BJOS
Brazilian Journal of Oral Sciences

Original Article

Volume 22
2023
e236508

The effectiveness of EDTA 17% as a cleaning solution for the fiber post space after filling with cements

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Aim: To evaluate the resistance of the union between a glass fiber post and radicular dentine after cleaning the root with 17% EDTA and filling with different endodontic cements. **Methods:** Forty uniradicular bovine incisors were removed to obtain root lengths of 18 mm. Endodontic treatment was performed on all roots using different filling cements (zinc oxide and eugenol-based, OZE; cement based on epoxy resin, AH) and cleaning solutions (saline, SA or EDTA), which made it possible to obtain four groups: OZE_{SA}, OZE_{EDTA}, AH_{SA} and AH_{EDTA}. Subsequently, 12 mm of filling material was removed from the roots, and they were prepared to receive fiber posts luted with resin cement. To execute the mechanical cycles ($2x10^6$ cycles, 90 N, 4 Hz), coronal reconstruction was performed with a silicon matrix. The roots were then sliced (2-mm thick) to perform the push-out test. The results were analyzed using analysis of variance (one factor and two factors) and Tukey's test (α=0,05). **Results:** Bond strength (Mpa) was significantly higher for OZE_{EDTA} (9,18) and AH_{EDTA} (8,70) than for OZE_{SA} (6,06) AH_{SA} (8,7). OZE_{EDTA} also presented the highest values in the cervical region (15,18) but was significantly lower in the apical region (2,99). However, AH_{EDTA} had a homogeneous bond strength in all thirds. **Conclusion:** Regardless of the endodontic cement used, EDTA was used as an irrigating solution, culminating in a higher bond strength between the glass fiber post and dentin.

Keywords: Tooth, nonvital. Endodontics. Zinc oxide-eugenol cement. Edetic acid.

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Editor: Dr. Altair A. Del Bel Cury

Received: July 7, 2021
Accepted: July 13, 2022

Introduction

Several clinical and in vitro studies have already highlighted the advantages of fiber glass posts over fused metallic cores and pre-fabricated metallic posts[1-3]. When adhesively cemented, these posts may show biomechanical properties similar to the dental structure, an elastic module, which favors the formation of a monoblock structure of the cement with the posts and root canal walls, providing a junction with high retention, equal stress distribution, low microleakage, and high root fracture resistance[2,4-6]. Moreover, they allow for a more conservative treatment of the dental structure, do not undergo corrosion, and present satisfactory esthetics[7].

However, this adhesion between glass fiber posts and root dentin may be compromised by the presence of remnants of the filling material, which is not totally removed during clearance[8-9]. One of the most common filling material is the zinc oxide and eugenol-based cements (OZE)[10]; however, the presence of eugenol in its composition prevents a complete polymerization of the resin cement, reducing the bond strength with the post[10-12]. Therefore, cements with other compositions, such as calcium hydroxide, tungsten hydroxide, or AH (AH), have been developed. Among them, AH Plus (AH; Dentsply Maillefer, Ballaigues, Switzerland) is one of the best endodontic sealers to fill the root canal before fiber post cementation[4,13]. This epoxy resin-based sealer has physicochemical properties that reduce viscosity and improve flow, adhesion, and capacity for filling empty space without compromising adhesion of the post[4].

After the removal of the gutta-percha and cement from the canal, a variety of solutions can be used in the chemical cleaning of the space before post luting. Sodium hypochlorite (NaOCl) and ethylenediaminetetraacetic acid (EDTA) are commonly used to remove dentinary remnants and smear layers from dentinal walls[14,15], which may increase the penetration of the adhesive in dentin and, consequently, increase the bond strength of the resin cement[16-18]. However, the application of EDTA for an exacerbated time can result in a dental demineralizing effect, widening the dentinal tubules, softening dentin, and denaturing the collagen fibers[14,19]. Nonetheless, while much is known about the influence of cleaning the post and the mode of cement application to optimize bond strength[9], little is known about the cleaning protocols prior to resinous cementation of the post. Therefore, it is important to investigate protocols that seek to effectively remove the remaining filling materials from the canals to ensure adequate retention of the glass fiber post to the dental root[20-22].

Therefore, the objective of this study was to evaluate the influence of 17% EDTA as a cleaning solution on the bond strength between a glass fiber post and a root filled with different endodontic cements subjected to mechanical aging. The null hypothesis was that the type of sealer (1) and use of 17% EDTA (2) did not influence the bond strength.

Methods and Materials

Tooth preparation

Forty uniradicular bovine incisors were randomly selected. The teeth were decoronated to their longitudinal axis using a slow-speed, water-cooled diamond disc; thus, the root length was standardized at 18 ± 0.2 mm. The canal diameter was measured with a digital caliper and must have equal or smaller dimensions to the post used in the study (ø= 2.0 mm, White Post DC #2, FGM, Joinvile, SC, Brazil).

A single operator, specialist in endodontics and who experienced all of the techniques used in this study, performed the root canal preparation, obturation, and post cementation procedures. The bovine roots were endodontically treated using Gates Glidden drills and hand files (Dentsply/Maillefer, Petrópolis, Brazil). A 2.5% NaOCl solution was used to irrigate the canals (10 mL in total), followed by drying using absorbent paper points (Dentsply Sirona, Rio de Janeiro, Brazil).

Randomization of the teeth in the different groups was performed using the "Random Allocator" software. The OZE (Endofill, Dentsply Maillefer, Petrópolis, RJ, Brazil) (n=20) and AH Plus (Dentsply/Maillefer, Petrópolis, Brazil) (n =20) sealers used to fill the root canals were prepared according to the manufacturers' recommendations, and the gutta-percha cone (Dentsply/Maillefer, Petrópolis, Brazil) was covered with a layer of sealer and inserted into the canal (with gentle brushing movements against the root canal walls). Subsequently, the gutta-percha cone was slowly positioned. Twenty teeth were filled with gutta-percha cones (Dentsply/Maillefer; Petrópolis, Brazil). The lateral condensation technique was used for thermofilling, with the foramen considered as the apical obturation limit. The entrance of the canal was closed with a Ketac™ Cem (3M, ESPE, St Paul, MN, USA) conventional restorative glass ionomer to avoid contamination. The roots were radiographed to evaluate the quality of the endodontic treatment and stored in distilled water at 37 °C for 7 days.

Cleaning protocols and fiber post luting

The teeth were mounted individually in plastic cylinders (Tigre, Rio Claro, SP, Brazil), and the roots were embedded in resin up to 3 mm below the coronary portion. The method used to reproduce the periodontal ligament was embedded in the root with a polyether impression material (Impregum F, 3M-Espe, Seefeld, Germany). The periodontal ligament was simulated by covering the root, including up to 3 mm of the coronary portion of the specimens with an elastomer (Impregum Soft, 3M-Espe, Seefeld, Germany), with a thickness of approximately 0.3 mm. They were then included in pipes with chemically activated acrylic resin (Dencrilay, Dencril, Caieiras, SP, Brazil), according to the methodology described by Junqueira et al.[23].

The filling material was initially removed from the root with heated Paiva condensers (Golgran, São Paulo, Brazil) supplemented with a drill corresponding to the White Post DC #2 glass fiber post (FGM, Joinville, SC, Brazil) in the first 12 mm. A total of 6 mm of the filling material remained in the apical third. After the preparation, the roots were irrigated according to their respective groups: saline solution (OZE_{SA} and AH_{SA}) or 17% EDTA (OZE_{EDTA} and AH_{EDTA}). For both groups, the dentinal walls were cleaned with 10 mL of the specific solution using a syringe and an irrigation needle (Ultradent Products, Indaiatuba, SP, Brazil) for 1 min and irrigated with 5 mL distilled water. The canal was then aspirated with microcannulas (Capillary Tips, Ultradent, São Paulo, Brazil) and dried using # 80 absorbent paper points (Dentsply Sirona, Rio de Janeiro, Brazil).

All glass fiber posts (White Post DC #2, FGM Produtos Odontológicos, Joinvile, SC, Brazil) were treated by conditioning with phosphoric acid 37% (15 s), washing with water (60 s), and air drying (15 s). Then, a silane agent (Silano, Ângelus Brazil) was applied using a brush, covering its entire surface (60 s), and laid on a glass plate for a period of 4 min and then air-dried (15 s).

Before adhesive cementation, the fiber posts were disinfected with alcohol 70° GL and then a silane layer (Prosil FGM, Joinville SC, Brazil) was applied on the post's surface using a micro-brush for 1 min and then dried by blowing compressed air for 20 s. After post preparation, the post space was etched with 37% phosphoric acid (FGM; Joinivile, SC, Brazil) for 15 s, rinsed with water for 60 s, and dried with #80 absorbent paper points. Subsequently, an adhesive (Ambar, FGM, Joinivile, SC, Brazil) was applied to the radicular canal using a microbrush (Cavibrush Longo, FGM) for 10 s, and the excess was removed with absorbent paper tips. Finally, the adhesive resin cement system (Allcem, FGM, Joinivile, SC, Brazil) was manipulated and placed in the post space with a Lentulo #40 tip. The endodontic post was also covered with a sealer and seated to full depth using finger pressure for 60 s. Light curing was performed for 40 s using halogen light at 1200 mW/cm^2 (Radii Cal; SDI, Melbourne, Australia).

For coronary reconstruction, the cervical dentin was etched for 15 s with 37% phosphoric acid (FGM; Joinivile, SC, Brazil), rinsed with water for 60 s, and dried with absorbent paper. Subsequently, a layer of dentin adhesive (Ambar, FGM) was applied to the dentin using a microbrush (Cavibrush, FGM, Joinivile, SC, Brazil) with a light air jet used to evaporate the solvent and homogenize the thickness of the adhesive. The cure was performed for 30 s (Radii Cal; SDI, Melbourne, Australia). Then, a standardized plastic matrix, obtained from a human central incisor with a 10 mm crown, was filled with micro-hybrid composite resin (Opallis, FGM, Joinville, Santa Catarina, Brazil), positioned on the post coronary surface, and light-curing was performed for 20 s on each surface of the tooth (Bergoli et al. 2014). The teeth were immersed in distilled water and placed at 100% relative humidity and 37 °C for 24 h.

Mechanical cycling

The teeth were placed at 45º in a mechanical cycling machine (ER 11000; Erios, São Paulo, Brazil). A 90 N load was applied with a piston (ø = 1.6 mm), 2 mm below the incisal edge on the palatal face of the specimen, for 1.2 x 10^6 cycles, at a frequency of 4 Hz in a humid environment. The piston (ø = 1.6 mm) was loaded 2 mm below the incisal border on the palatal face of each tooth.

Removal by extrusion (push-out)

Each root post space was cut into four 1.5-mm-thick slices using a cutting machine (Isomet 100 Precision Saw, Buehler Ltd., Lake Bluff, IL, USA) at a speed of 325 rpm and weight of 75 g under constant water irrigation. Two 1.0 ± 0.1-mm-thick slices were obtained from each third of the post. The cervical side of each test specimen was placed in contact with a support coupled to the base of a universal test machine (EMIC 2000, São José dos Pinhais, Paraná, Brazil). Loading was performed at a crosshead speed of 0.5 mm/min on the surface of the post, without reaching the cement and/or dentin, using a cylindrical tip with a 0.8-mm diameter until the post was completely dislodged from the root slice. Data were recorded in Newtons (N). The bond strength (BS) (Mpa) was calculated using the formula BS=N/π(R + r)√h2 + (R − r), where "π" is the constant 3.14, "R" is the measure of the radius of the resin cement/post junction in its coronal portion, "r" is the measure of the radius of the resin cement/post junction in its apical portion, and "h" is the height/thickness of the slice.

The obtained values were subjected to descriptive (mean and standard deviation) and inferential statistical analysis, using parametric ANOVA (one factor and two factors) and Tukey test (α = 0.05).

Failure analysis

The failure mode was determined using a stereomicroscope at 50x magnification (Vanox, Olympus, Tokyo, Japan) and classified as (1) adhesive between resinous cement and root dentin; (2) adhesive between the post and resinous cement; (3) mixed between post, resinous cement, and root dentin; (4) cohesive in dentin; (5) cohesive in post; and (6) cohesive in cement.

Results

When calculating the total bond strength, there was no statistical difference between the OZE_{SA} and AH_{SA} groups, demonstrating no influence of the sealing material. However, these values were significantly lower than those of OZE_{EDTA} and AH_{EDTA}, which demonstrates the influence of the irrigation solution on these results (Table 1).

Table 1. Mean and standard deviations (MPa) of the total teeth bond strength according to the filling cement and irrigation solution used for cleaning the post space. Lower case letters indicate differences pointed by the ANOVA 1-factor and Tukey test.

Groups	Average (±DP)	P Value				
		Groups	OZE_{SA}	OZE_{EDTA}	AH_{SA}	AH_{EDTA}
OZE_{SA}	6,06 (±0,80)[a]	OZE_{SA}	-	0,006	0,817	0,022
OZE_{EDTA}	9,18 (±0,76)[b]	OZE_{EDTA}	-	-	0,010	0,666
AH_{SA}	6,32 (±0,78)[a]	AH_{SA}	-	-	-	0,037
AH_{EDTA}	8,70 (±0,81)[b]	AH_{EDTA}	-	-	-	-

Table 2 compares the bond strengths of the root third of each sample. ZS and RS also did not show statistically significant differences between the thirds. In the ZE groups, it was observed that the cervical third presented union values that were significantly higher than those of the others.

Table 2. Mean and standard deviations (MPa) of bond strength of each root third. Upper case letters indicate difference on the same line. Lower case letters indicate difference in the same column pointed by ANOVA 2-factors and Tukey test (p<0,05).

Groups	Root thirds		
	Cervical	Middle	Apical
OZE_{SA}	7,60 (±1,40)^{Aa}	5,52 (±1,31)^{Aa}	5,06 (±1,46)^{Aab}
OZE_{EDTA}	15,18 (±1,31)^{Ab}	9,36 (±1,27)^{Bb}	2,99 (±1,35)^{Ca}
AH_{SA}	7,58 (±1,31)^{Aa}	5,63 (±1,52)^{Aab}	5,76 (±1,24)^{Aab}
AH_{EDTA}	10,91 (±1,35)^{Aa}	7,27 (±1,58)^{Aab}	7,92 (±1,24)^{Ab}

The frequencies of the failure modes are listed in Table 3. There was a predominance of adhesive between the resinous cement and root dentin, and mixed between the post, resinous cement, and root dentin.

Table 3. Frequency of failure mode between groups (%).

Groups	Failure mode					
	1	2	3	4	5	6
OZE_{SA}	60%	6.6%	18.4%	1.7%	13.3%	-
OZE_{EDTA}	63.3%	5.1%	16.7%	3.3%	11.6%	-
AH_{SA}	61.7%	6.6%	16.7%	1.7%	13.3%	-
AH_{EDTA}	63.3%	8.3%	13.5%	3.3%	11.6%	-

(1) adhesive between resinous cement and root dentin; (2) adhesive between the post and resinous cement; (3) mixed between post, resinous cement and root dentin; (4) cohesive in dentin; (5) cohesive in post and (6) cohesive in cement.

Discussion

Based on the results of this study, it was possible to answer our questions. The first null hypothesis, that the type of sealer would not influence the results of bond strength, was rejected because although the results of the totality of the dental root showed no differences between the OZE and AH groups, the results of the third cervical and apical regions showed differences when EDTA was used to irrigate the canals.

When analyzing the thirds, another interesting fact to note is that only the OZE_{EDTA} group showed a difference in bond strength between all its thirds, with the apical one showing statistically lower bond strength results between the post and dentin, which is also commonly observed in other studies[4,9], because the dentin tissue morphology in the cervical region shows a greater number of dentinal tubules and wider diameters than the others[4]. Thus, the cervical third is the most adequate for the adhesion of different types of adhesive sealers[4]. However, this is not always the case[15]. The AH_{EDTA} group, for example, presented homogeneity between its thirds, a fact that was also observed in previous studies[15,24]. This group also presented the highest bond strength in the apical third, which was statistically superior to the OZE_{EDTA} group. Normally, most studies show a better bond strength of the posts when the AH sealer is used at the expense of OZE[4,13,25], because zinc oxide eugenol-based sealer penetrates into the dentinal tubules[26], inhibits the polymerization of resin cement, significantly reducing bond strength, and is avoided in these cases[4,9,13,26-28]. Conversely, the epoxy resin, considered the gold standard[13], does not interfere with the free radicals that initiate the polymerization of the composite resin. The high BS between resin cement and endodontic epoxy resin sealer is due to the affinity between their components[4]. Thus, remnants of the epoxy resin sealer on the dentinal walls in prosthetic preparation may improve the adhesion of resin cement[4].

However, it is interesting to observe that in the other thirds, OZE_{EDTA} had the highest bond strength values in the cervical and middle thirds, but this result was statistically similar to that of AH_{EDTA}. The cervical OZE_{EDTA} results may be associated with easy access and application of adhesive materials and greater efficiency of the cement cure in this region, which is next to the light source[10,27]. Another fact that justified the higher values of bond strength found in cervical OZE_{EDTA} may be due to the short time between root canal filling and post placement[10,13]. Greater penetration of eugenol into the interior of the dentinal tubules may occur if the post luting was made after some days, which probably would cause a drop in the bond strength results[10,13,26]. Therefore, it is suggested that an OZE-based sealer would be better in the cervical third only; however, as it is not possible to use different sealers for each third of the root, an AH sealer may be more preferrable.

The second null hypothesis was also rejected because regardless of the analysis of the entire dental root or each third, the groups with irrigation performed with EDTA, except OZE_{EDTA} in the apical third, obtained better results. EDTA is also the most commonly used chelating solution to remove this smear layer created by post space preparation so that a hybrid layer is achieved to increase the retention when resin cement is used[14,15]. It has a calcium ion chelating capacity and is highly efficient in removing this smear layer[15] because EDTA reacts with calcium ions in the dentin and forms soluble calcium chelates[14]. The results found highlight the importance of cleaning the fiber post space in the cementation protocol.

The push-out test is a valid method to measure the adhesion of fiber posts to the root canal walls. It is considered more reliable than the conventional shear test and the micro-tensile test for evaluation of the bond strength of posts and allows the measurement of such values among different root regions[1]. It can be observed, when using this test, that there was a predominance of adhesive failure between resinous cement and root dentin, which represents the interface of interest to the study and can also be related to other studies[4,13,15].

Some limitations can be attributed to this study; however, the use of the bovine tooth is not among them, because previous studies have shown that the type of tooth (bovine or human) does not influence the bond between the post and radicular dentin[9,29]. However, although this methodology has proposed a mechanical fatigue test, it would be interesting to obtain clinical outcomes of the longevity of these treatments, which should be conducted in the future.

Given this, it may be suggested that the use of AH and subsequent use of EDTA for irrigation of dentinal tubules may be the most suitable protocol for the treatment of root canals prior to the adhesive cementation of the fiberglass posts; even in the cervical third, the use of OZE + EDTA has obtained better results, and the maintenance and predictability of post adhesion in the other thirds proved to be safer when using AH.

In conclusion, EDTA (17%) was an effective cleaning solution for the post space. Regardless of the endodontic cement, its use led to an increase in the bond strength between the glass fiber post and the dentinary structure.

Conflicts of Interest

The authors deny any conflicts of interest.

Author Contribuitions

Luciana Arruda Mendes de Paula: acquisition of data, drafting the article.

Lohara Campos de Abreu Reis: acquisition of data, drafting the article.

Jean Soares Miranda: acquisition of data, drafting the article.

Francielle Silvestre Verner: formal analyses, revising it critically for important intellectual content.

Rafael Binato Junqueira: conceptualization, revising it critically for important intellectual content, formal analyses.

Rodrigo Furtado de Carvalho: conceptualization, drafting the article, revising it critically for important intellectual content.

All authors actively participated in the discussion of the manuscript's findings, revised, and approved the final version of the manuscript.

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