BJOS Brazilian Journal of Oral Sciences

Original Article

Volume 18
2019
e191376

Bond strength at the cement/dentin interface according to protocols for final irrigation of post space

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Aim: The aim was to evaluate the bond strength at the cement/dentin interface in the post space, after specimens were treated with different final irrigation protocols, followed by cementation with a dual resin cement. **Methods:** Forty-eight extracted uniradicular human premolars were divided into four groups according to the irrigation (n = 12): control with distilled water; 2.5% sodium hypochlorite (NaOCl) + ethylenediaminetetraacetic acid (EDTA); 2.5% NaOCl + passive ultrasonic irrigation (PUI); and 2% chlorhexidine (CHX). The pull-out test was performed. The results were evaluated using ANOVA with Tukey's paired comparisons, with a significance of 5%. **Results:** When all groups were compared, significant difference occurred (p = 0.006), and in the paired comparison, NaOCl + PUI and CHX differed (p = 0.005). The CHX showed significantly higher adhesive defects between cement and dentin than other groups. **Conclusions:** The final irrigation protocol for cleaning after preparation for post space directly influences the bond strength at the cement/dentin interface.

Keywords: Dental cements. Dental pins. Smear layer. Sodium hypochlorite. Root canal irrigants.

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Received: August 13, 2018
Accepted: March 18, 2019

Introduction

In recent years, fiberglass posts have often been indicated for the reconstruction of endodontically treated teeth[1-3]. These posts have been increasingly used in the anterior teeth for aesthetic reasons, owing to their translucency, as well as because they facilitate polymerization of adhesive materials by allowing the passage of curing light[4]. With biomechanical properties equivalent to those of dentin, they allow distribution of stresses along the root canal in a more homogeneous manner when compared to metal posts. Furthermore, they minimize the steps in laboratory procedures, decreasing treatment time[4].

Even with the evolution of restorative materials, adequate adhesion to the root canals is still a challenge, since adhesives are limited with regard to their physicochemical properties[5]. In addition, the preparation of post space results in walls of the root canal becoming covered by a smear layer, which compromises the bonding of the cement to the dentin[4,6,7].

The cementation of endodontic posts through adhesive systems can generate some complications, such as the following: post detachment due to loss of retention, root fracture due to excessive wear, and endodontic lesions caused by maladaptation. Other factors that can lead to failure are the incomplete polymerization of adhesive materials and the adverse effects of endodontic cements[6,7].

For post cementation with adhesive systems, conventional or self-etching systems may be employed. Conventional adhesive systems depend on the removal of the smear layer through the use of acidic solutions that expose the collagen matrix for penetration of the adhesive[8,9]. In contrast, the self-etching adhesive systems partially infiltrate the dentin layer, demineralizing it by the use of an initiator containing an acid monomer, thus incorporating the smear layer[6,10]. The difference between the two approaches is in the requirement of a separate step when using the conventional adhesive system, involving the application of 35%-37% phosphoric acid gel, which is subsequently washed. The self-etching technique involves the use of a primer, followed by a jet of dry air, thus maintaining the modified smear layer[11]. Pashley et al.[8] pointed that acid-etching with 32%-37% phosphoric acid not only simultaneously etches enamel and dentin, but the low pH kills many residual bacteria.

With the evolution of restorative dentistry, resin cements are currently classified according to the form of their activation into chemical, physical (photoactivated), and dual activation[12].

Besides the variety of adhesive and cement systems, some irrigation solutions, commonly employed after preparation of post space, may compromise the retention of fiber posts[2]. Removal of debris using only irrigation with water jets, as recommended by some manufacturers, is difficult because the anatomy of the radicular space is narrow and deep[6]. In order to remove the smear layer of the root canal in endodontically treated teeth, chemical irrigation has been indicated. In view of the variety of commercially recommendations for irrigants, post-treatment irrigation and its effects on the bond strength of different adhesive systems are still of relevance[7].

Among the irrigation solutions used in endodontics, NaOCl solution is the most widely used because of its antimicrobial action and the ability to act in organic tissue[2]. But, when used in in the post space, the solution may adversely affect the bond strength of adhesive systems because it does not dissolve inorganic particles[13,14]. In contrast, EDTA acts as a calcium chelator, resulting in the demineralization of the root dentin, in addition to removing inorganic debris. The use of EDTA for one minute, followed by NaOCl, was considered effective and safe for the removal of smear layer[7]. However, such solutions, if used for an excessive period, can lead to erosion on the dentinal surface of the root canal[6].

As an alternative, a solution of 2% CHX, which has been advocated as the final irrigant, is indicated, owing to its antimicrobial activity and substantivity. In addition, it can remain active for up to 72 hours after its use, which is sufficient time for the cementation procedure of the post to be completed, thus guaranteeing the sealing of the root canal without recontamination by microorganisms[15]. However, unlike NaOCl solution, CHX cannot dissolve the residual necrotic tissue[7,15].

The combined use of PUI with the irrigation solution is considered an effective method by allowing the deep penetration into the dentinal tubules and other inaccessible areas, which cannot be achieved without agitation[16]. Zhang et al.[17] evaluated the effect of irrigation solution on the retention of fiber posts and found that, after irrigation with EDTA/NaOCl using PUI, there was an improvement in bond strength. Leal et al.[18] concluded that 5.25% NaOCl was the best solution for the final irrigation when combined with EDTA.

In order to determine the relationship between the bond strength and the maximum tensile force applied until failure occurs, the pull-out test was chosen. De Santis et al.[19] have pointed out that this test is reliable because it measures the bond strength between the post and the root dentin precisely, as well as distributes the stress uniformly.

The objective of this research was to evaluate the bond strength between the dual resin cement and root dentin after irrigation of the post space using four different irrigation protocols: distilled water, NaOCl associated with EDTA, NaOCl associated with PUI, and CHX solution.

The null hypothesis was that the irrigation protocols have no influence on the bond strength of the fiberglass posts.

Material and Methods

Sample selection

After approval of the Research Ethics Committee (no. 2.045.919), forty-eight extracted uniradicular human premolars with a single canal and complete apex formation, that were not treated endodontically, were selected for this study. The sample size was defined based on reference with a design and test similar to that of the present study[20].

The dental crown was removed at the cementoenamel junction under irrigation, and the mean root length was measured as 14 mm. The distribution of roots between the groups was performed randomly.

Preparation of the root canal

Endodontic treatment and all steps for the insertion of fiberglass posts were performed by a single trained operator.

In order to obtain the working length, visual odontometry was performed by verifying the passage of a #10.02 manual K-File (Dentsply Maillefer, Balaigues, Switzerland) via the apical foramen, and the real tooth length was determined. To calculate the working length, 1 mm of the real tooth length (RTL) was subtracted from RTL, so the instrumentation was performed 1 mm short of the apical foramen. The root canal was then prepared with the Reciproc® R50 instrument (VDW, Munich, Germany), as recommended by the manufacturer. Each instrument was discarded after use in four root canals.

Irrigation during the root canal preparation was done with 15 mL of 2.5% NaOCl. At the end of this stage, the canals were flooded with 3 mL of 17% EDTA (Biodynamic Chemistry and Pharmaceuticals, Ibiporã, Brazil) for 3 minutes, which was removed by further irrigation with 2.5% NaOCl. Subsequently, the root canal was dried by means of suction cannulas and tips of absorbent paper Reciproc® R50 (VDW, Munich, Germany).

Obturation

The obturation was performed with standard Reciproc® R50 gutta-percha (VDW, Munich, Germany) and AH Plus cement (Dentsply, York, USA), following the Modified Tagger´s Hybrid technique. After the adaptation of the master cone and accessory cones by lateral condensation, the Mc Spadden #60 compactor (Dentsply, York, USA) was used.

The samples were then kept in a humid oven at 37 ºC for seven days.

Preparation for the post

Seven days after the obturation, space preparation for the fiberglass post (White Post DC 1, FGM, Joinville, Brazil) was performed using a post-specific drill according to the manufacturer's recommendations. The gutta-percha was removed from the root canal, maintaining 4 mm of obturator material in the apical region. Next, the post was positioned and its adaptation was checked according to the length prepared.

The roots were divided equally among the four groups (n = 12) according to the irrigation protocol of the post space, as follows:

Control: final irrigation with 2 mL of distilled water, and suction and drying of the space;

NaOCl + EDTA: final irrigation with 2 mL of 2.5% NaOCl followed by 2 mL of 17% EDTA solution for 1 minute, and then suction and drying of the space;

NaOCl + PUI: final irrigation with 2 mL of 2.5% NaOCl with PUI in three cycles of 15 seconds each, with renewal of the irrigating solution at each cycle, and then suction and drying of the space;

CHX: final irrigation with 2 mL of 2% CHX solution and suction and drying of the space.

In the NaOCl + PUI group, the ultrasonic agitation was performed with the T0S-02 (CV-Dentus, São José dos Campos, Brazil) P1 programming, with 10% of the maximum power (10 W) of Ultrasound Dentsurg Piezo Clinic (CV-Dentus, São José dos Campos, Brazil).

After performing irrigation according to different irrigation protocols, all samples and posts were submitted to the following steps: application of 37% phosphoric acid (Condac®, FGM, Joinville, Brazil) in the entire post space as well as the post surface for 15 seconds and rinsing with water jets for 10 seconds. With the aid of a Cavibrush® microapplicator (FGM, Joinville, Brazil), the Amber adhesive (FGM, Joinville, Brazil) was then applied to the entire surface of the root canal and post, the first drop of the product being cured for 10 seconds, followed by a new layer of adhesive on the same surface cured for the same time. A jet of air was then applied for evaporation of the solvent.

Absorbent paper cones were used to remove excess adhesive in the most apical region of the root canal. The adhesive was photopolymerized by Poly Wireless photopolymerizer (KaVo, Joinville, Brazil) at 1100 mW/cm² per 20 seconds, both inside the canal and on the post surface, according to the manufacturer's recommendations. For evaluation of the light intensity, we used the Cure Rite digital radiometer (Dentsply/Caulk, Milford, USA). The Allcem® Core dual cement (FGM, Joinville, Brazil) was used, with the help of the Centrix® syringe (DFL, Rio de Janeiro, Brazil), in sufficient quantity to fill the entire root canal, thus avoiding the formation of void.

A small amount of cement was also applied to the surface of the post, which was introduced into the canal in tilting movements in order to remove possible voids. After insertion of the post, slight digital pressure was applied for 20 seconds to remove excess cement. Polymerization was carried out at a power of 1100 mW/cm² for 40 seconds on each side of the specimen (occlusal, buccal, lingual, mesial, and distal). The samples were kept in an oven at 37 °C for seven days for complete chemical activation of the cement.

Pull-out test

In order to evaluate the maximum tensile force required until post displacement occurs, the test specimens were subjected to the pull-out test in a universal test machine EMIC (Instron Brasil Equipamentos Científicos Ltda., São José dos Pinhais, Brazil) at a velocity of 0.5 mm/minute until failure occurred.

After the test, all the posts were removed from the canals. The types of joint failures were evaluated by a magnifying glass with a 5x magnification and classified as adhesive, cohesive, or mixed, taking into account the following criteria:

1. AD: adhesive failure occurred between dentin and cement.
2. AP: adhesive failure occurred between cement and post.
3. CC (cohesive): failure occurs within the cement.
4. M (mixed): more than one type of failure in the same sample.

Statistical analysis

The data were submitted to the normality test (Kolmogorov-Smirnov) and homogeneity of variance (Levene). Based on these assumptions, analysis of variance (one-way ANOVA) was performed with paired Tukey's comparisons, with a 5% level of significance.

Results

When the four groups were compared, the difference was found to be significant ($p = 0.006$) among all. In the paired comparison, the difference was statistically significant between NaOCl + PUI and CHX groups ($p = 0.005$) (Table 1).

The failure predominated in the groups was M type, being highest in the control. The AD type failure was more frequent in the CHX, and the AP failure occurred more commonly in the NaOCl + EDTA. The CC failure was similar between the control and NaOCl + EDTA groups, and the NaOCl + PUI and the CHX groups (Table 2).

Discussion

The null hypothesis of the present study was rejected, since the bond strength at the dual resin cement and dentin interface in the post space was significantly affected by the different irrigation protocols used.

In a previous study, two resin cements and four types of fiberglass post were evaluated by pull-out bond strength test using finite element analysis[19]. The authors pointed out that the test was reliable because it accurately measured the bond strength between the post and the root dentin, in addition to distributing the tensions more precisely[19]. In addition to the advantages cited, the choice of the test for the present study was due to the fact that sectioning of the teeth was not required, as in the push-out test,

Table 1. Mean and standard deviation of the groups subjected to the pull-out test according to the protocol of final irrigation

Group	Tensile force (MPa)
Control	$80.0 \pm 18.8^{a,b}$
NaOCl+EDTA	$60.8 \pm 18.7^{a,b}$
NaOCl+PUI	85.7 ± 13.4^{a}
CHX	53.3 ± 16.9^{b}

Note: Different letters indicate statistical significant difference, Tukey's test ($p<0.05$).

Table 2. Types of failures found in groups according to irrigation technique (in %)

Group	Failure			
	AD	AP	CC	M
Control	8.3	25.0	8.3	58.3
NaOCl+EDTA	16.7	41.6	8.3	33.3
NaOCl+PUI	16.7	16.7	16.7	50.0
CHX	33.3	8.3	16.7	41.7

since the vibration generated during sectioning could affect the adhesion and interfere with the results of the study.

Within the limitations of this in vitro study, in vitro, it was observed that a small percentage of adhesion failures occurred at the interface studied, between dentin and cement (AD), showing the importance of cleaning the post space prior to cementation.

Regarding the bond strength in terms of resistance to tensile force, the NaOCl + EDTA group presented lower resistance when compared to the control group and the NaOCl + PUI group, but with no statistically significant difference. This reduction in bond strength is probably due to the combination of the demineralizing effect of EDTA and the oxidizing action of NaOCl. Although this effect of EDTA is desirable for better NaOCl penetration, the interaction of phosphoric acid with the chemical structure of the underlying dentin altered by demineralization may compromise adhesion when using conventional adhesive systems, since the removal of dentin calcium may be detrimental to the adhesives that depend on the acidic corrosion of the dentin for their performance[21,22]. However, Haralur et al.[23] concluded, in a study evaluating the effect of irrigating agents on push-out bond strength of resin postcemented with adhesive systems, that the 17% EDTA had a negative influence on self-etch bond strength.

The NaOCl solution, when used in in the post space, may adversely affect the bond strength of adhesive systems[13,14]. Santos et al.[24] also found a lower bond strength in a study on bovine incisor teeth, but irrigation was performed with 5.25% NaOCl for 30 minutes and 17% EDTA for five minutes, longer than the recommended time of a minute, which can result in severe peritubular erosion, besides bovine and non-human teeth, triggering variations in the dentin substrates.

Regarding the frequency of adhesive failures between cement and post found in the NaOCl + EDTA group in this study, Balbosh and Kern[25] found a significant benefit in terms of retention after blasting of the surface of fiber posts, when they were cemented with resin cement. The type of failure observed between post/resin cement interface was largely detected on non-abrasive with aluminum oxide jet. However, blasting of fiberglass post is no longer performed because it is detrimental to its structure.

The NaOCl + PUI group presented the highest bond strength, although there was no significant difference when compared to the control group. Although it presents an oxidizing effect, apparently diminished by the use of PUI, irrigation with NaOCl using ultrasonic activation was observed to be effective in removing the smear layer, resulting in a good combination with the conventional adhesive system and the dual resin cement[2,7].

NaOCl, in addition to its strong bactericidal action, when in contact with the substances inside the root canal, releases chlorine and latent oxygen. This gaseous release of oxygen is possibly exacerbated by PUI, which leads to dislodgement of solid and semi-solid products present inside the root canal, such as remains of gutta-percha and obturation material, thus increasing its cleaning efficacy. Consequently, there is an increase in bond strength at the cement/dentin interface[26]. When used with EDTA, some authors concluded that the solution was the best solution for the final irrigation[18]. In addition, ultrasonic activation, when used to clean the post space, can potentiate the removal of the smear layer by virtue of its mechanism of action as follows: transient cavitation inside the canals; movement of the irrigating solution in circles; and increasing the temperature of the chemical, ensuring the efficiency of the irrigating liquid[27].

In the CHX group, where the majority of the defects were of M type and AD type, the lowest bond strength was observed when submitted to the tensile force testing, with a significant difference in comparison to the NaOCl + PUI group.

In a study by Wang et al.[28] irrigation of the post space with CHX was not effective in improving the bond strength of cement-bonding post with dual resin cement, besides having a negative effect on bond strength when using self-adhesive cement. Lindblad et al.[15] observed an increase in mixed and cohesive failures, suggesting an immediate effect of CHX.

However, in a study performed where the irrigation of the post space was done with 2% CHX gel, da Silva et al.[29] obtained higher bond strength when compared to groups irrigated with saline (control) and NaOCl + EDTA, probably due to their cleaning ability, creating better conditions for adhesion.

Bitter et al.[7] mentioned a positive action on bond strength, when they used CHX with the conventional adhesive system. However, CHX was used as a non-specific metalloproteinase inhibitor, i.e., applied after acid etching and not as a post space irrigant.

One of the possible causes of failures of the M type (majority in the present study) is that, although the capacity of photopolymerization of cements in anatomical regions where curing light does not completely reach the material is questionable, especially in more apical areas, studies prove that use of dual activation cements ensures uniform chemical polymerization. However, during the manipulation and insertion of the material, voids may be formed in the adhesive interface, because of air bubbles trapped in the material, compromising its effectiveness[12].

In addition, the failures may be attributed to the factor C of the post space inside the root canal, where the amount of non-adhered surfaces during the polymerization of a composite is inversely proportional to the material flow, resulting in stress relief related to the contraction of polymerization being unfavorable for the resin cement by maximizing the polymerization contraction tension along the walls of the root canal[30,31].

Another hypothesis related to the M type failure is related to the presence of residual acid monomers after the use of the self-etching adhesive system, which hinder polymerization by reacting with tertiary amines existing in the dual resin cement, leading to a decrease in bond strength[31]. This can also happen in conventional two-stage adhesive systems in areas where curing light for photopolymerization does not reach, triggering a chemical incompatibility between the cement and the adhesive system and, consequently, adhesion failures.

Further studies are required for evaluating the cleaning effectiveness of 2% CHX gel in comparison to the solution form in post space, as well as the effect of using this chemical prior to the conventional two-step adhesive system followed by cementation of the post with dual activation resin cement.

According to the results of the present study, we can conclude that the different protocols of final irrigation of the post space directly influence the bond strength at the cement/dentin interface.

Acknowledgements

The authors deny any conflicts of interest related to this study.

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