

Effect of endodontic chelating solutions on the bond strength of endodontic sealers

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Abstract: The purpose of this *in vitro* study was to evaluate the effect of various chelating solutions on the radicular push-out bond strength of calcium silicate-based and resin-based root canal sealers. Root canals of freshly-extracted single-rooted teeth ($n = 80$) were instrumented by using rotary instruments. The specimens were randomly divided into 4 groups according to the chelating solutions being tested: (1) 17% ethylenediaminetetraacetic acid (EDTA); (2) 9% etidronic acid; (3) 1% peracetic acid (PAA); and (4) distilled water (control). In each group, the roots were further assigned into 2 subgroups according to the sealer used: (1) an epoxy resin-based sealer (AH Plus) and (2) a calcium silicate-based sealer (iRoot SP). Four 1 mm-thick sections were obtained from the coronal aspect of each root ($n = 40$ slices/group). Push-out bond strength test was performed at a crosshead speed of 1 mm/min., and the bond strength data were analyzed statistically with two-way analysis of variance (ANOVA) with Bonferroni's post hoc test ($p < 0.05$). Failure modes were assessed quantitatively under a stereomicroscope. Irrespective of the irrigation regimens, iRoot SP exhibited significantly higher push-out bond strength values than AH Plus ($p < 0.05$). For both the sealers, the use of chelating solutions increased the bond strength, but to levels that were not significantly greater than their respective controls ($p > 0.05$). iRoot SP showed higher resistance to dislocation than AH Plus. Final irrigation with 17% EDTA, 9% Etidronic acid, and 1% PAA did not improve the bond strength of AH Plus and iRoot SP to radicular dentin.

Keywords: Root Canal Obturation; Root Canal Irrigants; Dentin.

Introduction

Three-dimensional obturation of the root canal is one of the key factors for success in endodontic therapy.¹ Today, most root canal filling methods continue to utilize different formulations of gutta-percha in conjunction with a minimum amount of sealer. This standard approach aims to provide a gap-free interface between root canal filling and dentin, which is essential for both establishing a fluid-tight seal,² and providing resistance for dislocation of the root filling during tooth flexure and operative procedures.³ To date, numerous materials have been developed to improve the sealing ability and stability of the root canal filling.

Among a wide spectrum of commercially available root canal sealers, a hydrophilic calcium silicate-based endodontic sealer, iRoot SP has gained popularity in recent years. iRoot SP exhibits excellent physical properties, antimicrobial activity, biocompatibility, and adhesion to root dentin under different moisture conditions.^{4,5,6} The composition of iRoot SP is similar to white mineral trioxide aggregate,⁴ which adheres to dentin through physicochemical reaction.⁷

Over the years, several methods have been tested to improve adhesion of root canal sealers to radicular dentin. Removal of the smear layer is one of these techniques,⁸ and numerous irrigation solutions/regimens have been recommended for that purpose.⁹ Chelating solutions are able to remove the smear layer, and expose a large number of dentinal tubules,¹⁰ which in turn may promote adhesion due to an increased contact area that would ensure a better adaptation between the sealer and root canal dentin.¹¹ However, chelating solutions are also capable of extracting major inorganic elements (e.g., calcium ions) from surface dentin, and partial demineralization may interfere with the bonding effectiveness of canal sealers that chemically adhere to root dentin.^{11,12,13} To date, little information exists on the effect of chelating solutions on the radicular push-out bond strength of calcium silicate-based and resin-based root canal sealers.

In the light of these observations, the aim of this *in vitro* study was to evaluate the effect of 17% ethylenediaminetetraacetic acid (EDTA), 9% etidronic acid, and 1% peracetic acid (PAA) chelating solutions on the bond strength of iRoot SP and a resin-based root canal sealer (AH Plus) to radicular dentin. The null hypothesis tested was that various chelating solutions would affect the bond strength of root canal sealers.

Methodology

Specimen Preparation

Eighty periodontally involved, freshly extracted single-rooted human teeth with straight roots, fully formed apices, free of previous root fillings, and root cracks were used. The crowns of all teeth were removed using a water-cooled, slow-speed diamond precision saw (IsoMet 1000; Buehler, Lake Bluff, USA),

so as to adjust the length of the roots to a standardized length of 16 mm. The root canals were prepared using ProTaper rotary instruments (Dentsply-Maillefer, Ballaigues, Switzerland) up to master apical rotary size F3 (#30), in conjunction with 2 mL of 5.25% sodium hypochlorite (NaOCl) irrigation between each file and a final rinse with 5 mL distilled water. The specimens were randomly divided into four groups (n = 20 each) according to the final irrigation solutions applied: 1.5 mL of 17% EDTA (Pulpdent Corporation, Watertown, MA) (pH = 7); 2.5 mL of 9% etidronic acid (Zschimmer & Schwarz Mohsdorf GmbH & Co. KG, Burgstädt, Germany) (pH < 2); 3.5 mL of 1% peracetic acid (Sigma-Aldrich, Steinheim, Germany); and distilled water (control). In each group, the specimens were further randomly divided into two groups (n = 10 each) according to root canal sealer used: (a) AH Plus (Dentsply DeTrey GmbH, Konstanz, Germany), and (b) iRoot SP (Innovative BioCeramix Inc., Vancouver, Canada). All materials were applied by one calibrated operator in strict adherence to the manufacturers' recommendations. Following root filling procedures, canal openings were sealed with CavitTM-G (3M ESPE, GmbH, Seefeld, Germany). The specimens were stored at 37°C and 100% humidity for 1 week to allow complete setting of the test materials.

Push-out bond strength test

Four 1 mm-thick horizontal sections were obtained from each specimen (n = 40 slices/group) consecutively from coronal-to-apical direction using a water-cooled precision saw (Isomet). Root sections demonstrating oval root canal form (*i.e.*, noninstrumented areas) were discarded and replaced with a new specimen prepared in accordance with the experimental protocol. Thereafter, the filling material was loaded with a stainless steel cylindrical plunger, which provided the most extended coverage over the filling material without contacting the surrounding dentin (Figure 1). Due to the convergence of the root canal sections, the push-out force was applied from apical-to-coronal. Loading was performed on a universal testing machine, (Lloyd Instruments Ltd., Fareham, United Kingdom) at a cross-head speed of 1 mm/min until bond failure occurred. The force was recorded by using Nexygen data-analysis software

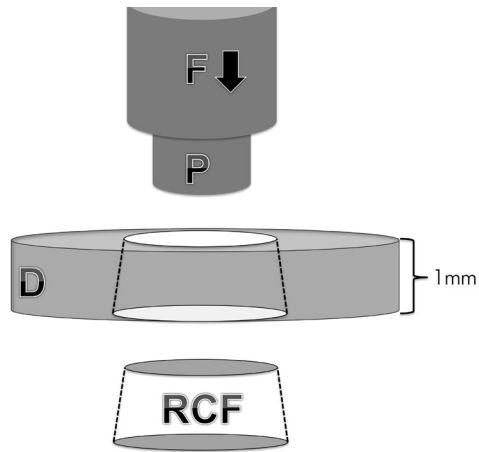


Figure 1. Schematic representation of the push-out test. (F) direction of force; (P) cylindrical plunger; (D) root dentin cylinder (1 mm thick); (RCF) root canal filling (sealer and core material).

(Lloyd Instruments Ltd., Fareham, United Kingdom), and the debonding values were used to calculate push-out strength in megapascals (MPa), according to the following formula: Push-out bond strength (MPa) = Maximum load (Newton)/ Adhesion area of root canal filling (mm^2).

The failure modes of each specimen on both surfaces were evaluated under a stereomicroscope (Olympus Optical Co. Ltd., Tokyo, Japan) at 40X magnification and classified into one of the following categories: I: Adhesive (failure at the sealer-dentin interface or sealer-core interface), II: Cohesive (failure within sealer or dentin), and III: Mixed (failure in both the sealer and dentin).¹⁴

Statistical evaluation

Push-out bond strength data were analyzed statistically in Statistical Package for Social Sciences V.11.5 software (SPSS, IBM, New York, USA) for Windows 2007 (Microsoft, New Mexico, USA) by two-way analysis of variance (ANOVA) with Bonferroni's post hoc test with the significance level of $p < 0.05$.

Results

The push-out bond strength values (MPa) are presented in Table 1 as mean \pm standard deviation.

In all experimental groups, iRoot SP yielded significantly higher push-out bond strength values

Table 1. Push-out bond strength values (MPa, mean \pm standard deviation) of the experimental groups.

	iRoot SP	AH Plus
17% EDTA	2.46 \pm 0.54	2.18 \pm 0.62
9% Etidronate	2.65 \pm 0.76	2.25 \pm 0.78
1% Peracetic Acid	2.54 \pm 0.60	1.98 \pm 0.80
Distilled Water (Control)	2.14 \pm 0.64	1.91 \pm 0.73

than AH Plus ($p < 0.05$). Although the tested chelating solutions improved the bond strength of both canal sealers, the increase was not significantly greater than their respective control values ($p = 0.296$). Likewise, the use of different chelating solutions had no significant effect on the debonding values in both the sealer groups ($p = 0.937$).

The failure modes of the test groups are listed in Table 2. The majority of specimens had adhesive failures along the sealer-dentin interface. Figure 2 depicts a representative image of adhesive failure (iRoot SP), which was the most frequent in the present study.

Discussion

The bond strength of root canal sealers to radicular dentin helps maintain the integrity of the sealer-dentin interface without being disrupted in long term.^{2,15} The push-out test is an efficient and reliable technique to assess bond strength of root canal filling materials to root dentin.¹⁶ Results obtained within the experimental conditions of the present study indicate that iRoot SP

Table 2. The failure modes (A: Adhesive, C: Cohesive, M: Mixed) distribution (%).

Irrigation solution	Failure modes	iRoot SP	AH Plus
17% EDTA	A	87.5	90
	C	10	10
	M	2.5	-
9% Etidronate	A	92.5	85
	C	7.5	10
	M	-	5
1% Peracetic Acid	A	85	92.5
	C	12.5	7.5
	M	2.5	-
Control	A	92.5	87.5
	C	5	12.5
	M	2.5	-

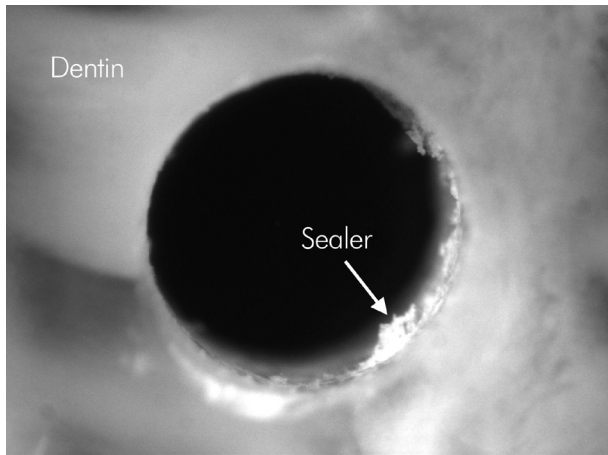


Figure 2. Representative image of adhesive failure.

yields higher dentin bond strength than AH Plus, regardless of the irrigation regimens employed. This finding may be explained in part by the calcium silicate composition of iRoot SP, which helps minimize shrinkage during the setting process.¹⁷ The extremely small particle size and the level of viscosity that enhances the flow of iRoot SP into dentinal tubules may have further enhanced its bonding effectiveness to root canal dentin, resulting in increased resistance to dislocation.¹⁸

In all groups, the predominant fracture mode was adhesive failure along the sealer-filling material interface. This observation of adhesive failures in the majority of the specimens may indicate inadequate level of chemical adhesion between sealers (iRoot SP and AH Plus) and the core material.

Several authors have recommended the sequential use of organic and inorganic solvents as endodontic irrigants, since no single solution has yet proved to be capable of removing the smear layer alone.⁸ A combination of NaOCl and EDTA solutions is recommended for the efficient removal of the smear layer from the surface of the root canal wall.¹⁹ However, this combined irrigation regimen can lead to erosion of the dentin surface.²⁰ Further, exposure of root dentin to EDTA for extended periods may decrease the modulus of elasticity and the flexure strength of dentin.²¹ Such decrease may adversely affect the physical and mechanical properties of dentin, and eventually increase the risk for root fracture.²² Hence, the use of alternative chelating

solutions with less or no detrimental effect on root dentin would be most desirable.²³ Among several decalcifying agents available, PAA appears to be a potential alternative to EDTA to dissolve the smear layer and disinfect the root canal system.^{23,24} A previous study has shown that 1% PAA had similar effect against *Enterococcus faecalis* compared with NaOCl and chlorhexidine.²⁴ PAA solution contains and liberates acetic acid, which is a weak chelating agent, but can reduce the smear layer as effective as EDTA.²³ The results of this study necessitates rejection of the null hypothesis, because the debonding values obtained after PAA treatment were statistically similar to those of EDTA-treated specimens.

Etidronic acid [also known as Etidronate or 1-hydroxyethylidene-1,1-bisphosphonate (HEBP)] is a non-toxic bisphosphonate, whose use as a soft chelating irrigation agent exerts minimal detrimental effect on root dentin while removing the smear.^{25,26} Here, in both iRoot SP and AH Plus groups, final irrigation with 9% etidronic acid resulted in slightly higher bond strength values than those achieved with 17% EDTA and 1% PAA irrigation. Despite some differences in the irrigation protocols, this finding is in agreement with a recent study,²⁷ which showed an improvement in the bond strength of AH Plus when a chelating irrigation protocol involving a mixture of 18% etidronic acid and 5% NaOCl was used in the instrumentation phase. The authors suggested that the use of 17% EDTA as a final rinse could better facilitate exposure of the collagen network and render the dentin substrate more conducive to bonding of AH Plus sealer, which can adhere to the organic phase of radicular dentin.²⁷ In the present study, EDTA was deliberately omitted from the experimental protocol to distinguish the effect of etidronic acid as a final irrigant. Further, a lower concentration of etidronic acid (9%) was utilized, since the demineralization kinetics promoted by both 9% and 18% etidronic acid have been shown to be similar.²⁵

Compared with the present results obtained using chelating solutions, significantly lower bond strength values were achieved in the NaOCl-treated (control) specimens. This finding strongly suggests that the presence of smear layer has a negative impact on the adhesion of iRoot SP and AH Plus. Thus, for

the tested sealers, the micromorphological pattern of the root canal surface achieved by removal of the smear layer maybe more relevant than the mineral content to explain the bonding effectiveness to dentin, though the latter definitely requires further confirmation.²⁸ The increase in surface roughness could be clinically beneficial, because retention is provided by the micromechanical interactions of the canal sealer with dentin tubules.^{29,30}

Conclusion

Within the experimental conditions of this *in vitro* study, it can be concluded that the tested chelating

solutions do not improve the bond strength of AH Plus and iRoot SP to the radicular dentin. From the clinical point of view, these results may indicate selection of a chelating solution that is capable of removing the smear with minimal adverse effect on dentin would be advantageous. Based on the present bond strength data, PAA appears to be a suitable alternative to EDTA, provided that its efficacy is further demonstrated with respect to several other properties including antibacterial effect, biocompatibility, alteration of the chemical composition of root canal dentin, and finally, its interaction with other endodontic sealers.

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