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Calcium Hydroxide improves epoxy sealer adhesion on root dentin

O hidróxido de cálcio melhora a adesão de selante resinoso à dentina radicular

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ABSTRACT

Objective: The aim of this study is to evaluate the adhesion of an endodontic sealer (AHPlus) in root canals after the use of different protocols of 17% EDTA and the use of intracanal medication (ICM) based on calcium hydroxide in aqueous vehicle. **Material and Methods:** For this, 72 single-rooted human teeth were instrumented up to #50 and divided into six groups (n = 12). Group 1: EDTA for 3 min; Group 2: 3 mL of EDTA + 3 min of EDTA; Group 3: 3 mL of EDTA + 3 min of EDTA + 30 seconds of ultrasonic agitation; Group 4: EDTA for 3 min + ICM; Group 5: 3 mL of EDTA + 3 min of EDTA + ICM; Group 6: 3 mL of EDTA + 3 min of EDTA + 30 seconds of ultrasonic agitation + ICM. The root canals were filled with endodontic sealer after each protocol and after 7 days they were prepared to the push-out test. The data were analyzed using two-way ANOVA ($p<0.05$). **Results:** It was observed no statistically difference in bond strength in the 3 different 17% EDTA protocols. However, the use of ICM increased significantly the resistance adhesion. **Conclusion:** Intracanal medication based on calcium hydroxide improved the bond strength of AHPlus to dentin walls, regardless of the EDTA protocol.

KEYWORDS

Calcium hydroxide; EDTA; Dentin.

RESUMO

Objetivo: O intuito deste estudo é avaliar a adesão de um cimento endodôntico (AH Plus) em canais radiculares após o uso de diferentes protocolos de EDTA 17% e o uso de medicação intracanal (ICM) à base de hidróxido de cálcio em veículo aquoso. **Material e Métodos:** Para isso, 72 dentes humanos unirradiculares foram instrumentadas até # 50 e divididos em seis grupos (n = 12). Grupo 1: EDTA por 3 min; Grupo 2: 3 mL de EDTA + 3 min de EDTA; Grupo 3: 3 ml de EDTA + 3 min de EDTA + 30 segundos de agitação ultra-sônica; Grupo 4: EDTA durante 3 min + ICM; Grupo 5: 3 ml de EDTA + 3 min de EDTA + ICM; Grupo 6: 3 ml de EDTA + 3 min de EDTA + 30 segundos de agitação-sônica + ICM. Os canais radiculares foram preenchidos com cimento endodôntico após cada protocolo e, após 7 dias foram preparados para o teste de push-out. Os dados foram analisados utilizando ANOVA dois fatores ($p < 0,05$). **Resultados:** Não foi observado diferença estatística na resistência de união nos 3 diferentes protocolos de EDTA a 17%. No entanto, o uso de ICM aumentou significativamente a resistência de adesão. **Conclusão:** a medicação intracanal à base de hidróxido de cálcio melhorou a resistência de união do AH Plus às paredes dentinárias, independentemente do protocolo de EDTA.

PALAVRAS-CHAVE

Hidróxido de cálcio; EDTA; Dentina.

INTRODUCTION

Complete removal of the pulp and aggressive agents through the mechanical, physicochemical and medications are extremely important to achieve endodontic success [1]. However, root canal preparation contributes to the formation of smear layer, reducing dentin permeability, which can interfere in penetration of antimicrobial agents and adhesion of sealers, compromising the quality of the sealing of the canal [2].

EDTA 17% has been widely used in endodontics by increasing dentin permeability [3]. Dentinal tubules are opened and demineralization occurs at intertubular and peritubular dentin, occurring exposition of a thin layer of collagen fibers, allowing the penetration of the sealer, promoting micromechanical bond between material and dentin [4].

Calcium hydroxide has been used as a intracanal medication for its broad antimicrobial potential. It has slow antiseptic action with ability to hydrolyze the lipid portion of the lipopolysaccharide [5] and also calcium hydroxide promotes denaturation of proinflammatory cytokines mediators [6]. It is known that the removal of the medication must precede the filling of the canal. For this, copious irrigation with sodium hypochlorite and final irrigation with 17% EDTA are indicated [7]. However, no technique has been very efficient in the removal of material, leaving approximately 45% of the surface covered by calcium hydroxide [8,9]. Some studies have shown that the presence of residues of calcium hydroxide in the canals and dentinal tubules may affect the penetration of sealers within them and hinder the desired sealing in the filling [10,11]. Curved canals, type of irrigant and depth of penetration of the irrigant are factors that can affect the removal of calcium hydroxide. The canal instrumentation with the use of ultrasound has been shown as an effective modality for the cleaning of pulp remnants, remnants of dentinal canals and isthmuses. The mechanical stirring and ultrasonic instrumentation from the use of lime or rotating together with the irrigation can enhance the removal of calcium hydroxide [8]. This study hypothesizes that bond strength of Epoxy sealer AHPlus, can be changed by varying EDTA protocols and with using intracanal calcium hydroxide medication in aqueous vehicle.

MATERIAL AND METHODS

Specimen Preparation

This study was conducted under the approval of our institutional ethics in research board (#409042). An in vitro study was performed using seventy-two extracted human single-rooted teeth. The crown portion of each tooth was removed by means of a water-cooled precision saw (LabCut 1010, Extec - Erios, São Paulo, Brazil). Working length of each tooth was determined by a K-file #10, until it reached the apical foramen, subtracting 1mm from this measurement. The teeth were endodontically treated according to a crown down rotatory system Reciproc® #25 up to #50 (VDW GmbH, Munich). For cleaning and shaping procedures were used 5ml of 2.5% sodium hypochlorite (Asfer Indústria Química Ltda, São Caetano do Sul, SP, Brazil). After the cleaning and shaping processes, the 72 teeth were randomly divided into six groups of 12 specimens each, according to three 17% Ethylenediaminetetraacetic acid (EDTA) (Asfer Indústria Química Ltda, São Caetano do Sul, SP, Brazil) protocols and intracanal medication with calcium hydroxide (UltraCal® XS, Ultradent, Salt Lake - USA).

In group 1, root canal was filling with 17% EDTA for 3 minutes.

In group 2, irrigation was performed with 3 mL of EDTA and root canal filling with EDTA for 3 minutes.

In group 3, irrigation was performed with 3 mL of EDTA and root canal filling with

EDTA with 30 seconds of ultrasonic agitation (CVDent1000 CVDentus, SP, Brazil).

After the use of EDTA, the root canals were rinsed with sterile saline and dried with the aid of Capillary Tips (Ultradent Brazil, SP, Brazil) and paper points (Dentsply Maillefer, Ballaigues, Switzerland). Root canal filling was performed with cement-based epoxy resin (AHPlus, De Trey Dentsply, Konstanz, Germany) handled according to manufacturer's instructions, using spiral lentulo #4.

In group 4, root canal was filling with 17% EDTA for 3 minutes; final irrigation was accomplished with sterile saline; and root canals were filled with UltraCal.

In group 5, irrigation was performed with 3 mL of EDTA and root canal filling with EDTA for 3 minutes; final irrigation was accomplished with sterile saline; and root canals were filled with UltraCal.

In group 6, irrigation was performed with 3 mL of EDTA and root canal filling with EDTA with 30 seconds of ultrasonic agitation; final irrigation was accomplished with sterile saline; and root canals were filled with UltraCal.

After 15 days, the groups that received intracanal medication, had the UltraCal removed using 5 mL of 2.5% sodium hypochlorite and K-file #50, followed by the respective EDTA protocol; and root canal was filling as described above.

Specimens were horizontally sectioned into approximately 2-mm-thick slices from the cement-enamel junction with a water-cooled precision saw after 7 days of the sealer cured. Three middle third slice per tooth was collected for push-out test and scanning electron microscopy (SEM) evaluation.

Push-out test

Push-out test was performed as described previously by Sly et al. [12]. Each slice's apical side was identified, and the perimeter and diameter of each obturation site were measured by a digital caliper. Each specimen was attached to a support jig with clearance for the dislodged material on the base of the universal testing machine (EMIC - DI -1000, Curitiba, Brazil) with the coronal end facing the support jig and the apical end facing the load cell (50kgf and 1mm of diameter) for the punch affixed to the crosshead to contact the part of the specimen. The punch moved downward at a crosshead speed of 1mm per minute until extrusion of the obturation from the specimen was performed, which generally resulted in an abrupt decrease in the applied load. The computer and software connected to the universal testing machine calculated the push-out bond strength value for each specimen from the average of the two perimeters (coronal and apical). The thickness of the specimen was calculated from the formula: Debond stress (MPa) = Debonding force (N) / Area (mm2), where debonding force is the maximum force before debonding and area (of the bonded interface) is the average value of the perimeter times the thickness. The values were subjected to parametric statistical analysis (ANOVA) with two factors, with a significance level of 0.05.

Scanning electron microscopy (SEM) was used to illustrate fractures between dentin and sealer.

RESULTS

Test results of bond strength in MPa are shown in Figure 1 and Table 1. Presence of intracanal calcium hydroxide positively influenced the bond strength of sealer to dentin walls ($p < 0.05$). However, no significant difference was observed when the comparison of different EDTA protocols was performed ($p > 0.05$).

ANOVA two factors (ANOVA 2) was used to test hypothesis of equality in the groups according to bond strength values obtained among different EDTA protocols and presence or absence of calcium hydroxide. It was

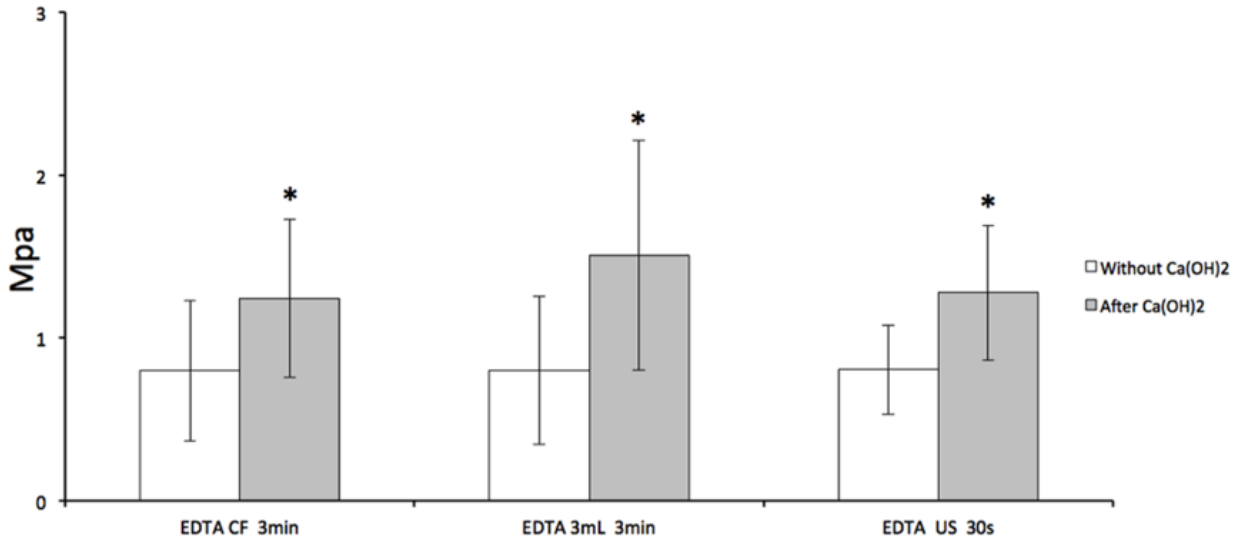

Figure 1 - Bond strength (media ± sd) in MPa of the 6 groups and 3 EDTA protocols (CF - canal filled, US - Ultrasound) n=12.

Table 1 - Descriptive statistics of push-out bond strenght for each group.

		3min EDTA			3mL + 3min EDTA		3mL EDTA + 30s agitation	
ICM		Mean	SD		Mean	SD	Mean	SD
Without	Group1	0.08	0.43	Group 2	0.08	0.45	0.08	0.27
With	Group 4	1.24	0.48	Group 5	1.51	0.70	1.27	0.41

observed that the interaction of these factors was significant (p=0.001).

Figure 2 demonstrates representative SEM images of fractures between dentin and sealer, suggesting that adhesive fractures occurred in the majority of the specimens, while few specimens presented sealer cohesive.

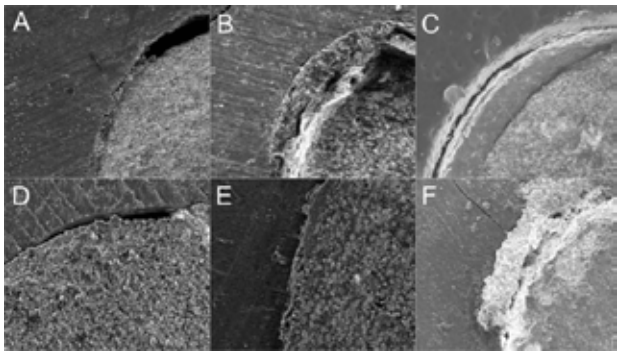

Figure 2 - Photomicrographs of sealer/dentin fractures in the 6 groups. (A) group 1; (B) group 2; (C) group 3; (D) group 4; (E) group 5; (F) group 6. (Original magnification, 200X).

DISCUSSION

Once AHPlus is the gold standard in endodontic obturation [13] and calcium hydroxide in aqueous vehicle is the most used medication worldwide [14], the present study evaluated the influence of calcium hydroxide as an intracanal medication on the bond strength between the sealer AHPlus and root dentin, after the use of different protocols of 17% EDTA to remove the smear layer.

The complete filling of the root canal is important to achieve the success of endodontic treatment. Some authors suggest that calcium hydroxide, used in previous sessions from obturation, can impair the quality of the sealing desired by forming a physical barrier on the tubules reducing dentin permeability [15-17]. Several studies demonstrate great difficulty in fully removing the calcium hydroxide from the root canal, which may contribute to the

presence of flaws in the filling, favoring bacterial recontamination and recolonization [7,9,18]. However, this study found that all root canals that were filled with intracanal medication based on calcium hydroxide had higher bond strength when compared to the groups that do not received intracanal medication. This result suggests that the presence of this material increases the adhesion of epoxy AHPlus to the dentinal walls. Similar results were obtained by Carvalho et al. [18] even using a different methodology.

EDTA acid is commonly used to remove the smear layer, and to remove intracanal medication. It is known that effectiveness of calcium hydroxide removal depends on the vehicle in which it is associated. Studies have shown that aqueous vehicle is removed easily [9,19]. In this study we evaluated three different EDTA protocols to remove smear layer and intracanal medication, ranging the time, volume, and the use of ultrasonic agitation. However, the results showed that these protocols had no influence on the adhesion of the endodontic sealer. Interestingly, the use of calcium hydroxide as intracanal medication significant increased the bond strength. One possible explanation for this positive influence of calcium hydroxide is based on its dimensional stability, given by the low polymerization shrinkage and subsequent volumetric expansion20. Another possible reason for this result is the formation of a covalent bond between the epoxy group from the sealer and amino group from the exposed dentin collagen [21].

According to Porkaew et al. [22] calcium hydroxide remaining in the root canal walls can enhance the sealing and the quality of filling with zinc oxide eugenol sealer. One possible explanation is that calcium hydroxide may be incorporated to the sealer, reducing permeability. We suggest that sealers based on epoxy resin, such as AHPlus, can also incorporate residues of calcium hydroxide to the sealer, thus, improving the bond strength, for example by expansion. Studies have also shown that AHPlus sealer has a better ability to penetrate dentinal tubules (approximately 1.4 mm) when compared to other materials, promoting a lower rate of apical leakage [23].

We decided to fill the root canals using only endodontic sealer, without gutta-percha, to obtain values more consistent with the aim of the study. Although this kind of procedure does not represent the clinical situation, it was chosen once the gutta-percha presents weak link and consequently is prone to failure first during the load test. [18] This methodology corroborate with the one performed by Jainaen et al. [24] who evaluated the influence of master cone in the bond strength of AHPlus or other methacrylates to root dentin and observed values of bond strength 2-8 times higher in the canals filled just with sealer. Previously, Souza et al. [25] also used only endodontic sealer.

Calcium hydroxide has been widely used as intracanal medication, even believing that the difficulty in its removal would be detrimental to the shutter. The results showed in this study indicates that its use is effective by the improvement in the bond strength of AHPlus.

CONCLUSION

Based on these results, we conclude that the bond strength of AHPlus to dentin walls was significantly improved in the groups that received calcium hydroxide as intracanal medication. Besides, the comparison among tree different protocols using EDTA, ranging in time, volume, and agitation had no significant influence on the bond strength.

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