

Effect of the application of different concentrations of EDTA on the adhesion of fiber posts using self-adhesive cements

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Abstract: The purpose of this study was to analyze the effect of the application of different concentrations of EDTA on the adhesion of fiber posts to root dentin using self-adhesive resin cements. After endodontic treatment, 78 single roots were randomly divided into six groups (n = 13) according to the combination of the following factors: surface dentin treatment – control (distilled water), 17% EDTA and 24% EDTA; and self-adhesive resin cement - RelyX U200 (RX); and Multilink Speed (ML). After fiber post cementation, six slices were obtained for each root. Ten roots of each group were used for bond strength (BS) and three for microhardness (MH) evaluations. Data obtained from BS and MH tests for each resin cement were subjected to two-way ANOVA (surface treatment vs. root region) and to a post-hoc Tukey's test ($\alpha = 0.05$). The lowest BS value was observed in the 24% EDTA group for RX cement, whereas the highest values for ML cement were observed for the control group in the middle and apical regions. In the MH test, the lowest value for RX was observed for 24% EDTA in the cervical region, whereas and the highest value for the ML cement was observed in the control group. Regarding both self-adhesive resin cements tested, the application of 24% EDTA was not able to improve the adhesion of fiber posts to root canal.

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Introduction

Fiber posts are commonly used in the reconstruction of teeth with extensive coronal destruction, since endodontically-treated teeth are more prone to fractures than are vital teeth.¹ Post systems minimize masticatory stress and serve as mechanical retention of the definitive restorations.² These retainers are cemented in the root canal by resin cements which require (or do not require) previous acid etching.³

Self-adhesive resin cements are commercially available and more easily applied than are conventional resin cements, in addition to combining characteristics of self-etching adhesive systems and composite resins.⁴ These cements contain functional acidic monomers capable of creating a double bonding mechanism between the dentin and the smear layer by the simultaneous demineralization and infiltration of the monomers,

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ensuring the bonding to the dental surface, as occurs with self-etching adhesives.^{4,5}

Since self-adhesive resin cements are in close contact with the dentin, that is, without the presence of an adhesive layer, surface treatment of the tooth substrate becomes an important step in cementing the fiber post to the root canal.³ The current literature suggests not only the care with the dentin surface treatment to avoid adhesive fractures, but also treatments that can improve bond strength.⁶

In crown dentin, ethylenediaminetetraacetic acid (EDTA) used as pre-etching agent has an inhibitory effect on dentin matrix metalloproteinases (MMPs),^{7,8} producing more stable adhesive interfaces in the healthy dentin⁹ and sclerotic dentin over time.¹⁰ Due to the chelating ability of EDTA, the increase in dentin permeability may favor the self-etching adhesion strategy.¹¹

However, there are few studies in the literature evaluating the benefits of EDTA, and the ones available present conflicting results regarding the use of EDTA prior to the application of self-adhesive cements in the radicular dentin.^{12,13,14} While there are authors who have concluded that EDTA application does not affect the adhesive properties of self-adhesive cements,¹² other authors have reported improved bonding.¹⁴

Because EDTA is a chelating and endodontic irrigating solution, attention is needed to its interaction with different adhesive systems and radicular dentin.¹⁵ The irrigation of the dentin substrate can directly interfere in the bond strength of fiber posts to the root canal¹⁶ and the choice of a correct protocol is crucial to maximize the bond strength between resin/dentin and resin/post.

Therefore, the objective of this study was to evaluate the influence of different EDTA concentrations on radicular dentin etching before bonding glass fiber posts with self-adhesive resin cements. The null hypothesis tested was that different concentrations of EDTA would not affect the bond strength of different self-adhesive resin cements.

Methodology

For this study, 78 extracted human single-rooted premolar teeth were selected after approval by the Research Ethics Committee of the School of Dentistry,

State University of Ponta Grossa (process number 1.795.649). The selected teeth should not present caries, root cracks, previous endodontic treatment, or incomplete root apex. The teeth should have a 14-mm root length measured from the cemento-enamel junction (CEJ).

Post space preparation

Dental crowns were removed at the CEJ using a low-speed diamond saw (Isomet 1000, Buehler; Lake Bluff, USA), and root spaces were endodontically shaped with nickel-titanium rotary files SX to F3 (Dentsply Maillefer; Ballaigues, Switzerland) using a low-speed handpiece (X-smart; Dentsply Maillefer; Ballaigues, Switzerland). Between files, the root canals were irrigated with 2% chlorhexidine solution (Biodinamica; Rio de Janeiro, RJ, Brazil). Only the apical 4 mm was filled with gutta-percha points (Tanari; Manacapuru, Brazil) and calcium hydroxide-based root canal sealer (Sealer 26, Dentsply Maillefer; Ballaigues, Switzerland).

Roots were stored in eppendorf tubes with a gauze pad soaked in distilled water at 37°C ± 1°C for one week. Thereafter, the root canals were prepared with the corresponding bur for shaping the Whitepost DC #2 fiber post (FGM; Joinville, Brazil); irrigated abundantly with distilled water and subsequently dried for 5 s with an air jet and two paper points #40 (Dentsply Maillefer; Ballaigues, Switzerland). The working length of the post space was 10 mm for all teeth.

Experimental groups and sample preparation

The roots were then divided into six groups (n = 13) by block randomization on a freely available website (www.sealedenvelope.com) according to the combination of the main factors: 1) Surface dentin treatment (distilled water [control], 17% EDTA [Biodinamica; Rio de Janeiro, Brazil] and 24% EDTA [Biodinamica]), and 2) self-adhesive resin cement (RelyX™ U200 RX, 3M ESPE, St. Paul, USA) and Multilink® Speed (ML, Ivoclar Vivadent; Barueri, Brazil). Ten teeth were used for bond strength evaluation and three were used for the microhardness analysis.

Root canals were irrigated with 2 mL of each irrigation solution agitated with a rigid microbrush (KG Sorensen, Cotia, Brazil) inside the root canal for 30 s and then washed with 2 mL of distilled water.

The root canals were dried with compressed air for 5 s at 2 cm and dried with two #40 paper points.

Glass fiber posts were horizontally sectioned in the coronal region with a water-cooled diamond cutting instrument to reduce post length to 13 mm (10 mm was cemented inside the root canal and 3 mm served as a guide for the light-curing protocol). After that, the posts were cleaned in 70% alcohol for 5 s.

The two self-adhesive cements were handled and inserted into the root canal using a Centryx syringe (Maquira, Maringá, Brazil), following the manufacturer's recommendations. Glass fiber posts were then placed with finger pressure (equivalent to 35 ± 5 g, when performed on a surface of an analytical balance); excess of resin cement was removed and light-cured under continuous mode for 40 s using a light-curing unit with a light intensity of 1200 mW/cm^2 (Radii Plus, SDI; Victoria, Australia). The posts were light-cured on the cervical surface at a standardized distance of 3 mm. All roots were stored in Eppendorf tubes with a gauze pad soaked in distilled water, at $37 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ for one week.

Six 1-mm-thick slices were obtained by horizontally cutting the roots with a low-speed diamond saw (Isomet 1000, Buehler; Lake Bluff, USA) to create two cervical, two middle, and two apical slices from each root.

Push-out bond strength test

All slices (10 roots per group) were photographed on both sides with an optical microscope at 40X magnification (Olympus BX 51 model, Olympus; Tokyo, Japan), and their individual bonding areas were calculated using the Image J software (University of Wisconsin; Wisconsin, USA). The area was calculated as the lateral surface of a truncated cone using the following formula:¹⁷

$A = \pi (R + r) [h^2 + (R - r)^2]^{0.5}$, where $\pi = 3.14$, R = coronal post radius, r = apical post radius, and h = root slice thickness.

Each slice was positioned with the cervical side down and subjected to constant compressive force (50 kg load cell at a crosshead speed of 0.5 mm/min) on a universal testing machine (Instron; Canton, USA). For this purpose, cylindrical tips with diameters compatible with the post at each third were used until debonding. The load for post dislodgement was

recorded in Newtons (N) and converted to MPa based on the bonding area (mm^2). After debonding, the failure modes were classified under a stereomicroscope (40X) as: adhesive (between resin cement and root dentin), cohesive (post, cement, or dentin), or mixed.

Microhardness test

The first slice of each third (cervical, middle, and apical) of the remaining three roots per group was used to evaluate the microhardness of the resin cement close to the root dentin. These sections were embedded in an acrylic resin with the cervical side facing up and polished down under wet conditions using 600-, 1200-, 1500-, 2000-, 2500- and 3000-grit silicon carbide papers (3M ESPE; St Paul, USA) for 30 s each. After storage for 24 h at 37°C , the samples were subjected to a microhardness test using a Vicker microhardness tester (Shimadzu HMV2, Newage Testing Instruments Inc., Southampton, USA) with a load of 100 g for 15 s. Four indentations per slice were performed as close to the dentin as possible in a clockwise direction (at 3, 6, 9, and 12 h). The length of the diagonal of the indentation was measured and Vickers values were calculated using the formula: $\text{VHN} = 1.8544 F/d^2$, where 1.8544 is a constant; F represents the force used in the test in kgf (0.1 kgf), and d represents the average of the diagonals of the indentation (mm).

Statistical analysis

The data on push-out bond strength and microhardness for each self-adhesive resin cement were subjected to two-way ANOVA (surface treatment *vs.* root region) and a post-hoc Tukey's test ($\alpha = 0.05$). All calculations were performed using Sigma Plot 11 statistical software (Systat Software, San Jose, USA). Data on the fracture pattern were only assessed qualitatively.

Results

Push-out bond strength

For RelyX U200, the two-way ANOVA demonstrated that the cross-product interaction (surface treatment *vs.* root region) was not significant ($p = 0.702$), and neither was root region ($p = 0.473$) (data not shown). Surface treatment was statistically significant (Table 1; $p < 0.001$). The lowest bond strength value was observed

in the 24% EDTA group, whereas the control and 17% EDTA groups were similar to one another.

For Multilink Speed, the two-way ANOVA demonstrated that only the cross-product interaction (surface treatment *vs.* root region) was statistically significant (Table 2; $p = 0.001$).

Failure pattern

The mixed failure mode was predominant in the control groups for both resin cements. For the groups with EDTA application, the adhesive failures between resin cement and root dentin were the most frequent, and more prevalent in the 24% EDTA groups. The

only cohesive failure occurred within the post, but at a lower rate (Figure).

Microhardness test

For RelyX U200, the two-way ANOVA demonstrated that the cross-product interaction (surface treatment *vs.* root region) was statistically significant (Table 3; $p = 0.001$). The highest microhardness value was observed in the cervical region for the control group and the lowest value in the cervical region for the 24% EDTA group.

For Multilink Speed, the two-way ANOVA demonstrated that the cross-product interaction (surface treatment *vs.* root region) was not significant (Table 4; $p = 0.683$), and neither was root region ($p = 0.234$). Only surface treatment was statistically significant ($p < 0.05$). The highest microhardness value was observed in the control group and the lowest value was observed in the 24% EDTA group, whereas the 17% EDTA group showed intermediate values.

Table 1. Mean values and standard deviation of bond strength (MPa) for self-adhesive resin cement RelyX™ U200.

Control	Surface treatment	
	17% EDTA	24% EDTA
14.5 ± 3.2 A	13.7 ± 3.8 A	7.8 ± 2.6 B

* Different letters indicate statistically significant differences ($p < 0.05$).

Table 2. Mean values and standard deviation of bond strength (MPa) for self-adhesive resin cement Multilink® Speed.

Root region	Surface treatment		
	Control	17% EDTA	24% EDTA
Cervical	8.7 ± 1.8 c	8.4 ± 2.6 c	5.8 ± 1.7 d
Middle	13.3 ± 1.7 b	7.1 ± 2.1 c	7.5 ± 2.1 c
Apical	16.8 ± 3.1 a	10.6 ± 2.6 b	7.7 ± 3.5 c

* Different letters indicate statistically significant differences ($p < 0.05$).

Table 3. Mean values and standard deviation of microhardness (Vickers) for self-adhesive resin cement RelyX™ U200.

Root region	Surface treatment		
	Control	17% EDTA	24% EDTA
Cervical	94.4 ± 6.4 a	70.6 ± 10.8 b	55.3 ± 5.8 d
Middle	73.8 ± 5.7 b	76.6 ± 9.6 b	58.8 ± 4.1 d
Apical	70.4 ± 4.7 b	70.0 ± 9.2 c	58.5 ± 2.0 d

* Different letters indicate statistically significant differences ($p < 0.05$).

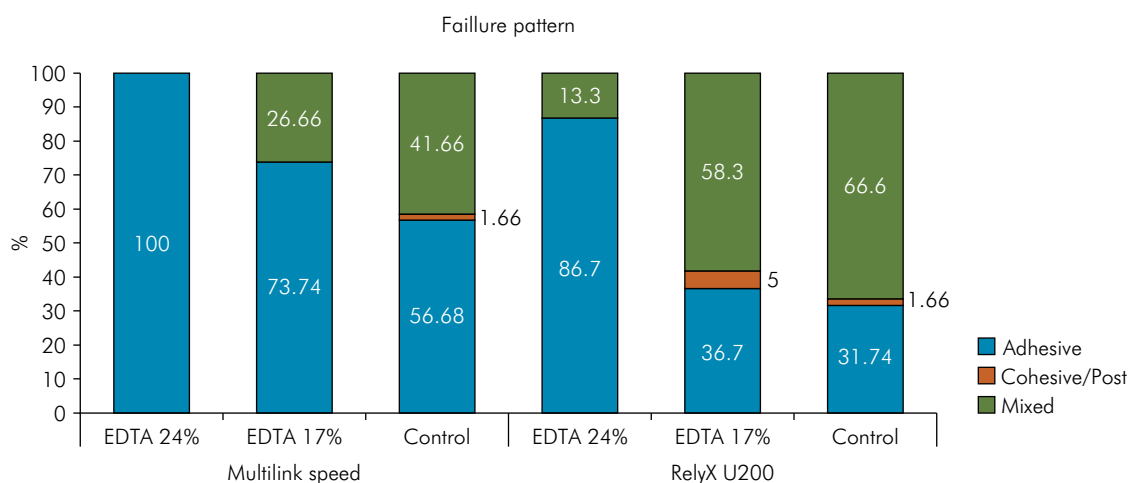


Figure. Representative graph of the fracture patterns observed after bond strength testing for the different experimental groups.

Table 4. Mean values and standard deviation of microhardness (Vickers) for self-adhesive resin cement Multilink® Speed.

Control	17% EDTA	24% EDTA
57.4 ± 7.1 A	54.4 ± 5.4 AB	51.8 ± 3.3 B

* Different letters indicate statistically significant differences ($p < 0.05$).

Discussion

The results of this study indicate that the tested EDTA concentrations interfere in the push-out bond strength and microhardness; thus, the null hypothesis was rejected. This whole study was performed by calibrated and experienced professionals; however, the results obtained for the push-out test do not match most of those described in the literature. The use of 24% EDTA compared to other lower concentrations of this chelating agent promotes a cleaner surface and significantly increases the contact area of the dentin substrate.¹⁸ However, the expectation of greater results was not fulfilled, since the use of these solutions did not positively interfere with the tests in this study.

For the RX group, 24% EDTA irrigation showed the lowest BS results. The literature has shown that 24% EDTA applied to root dentin was significantly more effective than all lower concentrations of EDTA with regard to smear removal capacity.¹⁹ The excessive removal of the smear layer by calcium-ion chelation theoretically is likely to reduce the chemical bonding of the self-adhesive cement to hydroxyapatite.^{20,21}

A relatively recent study has evaluated the bond strength of one of the resin cements assessed in the present study (RelyX U200) with the application of 17% EDTA and satisfactory results for BS.¹⁵ However, in that study, the EDTA solution was applied and subsequently activated by an ultrasonic tip, which may explain the conflicting results with the present research. Sonic vibration transmits energy to the fluids, allowing them to reach deeper areas than would manual application.^{22,23}

BS values were conflicting for ML cement. In most studies on the bond strength of fiber posts, the cervical portion has the highest BS values, almost unanimously.^{24,25,26} However; in this study, the middle and apical thirds presented higher values with distilled water. Because self-adhesive

resin cements show an affinity for the composition of composite resins, in addition to containing an unfavorable factor (e.g., C-factor).²⁷ The cervical dentinal tubules are larger than in the middle and apical portions, and root canal diameter is larger as well.²⁸ This may possibly explain the conflicting results with the literature.

Unlike BS values, MH results are in line with those described in the literature. For both self-adhesive resin cement systems, irrigation with 24% EDTA showed lower values. The MH test was carried out as close to the substrate as possible, and any type of negative adhesion can compromise complete polymerization and, consequently, the hardness of the cement. EDTA can partially remove hydroxyapatite and non-collagen proteins selectively, avoiding major changes in the dentin structure.²⁹ Probably, the higher concentration of 24% EDTA excessively removed this matrix, causing collagen fibrils to collapse and compromising the interaction of RelyX U200 and Multilink Speed cements with the radicular dentin.

Theoretically, the higher concentration of EDTA should facilitate the infiltration of resin cements into the dentinal tubules, since the chelating action of EDTA significantly promotes their cleaning.¹⁸ However, the solution excessively removes the smear layer, which is directly linked to the adhesion mechanism of the self-adhesive systems.³⁰ Our study suggests that self-adhesive resin cements not only depend on the chemical bonding to dentinal hydroxyapatite, but also on the modification and incorporation of the smear layer for complete adhesion.

Unlike most studies that evaluated the failure pattern after the push-out test, this study demonstrated the predominance of mixed and non-adhesive fracture patterns.^{31,32} Another recent study on self-adhesive cements corroborated the fracture pattern found in the present study.³³ Mixed fractures are interesting and promising in terms of bond strength, since they represent better interaction of the cement with the dentin, preventing the cement from being removed from the substrate as easily as in adhesive fractures.³⁴

The 24% EDTA solution is sold in more practical packaging (gel tube) than is conventional 17% EDTA (liquid), which may make it more attractive to dentists.

Therefore, companies should be cautious about contraindications to the use of this solution prior to self-adhesive cementation. Further studies are needed to evaluate adhesion over the long term.

Conclusion

Despite the limitations of this study, EDTA is not recommended as pretreatment for fiber post cementation using self-adhesive cements. The use of 24% EDTA affected the bond strength to the root dentin, microhardness of the cement, and the

failure pattern. The use of 17% EDTA influenced microhardness.

The use of 24% EDTA negatively affects the bond strength of fiber posts to the root canal with the use of self-adhesive cements.

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