA Fillapex		Sealapex		AH Plus	
Cervical	with	0.54	(0.22) ^c	1.08	(0.25) ^c	3.25	(1.69) ^b
	without	1.21	(0.42) ^c	0.92	(0.28) ^c	5.03	(1.87) ^a
Middle	with	0.46	(0.23) ^b	1.19	(0.30) ^b	3.15	(1.19) ^a
	without	0.90	(0.24) ^b	1.20	(0.37) ^b	3.65	(1.53) ^a
Apical	with	0.70	(0.29) ^c	1.02	(0.23) ^c	4.43	(1.65) ^b
	without	1.19	(0.56) ^c	1.36	(0.40) ^c	10.15	(4.36) ^a

Different superscript letters indicate a statistically significant difference at a 5% significance level, in each root third (ANOVA and Tukey test).

lapex ($p < 0.05$), regardless of prior application of calcium hydroxide dressing. Prior application of a calcium hydroxide dressing had no significant effect on the bond strength of MTA Fillapex and Sealapex ($p > 0.05$), which had similar bond strength values ($p > 0.05$), regardless of the root third.

Table 3 shows the incidence and frequency of each failure mode for each group. Adhesive failure

was most frequent for MTA Fillapex and Sealapex. Cohesive and mixed failures were most frequent for AH Plus. Figure 1 depicts a representative image of adhesive failure, which was the most frequent in the present study.

Discussion

Calcium hydroxide-based dressing had a negative effect on the push-out bond strength value of the AH Plus sealer, only in the cervical and apical root thirds, when compared to AH Plus without prior dressing. In addition, AH Plus presented higher push-out bond strength than MTA Fillapex and Sealapex, independently of the root third considered or whether or not a calcium hydroxide dressing was applied. The push-out bond strength values of MTA Fillapex and Sealapex were similar under all conditions.

Adhesion of endodontic sealers to root canal dentin has been routinely evaluated by push-out testing.^{7,11} This test is easily performed, allowing adequate evaluation of the bond strength of endodontic sealers to root

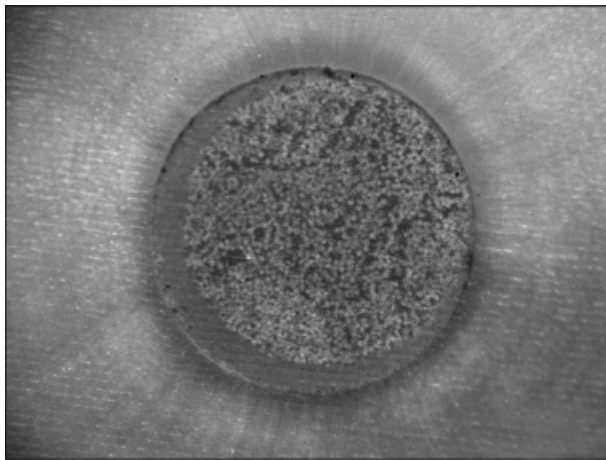


Figure 1. Representative image of adhesive failure.

Table 3. Incidence and frequency (%) of each failure mode for each endodontic sealer, with or without prior application of calcium hydroxide dressing.

Failure mode	Dressing	MTA Fillapex $n = 60$		Sealapex $n = 60$		AH Plus $n = 60$	
Adhesive	with	21	(35%)	23	(38.3%)	10	(16.7%)
	without	20	(33.3%)	18	(30%)	4	(6.7%)
Cohesive	with	0	(0%)	0	(0%)	0	(0%)
	without	0	(0%)	0	(0%)	14	(23.3%)
Mixed	with	10	(16.7%)	7	(11.7%)	20	(33.3%)
	without	9	(15%)	12	(20%)	12	(20%)

canal dentin.²² The main disadvantages of this method are non-uniform distribution of the shear stresses and deformation of the gutta-percha in response to compressive load application during the test.²³ To minimize these problems in the present study, the root canals of the root slices were previously standardized and only filled with an endodontic sealer, thereby ensuring full contact of the tested sealer with the entire dentinal surface of the root canal.^{7,24}

In order to more closely simulate a clinical situation, calcium hydroxide dressing was left in the canal for 21 days.^{25,26} Although the use of calcium hydroxide as an intracanal medicament is well established in endodontics, its complete removal from root canal dentin is a challenge, even using specific removal protocols, and dressing residue inevitably remains on the root canal dentin.^{13,15-17,27} Nevertheless, the effects of the persistence of this residue on the bond strength of the new endodontic sealers tested in the present study remain unknown.

The higher bond strength provided by epoxy-based sealers may be accounted for by the ability of an open epoxide ring to form a covalent bond with exposed amino groups of dentin collagen, as well as by the material's dimensional stability and low polymerization stress.²⁸ Because the adhesion of endodontic sealers to root canal dentin depends on the anatomy of the dentinal tubules and on the collagen fibers present, one possible reason for calcium hydroxide dressing having no negative effect on the bond strength observed in the middle root third is that the dentinal tubules and collagen fibers in this root third are more homogeneously distributed than in other root thirds.^{24,29} Calcium hydroxide residue may have adversely affected the bond strength of AH Plus in the cervical and apical thirds, by acting as a physical barrier between root dentin and the endodontic sealer, as also reported in previous studies.^{14,16,18}

Under certain conditions, MTA Fillapex has been reported to present lower bond strength values than AH Plus or iRoot and Endo-CPM sealers.^{6,11} Sealapex has also presented lower bond strength values than epoxy resin-based sealers.³⁰ According to the manufacturer's description, the chemical composition of MTA Fillapex is similar to that of Sealapex. This may explain why similar bond strength values

were found for these sealers, independently of prior application of calcium hydroxide dressing. The effect of calcium hydroxide dressing on the bond strength of these sealers to root dentin was considered insignificant, primarily because their adhesion to dentin is already very low.^{11,30}

Whereas the mixed failure mode (66.7%) was most frequently observed for AH Plus when a calcium hydroxide dressing was used, the adhesive failure mode was most frequently observed for MTA Fillapex (66.6%) and Sealapex (76.6%) under the same condition, demonstrating the higher degree of adhesion of AH Plus to root dentin compared to the other sealers. When evaluated without prior dressing, the cohesive (46.7%) and mixed (40%) failure modes were most frequent for AH Plus, contrasting with MTA Fillapex and Sealapex which presented a high incidence of adhesive failure (70% and 60%, respectively).

This study found a negative effect of calcium hydroxide dressing residue on the bond strength of the AH Plus sealer to dentin, at the cervical and apical root thirds. Although prior application of a calcium hydroxide dressing did not significantly affect the push-out bond strength values observed for MTA Fillapex and Sealapex, their bond strength was consistently lower than that of AH Plus. Therefore, further studies should be conducted to assess the effects of prior application of a calcium hydroxide dressing and its removal protocols on the bond strength of endodontic sealers to root canal dentin.

Conclusions

Prior application of a calcium hydroxide dressing only had a negative effect on the push-out bond strength of the AH Plus sealer, in the cervical and apical root thirds. Nevertheless, the push-out bond strength values observed for MTA Fillapex and Sealapex were lower than that of AH Plus, in all root thirds, independently of whether or not a calcium hydroxide dressing was applied.

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