

Drying protocol influence on the bond strength and apical sealing of three different endodontic sealers

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Abstract: The aim of this study was to evaluate the influence of drying protocols (DP) on the apical sealing (AS) and on the bond strength (BS) of teeth filled with different sealers. The root canals of one hundred and fifty-six roots of maxillary canines were prepared with Reciproc rotary files (R50). The teeth were randomly divided into four groups ($n = 39$), according to the DP: GI–paper points; GII–70% isopropyl alcohol + aspiration with NaviTip points; GIII–95% ethanol + paper points; GIV–EndoVac + paper points. Each group was divided into subgroups, according to the sealer used: AH Plus, Sealapex and MTA Fillapex, using a single-cone technique. Evaluation of AS and BS was performed with fluid filtration (FF) and push-out (PO) methods, respectively. The PO test consisted of sectioning the roots, and subjecting a single slice from each third to testing and analysis for failure type. The data was submitted to two-way and three-way variance analysis (ANOVA) and Tukey ($\alpha = 5\%$). The AS showed no drying protocol influence. The FF results revealed a statistically significant difference between MTA and Sealapex ($p < 0.05$) sealers. The BS test values showed that there was no statistical significant difference among the canal thirds ($p > 0.05$), but that there was such a difference among the sealers ($p < 0.05$), among the protocols ($p < 0.05$), and in the interaction between sealers and protocols ($p < 0.05$). AH Plus revealed the highest BS values among the sealers; the highest BS results for the sealers occurred with the specimens used with isopropyl alcohol, compared with ethanol and EndoVac.

Keywords: Root Canal Therapy; Wettability; Adhesiveness.

Introduction

Root canal sealers are primarily classified as resin, zinc oxide-eugenolbased, glass-ionomer, and calcium hydroxide. Those containing calcium hydroxide, such as Sealapex[®], were devised with the intention of combining the physical and chemical properties of the sealer with the biological properties of calcium hydroxide.¹ AH Plus[®] epoxy resin sealer has been used in research as a control,² due to its dimensional stability,³ microretention to dentin and reduced solubility.^{4,2} Special attention has been given by researchers to certain newly introduced sealers on the market, such as MTA Fillapex[®]. This material differs from conventional MTA, which contains calcium silicate. It was proposed for reducing the working time of the MTA, facilitating the manipulation phase,⁵ and improving adhesion strength.⁶



The literature has drawn attention to the importance of the adhesive properties of root canal sealers.^{7,8,9} If a material bonds to the root canal walls, it resists dislodgment of the filling.⁹ Adhesion capacity can be influenced by the drying protocol (DP) of the root canal, which interferes in the microleakage process.^{10,11} Although absorbent paper points are normally employed for drying, isopropyl alcohol has provided fillings with high bond strength (BS).² At the same time, mechanical devices such as the EndoVac[®] also allow drying of the root canal; however, there are still no reports on its performance.

The push-out (PO) test provided information that enabled better understanding of the BS of root canal sealers.^{12,13} This method has been used and recommended in the current literature.¹⁴ Fluid filtration (FF) is a method used to determine the sealing ability of the filling materials, and offers advantages such as: preserving the samples after testing,^{15,16} providing quantitative and volumetric data,¹⁷ and adjusting the sensitivity of the fluid transport system.¹⁵ The method has proved accurate and reproducible.¹⁶

This study examined the influence of different DPs of the root canal on the apical sealing (AS) and BS of epoxy resin-based sealers (AH Plus[®]), with calcium hydroxide (Sealapex[®]) and calcium silicate (MTA Fillapex[®]), based on FF and PO tests. The null hypothesis tested was that different drying protocols of the root canal would not affect the apical sealing and bond strength of calcium hydroxide-based and epoxy resin-based sealers.

Methodology

Sample selection

After approval of this study by the local Ethics and Research Committee (protocol no. 250.960), 156 human maxillary canine teeth were selected. They were selected by means of a stereomicroscope (25x), and included those with the apical foramen located in the apical vertex and having rounded shapes, and with similar characteristics. The roots were transversely sectioned in the cervical region, standardizing the length at 15 millimeters.

Biomechanical preparation

The working length (WL) was established by inserting a #15 K-file (Dentsply Maillefer, Ballaigues, Switzerland) in the root canal, until its tip was visualized in the apical foramen, and then retracting it 1 millimeter. The Reciproc[®] system (VDW-Silver, Munich, Germany) was used for instrumentation, with a #R50 file, and irrigation with 1% sodium hypochlorite. Afterwards, the canals were flooded with 5 mL of 17% EDTA for 3 min, followed by irrigation with 5 mL of distilled water.

Distribution of groups

The roots were randomly distributed into 4 groups, according to the DP (n = 39): GI-absorbent paper points; GII- 70% isopropyl alcohol + aspiration with NaviTip points; GIII-95% ethanol + paper points; GIV-EndoVac[®] + paper points. In GI, #50 paper points (Tanari, Manacapuru, Brazil) were successively inserted into the root canal, until the last one to be removed came out completely dry. In GII, 1 mL of 70% isopropyl alcohol was inserted into the root canal, with a 30-gauge blunt-tip syringe as close to the WL as possible. The solution was left in for 1 min, and then aspirated with NaviTip points (Ultradent, South Jordan, USA) for 5 seconds. In GIII, paper points were initially used to remove the distilled water excess, similar to GI. Afterwards, the root canal was flooded with 95% ethanol, by inserting a blunt-tip syringe as close to the WL as possible. The solution was left in for 10 s, and was dried immediately thereafter with paper points. The following procedure was used for GIV-EndoVac[®] (SybronEndo, Orange, USA): a macrocannula was introduced into the canal, at maximum depth in the apical direction, if no resistance was encountered. Then, a microcannula was placed at the WL, and, lastly, a single-cone paper point completed the drying process.

Each group was divided into 3 subgroups, according to the sealer used: AH Plus[®] (De Trey-Dentsply, Konstanz, Germany); Sealapex[®] (Kerr-Sybron, Orange, USA); and MTA Fillapex[®] (Angelus Ind. Prod. Odontológicos, Londrina, Brazil). The sealers were handled according to the guidelines set by their respective manufacturers. The roots were filled using the single-cone technique, and stored

in an incubator at 37°C with 95% humidity, for a period corresponding to three times the hardening time of the respective sealers. Eight of the thirteen roots belonging to each subgroup served to assess the AS, and five, to test the BS, using the FF and PO methods, respectively.

Fluid filtration method

The roots were covered with two coats of clear enamel and a cyanoacrylate ester (Henkel Ltd., Itapevi, Brazil), except for the apical millimeter and the upper inner surface of the cervical portion. Then, a Cralplast universal type 0–200 mL tip (Cral Artigos para Laboratório Ltda., Cotia, Brazil) was inserted into the apical root portion, and the root/tip interface was sealed with the same coating materials. The root assembly/plastic tubing was connected to the FF system, filled with distilled water, and submitted to a pressure of 10 psi. The measurement of the infiltrated volume of fluid inside the root was expressed as $\mu\text{L}/\text{minutes}$.

Push-out test

Initially, the roots were sectioned transversely by a cutting machine (Isomet 1000, Buehler, Illinois, USA), yielding 6 slices, each with a thickness of 2.0 millimeters. The first and last slices were discarded, and each second third was selected for testing. The Instron 4444 Universal Testing Machine was used (Instron Corporation, Canton, USA) with a speed of 1 mm/minutes. The force required for the displacement of the filling material, in kilonewtons (kN), was transformed into stress (σ) given in megapascals (MPa), by dividing the value of the force (F) of displacement by the adhesion area of the filling material (SL) in mm^2 . The following formula was used to calculate the approximate value of the area: $SL = \pi (R + r) g$, where: SL = adhesion area of sealer; $\pi = 3.14$; R = radius of the coronal portion of the slice, in mm; r = radius of the apical portion of the slice, in mm; g = relative height of the slice, in mm. After testing the slices, an analysis was made of the failure type displayed by each slice, using a stereomicroscope (25x) (Zeiss, Stemi 2000-C, Jena, Germany). The failures were classified according to a study by Saleh et al.,¹⁸ namely, adhesive to dentin, when the filling material

was detached from the dentin; adhesive to filling material, when the gutta-percha was detached from the root canal sealer; mixed, when the sealer became detached from both the dentin and gutta-percha; cohesive to dentin, when there was a fracture in the dentin; and cohesive to filling material, when there was a fracture in the root canal sealer.

Statistical analysis

The FF and BS data were subjected to the two-way ANOVA test, and the three-way ANOVA with a split plot, respectively, followed by the Tukey complementary test ($\alpha = 0.05\%$).

Results

Fluid filtration analysis

Only the root canal sealers showed a significantly statistical difference ($p = 0.029$) (Table 1). No difference was observed among the DPs ($p = 0.494$), or in the interaction between sealer and protocol ($p = 0.340$). The Tukey test (Table 2) showed a significant difference only between MTA Fillapex and Sealapex sealers ($p = 0.048$), and a similarity between AH Plus and MTA Fillapex ($p = 0.993$), and between AH Plus and Sealapex ($p = 0.063$).

Push-out analysis

The three-way ANOVA test with a split plot showed no statistically significant difference among the root canal thirds ($p = 0.316$), or interactions among sealers/thirds ($p = 0.630$) and protocols/thirds ($p = 0.888$). There were significant differences among the three different sealer fillings ($p < 0.001$), among the four different DPs ($p = 0.001$), and in the interaction between sealers and protocols ($p = 0.001$).

Based on the Tukey test, AH Plus (1.39 ± 0.60) ($p < 0.001$) was identified as presenting the highest BS values, as compared with Sealapex (0.83 ± 0.33) and MTA Fillapex (0.76 ± 0.34), with no difference between the last two ($p = 0.578$). Among the protocols, isopropyl alcohol (1.19 ± 0.58) promoted BS values similar to the paper points (1.04 ± 0.55) ($p = 0.358$). However, isopropyl alcohol was statistically different from ethanol (0.89 ± 0.35) ($p = 0.005$) and EndoVac (0.86 ± 0.53) ($p = 0.002$), with no significant difference

Table 1. Analysis of variance test.

Source of variation	SS	DF	MS	F	p
Sealers	0.255	2	0.127	3.679	0.029
Protocols	0.0837	3	0.0279	0.806	0.494
Sealers vs. Protocols	0.239	6	0.0399	1.152	0.340
Residual	2.910	84	0.0346	-	-
Total	3.488	95	0.0367	-	-

SS: sum of square; DF: degrees of freedom; MS: mean square; F: F-statistics. p: significance level.

Table 2. Tukey test.

Sealer	Difference in Means	q
MTA vs. Sealapex	0.112*	3.400*
MTA vs. AH Plus	0.00531**	0.161**
AH Plus vs. Sealapex	0.107**	3.239**

q: q test statistic.

*p < 0.05

**ns = not significance

between these last two (p = 0.991). The paper point protocol was similar to that of ethanol (p = 0.320) and of EndoVac (p = 0.190). Since there was no statistical difference among the root canal thirds (cervical, middle, and apical), and since this variance factor neither influenced the DP nor the sealers, a table was constructed with the BS mean values of the three root thirds, according to the sealers and the DP (Table 3).

In the sealer/protocol interaction, it was found that specimens filled with AH Plus sealer and dried with isopropyl alcohol showed higher BS, in comparison with the groups dried with ethanol (p < 0.001) and EndoVac (p = 0.001). Sealapex sealer did not undergo any interference from the DP. In the canals filled with MTA Fillapex and dried with isopropyl alcohol, the BS was significantly higher than those dried with EndoVac.

Fracture pattern analysis

Table 4 shows the incidence (%) of the failures types that occurred after the PO test for each root third.

Discussion

In the present study, the drying protocols did not affect the apical sealing; therefore, the null hypothesis was confirmed. The leakage observed in specimens

filled with AH Plus was similar to that observed in the fillings with Sealapex and MTA Fillapex. Xu et al.¹⁹ and Sagsen et al.²⁰ reported similar results. However, a previous study reported lower leakage values in specimens filled with Sealapex, compared with AH Plus, after 15 days of sealing.¹⁶ Nevertheless, a progressive increase in leakage was noted in the Sealapex group, over the course of this experiment, and the results for both sealers were similar at the end of 60 days. Sealapex underwent volumetric expansion because of water absorption during hardening. This expansion may cause an increase in solubility, with a consequent effect on sealing capability.²¹ The discrepancy in the results may be attributed to the difference in the methods used. Even when similar methods are adopted, the individual parameters of each method must be considered. The measurement period of the leakage, the hardening time of the sealer, and the pressure applied to the system may all influence the results.^{22,17}

There was similarity in the BS among the three root thirds. A previous study reported that even if tubule density is different throughout the root thirds, this factor is not enough to interfere with adhesion of the root canal sealer.²³

The null hypothesis was rejected, because the drying protocols affected the push-out bond strength differently. AH Plus presented the highest BS values, corroborating previous research findings.^{24,2} Several characteristics of this material promote its greater adhesion capability, in relation to Sealapex and MTA Fillapex. AH Plus is an epoxy resin-based sealer with high polymerization time and good flow capacity.²⁵ These characteristics favor its penetration through the dentinal tubules,²⁶ forming tags, similar to what occurs with dentin adhesions.^{27,28,13} Moreover,

Table 3. Mean \pm SD of the BS of the three thirds according to sealer and protocol used.

Protocol	Sealer			Pooled Average
	AH Plus	Sealapex	MTA	
Isopropyl	1.80 \pm 0.49	0.82 \pm 0.30	0.93 \pm 0.36	1.19 \pm 0.58 ^A
Paper point	1.53 \pm 0.61	0.78 \pm 0.27	0.81 \pm 0.35	1.04 \pm 0.55 ^{AB}
EndoVac	1.25 \pm 0.60	0.83 \pm 0.45	0.51 \pm 0.22	0.86 \pm 0.53 ^B
Ethanol	0.98 \pm 0.40	0.90 \pm 0.31	0.78 \pm 0.32	0.89 \pm 0.35 ^B
	1.39 \pm 0.60 ^A	0.83 \pm 0.33 ^B	0.76 \pm 0.34 ^B	-

Different superscript capital letters in the same column or the same line represent a statistically significant difference (SD) among the groups ($p < .01$).

Table 4. Frequency of failure types (%) by root thirds.

Type of Failure	Canal Third		
	Cervical	Middle	Apical
Cohesive to filling material	50	23.33	8.75
Cohesive to dentin	10	16.66	12.10
Adhesive to dentin	10	1.67	32.50
Adhesive to filling material	-	1.67	1.65
Mixed	30	56.67	45

a previous study has suggested that the adhesion of AH Plus to dentin is due not only to mechanical overlapping, but also to its potential to chemically bind to the collagen matrix of the dentin tissue.²⁹

The BS was influenced by the DP, as well as the sealer/protocol interaction. These results are consistent with the findings described in the current literature. Research showed that the moist conditions of the root canal directly interfere in the BS of root canal sealers.^{11,2} Therefore, neither completely moist nor completely dry canals favor adhesion to the sealer.^{10,11}

Regarding the influence of the DP in relation to the BS of the materials, specimens dried with isopropyl alcohol showed the highest BS values, compared with ethanol and EndoVac. Whereas the most favorable conditions for obtaining adhesion to the sealer was a root surface that was not completely dehydrated, it is believed that because ethanol (C_2H_5OH) presents a molecule with higher polarity, this could have favored greater water removal from dentinal tubules than isopropyl alcohol (C_3H_7OH), with lower polarity.^{30,10} The results for the isopropyl alcohol group were similar to those of the absorbent paper point group; this suggests that the paper point

promoted the drying of the canal, but not fully, thus retaining the moisture of the root dentin. The protocol/material interaction analysis showed that the use of paper points with AH Plus presented significantly higher BS values than Sealapex and MTA Fillapex with paper points, thus confirming the previous finding.

The BS values for Sealapex and MTA Fillapex were similar and significantly lower than those for AH Plus, in the specimens dried with isopropyl alcohol, paper points and EndoVac. Although the EndoVac system presents an interesting hydraulic drive, its dynamic as a drying system was not effective. Drying with one of the three previous groups probably retained excessive moisture within the canal, impairing adhesion of Sealapex and MTA Fillapex. This behavior can mostly likely be explained by these cements not having the hydrophilic characteristics displayed by AH Plus.

The analysis of the specimens showed that cohesive failures occurred in the cervical third, primarily in the cohesive material, regardless of the protocol or sealer used. This type of failure may be attributed to the greater amount of sealer in that region, depending on the adopted filling technique, according which a greater open area was noticed between the dentin and the gutta-percha cone in the cervical region than in the remaining thirds.

The results of this study revealed the importance of obtaining more information on DP, in regard to moist conditions, type of sealer favoring adhesion, and curbing of apical leakage.

Conclusion

Under the conditions of this study, DP did not influence the AS of the tested root canal sealers.

However, the highest BS values were observed in specimens dried with isopropyl alcohol, compared with ethanol and EndoVac.

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