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Effect of Intracanal Irrigants on Bond Strength of Fiber Posts Cemented With a Self-adhesive Resin Cement

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Clinical Relevance

Saline solution or sodium hypochlorite associated with ultrasonic activation seems to be an adequate solution for root canal cleaning before fiber post cementation. Chelating agents may cause decreased bond strengths when cementing with self-adhesive resin cement.

SUMMARY

Objective: The aim of this study was to evaluate the effect of five intracanal irrigants on bond strength of fiber posts cemented with newer self-adhesive resin cement.

Methods: A total of 60 extracted, single-rooted human premolars, sectioned at 14 mm, were prepared with the ProTaper Universal system with a size F3 instrument and filled with an F3 master cone and AH Plus. The root canal filling was partially removed, leaving 4 mm of apical gutta-percha. Specimens were randomly assigned to five groups (n=12), according to the solution used for dentin surface treatment before fiber post cementation, as follows: EDTA 17% (EDTA); QMix (QM); SmearClear (SC); 2.5% sodium hypochlorite (NaOCl), and 0.9% saline solution (SS). Ultrasonic activation was performed (three times, 20 seconds each), and root canals were dried with paper points. Fiber posts were cemented with RelyX U200. In one specimen per group, rhodamine B dye was mixed with RelyX U200 to provide adequate fluorescence for confocal laser scanning microscopy (CLSM) assessment. Specimens were

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transversally sectioned and three slices were obtained, one for each root third. Next, a push-out test was performed. A stereomicroscope and CLSM were used to analyze the failure modes and to illustrate the pattern of infiltration of RelyX U200 into dentinal tubules, respectively. Bond strength means were calculated, and analysis of variance and Bonferroni tests were used for statistical analysis.

Results: SS showed the highest mean bond strength values (11.5±5.3), superior to QM (5.1±3.1) and SC (5.1±3.3). NaOCl presented intermediary bond strength values (9.7±5.0), similar to EDTA (7.7±2.9) and SS. QM and SC showed the lowest mean bond strength (*p*<0.05). Adhesive failures between cement/dentin were predominant (53.9%).

Conclusion: SS and NaOCl associated with ultrasonic activation seem to be adequate solutions for root canal cleaning before fiber post cementation with self-adhesive resin cement, whereas chelating solutions, such as EDTA, QM, and SC, cause a decrease in bond strength.

INTRODUCTION

Endodontically treated teeth may exhibit pronounced coronal destruction, and the amount of residual coronal dentin can influence the clinical survival of posts and restorations.[1] The minimally invasive approach of associating adhesive techniques and posts with similar mechanical characteristics to dentin seems to contribute to a better clinical prognosis for endodontically treated teeth.[1,2] A direct relationship between fracture resistance and the amount of remaining tooth structure have been reported.[3] Some studies[4] have shown that the presence of substantial remaining coronal tooth structure reduces the risk of failure. Due to this fact, fiber post cementation is a viable alternative to restore weakened teeth.

Glass fiber posts were introduced as an alternative to cast metal posts, because glass fiber presents mechanical properties similar to dentin,[4,6] such as elastic moduli, which improves the distribution of functional loads to the root canal, instead of concentrating it, when compared with cast posts,[7] which might affect the risk and type of root fracture.[8] Several other factors led to the use of glass fiber posts, such as esthetic advantages, low costs, and being a simpler and less time-consuming technique.[6]

In terms of fiber post cementation, self-adhesive resin cements, such as RelyX U200 and RelyX U100 (3M ESPE, St Paul, MN, USA), have been introduced to reduce the sensitivity of pretreatment steps and to prevent application errors of cementation procedures. This factor may increase bond strength, as compared with a conventional three-step system.[9] According to Rodrigues and others[10] and Amaral and others,[11] self-adhesive resin cements are equally effective alternatives to conventional resin cement.

Ideally, a post cement system will provide a tight seal impermeable to oral bacteria; however, debonding failures have been reported.[4] Several variables may be associated with these failures, such as the action of irrigant solutions on dentin collagen (sodium hypochlorite [NaOCl], hydrogen peroxide, chlorhexidine [CHX]); the peculiar conditions of root canal dentin and the type of agent used to condition the substrate; the polymerization stress of resin cement; and the chemical and physical properties of the posts.[12]

To enable satisfactory adhesion of posts to root dentin, the smear layer has to be removed.[13] It consists of an agglomeration of dentin, irrigant solutions, and organic tissues poorly adhered to the root canal walls.[14] The smear layer is able to hinder penetration and adaptation of self-adhesive resin sealers inside dentinal tubules,[13] which may decrease bond strength.

In this sense, chelating solutions play an important role in removing debris and the smear layer.[15] Ethylenediaminetetraacetic acid 17% (EDTA) is the most widely used chelating solution because it enables dissolution of the inorganic portion of dentin and the smear layer.[16] On the other hand, EDTA can cause erosion of root canal dentin[17] and presents reduced antimicrobial action.[18]

With the aim of increasing antimicrobial activity without producing dentin erosion, new irrigants have been proposed. EDTA-based formulations have been developed as final-rinse solutions, such as SmearClear (SC) and QMix (QM).[19] SC (SybronEndo, West Collins, Orange, CA, USA) presents EDTA and cetrimide in its formulation, whereas QM (Dentsply Tulsa Dental, Tulsa, OK, USA) contains EDTA, CHX, and a surfactant agent. This one-step final rinse is supposed to combine the antimicrobial and substantivity properties of CHX with the smear-layer-removing properties of EDTA.[20]

Despite the reported literature, few studies have evaluated the effect of irrigating and chelating solutions on bond strength to root dentin. Moreover, information related to fiber posts cemented with RelyX U200 is still limited. Therefore, the main goal of this *ex vivo* study was to evaluate the effect of different, ultrasonically activated intracanal irrigants on the bond strength between root dentin and fiber posts cemented with self-adhesive resin cement.

The null hypothesis tested was that different irrigant solutions would have no influence on the bond strength.

METHODS AND MATERIALS

Experimental Design

First, the sample size was calculated, using the BioEstat 5.0 program (Fundação Mamirauá, Belém, Brazil), considering parameters that were based on Rosa and others:[21] minimum difference between groups of 1.65 MPa; standard deviation of 1.1 MPa; power of 80%; for five treatments. The program recommended 12 samples per group as the sample size.

Roots (N=60) were randomly allocated (http://www.random.org) to five groups (n=12), considering one factor (irrigant solutions) at five levels. The main outcome was push-out bond strength and the experimental unit was the root. The operators were blinded to the applications of intracanal solutions, post cementations, push-out tests, and failure analyses.

Tooth Selection

This study was submitted to and approved by the Ethical Committee of the Federal University of Santa Maria (No. 855,457). A total of 60 single-rooted human mandibular premolars with similar dimensions were selected and stored in a 0.9% saline solution at 4°C until use. Periapical radiographs were performed to confirm the presence of one root canal. All roots were observed at 8× magnification with a stereomicroscope (Zeiss Stemi SV6; Carl Zeiss, Jena, Germany) to exclude those with external cracks, incomplete root formation, root resorption, or coronal root canal diameter greater than 2 mm, as measured with a digital caliper (Starrett 727; Starrett, Itu, Brazil).

Specimens were decoronated at the cervical root third to standardize a remaining root length of 14 mm, using a diamond blade (Komet, Santo André, SP, Brazil) under cool water.

Root Canal Preparation

Canal patency was established with a size 10 K-file (Dentsply Maillefer, Chemin du Verger, Ballaigues, Switzerland), followed by PathFile 1, 2, and 3 (Dentsply Maillefer) instruments. The working length was set at 1 mm from the apex. Root canals were prepared by using the ProTaper Universal System (Dentsply Maillefer). Initially, the cervical and middle portions of the roots were prepared by using S1, SX, and S2 instruments. Later, S1, S2, F1, F2, and F3 files were sequentially used for all of the working lengths. Each canal was irrigated with 2 mL of a freshly prepared 2.5% NaOCl (Asfer, São Paulo, Brazil) between each instrument change. Specimens were irrigated with 5 mL of 17% EDTA (Biodinâmica, Ibiporã, Brazil) for three minutes and subsequently rinsed with 2 mL of NaOCl. Next, they were dried by using size 30 paper points (Dentsply Maillefer).

Root Canal Filling

AH Plus (Dentsply Maillefer) was mixed according to the manufacturer's instructions and placed in working length by using a 400-rpm lentulo spiral (Dentsply Maillefer) for five seconds. The single-cone technique was performed by using F3 (Dentsply Maillefer) main gutta-percha cones, coated with sealer and placed into root canals to the working length. The excess gutta-percha in the coronal portion was removed with a flame-heated plugger, and the access cavity was sealed with Filtek Z350 (3M ESPE) composite resin. Roots were stored for one week at 37°C and 100% humidity to allow the sealers to set.

Post Space Preparation

Root canal filling was partially removed using sizes 1, 2, 3, and 4 Largo drills (Dentsply Malleifer), alternating with 0.9% saline solution irrigation, in 10 mm length, leaving 4 mm of apical gutta-percha. Post space preparation was completed using the Exacto Translúcido Angelus N2 (Angelus, Londrina, Brazil) bur at 10 mm. Periapical radiographs were performed to confirm removal of the root filling.

Apical root portions were included in a chemically cured acrylic resin (Dencrilay Dencril, Pirassununga, Brazil) block. The specimens were fixed on a surveyor, with the long axes of the teeth and the resin block parallel to each other and perpendicular to the ground.

Irrigation Protocols

As mentioned, specimens were randomly assigned to five groups (n=12), according to final flushing after post space preparation, as follows: EDTA (EDTA

17%); QM (QMix); SC (SmearClear); NaOCl (2.5% NaOCl), and SS (0.9% saline solution).

EDTA, QM, SC, NaOCl, and SS were delivered into root canals using Ultradent syringes (Ultradent Products Inc, South Jordan, UT, USA) and 30G EndoEzeTip needles (Ultradent Products Inc).

The irrigation protocols for all experimental and control groups were as follows: 1) root canals were rinsed with 1 mL of the corresponding irrigant; 2) ultrasonic activation with a size 20/0.1 ultrasonic tip (Capelli e Fabris Ind, Santa Rosa do Viterbo, Brazil), attached to an NAC Plus ultrasonic device (Adiel LTDA, São Paulo, Brazil) was performed for 20 seconds, without touching root canal walls. This procedure was repeated twice and the irrigant was renewed;[22] 3) a final continuous irrigation with 2 mL of 0.9% saline solution was performed in all groups.

Specimens were dried with paper points. Exacto Translúcido N2 (Angelus) glass fiber posts were cleaned with ethyl alcohol 70%, coated with silane (Angelus) and put out to dry for five minutes, until solvent evaporation.

Rhodamine B dye in a ratio of 0.1%[23] was mixed with RelyX U200 (3M ESPE) in one specimen per group, to provide the fluorescence that enabled CLSM assessment. This procedure allowed the illustration of the patterns of self-adhesive resin cement distribution into the dentinal tubules.

RelyX U200 was mixed according to the manufacturer's specification, inserted into the root canal using Automix tips (3M ESPE), and immediately after, the fiber post was inserted by manual pressure. The cement was light-cured for 40 seconds using a previously calibrated LED light-curing unit (Radii Cal; SDI, Melbourne, Australia), maintaining the light guide tip of the light-curing unit placed perpendicular to the post. A single operator performed all procedures. The coronal access was sealed with composite resin (Filtek Z350; 3M ESPE). Roots were stored for one week at 37°C.

Sample Preparation for CLSM Analysis

To illustrate the pattern of distribution of RelyX U200 inside dentinal tubules of each group, one specimen per group was prepared for CLSM.

A cutting machine (Extec Labcut 1010, Enfield, CT, USA) was used for sectioning transversally the roots, providing three slices, one for each root third. For CLSM assessment, surfaces were polished with Arotec paste (Arotec, Cotia, Brazil) to eliminate dentin debris generated during the cutting procedures. The coronal surface of one sample per group was examined with the Olympus FluoView Confocal Laser 1000 Microscope (Olympus Corporation, Tokyo, Japan). The absorption and emission wavelengths for rhodamine B were 540 nm and 494 nm, respectively. Dentin samples were analyzed using the 10× oil lens.

Push-out Test

The first cervical slice (approximately 1 mm thick) was discarded due to excess cement, which could influence the adhesive resistance. Three other slices per specimen (thickness: 2±0.3 mm) were obtained. Each slice was positioned on a metallic device with a central opening (Ø=3 mm) larger than the canal diameter. The most coronal portion of the specimen was placed downward. For the push-out test, a metallic cylinder (Ø extremity=0.8 mm) induced a load on the post in an apical to coronal direction, without applying any pressure on the cement and/or dentin.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, Sao Jose dos Pinhais, Brazil) at a speed of 1 mm/min. Bond strength values (σ) in MPa were obtained as follows: $\sigma = f/a$, where f = load for specimen rupture (N) and a = bonded area (mm^2). To determine the bonded interface area, this formula was used: $A = 2\pi g (R^1 + R^2)$, where $\pi = 3.14$, g = slant height, R^1 = smaller base radius, and R^2 = larger base radius. To determine the slant height, the following calculation was used: $g^2 = (h^2 + [R^2 - R^1]^2)$, where h = section height. R^1 and R^2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, which corresponded to the internal diameter between the root canal walls. The diameters and h were measured using a digital caliper (Starrett 727, Starrett).[2,24]

Failure Mode Analysis

Dentin slices were analyzed first in a stereomicroscope (Zeiss Stemi SV6), and some samples were selected for scanning electron microscopy (JEOL 6060, JEOL, Tokyo, Japan) in order to categorize and illustrate the failure modes, respectively. The failure modes were categorized as follows: Ac/d = predominant adhesive at cement/dentin interface failure; Ac/p = predominant adhesive at cement/post interface failure; CC = cement cohesive failure; DC = dentin cohesive failure; PC = post cohesive failure. Specimens presenting cohesive fracture of the fiber post or dentin were excluded from the study given

Table 1: *Mean of Bond Strength Values and Failure Modes Distribution After Push-Out Test for the Irrigants Used Prior to the Post Cementation*

Groups	Bond Strength	Failure					
		Ac/d	Ac/p	DC	PC	CC	Total
SS	11.5 ± 5.3 ^{a}	19	4	13	—	—	36
NaOCl	9.7 ± 5.0 ^{ab}	18	8	10	—	—	36
QM	5.1 ± 3.1 ^{c}	16	15	5	—	—	36
SC	5.1 ± 3.3 ^{c}	18	6	12	—	—	36
EDTA	7.7 ± 2.9 ^{bc}	26	2	8	—	—	36
	Total	97 (53.9%)	35 (19.5%)	48 (26.6%)	0 (0%)	0 (0%)	180 (100%)

Abbreviations: Ac/d, predominant adhesive at cement/dentin interface failure; Ac/p, predominant adhesive at cement/post interface failure; CC, cement cohesive failure; DC, dentin cohesive failure; NaOCl, sodium hypochlorite; PC = post cohesive failure; QM, QuickMix; SC, SmearClear; SS, 0.9% saline solution.
** Different superscript letters identify statistically significant differences at the $p < 0.05$ level.*

that these types of failures did not represent real push-out bond strength.

Data Analysis

The κ test was used to analyze the intraexaminer agreement regarding failure modes. The mean of bond strength distribution was checked with the Shapiro-Wilk test. One-way analysis of variance (ANOVA) and Bonferroni tests (SPSS 12.0; SPSS Inc., Chicago, IL, USA) were used for statistical analysis. The significance level was set at 5%.

RESULTS

The κ value was 0.84. After the push-out test, some dentin cohesive failures were observed and those specimens were excluded from the bond strength calculations.

One-way ANOVA revealed a significant difference among the groups (p=0.0009). SS showed the highest mean bond strength values, superior to QM and SC (p<0.05). NaOCl presented intermediary bond strength values, similar to EDTA and SS (p>0.05). QM and SC showed the lowest mean bond strength (p<0.05) (Table 1).

Table 1 also presents failure mode distribution. Adhesive failures between cement/dentin (Ac/d) were predominant (53.9%), followed by adhesive failures between cement/post (Ac/p) (19.5%), whereas dentin cohesive (DC) failures represented 26.6% of the specimens. Post cohesive failures (PC) and cement cohesive failures (CC) were not observed. Figure 1 represents the failure modes.

Figure 2 shows the penetration patterns of RelyX U200 into dentinal tubules. Images A (EDTA), C (NaOCl), and E (SS) showed the penetration of RelyX U200 into dentinal tubules. SS presented the most homogeneous penetration into dentinal tubules, along the entire perimeter of the root canal. Images B (QM) and D (SC) showed RelyX U200 limited to the root canal perimeter and not in the dentinal tubules.

DISCUSSION

This investigation indicated that the tested intracanal irrigants promoted different push-out bond strengths; thus, the null hypothesis was rejected. Higher means of bond strength were found for the SS and NaOCl groups, whereas SC and QMix presented the lowest means and EDTA presented intermediary mean values.

This study tested the RelyX U200 resin cement, and the manufacturer recommends the use of NaOCl flushing prior to fiber post cementation; however, the criteria used for choosing this irrigant is not clear. At the moment, no study supports this indication, nor is the NaOCl concentration clear.

It is known that NaOCl acts upon organic components of the dentin and improves the penetration of monomers into the dentinal structure.[25] However, NaOCl presents the potential for collagen degradation, which could affect bond strength to root dentin.[12] A negative correlation was found between dentin exposure time to NaOCl and bond strength.[26] Zhang and others[27] stated that a long period of dentin exposure to a high concentration of NaOCl is able to reduce the bond strength and could result in root fracture. However, in this study the high bond strength values achieved in the NaOCl group may be explained because an intermediary concentration was used (2.5%) just for 60 seconds, contributing to root canal cleaning, reversing possible bond strength loss caused by chelating solutions used during root canal treatment, not resulting in collagen degradation.

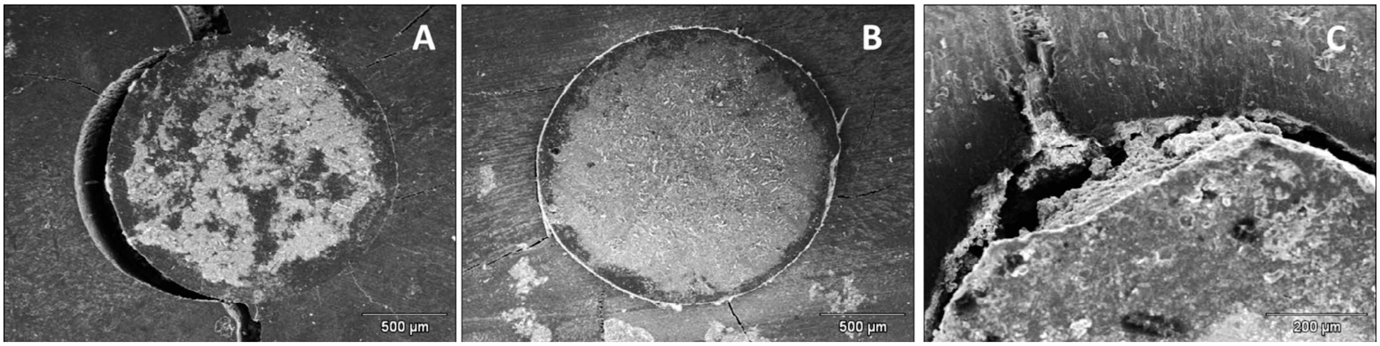

Figure 1. *Scanning electron microscopy photomicrographs of fiber post/self-adhesive resin cement and self-adhesive resin cement/root dentin interfaces. Failure modes are illustrated as follows: (A): Dentin cohesive (DC) failure (50×). (B): Predominant adhesive at cement/dentin (Ac/d) failure (50×). (C): Predominant adhesive at cement/post interface (Ac/p) failure (150×).*

Saline solution presents no antimicrobial and chelating properties. In endodontics, it is frequently associated with CHX 2% gel, for root canal preparation. SS is not able to dissolve organic tissue; however, its ultrasonic activation resulted in higher bond strength values. Such results are also associated with a nondeleterious effect of SS over root dentin. The cleaning efficacy of passive ultrasonic irrigation (PUI) implies the effective removal of dentin debris, microorganisms (planktonic or in biofilm), and organic tissue from the root canal.[22] The cleaning effect of PUI associated with SS can be attributed to cavitation and microstreaming effects of PUI to root canal walls.[22] In this sense, PUI was used to enhance the irrigating and chelating potential of the solutions, exposing dentinal tubules for cement penetration, which could contribute to higher bond strength values. When self-adhesive resin cement is used, a chelating solution plays an important role in smear layer removal,[28] given that no previous acid etching is indicated in the resin cementation technique. This acid etching in a conventional three-step system is responsible for smear layer removal. Rodrigues and others[10] observed that 37% phosphoric acid etching prior to self-adhesive resin cement did not improve the bond strength values.

Chelating agents are widely used in endodontic routines, especially EDTA. It is in a salt obtained

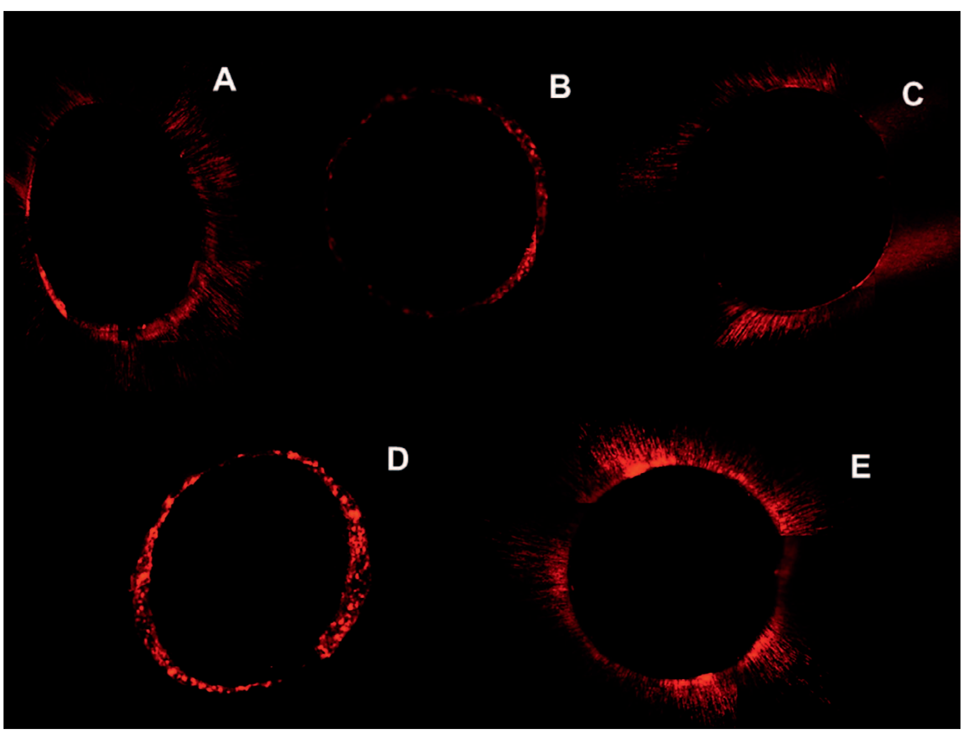

Figure 2. *Qualitative analysis under CLSM (10×) of self-adhesive resin cement penetration into dentin tubules. (A): EDTA; (B): QM; (C): NaOCl; (D): SC; (E): SS. Red: RelyX U200 stained with rhodamine 0.1%. Note that in B and D, the self-adhesive resin cement is confined to the root canal perimeter with absence of tubule penetration.*

from a weak acid that promotes chelation of dentin calcium ions. In an alkaline pH, EDTA selectively removes the hydroxyapatite and noncollagenous protein.[16] On the other hand, the adhesive properties of RelyX U200 depend on a chemical interaction between its acidic monomers and the amount of calcium hydroxyapatite presented in root dentin, providing micromechanical retention.[10,11] Therefore, excessive removal of calcium hydroxyapatite is deleterious for the bond strength of self-adhesive resin cements to root dentin. Intermediary bond strength values were achieved in the EDTA group in this study, contrasting with the findings of Gu and others.[28] These authors stated that EDTA effectively removed the smear layer and increased the bond strength when compared with NaOCl and SS. However, Gu and others[28] activated the EDTA manually for 60 seconds. In the current study, the use of chelating solutions during root canal treatment associated with ultrasonic activation as a dentin pretreatment may lead to dentin erosion and may explain the bond strength reduction.

According to the manufacturer's brochure, QM is a proprietary blend of 2% CHX, EDTA, and a surfactant. SC, in turn, contains EDTA, cetrimide, and a special surfactant. Cetrimide presents low detergent capacity and high antimicrobial activity. The presence of a surfactant aims to enhance the wettability of the irrigant, enabling penetration into root canal anfractuosities. However; Ulusoy and others[29] showed that the presence of surfactants in SC did not improve its efficiency in removing the smear layer. In addition, information related to the influence of SC and QM as irrigants on bond strength of fiber post to root dentin is still poor. An interaction among the three different substances in the formulations of SC and QM and, as mentioned, the presence of EDTA in the formulation, may be responsible for the lower bond strength values obtained in these groups.

In endodontics, CLSM is used to determine the degree of adaptation and penetration of the root canal filling to dentin walls and into dentinal tubules, respectively.[18] Rhodamine dye must be incorporated with cement in a ratio of 0.1%. It did not interfere with the physical-chemical proprieties of endodontic sealers[23,30]; however, it is not clear whether this dye is able to affect the behavior of resin cements used in fiber post cementation. In this study, CLSM was used to illustrate the penetration pattern of self-adhesive resin cement into dentinal tubules. Figure 2 shows that SS, NaOCl, and EDTA presented self-adhesive resin penetration into dentinal tubules. In the QM and SC groups, resin cement remained restricted to root canal space. Considering bond strength and CLSM findings, it can be stated that groups presenting self-adhesive resin penetration into dentinal tubules resulted in higher bond strength values. For instance, SS showed more evident and homogeneous penetration of resin cement into dentinal tubules and also resulted in the greatest bond values. However, as a limitation, just one specimen per group was prepared for CLSM; this finding is suggestive but not conclusive. More studies have to be conducted to establish the relationship between depth-continuity of self-adhesive resin cement penetration and bond strength outcomes.

When new products are investigated, *in vitro* mechanical tests should be conducted to examine experimental variables and test behavioral conditions. Thus, push-out tests are recommended to determine the bond strength of fiber posts to root dentin[31] because they are able to distribute stress more homogeneously; produce less variability in mechanical testing results, fewer pretest failures, and lower standard deviation.[32]

Predominant failures of bonding occur at the cement-dentin interface, which represents a critical interface.[33] In the current study, adhesive failure between cement/dentin (Ac/d) was predominant (53.9%), as expected, followed by adhesive failure between cement/post (Ac/p) (19.5%). Dentin cohesive (DC) failures represent 26.6% of the specimens. Bonding to root canal dentin might be a challenge due to the anatomy of the root, handling characteristics of the adhesive systems, and adhesive procedures.[5] In a root canal, the factor cavity configuration is critical, increasing the stress polymerization of resin cements.[31] The force of polymerization shrinkage into a root canal may be greater than the adhesion of the cement to dentin, resulting in gaps that affect the adhesive interface and may compromise the restoration longevity.[34]

Some limitations of this current study can be depicted as follows: no aging condition (mechanical or thermal cycling) was performed on the tooth with crown restoration, which could depict more realistic clinical behavior. Also, a control group without PUI was not performed. Thus, the isolated action of PUI cannot be stated.

CONCLUSIONS

SS and NaOCl 2.5% associated with PUI promoted the highest bond strength values of RelyX U200.

However, intracanal chelating solutions, such as EDTA, QM, and SC, decreased the bond strength of RelyX U200.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethical Committee of the Federal University of Santa Maria, Brazil. The approval code for this study is 855.457.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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