Longevity of Bonding of Self-adhesive Resin Cement to Dentin

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Clinical Relevance

Regardless of the cementing strategy, the durability of bonding to root canal dentin may be influenced by the dentin treatment protocol.

SUMMARY

Objective: To evaluate the effect of root dentin treatment on the bonding of self-adhesive resin cement after 24 hours and after 6 months.

Methods: A total of 48 single-rooted premolars were endodontically treated and divided into four groups (n=12): Adper Scotchbond Multi-Purpose + RelyX ARC (ARC); RelyX U200 (U200); EDTA + RelyX U200 (EU200); and phos-
phoric acid (H$_3$PO$_4$) + RelyX U200 (HU200). After filling the roots, an Exacto No. 2 fiber post was cleaned, treated with silane (60 seconds), positioned, and light cured (LED; 60 seconds at 1200 mW/cm^2). After storage (37°C/24 h), the roots were cut to obtain two discs (1 mm) of each third. They were stored in distilled water (24 hours at 37°C); one disc of each root-third was subjected to the push-out test (0.5 mm/min) at 24 hours and the other disc after six months of water storage (37°C). The data on the root-thirds were averaged for statistical purposes. The average values of bond strength (MPa) were analyzed by two-way analysis of variance and post hoc Student-Newman-Keuls (5%).

Results: There were statistical differences for the treatment of dentin (*p*<0.001), for time (*p*=0.003), and the interaction of treatment and time (*p*=0.017). After 24 hours, we observed lower bond strength in the HU200 group when compared with other groups (ARC, U200, and EU200). After six months, HU200 showed the lowest bond strength. Higher strengths were observed for EU200 and U200 similarly, which were higher than ARC.

Conclusion: The bonding of the self-adhesive resin cement varied over time in the tested groups.

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INTRODUCTION

Fiber posts have several properties (eg, esthetics and elastic modulus) that are similar to those of tooth structure, which provides excellent biomechanical behavior and better load dissipation, reducing the risk of radicular fracture.[1,2] A post's association with the adhesive system and resin cement improves the performance of their mechanical properties.[3,4]

Resin cements are recommended for cementing the fiberglass posts in the root canal and are divided into self-adhesive and adhesive systems according to the cementing strategy.[5] Self-adhesive cement was developed a decade ago, with the purpose of simplifying the cementation process by assembling all the components into a single product.[6] This combination has resulted in a material that self-adheres to dentin, does not require pretreatment of the surface of the tooth, is simple to implement, and can be performed in a single step.[5,7] Given that the removal of the smear layer is not recommended with most self-adhesive cements, there is increased tolerance to moisture and the release of fluoride ions.[5]

Some studies have indicated that retaining the smear layer on the dentin could interfere with the adhesion of self-adhesive materials because it may hinder the adaptation and bonding of the resinous material to the walls of the root canal.[8,9] Others have indicated its retention because some solutions used to remove it could modify the structure of the dentin, increase water flow, and compromise the bonding with the resinous monomers by interfering with the polymerization.[10]

There is no consensus in the literature regarding the treatment of dentin in preparation for self-adhesive resin cement. Some studies have suggested that the cement is able to cross the smear layer and bond to the dentin.[5-7] The simplification of the cementation technique has been welcomed by clinics, and according to a recent study[11] has demonstrated bonding similar to the resin cement associated with the adhesive system because the latter system is more sensitive due to the various steps involved and can incur operator errors.[12] However, to our knowledge, the durability of the bond strength of self-adhesive resin cements is still unknown.

This investigation was conducted to test the hypotheses that 1) there is no difference between treating the dentin with ethylenediaminetetraacetic acid (EDTA) at a concentration of 17% or phosphoric acid (H_3PO_4) at a concentration of 35% in the strength of the push-out bond strength to dentin when resin cement is applied; 2) there is no difference at 24 hours and after six months in the strength of the push-out bond strength to dentin when resin cement is applied.

METHODS AND MATERIALS

A total of 48 single-rooted human premolars, with complete root apex formation, that were free of caries, fractures, root lacerations, and previous endodontic treatment and had at least 14 ± 1 mm between the cementoenamel junction (CEJ) and the root apex were disinfected in a 0.5% solution of chloramine-T for seven days at 4°C.

Preparation of the Teeth

The dental crowns were sectioned in a direction perpendicular to the long axis at the height of the CEJ to obtain roots of a minimum length of 14 mm and to create access to the root canal. The patent canal length was the established working limit and the instrumentation was performed up to the apical foramen. The roots were filled using the hybrid trigger technique, maintaining a distance of 1 mm from the apex with the endodontic cement, Sealer 26 (Dentsply, Dentsply DeTrey, Konstanz, Germany). The cones were condensed using a McSpadden device (Easy Dental Equipment, Belo Horizonte, Brazil). After allowing for the cement setting time, the 48 roots were unobstructed using the equipment Termo Pack II (Easy Dental Equipment, São Paulo, Brazil). The specimens were wrapped in sterile gauze soaked in a solution of 0.9% sodium chloride (NaCl), packed in individual containers, and stored for 24 hours at room temperature. For the final calibration of the conduit a No. 2 Exacto drill (1.6 mm in diameter; Angelus, Londrina, Brazil) was used, coupled to a low-speed turbine (Koncept, KaVo, Joinville, Brazil) to a maximum depth of 9 mm from the CEJ.

Experimental Groups

The composition and application mode of the materials used are presented in Table 1. The prepared roots were irrigated with a solution of 0.9% NaCl and dried with absorbent paper towels before being categorized from one to 48 and randomly allocated to four treatment groups (n=12). Each specimen received a translucent fiberglass post Exacto No. 2 (Angelus) with a conical shape, smooth surface, diameter greater than 1.6 mm, and length of 15 mm. Its surface was cleaned with sterile gauze soaked in absolute ethanol (F Maia, Cotia, Brazil) and dried with jets of air. The binding agent, Silane

Table 1: *Composition and Application Mode of the Materials Used in the Study*		
Material / Manufacturer	**Composition**	**Application**
Ethylenediaminetetraacetic acid (EDTA) / Biodinâmica	17% aqueous solution of tetrasodium salt (1 M; pH 7.2)	Apply with 5 mL on the surface (3 min), Wash (0.9% NaCl for 30 s), dry with absorbent paper points.
Phosphoric acid conditioner Scotchbond / 3M ESPE	35% phosphoric acid	Apply (15 s) on dentin, wash with water (30 s), dry with absorbent paper points.
Silane / Angelus	monofunctional γ-methacryloxypropyltrimethoxysilane (MPS) and ethanol	Apply for 60 s and lightly air dry.
RelyX U200 Automix / 3M ESPE	Paste A—amine, bisphenol A glycidyl methacrylate (bis-GMA), triethyleneglycol dimethacrylate (TEGDMA), photoinitiators, inorganic particles of silica and zirconia (68% by weight), and pigments.	Apply (Automix syringe), wait the setting time.
	Paste B—TEGDMA, bis-GMA, inorganic particles of silica and zirconia (67% by weight), benzoyl peroxide.	
RelyX ARC / 3M ESPE	Silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer.	Mix equal parts of Paste A and Paste B (10 s)
		Apply (Centrix syringe), wait the setting time
ScotchBond Multi-Purpose Plus (SBMP) / 3M ESPE	35% phosphoric acid	Acid etching (15 s); wash (30 s); dry (absorbent paper); apply activator (one coat); apply primer; apply catalyst
	SBMP activator: sulfinic acid salt ethanol based solution, photoinitiators	
	Primer: 2-hydroxyethylmethacrylate (HEMA), polyalkenoic acid copolymer	
	SBMP catalyst: bis-GMA, HEMA, benzoyl peroxide	
Fiberglass post Exacto / Angelus	80% glass fiber	Place the post in the root
	20% epoxy resin	

(Angelus), was applied for 60 seconds with an extra-fine disposable brush (KGbrush, KG Sorensen, Barueri, Brazil), and the excess removed with jets of air.

The root dentin was conditioned according to experimental group as follows: ARC group, 35% H_3PO_4 gel (3M ESPE, St Paul, MN, USA) for 15 seconds; U200 group, not conditioned; EU200 group, conditioned with a solution of 17% EDTA (Biodinâmica, Ibiporã, Brazil) for three minutes; and the HU200 group, conditioned with 35% H_3PO_4 for 15 seconds.

Following this, the root dentin was washed with water for 30 seconds by means of a triple syringe and dried with absorbent paper, leaving the canal slightly damp. In the ARC group, the posts were cemented after application of the adhesive system Adper Scotchbond Multi-Purpose (3M ESPE) with resin cement RelyX ARC (3M ESPE) by means of a Centrix syringe (Nova DFL, Rio de Janeiro, Brazil). In the U200, EU200, and HU200 groups, the posts were cemented with ready-mixed self-adhesive resin

cement, RelyX U200 Automix (3M ESPE), inserted directly into the canal. In all groups, the cements were light-cured using a LED device (Radii-Call, SDI, Bayswater, Australia) with an irradiance of 1200 mW/cm^2 for 60 seconds from the coronal direction.

After preparation of the 48 specimens according to their experimental group, the specimens were stored for 24 hours in humidified individualized containers in an oven at 37°C. With the aid of a diamond-cutting disc (Extec 12205, Erios, São Paulo, Brazil) coupled to a cutting machine (model Isomet 1000, Buhler Ltd, Lake Bluff, IL, USA) at a speed of 200 rpm under constant cooling with distilled water, two 1-mm thick slices of each root-third of the specimens were obtained (which means that six discs were obtained from each root). They were stored in distilled water (37°C/24h). At 24 hours, three discs from each root (cervical, medial, apical) were subjected to a push-out test, and the other three discs from each root (cervical, medial, apical) were kept stored in distilled water at 37°C to be tested

Time	Surface Treatment				
	ARC	**U200**	**EU200**	**HU200**	**p value**
24 h	4.99 (1.79) Aa	5.04 (1.36) Aa	3.84 (1.64) Aa	2.38 (1.63) Bc	0.001
6 mo	4.54 (2.40) Aa	6.79 (2.41) Ab	7.11 (1.83) Cb	3.19 (2.48) Bc	
p value	0.03				

Table 2: *Mean (Standard Deviation)in MPa of Bond Strength to Dentin According to the Experimental Conditions[a]*

[a] *Lowercase letters show comparison inside the rows whereas uppercase letters indicate comparisons among columns.*

after six months. The storage medium was changed weekly.[13]

Mechanical Testing

Each disc was fixed in a device and a compression load applied to the slice in the apical-coronal direction so as to push the post, respecting the taper of the root canal, by means of a 1-mm-diameter cylindrical punch connected to a universal testing machine (Emic DL 2000, São José dos Pinhais, Brazil) at a speed of 0.5 mm per minute, using a load cell of 50 kgf until the post was displaced in the root canal. The bond strength was obtained in newtons and converted to MPa by dividing the maximum load failure obtained by the area of the bonded interface. After being subjected to the push-out test, the dimensions of the specimen were measured using a digital caliper, Digimess (Digimess Precision, São Paulo, Brazil), with an accuracy range of 0.02 mm.

Analysis of Fracture Pattern

The failure surfaces were examined with an optical microscope (Bel MicroImage Analyzer, Bel Photonics, Monza, Italy) with a magnification of 40× to determine the type of failure, categorized as follows: 1) adhesive failure between the post and cement, 2) adhesive failure between the cement and root dentin, 3) cohesive failure of the post system, 4) cohesive failure of the cement, and 5) mixed type, a combination of the two aforementioned failures. Representative specimens were selected and analyzed with a scanning electron microscope (SEM; SSX 550 EDX, Shimadzu, Bangkok, Thailand).

Dentin Micromorphology

The same SEM was used to characterize the root dentin surface. After preparation of the teeth in the same way as previously described, each root was cut in the direction of the long axis of the buccal and lingual surfaces of each root with the aid of a diamond disc at a speed of 200 rpm under constant cooling with distilled water (Extec 12205, Erios)

coupled to a cutting machine (model Isomet 1000, Buhler Ltd). Following that, the conditioning protocols of each experimental group were carried out; next, the roots were broken at the end to obtain two pieces of each root, which were then fixed with paraffin in aluminum stubs and left in a dry oven with silica for 24 hours to lose all moisture. Next, they received gold metallization in a vacuum chamber (Sputtering SCD050, Bal-Tec, Balzers, Liechtenstein) and were taken to the SEM for observation at 12 kV operating in secondary electron mode. Images of low (30×) and high (1200×) amplitude were obtained of the coronal, middle, and apical thirds of each root, and each one was qualitatively evaluated.

Statistical Analysis

For statistical purposes, the data of the root-thirds were grouped and a root considered as the experimental unit.[12,14] The mean values of bond strength of the roots were transformed into square roots and subjected to a two-way analysis of variance (Treatment × Time). All pairwise multiple comparisons were performed using the Student-Newman-Keuls method with a significance level of $\alpha = 0.05$. The statistical software Sigma-Stat 3.5 (San Jose, CA, USA) was used.

RESULTS

Table 2 presents the mean values of bond strength in the experimental groups. A statistically significant difference was observed for the treatment of the root dentin ($p < 0.001$; power of test = 1), for the time ($p = 0.003$; power of test = 0.83), and for the interaction of treatment and time ($p = 0.017$; power of test = 0.609). The results demonstrated that conditioning the dentin with 35% H_3PO_4 statistically decreased the bond strength at 24 hours when compared with U200, EU200, or ARC. With regard to the six-month results, HU200 showed the lowest bond strength. Higher bond strength was observed for U200 and EU200 similarly, which was higher than ARC.
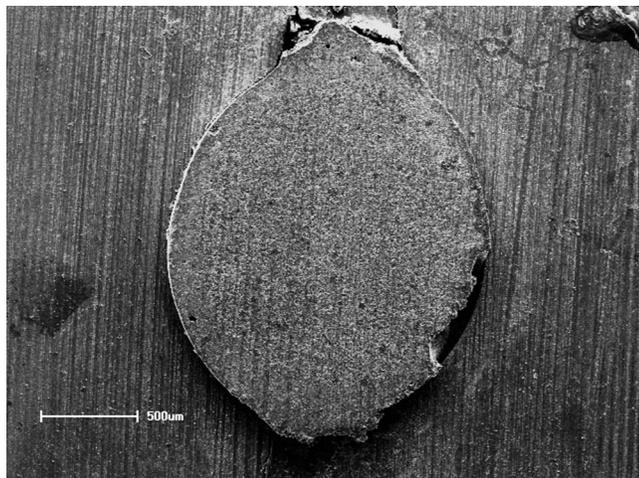
Figure 1. *Scanning electron microscopy of adhesive fracture mode.*

Observation of the fracture pattern showed the predominance of adhesive failure between the cement and root dentin in all the experimental groups, followed by a lower rate of adhesive failure between the cement and root. There was no cohesive failure reported in the present study (Figure 3). A representative image of the predominant failure mode can be seen in Figure 1.

Figure 2 presents SEM imagery of the root dentin treated with acidic solutions, in which the root covered by the smear layer in the coronal (A), middle (B), and apical (C) thirds in the group that did not receive acid conditioning is noted. After conditioning with 17% EDTA (coronal [D], middle [E], apical [F]) and 35% H_3PO_4 (coronal [G], middle [H], apical [I]), there was exposure of the dentinal tubules.

DISCUSSION

The objective of this study was to evaluate the influence of root dentin treatment prior to cementing a fiberglass post with self-adhesive resin cement and a resin cement associated with the adhesive system after 24 hours and after six months. The results showed that the bonding between the dentin and the cement was influenced by the dentin treatment and the storage time.

After 24 hours, the adhesion of the U200 was similar to that of the EU200 and ARC, and all three were greater than the HU200, rejecting the first hypothesis of this study. The results corroborate a systematic review[11] that observed similar adhesion between resin cements, regardless of the cementing strategy. The similarity could be explained by the conditioning of the dentin having been performed with a weak acid such as 17% EDTA, which

superficially exposed the dentinal tubules[9,10] of the root canal (Figure 2) and enabled the interaction of the self-adhesive cement with the dentin underlying the smear layer. Promising results were reported after the etching of the dentin with EDTA[15-17] by greater unobstruction of the dentinal tubules and the formation of the hybrid layer.

Despite the self-adhesive strategy theoretically eliminating the need for any surface treatment of the dentin, there are doubts about retaining the smear layer, which, according to some authors,[16,18] could interfere with the bonding to the root canal, and therefore propose its removal with chemical solutions such as polyacrylic acid, EDTA, NaCl, and sodium hypochlorite (NaOCl). However, one review[9] showed that the removal of the smear layer has not been standardized among different studies and cannot be removed uniformly along the full extent of the root canal, given that switching solutions of 17% EDTA with 5.25% NaOCl led to greater removal of the smear layer. Figure 2 shows the removal of the smear layer and unobstruction of the dentinal tubules by 17% EDTA, partially agreeing with the results of this review, because in this study EDTA was not switched with another substance. Conditioning of the dentin with 35% H_3PO_4 also demonstrated a similar result.

This study also showed that the etching of the dentin with 35% H_3PO_4 resulted in lower bond strength after 24 hours and after six months. But when the adhesive system was applied after etching with H_3PO_4 as in the ARC, the formation of the hybrid layer ensured bonding similar to the U200. Studies within the scope of the adhesive cementation of posts usually use the ARC as a comparison group[19] and promising results have been observed; however, few of these assessed bond strength after 24 hours.[20]

Thus, the second objective of this investigation was to evaluate the longevity of bonding of the resin cement/dentin after six months, and the results showed that the storage time influenced the adhesion, rejecting the second hypothesis of this study. After six months, the adhesion of the EU200 group increased, and it was higher than that of all the other groups. These results can be explained by the use of EDTA, a molecule containing four carboxylic acid groups, which acts as a light chelator of calcium (Ca) in selective dissolution of hydroxyapatite.[21] Unlike phosphoric acid etching, which is deep, with EDTA the dentin is superficially demineralized.[22] It can be understood that the adhesion of the EU200 group was higher than the ARC at six months due to the interaction of the resin cement and the dentin, with
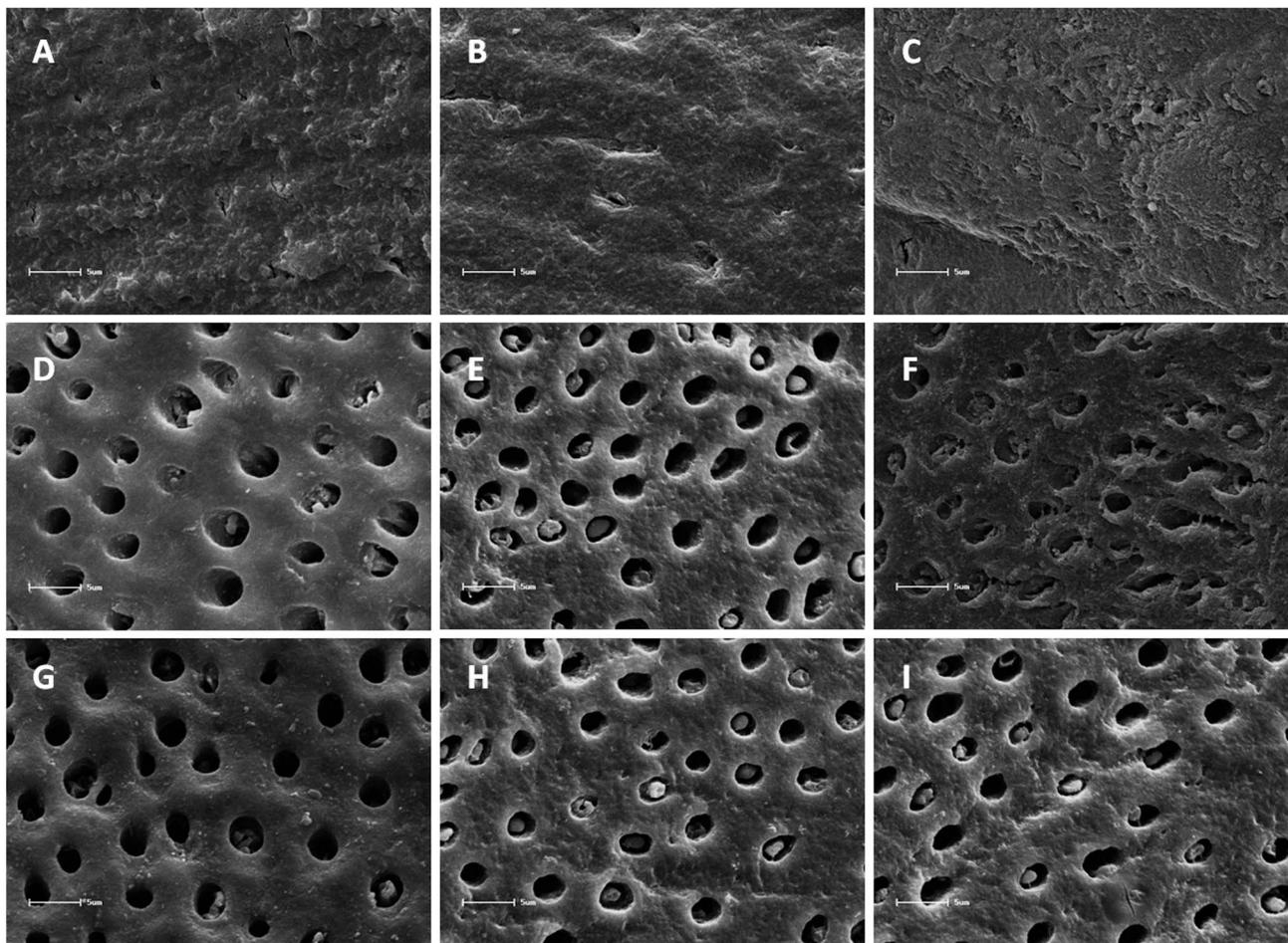
Figure 2. *Scanning electron microscopy of radicular dentin after surface treatments. Without acid etching: (A) cervical, (B) middle, (C) apical; After etching with 17% EDTA: (D) cervical, (E) middle, (F) apical; After etching with 35% H_3PO_4 (G) cervical, (H) middle, (I) apical.*

the latter having been more uniformly demineralized by the EDTA compared with the HU200 group.

The preservation of the longevity of adhesive restorations was described when the conditioning of the dentin with EDTA was carried out,[22,23] where surface removal of the hydroxyapatite ensured that the collagen fibers would not be exposed or suffer structural changes[24] or dehydration.[25] Another aspect to be considered is that EDTA is a potent inhibitor of matrix metalloproteinases (MMPs), collagenolytic enzymes related to the degradation of the organic matrix.[24,26] The chelating activity of EDTA promotes the sequestration of Ca^+ ions present in the dentin,[21] which together with zinc, act as potential activators of MMPs.[26,27] When performing the conditioning with phosphoric acid etching, greater demineralization and exposure of collagen occurs, activating MMPs.[24] These aspects may also explain the results observed at six months in this study.

The bonding of resin cement to dentin will occur if there is an interaction between the surfaces.[28] Although the pH of some self-adhesive cements is initially acidic, it is unable to completely remove the smear layer as a strong acid such as H_3PO_4 would.[7,29,30] For some authors[31] this limitation could be overcome by the possible chemical bond between the dentin and some self-adhesive resin cements that contain the functional monomer. In the case of the U200, according to the manufacturer, the chemical adhesion is ensured by the presence of 4-methacryloxyethyl trimellitic acid monomers and phosphoric acid esters.

Conditioning the root dentin with H_3PO_4 before cementation seems to hinder adhesion to the dentin[4,7,30] by exposing the collagen, deeply removing the hydroxyapatite, and changing the water flow.[32,33] The results of the HU200 group in this study agree with these assertions. However, the application of the adhesive system after conditioning
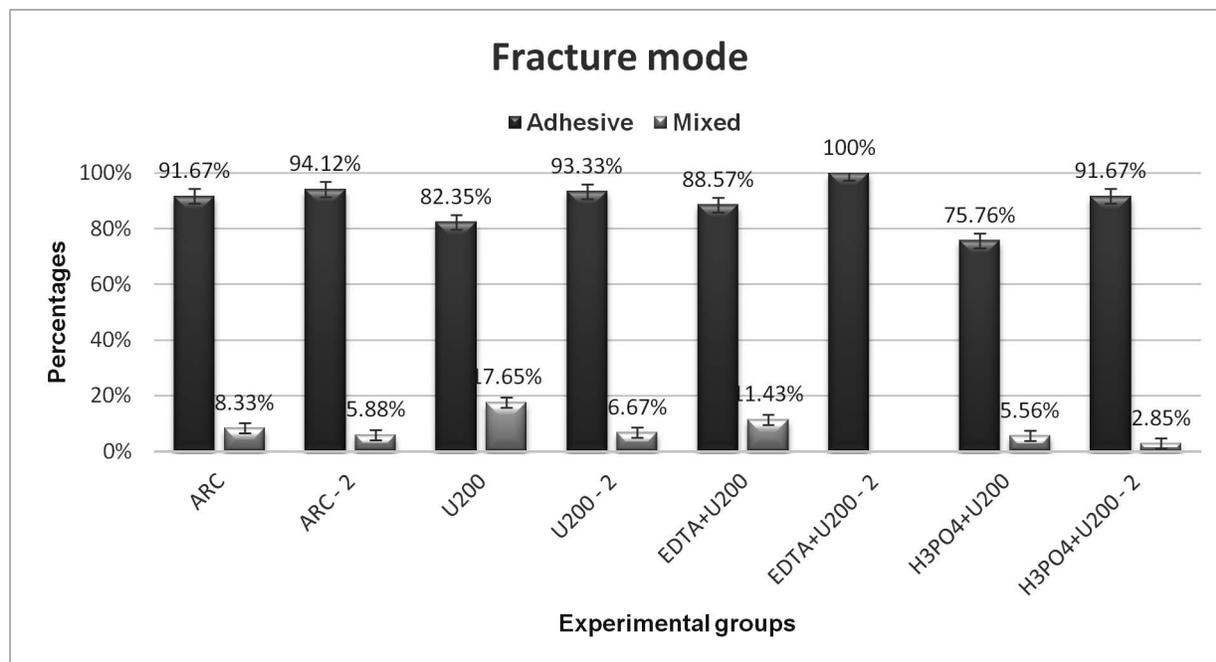
Figure 3. *Percentages of fracture modes according to the experimental conditions.*

with this acid ensured adhesion in the ARC group after 24 hours and after six months. Thus, the result of this study demonstrated that phosphoric acid should not be used in isolation as a dentin conditioner, prior to the self-adhesive cement studied, because RelyX U200 contains hydrophilic monomers that can interfere with the cement polymerization process, compromising the bonding.[17]

The adhesion of the fiberglass post to the root dentin can be influenced by the difficulty in controlling the dentin moisture when used with a resin cement adhesive system, by visualizing the canal along its entire length,[29,33] and by the formation of bubbles in the cement interfaces.[34] In the present study this aspect was minimized because the RelyX U200 cement was inserted with the adapted tips that accompanied the Automix kit (3M ESPE) and for the RelyX ARC cement a Centrix syringe (Nova DFL) was used. This detail of the methodology and the silanization of the fiberglass posts favored the prevalence of adhesive fractures between the cement and root dentin, agreeing with previous studies that found the majority of fractures in the same place[33,34] and disagreeing with a study that observed a predominance of fractures in the cement-post interface.[32]

According to Heintze and Zimmerli,[35] the push-out test is commonly used to analyze the post adhesion of roots, and well-designed and conducted *in vitro* bond strength tests are useful as a screening test if the specimens are tested after one day and after at least three months of water storage. In this study, the bonding of fiber posts to dentin was analyzed after 24 hours and after six months of water storage, more than suggested in the literature.[35] The present investigation analyzed the bonding of RelyX U200, a self-adhesive resin cement, comparatively with that of RelyX ARC, a gold standard used on cementation of fiber posts to dentin, using natural teeth, which gives results close to the clinical outcome.[36] Further research on self-adhesive cementation is needed, especially randomized controlled clinical trials.

CONCLUSIONS

Given the limitations of this study, it may be concluded that the bonding durability of resin cements to root dentin varied over time, according to the surface treatment of dentin.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee

guidelines and policies of the Ethics Committee of Unopar. The approval code for this study is 95060.

Conflict of Interest

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

(Accepted 4 December 2014)

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