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Effect of Post-Space Irrigation with NaOCl And CaOCl at Different Concentrations on the Bond Strength of Posts Cemented with a Self-Adhesive Resin Cement

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The aim of this study was to evaluate the effect of post-space irrigation with NaOCl and CaOCl at different concentrations on the bond strength of posts cemented with a self-adhesive resin cement. Eighty premolars were sectioned 14 mm from the apex, and endodontically treated. The root canal filling was partially removed. Specimens were randomly assigned into 8 groups (n=10), according to the irrigant for post-space irrigation: SS – 0.9% saline solution (control group); CHX – 2% chlorhexidine; 1% NaOCl – 1% sodium hypochlorite; 2.5% NaOCl – 2.5% sodium hypochlorite; 5% NaOCl – 5% sodium hypochlorite; 1% CaOCl – 1% calcium hypochlorite; 2.5% CaOCl – 2.5% calcium hypochlorite; and 5% CaOCl – 5% calcium hypochlorite. For each group, irrigation was performed continuously with 2 ml of solution. The post-spaces were dried with paper points (#80), and glass fiber posts were cemented using a self-adhesive resin cement. The specimens were sectioned perpendicularly and the push-out test was performed. Optical microscopy was used to analyze the failure mode. ANOVA and Bonferroni tests analyzed the bond strength data. NaOCl and CaOCl presented similar bond strength regardless the concentration used to irrigate the post-space ($p>0.05$). SS showed the highest bond strength (11.47 MPa) ($p<0.05$). Adhesive failures at the cement/dentin interface were predominant (58.33%). Saline solution should be irrigant of choice to irrigate the post-space before fiber post cementation with self-adhesive resin cement. NaOCl and CaOCl negatively affect the bond strength values.

Key Words: Endodontics, bond strength, root canal irrigants, self-adhesive resin cement.

Introduction

Restorative strategies of endodontically treated teeth still generate discussion in dentistry, especially when it comes to difficulties in obtaining adhesion between a fiber post and root dentin. The difficulties in removing the smear layer, of drying excess moisture from the root canal, as well as the complete cure of the adhesive system and resin cement are inherent challenges in obtaining high adhesion of the fiber post to root dentin (1). Additionally, the endodontic treatment (e.g., gutta-percha and endodontic sealer adhered to canal walls), post pretreatment and method of cement application affect the bond strength of fiber posts to root dentin (1). Therefore, self-adhesive resin cements are intended to simplify the technique, as they are easy to handle and timesaving since pre-treatment of the root surface is not required (2).

The mechanism of adhesion of self-adhesive resin cements is based on micromechanical retention and chemical bonding between the cementing agent and the dental substrate. The cement acidity is high enough to promote hybridization with the tooth structure (3). To obtain sufficient adhesion, the removal of the smear layer, which contains inorganic and organic components, sealer and gutta-percha remnants, microorganisms, and infectious deteriorated dentin is necessary for the penetration of resin cement into the dentin tubules. Root canal irrigation is mandatory for eliminating debris and bacteria from the root canal system (4).

Sodium hypochlorite (NaOCl) is the most commonly used irrigant in the chemical-mechanical preparation of root canals. However, NaOCl is an oxidizing agent that may create an oxygen-rich layer on the dentin wall that inhibits resin polymerization and increases microleakage, resulting in lower bond strengths of the various adhesive systems to the root canals (5).

Chlorhexidine gluconate (CHX) has been recommended as an alternative irrigant with antimicrobial action, low toxicity, and ability to remain active at the site of action (6). CHX preserves the durability of the bond strength up to 12 months, inhibiting collagen-degrading enzymes metalloproteinases (MMPs) (7).

A recently introduced solution for irrigating root canal systems is calcium hypochlorite (CaOCl). This solution exists as a relatively stable substance; generally used for industrial sterilization and water purification treatments. CaOCl is an antibacterial substance with acceptable cytotoxicity and biocompatibility and promotes soft-tissue dissolution, equivalent to NaOCl (8,9). It presents high alkalinity and has a more significant amount of available chlorine than sodium hypochlorite (10,11).

NaOCl and CaOCl have similar potentials as root canal irrigant: the available chlorine in those solutions may help to explain their antimicrobial activity and ability to alter the dentinal structure, mainly collagen (12). However, CHX does not promote morphologic structure alterations of the organic matrix and has been used owing to its substantivity (11). For these reasons, it is relevant to assess whether these properties could affect the bond strength between root dentin and a fiber post. Therefore, the primary goal of this *ex vivo* study was to evaluate the effect of post-space irrigation with NaOCl and CaOCl at different concentrations (1%, 2.5% and 5%) on the bond strength of posts cemented with a self-adhesive resin cement. The null hypothesis was that different irrigants and concentrations would not influence the bond strength.

Material and Methods

Experimental design

Roots (N = 80) were randomly allocated (www.random.org) into 8 groups (n=10), considering 1 factor (irrigant solutions) at eight levels: SS (control group – 0.9% saline solution), CHX (2% chlorhexidine gluconate), 1% NaOCl (sodium hypochlorite), 2.5% NaOCl, 5% NaOCl, 1% CaOCl, 2.5% CaOCl and 5% CaOCl (Farmácia Marcela, Porto Alegre, RS, Brazil). The main outcome was the push-out bond strength, and the experimental unit was the root. The operators were blinded for irrigants, push-out tests, and failure analyses.

Tooth selection

This study was appreciated and approved by the Ethical Committee of the Federal University of Santa Maria (number *#984.334*).

Eighty single-rooted premolars with similar dimensions were selected and stored in a 0.9% saline solution at 4°C until use. Periapical radiographs were taken in order to confirm the presence of one root canal and to exclude incomplete root formation, root resorption, external cracks and a coronal root canal diameter greater than 2 mm, as measured with a digital caliper (Starrett 727; Starrett, Itu, Brazil).

The dental crowns were sectioned using a diamond blade (Komet, Lemgo, Germany) under water irrigation and the remaining root of each specimen was 14 mm long.

Root Canal Preparation

Working length (WL) was established with a size 10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) and was set at 1 mm from the apex, followed by PathFile 1, 2, 3 (Dentsply Maillefer) instruments. Root canals were instrumented with the ProTaper Universal System (Dentsply Maillefer). Initially, S1, SX, and S2 files were inserted to 2/3 of the WL. Afterwards, S1, S2, F1, F2, and F3 instruments were sequentially used to the full WL.

All root canals were irrigated with NaOCl 2.5%. The irrigant was delivered to the canals using Ultradent syringes (Ultradent Products, South Jordan, USA) and 30G EndoEzeTip needles (Ultradent). The irrigation protocols for all experimental and control groups were: (1) 2 ml of NaOCl 2.5% after each instrument change; (2) continuous irrigation for 3 minutes using 5 mL of 17% EDTA in all groups; (3) 2 ml of 0.9% of saline solution. The specimens were dried using size 30 paper points (Dentsply Maillefer).

Root Canal Filling

After root canal preparation, all roots were filled using the single cone technique and size F3 ProTaper gutta-percha points (Dentsply Maillefer), and AH Plus (Dentsply Maillefer). The endodontic sealer was mixed according to the manufacturer's instructions and placed to the working length by using a 25 mm Lentulo spiral (Dentsply Maillefer), followed by removal of excesses of gutta-percha with a heated plugger. The canal opening and remaining dentin were conditioned with phosphoric acid 37% for 30s (Condac 37; FGM, Joinvile, Brazil), rinsed and dried with absorbent paper. Next, these structures were treated with a multiple-bottle total-etch adhesive system (ScotchBond Multi-Purpose, 3M ESPE, Saint Paul, USA). The bonding agents (e.i., Primer and Catalyst) from the adhesive system were then separately applied to the root dentin as recommended by the manufacturer, using a microbrush (Cavibrush, FGM). Finally, the specimens were restored with Filtek Z350 composite resin (3M ESPE). Roots were stored for one week at 37°C and 100% humidity to allow the sealer to set completely.

Cementation Procedures

First, each pulp chamber was re-accessed. Resin composite was removed (without removing dentin) using a spherical diamond bur (#1014; KG Sorensen, Barueri, Brazil) coupled to a high-speed motor (KaVo Dental, Biberach, Germany) under continuous water-cooling. The gutta-percha was then partially removed using sizes 2, 3, 4 Largo drills (Dentsply Mailleifer), keeping 4 mm of gutta-percha at the apical region. Periapical radiographs were taken to confirm the gutta-percha removal. Next, post space was prepared using the Exacto Translúcido

Angelus N2 (Angelus, Londrina, Brazil) bur at 10 mm.

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

The tested irrigant was delivered into the canals using Ultradent syringes (Ultradent) and 30G EndoEzeTip needles (Ultradent). The final irrigation was performed for 3 minutes, using 5 ml of the appropriate solution. The root canals were dried with paper points (#80).

Exacto Translúcido N2 (Angelus) fiber posts were cleaned with 70% ethyl alcohol and coated with a silane-based primer (Prosil; Angelus). The resin cement (RelyX U200; 3M ESPE) (Table 1) was mixed and inserted into the root canals using automix tips and the fiber post was immediately inserted into the root canal. The excess cement was removed using a microbrush (KG Sorensen). The resin cement was light-cured for the 40s using a previously calibrated LED light-curing unit (Radii Cal; SDI, Bayswater, Australia). A single operator performed all procedures. The coronal access was sealed with composite resin (Filtek Z350; 3M ESPE). The specimens were stored for 48 hours at 37°C.

Push-Out Bond Test

A cutting machine (Extec Labcut 1010, Enfield, USA) was used to transversally cut the roots. Three slices per specimen (thickness: 2 ± 0.3 mm) were obtained, one for each root third. 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 marked and placed downward. For push-out testing, a metallic cylinder with a flat tip ($Ø_{tip}$ = 0.8 mm) induced a load on the post in an apical to coronal direction, without any pressure to the cement and dentin.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, São Jose dos Pinhais, PR, Brazil) at a speed of 0.5 mm/min. Bond strength values (σ) in MPa were obtained as follow: $d\sigma = F/A$, where **F** = load for specimen rupture (N) and **A** = bonded area (mm²). To determine the bonded interface area, a formula was used: $A = 2 \times \pi \times g(R1 + R2)$, where π = 3.14, **g** = slant height, **R1** = smaller base radius, **R2** = larger base radius. The following calculation was used to determine the slant height: $g^2 = (h^2 + [R2 - R1]^2)$, where **h** = section height. **R1** and **R2** 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. A single-blinded operator performed all the tests and measurements, to prevent bias by operator.

Table 1. Main composition of RelyX U200 self-adhesive resin cement (3M ESPE)

Base paste	Catalyst paste
- Methacrylate monomers containing phosphoric acid groups - Methacrylate monomers - Silanated fillers - Initiator components - Stabilizers - Rheological additives	- Methacrylate monomers - Alkaline (basic) fillers - Silanated fillers - Initiator components - Stabilizers - Pigments - Rheological additives

Failure Mode Analysis

Specimens were selected and analyzed using stereomicroscope (StereoDiscovery V20, Carl-Zeiss, Gottingen, Germany), up to 150 magnification, to determine the failure modes. The failure modes were labeled as follows: Ac/d = Predominant adhesive at cement/dentine interface; Ac/p = Predominant adhesive at cement/post interface; C = cohesive of dentine. Specimens presenting cohesive failures were excluded from the study since this failure mode does not represent a real push-out bond strength (13).

Data Analysis

The Kappa test was used to analyze the agreement between the readings of the examiner regarding failure mode at different times (two weeks interval).

The distribution of the bond strength data was checked using the Shapiro-Wilk test. One-way 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 Kappa test value was 0.84. After push-out testing, some dentin cohesive failures were observed (22.92%), and those specimens were excluded from the bond strength calculations.

Table 2 presents the mean bond strength values and failure mode distribution. One-way ANOVA revealed a significant difference among the groups (p = 0.0023). SS showed the highest mean values of bond strength ($p<0.05$). NaOCl and CaOCl presented similar bond strength values regardless the concentration used to irrigate the post-space (1%, 2.5% and 5%) ($p>0.05$). When post-space was irrigated with CHX, the bond strength values were similar to those obtained in NaOCl and CaOCl ($p>0.05$).

Adhesive failures between cement/dentin (Ac/d) were predominant (58.33%), followed by cohesive (C) failures of dentin, cement or post (22.92%) and adhesive failures between cement/post (Ac/p) (18.75%). Figure 1 represents the failure modes using optical microscopy at 7.5 magnification. Image A shows adhesive failure at the cement/dentin interface (type Ac/d), image B presents adhesive failure at the cement/post interface (type Ac/p) and image C shows cohesive failure of the dentin substrate (type dc).

Discussion

The present study found that SS had the highest mean bond strength values when compared to the NaOCl, CaOCl, and CHX. Thus, the null hypothesis was rejected.

Bond strength values promoted by self-adhesive resin cements are similar to those obtained with conventional resin cements with the advantage that they exempt dentin pretreatment with greats results of adhesion similar to those obtained with conventional resin cements (14-16). However, the post space must be cleaned before fiber post cementation to promote smear layer and debris removal (13). Interestingly, the manufacturer of the self-adhesive resin cement RelyX U200 recommends the use of NaOCl before fiber post cementation; however, at the moment, the criteria used for choosing this irrigant is not clear.

The present study stated that the concentrations of both irrigants (NaOCl and CaOCl) affected the bond strength similarly. It is likely that they promoted similar dentin alterations and provided a negative influence on adhesion, independent of their concentrations. The results obtained after irrigation of post space prepare with NaOCl can be explained because it interacts with the organic portion of the dentin and it occurs a collagen degradation potential, which could adversely affect the bond strength. This fact was observed by Katalinic *et al.* (17) where NaOCl was associated with lower bond strength values, but there was no difference between this solution, chlorhexidine and ozone gas.

Recent studies have shown that CHX improves the longevity of adhesion of composite adhesive bonding to dentin by inhibiting hybrid layer collagen-degrading enzymes called MMPs (6,7,18). According to Lindblad *et al.* (18), CHX enhanced the bond strength of fiber posts to root dentin and

Table 2 Mean values of bond strength (standard deviations, in MPa) and failure modes (%) distribution for different groups

Groups	Bond strength*	Failure modes**			
		Ac/d	Ac/p	C	Total
SS	11.5 (5.3)[a]	17	2	11	30
CHX	7.6 (2.5)[b]	18	5	7	30
1% CaOCl	6.0 (2.6)[b]	21	6	3	30
1% NaOCl	5.7 (5.2)[b]	15	3	12	30
2.5% CaOCl	5.2 (3.4)[b]	17	13	0	30
2.5% NaOCl	6.1 (2.6)[b]	17	6	7	30
5% CaOCl	5.0 (2.4)[b]	20	7	3	30
5% NaOCl	5.8 (4.1)[b]	15	3	12	30
		140 (58.33 %)	45 (18.75 %)	55 (22.92%)	240 (100%)

*Different superscript letters identify statistically significant differences (p<0.05). ***Failure modes*: Ac/d = mainly adhesive failure at cement/dentine interface; Ac/p = mainly adhesive failure at cement/post interface; C = cohesive failures of the dentine.

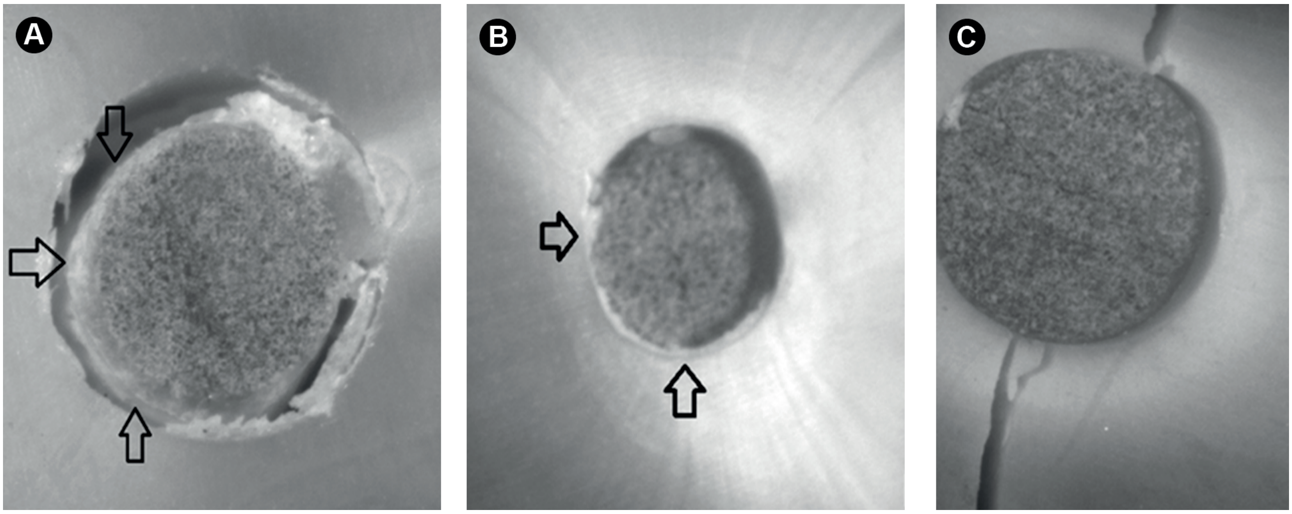

Figure 1. Optical microscopy (7.5× magnification) of the failure modes of the specimens (root dentin - resin cement - fiber post). (A) Mainly adhesive failure at cement/dentine interface; (B) Mainly adhesive failure at cement/post interface; (C) Cohesive failure in the dentine.

significantly reduced the number of adhesive failures. However, Leitune *et al.* (19) stated that CHX did not influence the bond strength of glass fiber posts cemented with resin cement. And finally, Angeloni *et al.* (20) found that CHX pretreatment did not prevent bond strength degradation of fiber posts luted with self-adhesive cements when they were exposed to artificial saliva up to one year.

The CaOCl's mechanism of action is given by the dissociation of calcium hydroxide and HOCl, suggesting that the high pH of this irrigant is due to the dissolution of precipitated particles of calcium hydroxide (10). CaOCl shows greater stability when compared to NaOCl because it presents a higher concentration of active chlorine (19). Moreover, the released ions and the high chlorine level appear to explain its high antimicrobial effect (8). Nevertheless, there is a lack of consistent information regarding the chemical properties of CaOCl at different concentrations, as well as their interaction with dentinal properties. CaOCl modifies the root canal dentin roughness similarly to NaOCl (21). These alterations promoted by both irrigants can explain the similar bond strength values obtained in this study irrespective of the concentration of NaOCl or CaOCl employed. Comparisons with previous studies cannot be performed because this is the first one that evaluated the association between CaOCl and bond strength values of fiber posts.

SS presents no antimicrobial or chelating properties. Therefore, it is used with CHX during root canal preparation in Endodontics. Da Silva et al. (6) used SS associated with 2% CHX gel and obtained the highest bond-strength values, which were statistically different to the association of NaOCl and EDTA and control group (SS). The present study used SS before fiber post cementation and obtained the highest bond strength values, indicating that SS does not contribute to collagen degradation. In line with the present study, Barreto et al. (13) found the highest bond strength values when fiber posts were cemented with self-adhesive resin cements after post space irrigation with saline solution and 2.5% NaOCl. However, the authors promoted passive ultrasonic irrigation of the irrigant for three intervals of 20 s previously post cementation.

A negative factor associated with the bond strength in root dentin is the limited capacity of the resin cements to dissipate tensions generated during polymerization shrinkage. The cavity configuration factor (C-factor) is related to the ratio of the adhesion area to the free area, which is unfavorable in the root canal, responsible for creating gaps between cement and dentin and affecting the sealing ability of resin cements (22). Therefore, the cement-dentin interface is more susceptible to dentin/adhesive failure when compared to the cement-post interface. As expected, this fact was observed in this study, since it found high percentage of adhesive failure at the cement-dentin interface (58.33%), followed by cohesive ones (22.92%). Cement-post failures represented 18.75% of the specimens. Representative specimens of adhesive and cohesive failures were illustrated by optical microscopy (Fig. 1).

Based on the results, it can be concluded that NaOCl and CaOCl negatively affected the bond strength between root dentin and fiber posts when cemented with self-adhesive resin cement and should not be used to irrigate the post-space. On the other hand, saline solution promoted the highest bond strength and could be the preferred intracanal solution before fiber post cementation when self-adhesive resin cement will be used, despite its lack of disinfection.

Resumo

O objetivo deste estudo foi avaliar o efeito da irrigação do espaço preparado para o pino com NaOCl e CaOCl em diferentes concentrações na resistência adesiva de pinos cimentados com um cimento resinoso autoadesivo. Oitenta pré-molares foram seccionados 14 mm do ápice e endodonticamente tratado. As obturações foram parcialmente removidas. Os espécimes divididos randomicamente em 8 grupos (n=10) de acordo com o irrigante usado para irrigação do espaço preparado para pino: SF – soro fisiológico 0,9% (grupo controle); CHX – clorexidina 2%; NaOCl 1% – hipoclorito de sódio 1%; NaOCl 2,5% – hipoclorito de sódio 2,5%; NaOCl 5% – hipoclorito de sódio 5%; CaOCl 1% – hipoclorito de cálcio 1%; CaOCl 2,5% – hipoclorito de cálcio 2,5%; CaOCl 5% – hipoclorito de cálcio 5%. Para cada grupo, irrigação foi realizada com 2 mL de solução. O espaço preparado para pino foi seco com cone de papel absorvente (#80) e os pinos de fibra foram cimentados com cimento resinoso autoadesivo. Os espécimes foram seccionados perpendicularmente e o teste de push-out foi realizado. Microscopia óptica foi usada para analisar o padrão de falha. Os testes de ANOVA e Bonferroni analisaram os dados de resistência adesiva. NaOCl e CaOCl apresentaram similar resistência adesiva, independentemente da concentração usada para irrigar o espaço preparado para pino ($p>0,05$). SF apresentou os maiores valores de resistência adesiva (11,47 MPa) ($p<0,05$). Falhas adesivas na interface cimento/dentina foram predominantes (58,33%). Soro fisiológico deve ser o irrigante de escolha para irrigar o espaço preparado para pino antes da cimentação de pinos de fibra com cimentos resinosos autoadesivos. NaOCl e CaOCl afetam negativamente os valores de resistência adesiva.

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