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Do Irrigation Solutions Influence the Bond Interface Between Glass Fiber Posts and Dentin?

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The aim of this study was to evaluate the push-out bond strength and interface permeability of glass-fiber posts in different regions of post space (cervical, middle, and apical) submitted to different irrigation solutions. Ninety single-rooted human teeth were submitted to endodontic treatment and divided into five groups, according to irrigation solutions: distilled water, 5.25% NaOCl, 25% polyacrylic acid, 2% chlorhexidine, and 23 ppm Ag NP dispersion. Each group was divided in 3 subgroups (n=6) according to cementation: SBU: Adper Scotchbond Universal + RelyX ARC; U200: RelyX U200; MCE: MaxCem Elite. Bond strength and sealing ability were measured in different areas of post space. The data was subjected to ANOVA and Fisher's test (α=0,05). The silver nanoparticle solution showed highest bond strength values and lowest interface permeability in all thirds analyzed for SBU group. In the U200 group, the highest bond strength values were found for sodium hypochlorite solution, with significant difference between this solution and polyacrylic acid. A decrease in bond strength values in cervical to apical direction was found for MCE group and the same behavior were found for others groups. Regarding interface permeability, use of silver nanoparticle solution resulted in lower values in cervical and apical thirds. There was a decrease in bond strength in cervical to apical direction. Different irrigation solutions and intraradicular depth influenced the bond strength and interface permeability of adhesive material to dentin substrate. Silver nanoparticle solution can effectively be used as an irrigation agent in post space prior to fiber post cementation process.

Key Words: resin cement, dentin, nanoparticles, silver, push-out strength.

Introduction

With the increase in demand for aesthetic restorations, endodontically treated teeth with little coronal structure have been restored using fiber posts (1). The advantages of fiber posts are related to their mechanical properties, including an elastic modulus which is close to dentin (2), high flexural strength, compatibility with Bis-GMA-based resin, biocompatibility and resistance to corrosion (3).

However, the success of these intraradicular restorations is dependent upon adequate bonding at the post-dentin interface (1). The most common failure associated to fiber posts is their debonding (2) in this interface resulting from problems with dentin hybridization (4). Studies have shown that this hybridization can be affected by irrigants, root regions, post space preparations and adhesive systems (5,6).

Among the irrigating solutions, sodium hypochlorite (NaOCl) is one of the most popular and is widely used in root canal and post space preparation because of its antimicrobial properties and its capacity for dissolving organic tissue (7,8) When NaOCl interact with microorganisms and organic tissue it causes saponification reactions leading to antibacterial and tissue-dissolving effects (8). Another solution widely used is chlorhexidine because of its antimicrobial properties (9), substantivity, and effect on the longevity of the bonding interface (2,9,10). Additionally, in vitro an in vivo investigations have been evidenced that chlorhexidine postpone the degradation of this interface due to its ability to inhibit dentin matrix metalloproteinases (MMP) (11) and/or cysteine-cathepsins (CCs) collagenolytic activity in the hybrid layer (12). Polyacrylic acid has been used to condition the dentin to improve the adhesion of glass ionomer cement. According to Pavan et al. (13), this solution also enhances the bonding ability of self-adhesive resin cements. Polyacrylic acid is not able to remove smear plug, it just removes partially the smear layer, leaving free calcium and phosphate ions on dentin surface (14).

Recently, silver nanoparticles (Ag NPs) have gained popularity in many health care fields because of their broad-spectrum bactericidal and virucidal properties (8,15). They have been widely used in medicine (15), for example in nanofiber mats, bandages, wound dressings and ointments. Ag NPs also serve to prevent bacterial colonization on various surfaces such as catheters, clothing and prostheses (7).

However, Ag NPs have not yet been evaluated for use in post space preparation especially as an irrigant solution. The aim of this study was to determine the push-out bond strength and interface permeability of glass-fiber posts in different regions of the post space (cervical, middle, and apical) subjected to different conditioning agents (distilled water, 5.25% sodium hypochlorite, 25% polyacrylic acid, 2% chlorhexidine and 23 ppm silver nanoparticles dispersion). The null hypotheses tested were: [1] the type of conditioning agents do not affect the bonding effectiveness and [2] the sealing ability; there are not difference in sealing ability [3] and bond strength [4] of glass fiber posts in different thirds of intraradical dentin (cervical, medium and apical).

Material and Methods

In this study, one universal adhesive system and three resin cements (one etch-and-rinse and two self-etch adhesives) were used (Table 1). Ninety single-rooted human upper premolars from different individuals, which were extracted for orthodontic or periodontal reasons, were used in this study. The project was approved by the Research and Ethics Committee of the Araçatuba School of Dentistry, UNESP (Protocol #05142812.4.0000.5420). Before use, the teeth were cleaned and kept frozen (-20 °C). All teeth exhibiting evidence of caries, root resorption, cracks, or fractures were excluded from the study.

The anatomic crowns of all teeth were removed 1 mm above the cementum-enamel junction with a low-speed diamond saw (Isomet 2000; Buehler Ltd., Lake Bluff, IL, USA) under water cooling. (Fig. 1) The specimens were then subjected to endodontic treatment. During preparation, the root canals were irrigated with 2.5% NaOCl. The root canals were then dried with sterile paper points and immediately obturated by lateral condensation using gutta-percha cones (Dentsply-Maillefer, Ballaigues, Switzerland) and calcium hydroxide cement (Sealapex; Kerr, Orange, CA, USA). Coronal access was sealed with zinc oxide/zinc sulfate cement (Coltosol; Vigodent, Rio de Janeiro RJ, Brazil). The endodontically treated teeth were stored in water at 37 °C for 7 days.

A #3 glass fiber post system (Reforpost; Angelus, Londrina, PR, Brazil) was used in this study. The post spaces of all the specimens were prepared using a # 2, 3, 4 and 5 low-speed drill bit (Dentsply Maillefer) sequentially. The gutta-percha cones were removed to a depth of ±9 mm with reference to the working length of the tooth. After the completion of the preparation process, adaptation of the fiber posts was confirmed by placing them in the post space. If the posts penetrated to the 9 mm depth, they were considered to have adapted.

Before the adhesive procedure, the surfaces of the glass-fiber posts were treated with 35% phosphoric acid (3M ESPE, ST. Oaul, MN, USA) for 60 s, washed and air dried. The post surfaces were silanized for 60 s (Angelus) and gently dried with air-stream jet. Finally, the adhesive system to be used on the dentin surface was first applied to the post surface if required. Thereafter, the post was not again manipulated to prevent contamination.

The post spaces were irrigated with 2.0 mL distilled water to remove any gutta-percha debris and to maintain the humidity of the environment. The root canal was dried with air and sterile paper points before the bonding procedures. The specimens were divided by drawing lots into the

Table 1. Adhesives and resin cements used in study

Trademark	Product	Batch #	Composition*	Manufacturer
Adper Scotchbond Universal Adhesive	Total-Etch, Selective Etch, Self-Etch adhesive	1314900467	MDP Phosphate Monomer, dimethacrylate resins, HEMA, Vitrebond™ Copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St. Paul, MN, USA
Adper Scotchbond Universal DCA	Dual Cure Activator	1312701068	Sodium toluene sulfinate, ethanol	3M ESPE, St. Paul, MN, USA
RelyX ARC	Conventional resin cement	1309200341	*Base paste*: Bis GMA,TEGDMA, Benzoyl peroxide *Catalyst paste*: Bis GMA, TEGDMA, photoinitiator system, amine, peroxide, zirconia/silica filler 67.5% by weight	3M ESPE, St. Paul, MN, USA
RelyX U200	Self-adhesive resin cement	491941	*Base paste*: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives *Catalyst paste*: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	3M ESPE, St. Paul, MN, USA
Maxcem Elite	Self-adhesive resin cement	4771220	Uncured methacrylate ester monomers, non-hazardous inert mineral fillers, ytterbium fluoride, activators, stabilizers, colorants	Kerr, Orange, CA, USA

*According to the manufacturer.

following 5 groups (n=18) on the basis of the conditioning agents used: distilled water, 5.25% sodium hypochlorite (16), 25% polyacrylic acid (13), 2% digluconate chlorhexidine (11) and 23 ppm silver nanoparticles dispersion (7). The post spaces were prepared for cementation using 5ml of each irrigation solution for a period of 60 s.

After irrigation, the specimens were divided by drawing lots into the following 3 subgroups (n=6), according to the luting procedure used:

SBU group: After application of the irrigation solution, the post spaces were dried with sterile paper points then ScotchbondTM Universal Adhesive (3M ESPE) was applied along with Scotchbond™ Universal Dual Cure Activator. One drop of each solution was mixed for 5 s and then the preparation was applied actively to the post space with microbrush for 20 s. The adhesive was gently air dried for approximately 5 s to allow evaporation of the solvent, and light polymerized with a light-emitting diode (LED) (Dabi Atlante, Ribeirão Preto, SP, Brazil) for 10 s. The RelyX ARC conventional dual cure resin cement (3M ESPE) was mixed for 10 s then inserted into the root canal with a #15 endodontic file. The resin cement was also applied to the post surface before it was brought into position within the post space; any excess cement was removed. The resin cement was light polymerized for 40 s.

U200 group: After application of the irrigation solution, the root canals were dried with sterile paper points. RelyX U200 self-adhesive resin cement (3M ESPE) was manipulated and inserted into the post space with a #15 endodontic file. The cement was also applied to the post surface, the post was properly positioned, and any excess cement was removed. Finally, the resin cement was light polymerized for 40 s.

MCE group: After application of irrigating solution, the root canals were dried with sterile paper points and Maxcem Elite (Kerr) was applied directly in the post space with the aid of a mixing tip. The post was placed in position, mild vibratory movements were made to avoid the possibility of air trapping, and any excess cement was removed. Finally, the resin cement was light polymerized for 40 s.

After the bonding processes were complete, the coronal portions of the teeth were sealed with Z350 XT composite

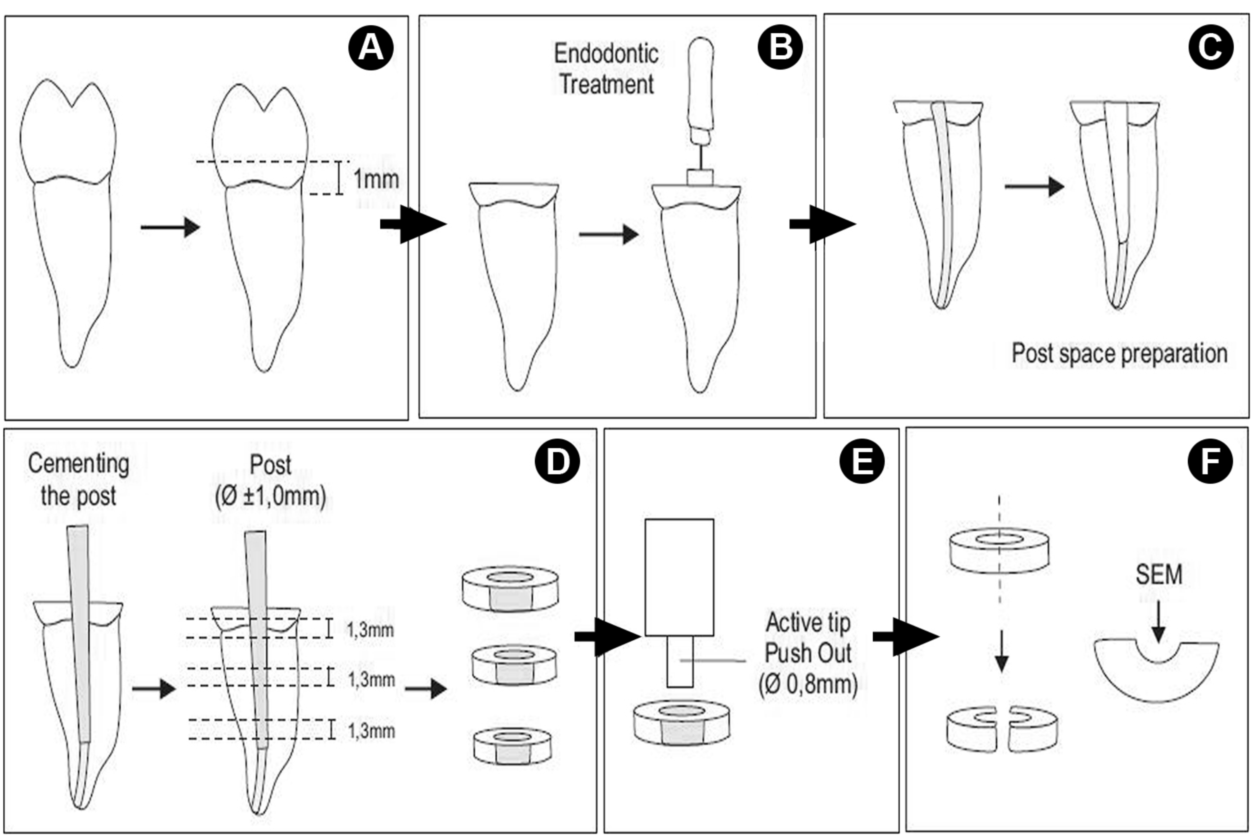

Figure 1. Specimen preparation: (A) Removal of anatomical crown of tooth 1 mm above cementum-enamel junction (B) Endodontic treatment performed with gutta-percha cones and calcium hydroxide cement (C) Intraradicular post space preparation (D) Intraradicular dentin treatment with irrigation solutions and post-luting procedure. After 7 days, slices of ± 1.3 mm were obtained from each third of the canal space to be analyzed (cervical, middle, and apical). (E) Extrusion shear test (push-out) performed with active tip 0.8 mm in diameter and crosshead speed of 0.5 mm/min. (F) Specimens were sectioned to obtain SEM images.

resin (3M ESPE). Then all of the teeth were stored for 7 days (17). The teeth were submitted to thermal cycling (12,000 cycles; 5 to 55 °C; dwell time: 30 s, transfer time: 2 s) using a thermal cycle simulation machine MSCT- 3 Plus (18).

After cycling, the teeth were embedded in an acrylic resin (Clássico, São Paulo, SP, Brazil) and sectioned perpendicular to their long axis with a low-speed diamond saw under water cooling. An Isomet 2000 (Buehler) was used to obtain one slice of approximately 1.3 mm thickness from each third being analyzed (cervical, middle, and apical). The thickness was measured using a digital caliper (Mitutoyo, Tokyo, Japan) and the coronal side was marked with insoluble ink. Each specimen was connected to a fluid infiltration system (Flodec machine; De Marco Engineering) in order to measure the permeability of post-cement-dentin interface, and was kept under a constant deionized water pressure of 140 cm (2 psi). The movement of the bubble inside a capillary glass (inside diameter, 0.83 mm; outside diameter, 4 mm; detectable volume by step, 2.71 nL) in the Flodec system was continuously recorded for 5 min. The last 3 min were used to calculate the interface permeability for each experimental condition. The displacement in mm was measured and converted into µL/min (19).

After the fluid infiltration test, the push-out bonding strengths were measured using a universal testing machine (Model DL3000; EMIC, São José dos Pinhais, PR, Brazil). The post segment was loaded with a cylindrical plunger (0.8 mm in diameter), which was centered on the post segment without any contact with the surrounding dentin surface. A load was applied to the plunger with a universal testing machine in an apical-to-cervical direction. It was tested at a crosshead speed of 0.5 mm/min until the post was dislodged. The push-out bond strength was calculated for each specimen, using the following formula: $Bs=N/2\pi rh$. Bond strength (Bs) equal to maximum force (N) divided by area ($2\pi rh$), where (r) is radius, (h) is the post height, and (π) is 3.14.

After the bond strength test, the specimens were sectioned to expose the adhesive interface, coated with gold (Q150T; Quorum Technologies). The specimens were also observed using scanning electron microscopy (EVO-LS15; ZEISS) to characterize the dentinal structure after bond failure.

Bond strength and interface permeability data were submitted to normality test and the means were compared by 3-way ANOVA for repeated measures (irrigation solution, adhesive technique and root third) and PLSD Fisher test ($\alpha=0.05$).

Results

Push-out Bond Strength

The results of the 3-way ANOVA for bond strength showed differences among the adhesive materials ($p<0.0001$), the irrigation solutions ($p=0.0009$), the root thirds ($p<0.0001$), and the interaction of between these factors ($p<0.05$).

In Table 2, referring to the SBU group, it can be observed there is no statistically significant difference between irrigation solutions in the cervical third ($p=0.34$) of the root. In middle and apical thirds, in general, the highest values for push-out bond strength were found for Ag NPs solution. In these same section, chlorhexidine showed the lowest bond strength values, and there were no statistically significant differences for NaOCl ($p=0.05$ in middle third and $p=0.28$ in apical third). A decrease in the push-out bond strength values in the cervical to apical direction was found for all groups ($p<0.05$).

For the U200 group (Table 3), in general, the highest bond strength values were found for the NaOCl solution. There were no statistically significant differences for distilled water, chlorhexidine and Ag NPs for all experimental conditions analyzed ($p=0.05$). The lowest values were found for polyacrylic acid for all thirds analyzed. As was observed with the UBS group, there was a decrease in bond strength values in the cervical to apical direction for all groups ($p=0.05$).

For MCE group (Table 4), the highest bond strength values were found for the cervical third, with a statistically significant difference for the middle and apical thirds, independent of the irrigation solution used ($p<0.05$). In the cervical third, the highest push-out bond strength values were found for the control group (distilled water) with a significant difference between this and the other solutions ($p<0.05$). In the middle third, the highest values were found for the Ag NPs solution, with a significant difference found only for NaOCl solution ($p=0.04$). In the apical third, the highest values were found for chlorhexidine, with no significant difference for NaOCl ($p=0.15$).

Sealing Ability

Three-way ANOVA for sealing ability showed significant differences among the adhesive materials ($p<0.0001$), the irrigation solutions ($p=0.0001$), the thirds ($p<0.0001$), and the interaction between these factors ($p<0.0009$).

For SBU group (Table 2), the cervical third showed lower permeability than middle and apical thirds ($p<0.05$) for all treatments. Higher permeability was found in the apical third in all treatment groups, except for the group treated with polyacrylic acid. There was no statistically significant difference found between the middle and apical thirds ($p>0.05$) only for polyacrylic acid. Analysis of the cervical third revealed the lower permeability in the group treated with Ag NP, but statistical significance was only achieved for the group treated with polyacrylic acid ($p=0.0027$).

In the middle third, lower permeability was observed for Ag NP, with no statistically significant difference versus distilled water ($p=0.2459$). Chlorhexidine was the treatment that showed significantly higher permeability ($p<0.05$). In the apical third, the Ag NP showed significantly lower permeability values ($p<0.05$).

For the U200 group (Table 3) and the comparison between the thirds, an increase in permeability was noted in the cervical apical direction. The cervical third showed the lowest permeability. There was no statistically significant difference between the cervical and middle thirds when distilled water was used for irrigation. There was no statistically significant difference between the cervical third and the middle and apical thirds when polyacrylic acid was used for irrigation ($p>0.05$). In general, the highest permeability values were found in the apical third. In comparing the various irrigation solutions for the cervical third, use of the Ag NP solution resulted in lower permeability values. However, these values were only significant different from those obtained for the polyacrylic acid group ($p=0.0093$). In the middle third, the lowest permeability values were obtained for chlorhexidine.

Table 2. Push-out bond strength [BS] (MPa) and sealing ability [SA] (μL/min) in designated thirds of intraradicular dentin treated with different irrigation solutions in the SBU group.

	Third	Distilled water	Chlorhexidine	Sodium hypochlorite	Polyacrylic acid	Silver nanoparticles
BS	Cervical	4.72 (1.22) Aa	6.12 (2.37) Aa	7.62 (3.19) Aa	5.03 (2.62) Aa	6.63 (1.85) Aa
	Middle	3.27 (0.92) Ba	0.24 (0.27) Bc	1.52 (1.10) Bbc	1.58 (1.13) Bb	3.01 (1.53) Ba
	Apical	0.27 (0.29) Cb	0.03 (0.08) Bb	0.65 (0.61) Bab	1.54 (1.34) Ba	1.76 (1.56) Ba
SA	Cervical	12.29 (2.67) Aab	8.99 (1.63) Aa	7.98 (1.23) Aa	19.89 (22.64) Ab	3.33 (1.30) Aa
	Middle	35.74 (7.98) Ba	72.92 (13.76) Bc	54.77 (7.10) Bb	59.61 (11.50) Bb	29.96 (6.66) Ba
	Apical	91.29 (16.90) Cbc	104.81 (22.67) Cc	97.36 (32.10) Cc	73.27 (13.68) Bb	52.00 (8.73)Ca

Different capital letters in columns and lower case letters in rows are significantly different (5%).

Table 3. Push-out bond strength [BS] (MPa) and sealing ability [SA] (μL/min) in designated thirds of intraradicular dentin treated with different irrigating solutions in the U200 group.

	Third	Distilled water	Chlorhexidine	Sodium hypochlorite	Polyacrylic acid	Silver nanoparticles
BS	Cervical	5.70 (2.65) Aab	6.21 (2.89) Aab	7.73 (2.44) Aa	3.33 (3.16) Ab	7.28 (2.67) Aa
	Middle	3.75 (1.22) Aa	3.80 (1.00) Ba	4.42 (0.80) Ba	1.17 (0.61) Bb	3.44 (2.46) Ba
	Apical	2.34 (0.97) Ba	0.89 (0.68) Cb	2.01 (1.47) Cab	0.86 (0.76) Bb	1.29 (0.75) Bab
SA	Cervical	44.89 (7.19) Aab	11.81 (5.00) Aa	31.73 (3.35) Aab	79.98 (112.53) Ab	10.42 (2.70) Aa
	Middle	49.95 (10.42) Aab	46.54 (8.75) Ba	47.36 (10.57) Ba	67.42 (11.92) Ab	47.16 (34.71) Ba
	Apical	85.86 (18.22) Bab	74.13 (20.54) Cab	89.48 (14.38) Cb	78.71 (22.82) Ab	51.44 (7.59) Ba

Different capital letters in columns and lower case letters in rows are significantly different (5%).

Table 4. Push-out bond strength [BS] (MPa) and sealing ability [SA] (μL/min) in designated thirds of intraradicular dentin treated with different irrigating solutions in the MCE group.

	Third	Distilled water	Chlorhexidine	Sodium hypochlorite	Polyacrylic acid	Silver nanoparticles
BS	Cervical	4.63 (2.23) Aa	2.93 (1.39) Ab	2.23 (0.75) Ab	2.48 (0.59) Ab	2.63 (1.06) Ab
	Middle	0.93 (0.62) Bab	1.11 (0.86) Bab	0.68 (0.63) Bb	1.07 (0.44) Bab	1.42 (0.33) Ba
	Apical	0.63 (0.42) Bb	1.35 (0.93) Ba	0.84 (0.71) Bab	0.46 (0.39) Bb	0.63 (0.32) Bb
SA	Cervical	25.43 (7.82) Aa	50.48 (9.09) Ab	58.74 (8.42) Ab	55.98 (8.73) Ab	54.72 (7.00) Ab
	Middle	94.51 (23.03) Bb	89.86 (20.70) Bb	83.91 (18.02) Bb	84.04 (23.60) Bb	46.91 (7.26) Aa
	Apical	108.20 (35.40) Bb	94.24 (27.75) Bb	112.63 (38.35) Bb	118.70 (29.32) Cb	57.76 (8.05) Aa

Different capital letters in columns and lower case letters in rows are significantly different (5%).

These values were significantly different only from those obtained for polyacrylic acid (p=0.2773). In the apical third, the lowest permeability values were again found for the Ag NP solution. A statistically significant difference was evidenced between Ag NP and both NaOCl and polyacrylic acid (p=0.001 and p=0.0037, respectively).

For the MCE group (Table 4), only the Ag NP treatment resulted in similar permeability for all thirds (p>0.05). Following irrigation with the other solutions, the cervical third showed significantly lower permeability than the middle and apical thirds (p<0.05). In the cervical third, irrigation with distilled water resulted in lower permeability values. These values were significantly different from those obtained for the other solutions (p<0.05). In the middle and apical thirds, treatment with the Ag NPs solution resulted in a significantly lower permeability compared to other treatments (p<0.05).

Discussion

In this study, the bond strength and the sealing ability of the glass fiber post bonding interface in different thirds of the intraradicular dentin was evaluated after being subjected to different irrigation solutions and luting strategies. The interface permeability is as important as the bond strength; even if a material has relatively low bonding strength, it could still be effective in preventing microleakage. Based on this rationale, both tests were performed in this investigation evidencing overall coherent outcomes.

The irrigation solutions evaluated are commonly used in dentistry mainly because of their antibacterial properties and/or their ability to condition the dentin. As root canal offer technical difficult of adequate mechanical assessment, the adjunctive use of a chemical agent is interesting to assure minimal or no microorganisms. The results showed that the solutions both interfered with the bond strength and the interface permeability, resulting in the rejection of the first null hypothesis of the study.

The 23 ppm silver nanoparticles dispersion is a solution whose ability to reduce bacterial adhesion and prevent biofilm formation has already been proven. It could potentially be used as an antimicrobial solution in preparation of the root canal for endodontic treatment (7). However, the effect of this solution on the adhesion between resin materials and intraradicular dentin has not been evaluated. The 23 ppm Ag NPs dispersion could be particularly useful in cases where removal of the conduit is not performed in the same session of intraradicular post cementation. The results revealed that for the SBU group, which was treated with Scotchbond™ Universal self-etching adhesive and RelyX ARC conventional resin cement, and the U200 group, which was treated with RelyX U200 self-adhesive resin cement, use of the Ag NP solution resulted in higher bond strengths and lower permeability (Tables 2 and 3). No infiltration of Ag Np solution was noted in many specimens and when it was seen, the total amount of penetration was limited. There was no statistically significant difference in bond strength and interface permeability when Ag NP solution was compared to distilled water (control) in the cervical and middle thirds of the SBU group (Table 2) and in all thirds of the U200 group (Table 3), probably there would be a clinical benefit to using the Ag NP solution before the bond process because of its antibacterial properties and because it would not interfere with the adhesion of the glass fiber post. Comparing the scanning electron microscopy (SEM) images of distilled water (Fig. 2) and Ag NP solution (Fig. 3) one can observe the same pattern of intraradicular dentin. Thus, the bonding process following irrigation with Ag NP solution likely occurred in the same way as it did for the control group. Incorporation of these particles into orthodontic adhesives was previously studied and was proven not to affect bond strength, while still maintaining an antimicrobial effect (7,15).

The same antibacterial effect has also been found with prior application of digluconate chlorhexidine solution (2). Chlorhexidine is also a commonly used because of its antimicrobial properties, substantivity and its positive effect on the longevity of the bond interface between the adhesives composite and dentin (10). However, the results showed that for SBU groups (middle and apical thirds) and U200 group (apical third), specimens treated with chlorhexidine solution presented lower bond strength values and higher permeability values (Tables 4 and 5). These findings corroborated those of previous studies (5). The interface permeability of these specimens was significantly worse. A reaction between the phosphate in dentin and chlorhexidine results in precipitation, which could be responsible for the observed decrease in bond strength (Fig. 4) (20). These precipitates could create a physical barrier, thus reducing the interaction between the luting materials and the dentin surface (9).

The effect of the application of different irrigation solutions was dependent upon the type of adhesive material used. NaOCl, for example, had no significant effect on bond strength and interface permeability as compared to the control group (distilled water) for the U200 group only. For the other adhesive materials, this effect was not so clearly observed (Tables 2 and 4). NaOCl is one of the most popular irrigation solutions and it widely used because of its antibacterial activity and its ability to dissolve necrotic tissue debris (7). Unfortunately, it is also apt to cause dissolution of collagen through breaking of the carbon bonds and disorganization of the primary structure of the protein, resulting in degeneration of the dentin (6). NaOCl that is in contact with the dentin surface, decomposes into sodium, chloride and oxygen which may cause inhibition of polymerization of the resin materials (12). In the SEM image of the tooth that was previously irrigated with NaOCl (Fig. 5), a resin cement layer is evident on the surface of the intraradicular dentin. Previous studies evaluating irrigation with 5% NaOCl, showed that there was a decrease in the bond strength between the resin cements and the dentin (6). Rinsing of the solution from the deepest regions (middle and apical thirds) is more difficult, and could have resulted in increased contact time between the NaOCl and the

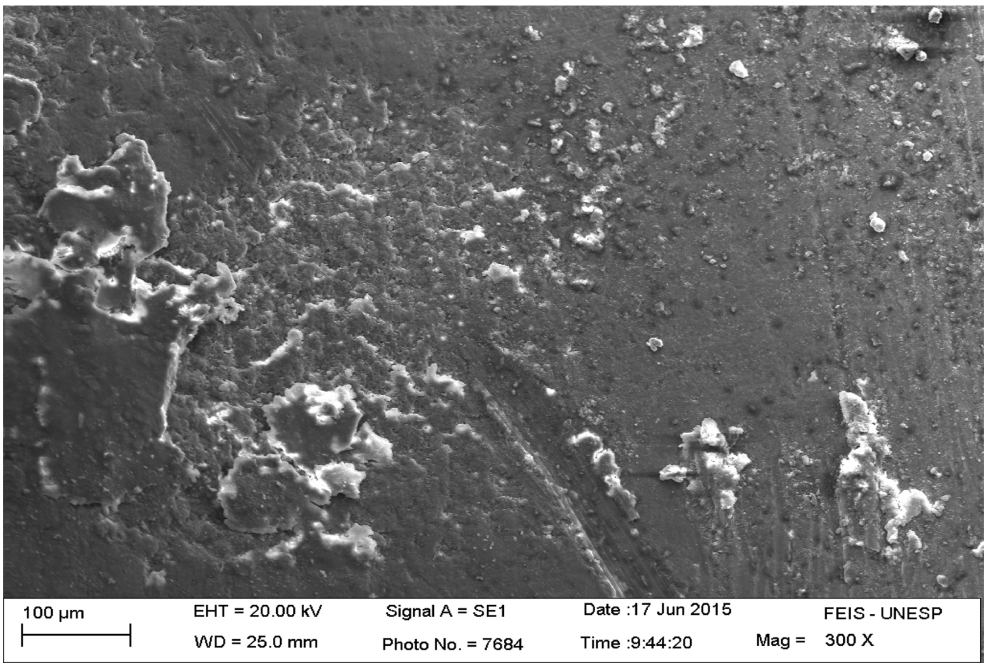

Figure 2. Representative specimen of intraradicular dentin irrigated with distilled water. Obtained from the SBU group in the cervical third.

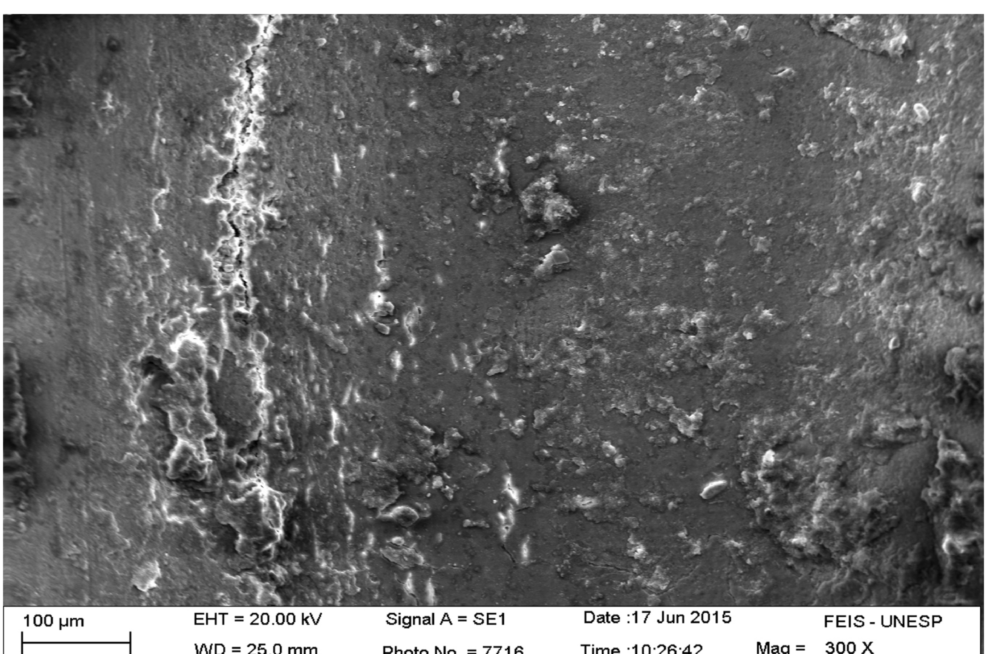

Figure 3. Representative specimen of intraradicular dentin irrigated with silver nanoparticles solution. Obtained from the SBU group in the cervical third.

intraradicular dentin in these regions (3). Demiryürek et al. (3) observed that increased application time of NaOCl resulted in a progressive decrease in bond strength due to the residual effects of the solution on the dentin.

The effect of polyacrylic acid application was also dependent on the resin material used. In this case, some slices presented higher infiltration. Polyacrylic acid is commonly used in combination with glass ionomer cement to improve the interaction between this cement and the substrate (21). When polyacrylic acid is applied to dentin, it removes the smear layer while maintaining the smear plugs within the dentinal tubules. Previous studies have shown

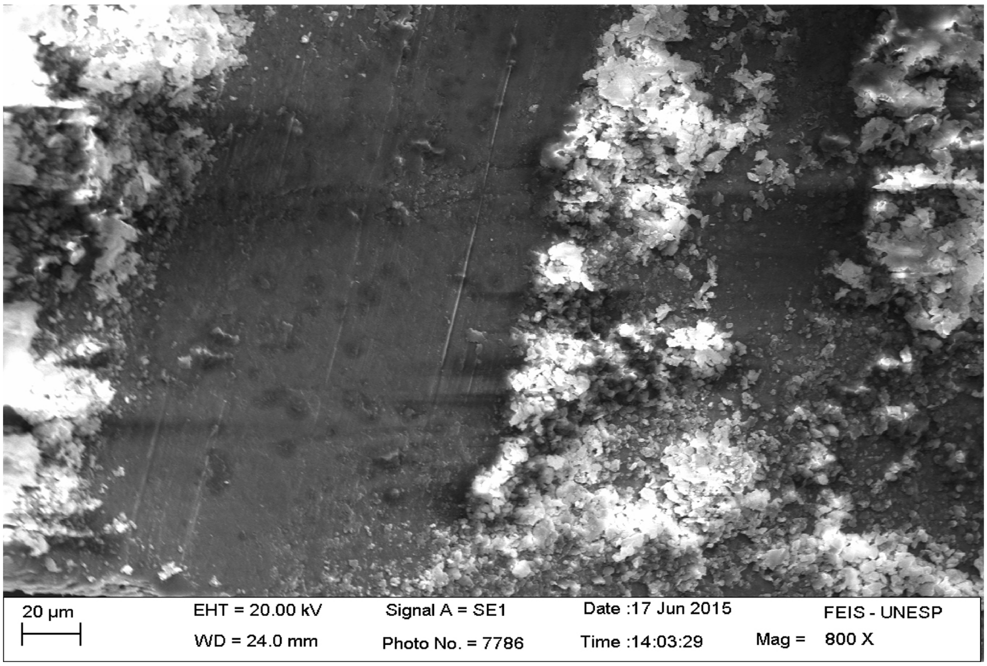

Figure 4. Representative specimen of intraradicular dentin irrigated with chlorhexidine. Obtained from the U200 group in the apical third.

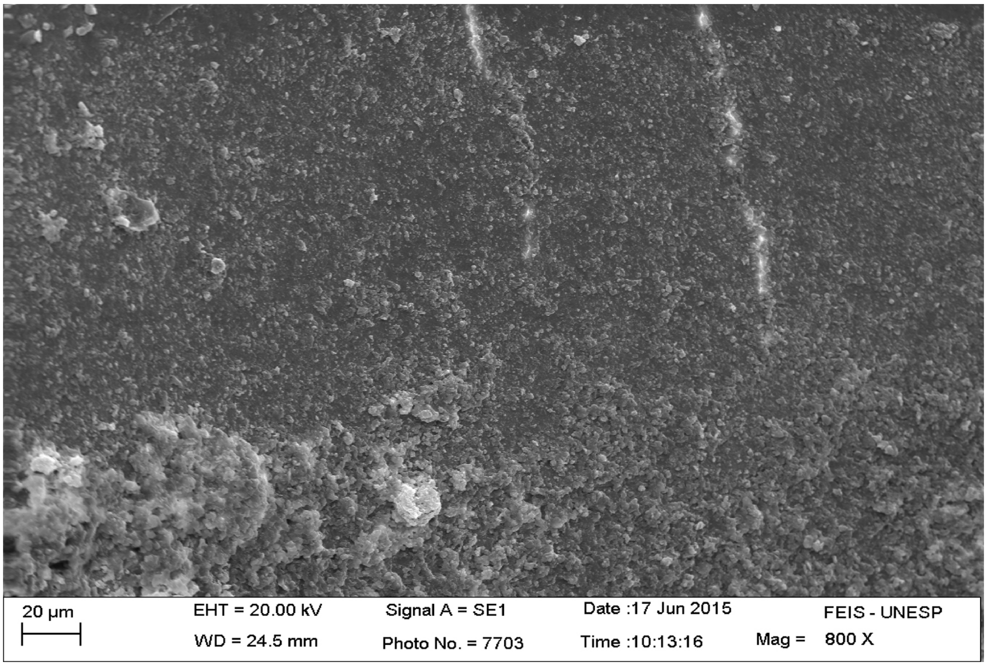

Figure 5. Representative specimen of intraradicular dentin irrigated with sodium hypochlorite. Obtained from the U200 group in the middle third.

that the effect of this acid is material-dependent (13). In this study, in general, irrigation with polyacrylic acid seems to have contributed to the improvement in bond strength values, especially for the U200 group (Table 3). Figure 6 is a representative sample of the U200 group that has been conditioned with polyacrylic acid. In this image, dentinal tubules with resin cement on their surface can be observed. In comparing the adhesives used, specimens in the MCE group showed, in general, lower bond strength values and higher permeability values than specimens in the SBU and U200 groups. These findings resulted in rejection of second null hypothesis. The Maxcem Elite resin cement used in MCE group contains HEMA (2-hydroxyethyl methacrylate) which makes the material more prone to water absorption. The increased water permeability accelerates hydrolytic degradation of the cement (4). Owing to the lower bond strength values and the higher permeability values found for this group, the effect of the different irrigation solutions was insignificant effect for this material (Table 4). The U200 group utilized the RelyX U200 self-adhesive resin cement, which offers, according to the manufacturer, an additional monomer, new rheology, and optimization of the filler particles when compared to its predecessor, RelyX Unicem. Furthermore, the decreased hydrophilicity of the RelyX U200 resin cement as compared to the Maxcem Elite resin (MCE group) seemed to result in more favorable bonding results. This suggests that hydrophilic components deteriorate the bond strength, and result in higher water absorption (19). In addition, the Scotchbond™ Universal adhesive used in the SBU group has an acidic pH which is able to partially demineralize dentin, leaving a significant number of hydroxyapatite crystals surrounding the collagen fibrils (22). This adhesive presents MDP in its composition that interacts chemically with the hydroxyapatite of dentin (23), and forms a stable nanolayer which could result in a stronger bond at the adhesive interface (24).

In comparing the different thirds of the intraradicular dentin, in general, higher bond strength values and lower interface permeability values were found for the cervical third, with a statistically significant difference for the other thirds, rejecting the third null hypothesis of this study (Tables 2,3 and 4). The SBU group used the Scotchbond™ Universal Adhesive with the Scotchbond™ Universal Dual Cure Activator and RelyX ARC dual-cured resin cement. According to the manufacturer, addition of the optimizer allows the adhesive system has compatibility with both dual and the chemical resin cements, enabling polymerization of the resin cement, but does not change the adhesive that is exclusively photoactivated on a dual adhesive. Thus, at the time of photoactivation, the light intensity is decreased during the root canal, and the cervical third is the portion that more receives light irradiation, which would prejudice the polymerization of the adhesive in the deepest thirds (1). The self-adhesive resin cements, even though dual activation, their values were also decreased in the course of intraradicular thirds (Tables 3 and 4). In the deepest thirds (middle and apical), which could not have received an adequate light energy when compared to the cervical

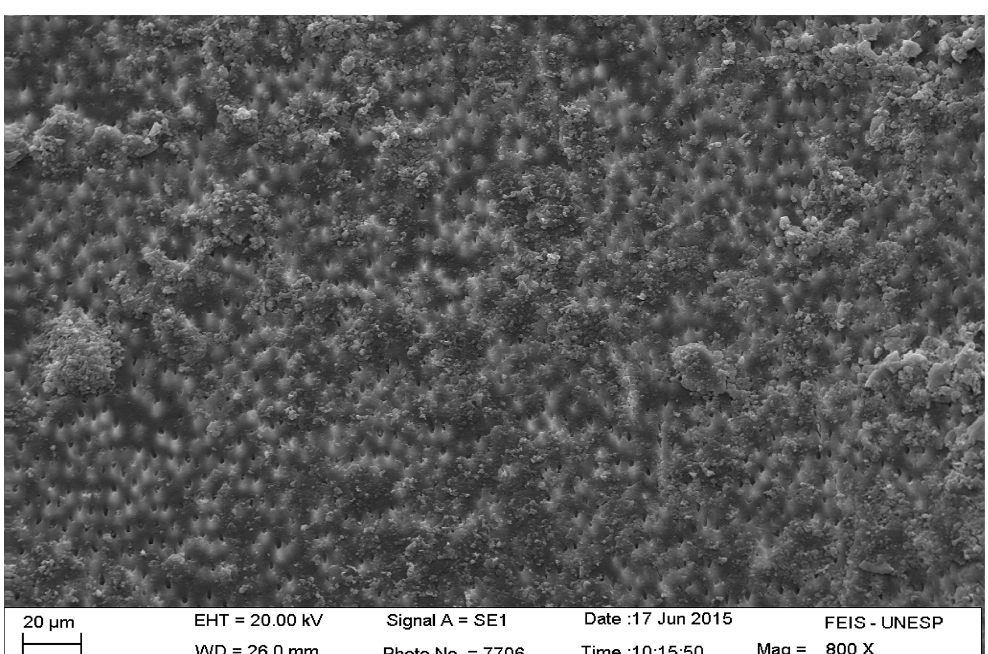

Figure 6. Representative specimen of intraradicular dentin irrigated with polyacrylic acid. Obtained from the U200 group in the middle third.

third, these cements depend almost exclusively on chemical activation of the material. However, when comparing dual conventional resin cement and self-adhesive cement, when performed only chemical activation of the materials, the self-adhesive cement had the lowest bond strength values (25). This finding may be related to the reduced degree of conversion of the resinous monomers, when cement photoactivation is not performed (26).

Thus, the results of this study show greater difficulties in obtaining a reliable bonding process with some of the luting materials in the apical region. Further studies are needed in order to improve the adhesive procedures. A shorter, more conservative preparation of the intraradicular area, ensuring adhesion of the cervical third, may be indicated. The use of glass ionomer cements instead of composite resin based cements could facilitate all process of cementation (27,28). Furthermore, the use of substances with adequate antimicrobial properties that do not cause interference with the bonding process is recommended. Ag NP solution appears to be a viable option, but additional studies, particularly evaluating the long-term effects, are required.

Based on the results obtained in this study, it was concluded that the bond strength and interface permeability of luting cement agents is influenced by the previous application of irrigation solutions and the intraradicular depth analyzed. The 23 ppm silver nanoparticles dispersion is a viable treatment option, since no negative effects on the adhesion and interface permeability values between the glass fiber posts and the intraradicular dentin were observed.

Resumo

O objetivo deste trabalho foi avaliar a resistência de união e permeabilidade da interface de pinos de fibra de vidro nos diferentes terços da dentina intrarradicular (cervical, médio e apical) submetidos a diferentes agentes irrigantes. Noventa dentes unirradiculares humanos foram submetidos ao tratamento endodôntico e foram divididos em cinco grupos de acordo com os agentes irrigantes: água destilada, hipoclorito de sódio 5,25%, ácido poliacrílico 25%, clorexidina 2% e dispersão de nanopartícula de prata à 23 ppm. Os grupos foram divididos em 3 subgrupos (n=6) de acordo com a técnica adotada para cimentação adesiva dos pinos de fibra de vidro: Grupo SBU: sistema adesivo ScotchbondTM Universal + cimento resinoso RelyX ARC; Grupo U200: cimento resinoso autoadesivo RelyX U200; Grupo MCE: cimento resinoso autoadesivo Maxcem Elite. A resistência de união e a permeabilidade da interface foram mensurados em diferentes áreas da dentina intrarradicular. Os dados foram submetidos a ANOVA e teste de Fisher (α=0,05). Amostras representativas foram levadas à microscopia eletrônica de varredura. A solução de nanopartícula de prata apresentou os maiores valores de resistência de união em todos os terços analisados para o grupo SBU. No grupo U200, os maiores valores foram encontrados para a solução de hipoclorito de sódio, com diferença para o ácido poliacrílico. Observou-se uma diminuição nos valores de resistência de união na direção cérvico-apical para o grupo MCE e o mesmo comportamento foram encontrados para os demais grupos. Com relação a permeabilidade da interface, o uso da solução de nanopartícula de prata resultou em menores valores nos terços cervical e apical. Houve diminuição dos valores de resistência de união no sentido cérvico-apical. As diferentes soluções irrigantes, bem como a profundidade intrarradicular influenciaram a resistência de união e permeabilidade da interface dos materiais adesivos ao substrato dentinário. A nanopartícula de prata pode ser utilizada como agente irrigador do conduto radicular previamente à cimentação de pinos de fibra de vidro.

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