Effect of Bleaching and $Ca(OH)_2$ Dressing on the Bond Strength of Fiberglass Posts to Root Dentine

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Abstract

Objective The purpose of this study was to evaluate the effect of the intracoronary bleaching and calcium hydroxide ($Ca(OH)_2$) dressing use, on the bond strength (BS) of fiberglass posts to root dentine.

Materials and Methods After root canal filling of 40 bovine incisors, a 2-mm thick cervical plug was fabricated 2 mm below the cementum–enamel junction. Seven days later, teeth were randomly distributed into four groups (n = 10), as follows: G1 no bleaching, followed by immediate post cementation; G2 bleaching and immediate post cementation; G3 bleaching, dressing with $Ca(OH)_2$ for 7 days, and post cementation; and G4 bleaching, no dressing, and post cementation after 7 days. The roots were transversally cut into 1-mm thick slices to perform the push-out test (0.5 mm/min). Failure modes were assessed under scanning electron microscopy.

Statistical Analysis The analysis of variance (two-way ANOVA) was performed, followed by the supplementary Tukey multiple comparison test (α = 5%).

Results No significant difference for BS was observed among groups. Considering the different root thirds, G1 had higher BS values for the cervical third in comparison with the apical one ($p < 0.05$). The most frequent failure modes were adhesive between cement/dentine (G1); cohesive at the post (G2 and G3), and mixed (G4).

Conclusions The BS of the fiberglass posts to root dentine was not affected by the intracoronary bleaching and the use of $Ca(OH)_2$ dressing.

Keywords
- bond strength
- calcium hydroxide
- fiberglass posts
- intracoronary bleaching

Introduction

The discoloration of nonvital teeth, especially in aesthetic areas, cause great social and psychological discomfort.[1] Such chromatic change may occur due to the presence of noxious by-products after pulp tissue necrosis, which may penetrate into dentinal tubules and discolor the surrounding dentine.[2] Furthermore, the diffusion of blood components into dentinal tubules, incomplete removal of pulp tissue, or the presence of endodontic filling materials close to cervical dentine may also promote tooth discoloration.[2] In these cases, the intracoronary bleaching is considered a conservative treatment option in comparison with other more invasive treatments, such as full crowns or dental veneers.[3]

The chemical agents commonly used to bleach discolored teeth are hydrogen peroxide (H_2O_2), carbamide peroxide, and sodium perborate.[4] The active ingredient is H_2O_2, which acts as a strong oxidant agent through the formation of free radicals (oxygen, perhydroxyl, and hydroxyl).[4] The tooth whitening effect of this oxidant agent has been attributed to its ability to break longer-chained chromophores into shorter-chained colorless compounds.[2]

However, the use of bleaching agents has been related to undesired effects to hard dental tissues, such as dentinal morphology and chemical composition changes.[5] In addition, bleaching agents promote reduction of dentinal microhardness,[6] reduction of the bond strength (BS) of restorative materials to dentine,[7,8] and external cervical resorption.[9]

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To reduce the occurrence of external root resorption, calcium hydroxide ($Ca[OH]_2$) dressing has been used on the pulp chamber after completion of bleaching procedures.[10] The diffusion of hydroxyl ions within the dentinal tubules promote a higher pH in dentine and prevents progression of external resorption.[4] Also, some studies reported that the use of $Ca(OH)_2$ dressing after bleaching may reduce,[10] or not increase, the harmful effect of the bleaching agent in the posterior use of resinous materials.[11]

Researchers have observed that intracoronary bleaching reduces the adhesion of resinous materials to the dentine, especially when the adhesive procedure is performed immediately after the bleaching treatment.[7] The presence of residual oxygen in the dentine may inhibit the full polymerization of resinous materials and jeopardize adhesion.[12] Consequently, after bleaching treatment, a waiting period is recommended to perform definitive restorations.[8-13]

However, the literature has few studies that evaluate the bond strength (BS) of restorative materials to root dentine after intracoronary bleaching process in endodontically treated teeth. Since these teeth usually have insufficient coronary structures to retain the restoring material, the use of intraradicular posts becomes necessary to support the crown restoration.[14] In these cases, the use of a fiberglass post combined with adhesive cementation is appropriate because it presents an elasticity module that is similar to the dentine, which provides a better load distribution along the root wall.[15]

The BS reduction of restorative materials to dentine may compromise the clinical longevity of restorations, with occurrences of infiltrations, discoloration, secondary caries, and root canal system recontamination.[14,15] Additionally, there is also the possibility of detachment of the cement/post system from the root canal space, which may result in the extrusion of the post, or root fractures.[16] Therefore, laboratory mechanical tests should be performed to assess the BS of adhesive systems to the dental structure besides testing new products and investigating experimental variables.[17]

The present study evaluated the effect of intracoronary bleaching and the use of the $Ca(OH)_2$ dressing on the BS of fiberglass posts to root canal dentine by push-out test. The null hypotheses tested was that the intracoronary bleaching and the $Ca(OH)_2$ dressing have no influence on the BS of cement/post system to root dentine, regardless of the post-bleaching waiting time or the root canal third to be assessed.

Materials and Methods

Sample Selection and Preparation

Forty bovine incisors were selected based on the radiographic analysis of 200 teeth. The inclusion criteria were: recently extracted teeth of animals aged between 24 to 48 months; roots anatomically similar in size and shape; single and straight root canals; and fully formed apex. Root canals with larger diameter were discarded from the final sample. First, the teeth were cleaned with periodontal curettes (Hu-Friedy; Chicago, Illinois, United States) and kept in 0.1% thymol solution for disinfection until use. Next, the crowns were transversely cut at 2 mm bellow the cementoenamel junction (CEJ) in coronal direction, using a double-sided diamond disc (KG Sorensen; Cotia, São Paulo, Brazil) under copious air/water spray cooling. The root canal access was complemented with a conical truncated diamond tip (FG 2082; KG Sorensen).

Endodontic Treatment

The root canal length was determined by introducing a size 15 Flexofile instrument (Dentsply Maillefer; Tulsa, Oklahoma, United States) into the canal to the point of displaying its tip at the apical foramen. The working length (WL) was then obtained by subtracting 1 mm from the root canal length. The same operator performed the root canal preparation using nickel-titanium rotary instruments (ProTaper Universal; Dentsply Maillefer) up to the F5 file. Patency of the apical foramen was maintained with the use of a size 15 Flexofile instrument (Dentsply Maillefer). Between the uses of each instrument, the root canals were irrigated with 2 mL of 1% NaOCl solution and, at the end, with 3 mL of 17% ethylenediaminetetraacetic acid (EDTA), followed by a final irrigation with 3 mL of 1% NaOCl solution. After, root canals were completely dried with absorbent paper points (Dentsply Maillefer) and then, filled with AH Plus sealer (Dentsply DeTrey GmbH, Germany) and gutta-percha cones (F5 main cone and fine points; Dentsply Maillefer) using the hybrid technique of Tagger.

After the thermomechanical compaction, the gutta-percha was removed up to 4 mm below the CEJ (►**Fig. 1A, 1B**) using a heated plugger (Buchanan Plugger 0.06 Taper; SybronEndo Corporation, Orange, California, United States). A 2-mm thickness of zinc oxide/zinc sulfate cement (Citodur; Dori-Dent, Wien, Austria) was placed over the filling material as a temporary restorative material (►**Fig. 1C**). The specimens were kept at 37°C and relative humidity of 100% for 7 days. After this period, the specimens were randomly distributed into four groups (n = 10, ►**Table 1**), as follows: G1 (control group)—no bleaching, immediate fiberglass post cementation; G2—intracoronary bleaching and immediate post cementation (same session); G3—intracoronary bleaching, dressing with $Ca(OH)_2$ paste for 7 days, and then, post cementation; and G4—intracoronary bleaching, no $Ca(OH)_2$ dressing and post cementation after 7 days.

Intracoronary Bleaching

In the samples of G2, G3, and G4, the 35% H_2O_2 bleaching agent (Whiteness HP Maxx; FGM, Joinville, Santa Catarina, Brazil) was used. The different stages of the bleaching agent were mixed in the proportion of three drops in stage 1 (peroxide) to one drop in stage 2 (thickener). After the mixture, a calibrated syringe was used to inject 0.10 mL of the bleaching agent into the access cavity (►**Fig. 1D**). A brush was then used, to create a layer of 1 mm thickness. The material remained in contact with the surrounding dentine walls for 45 minutes. After this period, the bleaching agent was removed through an abundant rinsing with distilled water.

In the G2 samples, the fiberglass posts were cemented immediately after the conclusion of the intracoronary

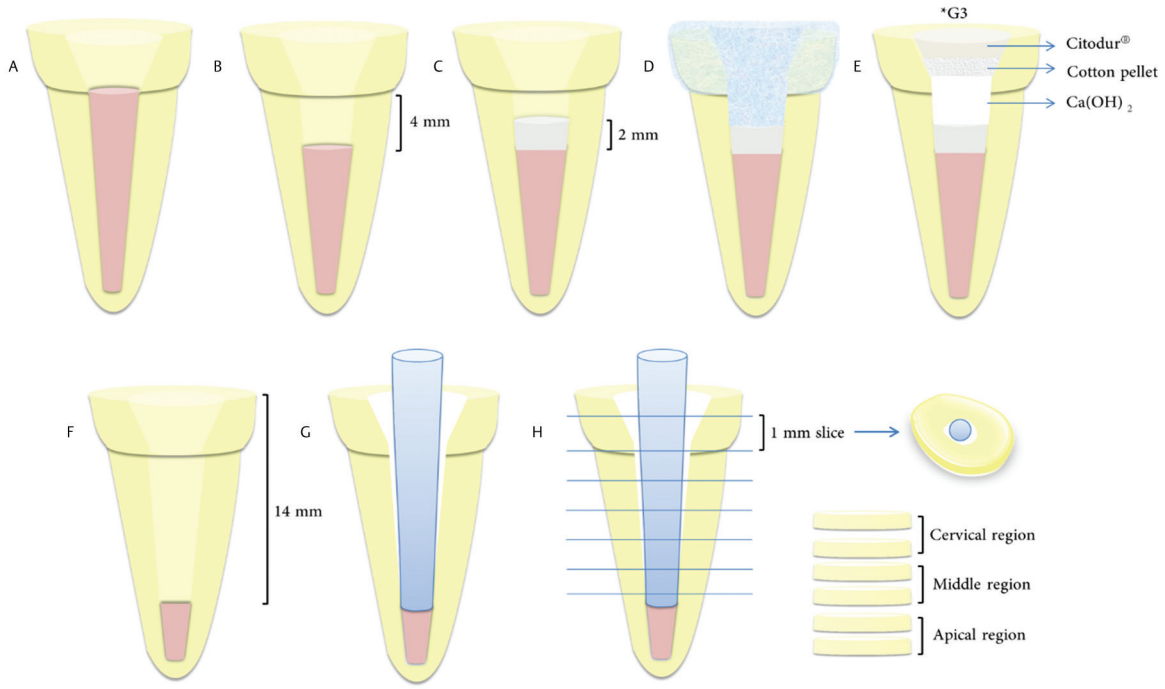

Fig. 1 Diagram of the methodological steps. Completed endodontic treatment (**A**); Gutta-percha removal 4 mm below the CEJ. (**B**) Filling material sealing with 2-mm thick zinc oxide/zinc sulfate cement (**C**). Bleaching procedure (product inserted into the access cavity for 45 minutes) (**D**). Pulp chamber filled with $Ca(OH)_2$ dressing for 7 days (G3 group; **E**); filling material removal up to a length of 14 mm (**F**); fiberglass post cementation (**G**); root sectioned perpendicular to the long post axis (1.0-mm-thick slices obtained from the cervical, middle, and apical thirds (**H**).

Table 1 Description of the experimental groups

Groups	Intracoronary bleaching	Dressing with $Ca(OH)_2$ paste	Fiberglass post cementation
G1 (Control)	No	No	After 7 days of endodontic procedures
G2	Yes	No	Immediate after intracoronary bleaching (same session)
G3	Yes	Yes	After intracoronary bleaching and dressing with $Ca(OH)_2$ paste for 7 days
G4	Yes	No	After 7 days of intracoronary bleaching

bleaching process. In G3, the samples had their pulp chamber filled with $Ca(OH)_2$ dressing (►**Fig. 1E**), which was prepared by mixing 0.712 g of $Ca(OH)_2$ powder (Merck KGaA; Darmstadt, Germany) with 0.4 mL of propylene glycol. The chambers were sealed with Citodur (DoriDent), and the teeth were stored by 7 days at 37°C and 100% relative humidity. After this period, the $Ca(OH)_2$ dressing was removed with distilled water rinsing. In the G4 samples, after the bleaching process, the pulp chamber was filled with cotton pellets and sealed with Citodur (DoriDent), and the teeth were also stored by 7 days at 37°C and 100% relative humidity.

Post Space Preparation and Cementation

The filling material was removed with the aid of Largo drills (Dentsply Maillefer) up to a length of 14 mm (►**Fig. 1F**). At the end of the post space preparation, the root canals were irrigated with distilled water and dried with absorbent paper points (Dentsply Maillefer). Fiberglass posts of double taper (Whitepost DC size 3, FGM), with apical and cervical diameters of 1.25 and 2.0 mm, respectively, were used. Before cementation, the posts were cleaned with alcohol 70% and dried with air jets. Then, the bonding agent (Silano Prosil; FGM) was applied with a microbrush (Cavibrush; FGM) on the post surface. A reaction time of 60 seconds was respected, followed by a careful drying process with air jets. The intraradicular dentine was etched with 37% phosphoric acid (Condac; FGM) for 15 seconds, followed by rinsing with water by 30 seconds. The water excess was removed with a light air jet and absorbent paper points (Dentsply Maillefer), resulting in a lightly moist dentine substrate. The adhesive system (Adper Single Bond; 3M/ESPE, St. Paul, Minnesota, United States) was applied according to the manufacturer's recommendations and photoactivated by 15 seconds, with a 1200 mW/cm^2 LED photo activator (Radii-Cal; SDI, Bayswater, Australia).

To perform post cementation, similar amounts of the base and catalyst pastes of the resinous cement dual (RelyX ARC, 3M/ESPE) were mixed for 10 seconds. The resinous cement was inserted into the canal with a size 40 Flexofile instrument

(Dentsply Maillefer), and then, the post, wrapped in cement, was inserted into the canal and pressed by 40 seconds. After the cement excesses removal, the set was photoactivated for 40 seconds on the incisal, buccal, and lingual faces, totaling 120 seconds by root. Immediately after post' cementation, the samples were placed in distilled water at 37°C and relative humidity of 100% for 24 hours, until the push-out test to be perform (►**Fig. 1G**).

Sample Preparation and Push-out Test

The roots were fixed to a flat metallic surface attached to the cutting machine (Isomet 1000; Buehler, Lake Forest, Illinois, United States), and serially sectioned perpendicular to the post long axis, by using a water-cooled diamond saw (South Bay Technology Inc.; San Clement, California, United States). Dentine slices of 1.0-mm-thick (± 0.1 mm) were obtained from the cervical, middle, and apical thirds of the post (►**Fig. 1H**). Two slices for root third were selected to perform the push-out test.

Each slice was attached to a metallic surface containing a 2.5 mm diameter hole at its center, adapted to the lower portion of the Universal Testing Machine (Instron; Model 4444, Instron, Canton, Massachusetts, United States). A compressive load was applied by using a 1 mm diameter cylindrical plunger attached to the upper portion of the Instron machine. A crosshead speed of 0.5 mm/min was applied until bond failure occurred.

The load at failure (F) recorded in Newtons (N) was divided by the bonded area of the cement post/dentine interface (SL) in mm^2 to obtain the bond strength (BS) in Megapascal (MPa) using the following equation: BS = F/SL. The bonded area represents the lateral surface of a truncated cone and was calculated by the formula of a conical frustum,[18] considering the top and bottom circles of the dislodged bonded assembly along with the height of the slice.

Failure Mode Analysis

For the failure mode analysis, the debonded area was initially assessed under stereomicroscope (SteREO Discovery. V12; Carl Zeiss, Jena, Germany) at 15× to 50× magnifications. The failures were classified according to the following criteria: post adhesive failure (cement-free post); dentine adhesive failure (cement-free dentine); mixed failure (partially free and partially cement-covered dentine); post cohesive failure (post fracture); and dentine cohesive failure (dentine fracture).

The representing samples of each group were prepared for analysis under Scanning Electron Microscope (SEM) (JEOL; JSM 6390 LV, Peabody, MA, USA). The samples were dried, mounted on aluminum stubs, placed in a vacuum chamber and sputter-coated with a gold layer of 300 Aþ (Bal-Tec SCD 005; Bal-Tec Co., Balzers, Liechtenstein). The failure modes analysis was performed under SEM operated at accelerating voltage of 15 kV, at 20×, 50× and 100× magnifications.

Statistical Analysis

Means and standard deviations were calculated, and data homogeneity and normality were tested by Levene's and Kolmogorov-Smirnov's tests, respectively. Data were analyzed by using analysis of variance (two-way ANOVA), and post hoc tests were calculated by using Tukey's multiple comparison test. The significance level for all of the tests was 5% ($a = 0.05$). The failure modes were analyzed by the frequency distribution. The statistical tests were performed with the aid of the SPSS 21 software (IBM SPSS Inc.; Chicago, Illinois, United States).

Results

►**Table 2** shows the results of the push out test. Comparison among groups regarding the BS resulted in no statistically significant difference ($p = 0.44$). Neither the post/root canal third (cervical, middle, and apical), or the interaction between the groups and thirds had significant influence on the results ($p > 0.05$), except for G1 (control group: no bleaching), where the BS in the cervical third had greater value in comparison with the apical third ($p < 0.05$).

The failure mode analysis (►**Table 3**) showed predominance of adhesive failure in the cement/dentine interface in the control group (G1). For G2 and G3 groups, the post cohesive failure occurred more frequently. In G4, the mixed failure was the most frequent one. ►**Fig. 2** presents photomicrographs of the predominant failure mode in each group after the push-out test.

Table 2 Bond strength mean values (MPa) and standard deviation after push-out test, according to the post/dentine areas in each evaluated group

Groups	Post/dentine area		
	Cervical	Middle	Apical
G1	4.72 ± 2.73^{aA}	$3.91 \pm 2.53^{a,bA}$	1.66 ± 1.33^{bA}
G2	3.26 ± 1.48^{aA}	2.58 ± 1.54^{aA}	3.05 ± 1.59^{aA}
G3	3.31 ± 1.54^{aA}	4.22 ± 1.79^{aA}	3.33 ± 1.59^{aA}
G4	3.40 ± 1.66^{aA}	2.37 ± 1.05^{aA}	2.16 ± 1.23^{aA}

Different lowercase letters mean significant statistical difference in the lines (Tukey's $p < 0.05$). Different uppercase letters in the same column mean significant statistical difference (Tukey's $p < 0.05$). G1: no bleaching; G2: bleaching and immediate post cementation; G3: bleaching, post cementation after 7 days of intracanal dressing with $Ca(OH)_2$; G4: bleaching, and post cementation after 7 days.

Table 3 Failure modes (%) distribution after the push-out test in the different groups

Groups	Failure modes				
	Adhesive post/ cement	Adhesive cement/ dentine	Mixed	Cohesive post	Cohesive dentine
G1	13.3	51.6	16.6	18.3	0.0
G2	5.0	25.0	30.0	38.3	1.66
G3	0.0	25.0	21.6	45.0	8.3
G4	3.3	30.0	40.0	25.0	1.6

Note: G1: no bleaching; G2: bleaching, and immediate post cementation; G3: bleaching, post cementation after 7 days of intracanal dressing with $Ca(OH)_2$; G4: bleaching, and post cementation after 7 days.

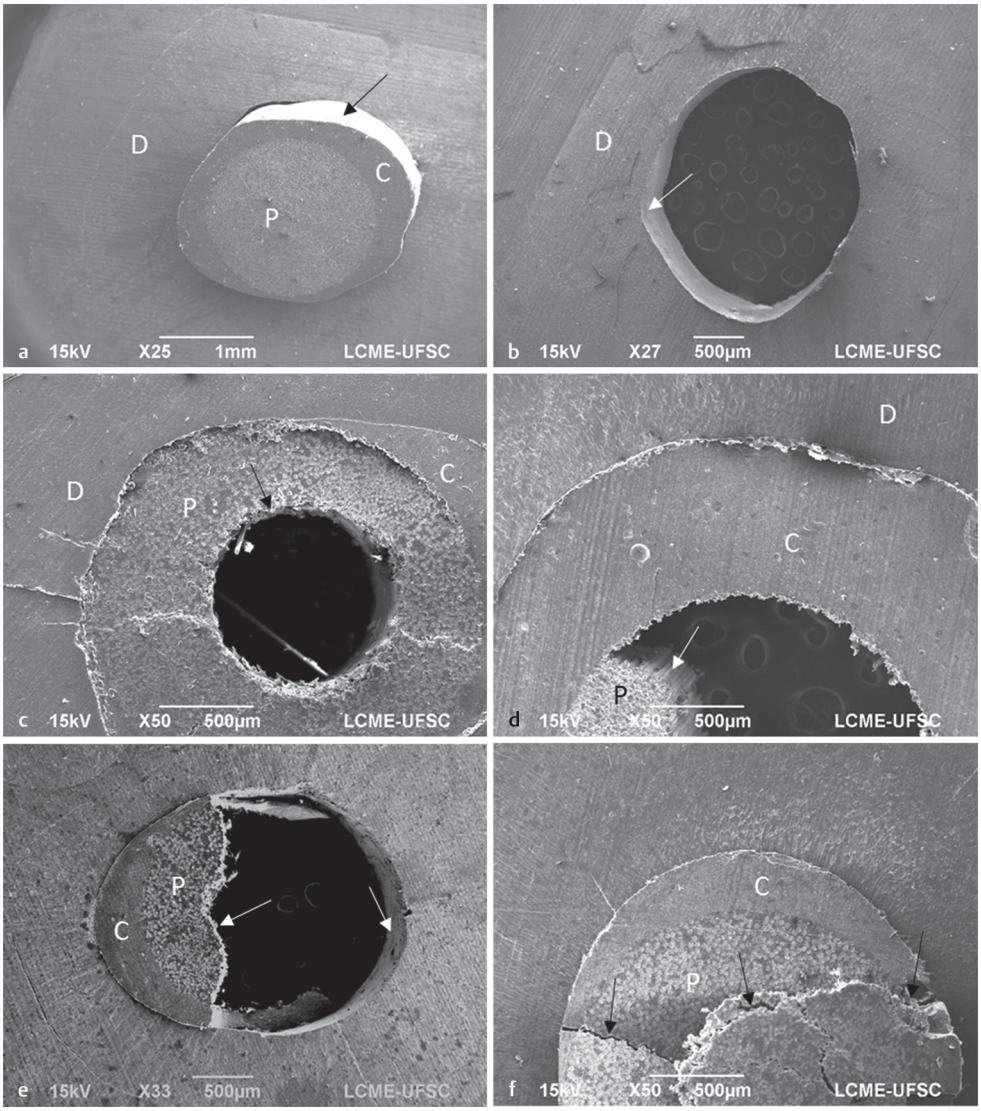

Fig. 2 Representative SEM micrographs of the most predominant failure mode in each group after the push-out test. G1: adhesive failure in the cement/dentine interface (*arrows*) (**A** and **B**); G2 and G3: post cohesive failure (*arrow*) (**C** and **D**); G4: mixed failure, part of the dentine has no cement, and part is covered with fractured cement/post (*arrows*) (**e**); G4: mixed failure showing fracture of cement and post (*arrows*; **F**). D: dentin; P: post; C: cement. SEM, scanning electron microscope.

Discussion

In the present study, bovine teeth were used to perform the tests because of the easy acquisition of teeth with no caries, restorations, or other flaws,[18-20] and the possibility of age standardization.[21] Bovine teeth aging from 24 to 48 months were used, considering that older bovine teeth are more similar to human teeth.[22] According to Camargo et al[21] the number of dentinal tubules within the root canal is significantly greater in bovine teeth in comparison with human

teeth. However, when the diameter of the tubules was compared between themselves, there was no significant difference. Other studies have also reported significant similarities between human and bovine teeth.[23,24]

The internal bleaching of the teeth was performed with a 35% H_2O_2 chemical agent, which was kept in the pulp chamber for 45 minutes, with no exposure to a light source. Caneppele et al[25] demonstrated that the use of H_2O_2 in higher concentrations (35 to 38%), combined with the use of different protocols (continuous application or with reposition), promotes similar bleaching effect. Also, a recent systematic review verified that the association of light with bleaching agents does not increase the effectiveness of bleaching.[26]

Despite the considerable variation in the push-out bond test design used in other endodontic researches,[27] this test is still valid due to its effectiveness and lower data variability in comparison with other mechanical tests.[28] The use of 1-mm-thick slices allows a uniform application of the shearing forces on the adhesive interface, simplifies the adhesion area calculation, and is less sensitive to stress distribution variation during strength applications.[29]

Based on the results obtained in our study, the null hypothesis tested was accepted, as no significant difference in the BS values among groups was observed. Our results demonstrated that fiberglass posts cementation immediately after internal bleaching process, or after 7 days, regardless the use of $Ca(OH)_2$ dressing, had BS values to dentine similar to the control group (no bleaching).

The lack of studies assessing the effect of intracoronary bleaching period on the BS of fiberglass posts to dentine, restrains a deeper discussion of the results obtained. However, other studies evaluated the influence of bleaching treatment on the BS of resinous materials to root dentine through several mechanical tests.[8,13] Vieira et al[8] reported that the use of a high-concentration bleaching agent, after 7 days, negatively affected the BS of the resin/dentine interface and dentine flexural strength. Similarly, Souza-Gabriel et al[13] noticed a BS reduction of the resinous materials to dentine, when restoration was performed immediately, 3 and 7 days after the intracoronary bleaching treatment with 38% H_2O_2. According to these authors,[13] restorative procedures must be performed after 10 or more days of finishing the bleaching treatment. However, the study of Bittencourt et al[7] reported that after 7 days of bleaching treatment with 35% H_2O_2, the BS of composite resin to dentine increased and, it was similar to the no-bleaching group. In addition to the bleaching agents, other substances, such as photosensitizers (curcumin and methylene blue), may influence the mechanical properties of intraradicular dentin and the bond strength of glass-fiber posts.[19,20]

Considering the different root thirds, the other null hypothesis tested in the present study was rejected. The BS observed for G1 (no bleaching) in the cervical third was significantly higher in comparison with the apical third of the post, as found in other studies.[18,30-32] This outcome may be related to the greater density of the dentinal tubules in the cervical third, which results in greater adhesion values in this area, followed by the middle and apical thirds.[33] Also, the limited access in the deeper areas of the root canal is challenging to professionals, hindering the acid etching, the adhesive system application, the proper light transmission during photoactivation, and the distribution of the resinous cement.[32,34] However, no BS difference was noticed among root canal thirds for the bleaching treatment-groups.

The absence of difference among these groups might be explained by the efficacy of the cervical sealing performed with zinc oxide/zinc sulfate cement.[35] The existence of this barrier effectively prevented access of the bleaching agent to the inner portion of the root canal. The study of Smith et al[35] evaluated the effectiveness of a cervical barrier with zinc oxide/zinc sulfate cement in the intracoronary bleaching treatment and noticed that the use of 2 mm of the material was enough to significantly reduce the dentinal infiltration and penetration of the bleaching agents. Furthermore, in the study of Hosoya et al,[36] the zinc oxide/zinc sulfate cement had a better performance as a cervical barrier to prevent infiltration when compared with a photoactivated temporary restorative material, zinc phosphate cement, and zinc oxide/eugenol cement.

The use of $Ca(OH)_2$ dressing after intracoronary bleaching treatment (G3) did not result in a significant change in the BS of the post/cement to dentine in comparison with the groups that did not use $Ca(OH)_2$ (G1, G2, and G4). The residues of the $Ca(OH)_2$ dressing were probably removed during root canal preparation for post cementation, as 37% phosphoric acid etching was performed.[10]

Several studies had reported that the use of a bleaching agent increased the microleakage in composite resin restorations.[10,11] However, the short-term use of $Ca(OH)_2$ dressing did not increase such microleakage.[10] Therefore, as performed in previous studies,[8,13] in our study, the 35% H_2O_2 gel was applied for 45 minutes, corresponding to the period of a bleaching clinical session, although three or four sessions might be acceptable as well. Such fact might have been one of the factors that contributed to the similarity found in the BS values for the bleaching- and no bleaching-groups. The use of a single bleaching session and, therefore, the reduced time in which the bleaching agent remained in contact with the dentine substrate promoted such findings.[8,13]

The mean BS values (MPa) obtained in this study were similar to those observed in other researches,[37,38] which also used the conventional dual resinous cement RelyX ARC (3M/ESPE). Such values may be considered low and justified by some possible reasons. The elevated polymerization contraction of the cement generates stress that might damage the set post/cement bonding to the root dentine.[39] Despite the fact that this material has a dual polymerization, at the deeper portions of the root canal, the access of light is limited, making the cement dependent of its chemical curing.[39] This fact results in the reduction of the cement's conversion degree and, consequently, it compromises its mechanical properties.[40] Conventional resinous cements, such as RelyX ARC, require a previous application of adhesive system, in this case, based on total acid etching.[39] Therefore, the cementation technique requires several stages, is more complex, extremely sensitive, and may affect the adhesion quality.[37,41]

Although the BS values were similar, the failures presented a higher variation among groups. G1 and G4 had predominance of adhesive failures, in the cement/dentine and post/cement/dentine interfaces, respectively. Cohesive failures in posts were observed in all groups, especially in G2 and G3. The most frequent cause of the adhesive or cohesive failures has been related as a result of an overside post space preparation,[27] which may have occurred in the present study despite of the efforts to avoid larger root canals. Curiously, dentine cohesive failures were observed only in the specimens of the bleaching-groups, with higher percentage in the group submitted to $Ca(OH)_2$ dressing. Further studies must be conducted to evaluate if the use of hydrogen peroxide in longer periods or in different concentrations affect the BS of fiberglass posts to the intraradicular dentine.

Conclusion

Despite the limitations of this ex vivo study, it may be concluded that intracoronary bleaching did not reduce the BS of fiberglass posts to the root dentine, regardless of the post-bleaching waiting time. In addition, the use of $Ca(OH)_2$ dressing had no effect on the BS of fiberglass posts to the dentine.

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Conflict of Interest
None declared.

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