

RESEARCH AND EDUCATION

## Bonding properties of universal adhesives to root canals prepared with different rotary instruments



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Glass fiber posts have been widely used as intracanal retainers in endodontically treated teeth. These posts must be cemented adhesively to the root canal, and their success depends on the quality of the bonding between the post, resin cement, and root dentin. Even with challenges, the restorations retained by fiber posts present adequate clinical performance when correctly indicated.<sup>1-6</sup>

Before the cementation of glass fiber posts, the root canals are usually instrumented to receive the appropriate post, typically with tungsten carbide burs provided by the manufacturer of the posts. During cutting or abrasion procedures in dentin, considerable organic and inorganic residue, also known as smear layer, is produced, and this becomes embedded in the surface of the root dentin.<sup>7-10</sup>

The type of rotary device used to prepare coronal dentin

### ABSTRACT

**Statement of problem.** Preparation of coronal dentin by using a diamond rotary instrument usually results in higher bond strength values than preparation with tungsten carbide burs, but information is lacking about the influence of rotary instruments on root canals before the bonding of fiber posts.

**Purpose.** The purpose of this in vitro study was to evaluate the influence of the rotary instrument used to prepare the root canal and bonding strategies on the adhesion of fiber posts to root dentin with universal adhesive systems.

**Material and methods.** Human premolars were used and divided into 8 groups according to the combination of the following factors: rotary instrument (tungsten carbide bur versus diamond rotary instrument), cementation system (single-bond versus prime and bond), and bonding method (etch-and-rinse versus self-etch). Eight teeth per group were evaluated by push-out bond strength, 4 teeth were evaluated for nanoleakage by using scanning electron microscopy (SEM), and 2 teeth were evaluated for shape by SEM. Data for bond strength and nanoleakage for each cementation system were subjected to 2-way ANOVA and Tukey honest significant differences tests ( $\alpha=.05$ ).

**Results.** The highest bond strength values were observed for preparation using a diamond rotary instrument for both cementation systems. For prime and bond, the highest bond strength values were observed with the self-etch adhesion strategy, and the self-etch strategy was better for the single-bond; and the self-etch strategy was better than etch-and-rinse just after the use of a diamond instrument. In relation to nanoleakage for the cementation system, the prime and bond had the lowest values for groups using a diamond instrument and self-etch strategy. For the single bond, the adhesion strategy did not influence nanoleakage, but the rotary instrument did, with diamond rotary instruments resulting in lower values. SEM analyses showed a greater number of unobliterated tubules in the self-etch mode and a more regular surface when prepared with a diamond rotary.

**Conclusions.** To improve the adhesion of fiber posts to root canal, a diamond rotary instrument should be used, and generally, universal adhesive systems must be used in self-etch mode. (J Prosthet Dent 2019;121:298-305)

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## Clinical Implications

Diamond rotary instruments used for the preparation of root canal space and universal adhesive applied in the self-etch mode provide better adhesion of glass fiber posts to the root canal.

has been shown to produce different patterns of smear layer, and this may play a role in strengthening the bonding between adhesives and dentin.<sup>11-16</sup> Preparation of coronal dentin with a diamond rotary instrument usually results in higher bond strength values.<sup>15,17</sup> However, studies of the rotary instruments used for root canal preparation and their influence on the adhesion of glass fiber posts are lacking.

The smear layer formed during the preparation of the root canal must be removed or modified before cementation<sup>18-22</sup> by using acid conditioning or applying a self-etching adhesive system. Universal adhesive systems can be applied in both etch-and-rinse and self-etch modes,<sup>23</sup> but clinicians need to know which bonding strategy is the best for root canals. Studies have been recently published with universal adhesive systems in coronal dentin<sup>24-26</sup>; however, the reports are still scarce when it comes to the performance of these universal adhesives in root dentin.

Therefore, the purpose of this study was to evaluate the effect of rotary instruments and the bonding strategy of the universal adhesive systems used for root preparation before fiber post cementation on the smear layer pattern, nanoleakage, and bond strength of fiber posts to root dentin. The null hypothesis was that neither the different rotary instruments nor bonding strategy of the universal adhesive systems influence the smear layer pattern, push-out bond strength, and nanoleakage.

## MATERIAL AND METHODS

The ethics committee of the local university approved this study. A total of 112 sound human mandibular premolars without previous endodontic treatment were selected. The teeth were transversally sectioned at the cementoenamel junction, and roots were endodontically treated with a vertical condensation technique,<sup>27</sup> by using gutta percha points (Maillefer; Dentsply Sirona) and canal sealer (Sealer 26; Dentsply Sirona).

After 1 week, the root canals were randomly divided by block randomization ([www.sealedenvelope.com](http://www.sealedenvelope.com)) into 8 groups (n=14) according the combination of the following factors: rotary instrument used to prepare the root canal (tungsten carbide bur versus diamond rotary instrument), cementation system (Single Bond Universal/RelyX ARC versus Prime & Bond elect/Enforce), and

bonding strategies of the universal adhesives (etch-and-rinse versus self-etch).

In the tungsten carbide group, the post space was prepared with the corresponding tungsten carbide bur of the glass fiber post (Whitepost DC #2; FGM). In the diamond rotary instrument group, the post space was prepared by using a diamond rotary instrument (#4138 FF; KG Sorensen) in a low-speed handpiece. The working length was 10 mm for all roots, leaving a 4-mm gutta percha obturation on the apical end of the root canal. After the mechanical preparation, the root canals were irrigated with distilled water and dried.

For morphological ultrastructural evaluation of the smear layer, 2 roots of each group were randomly selected by lottery after the preparation of root canals by using the different rotary instruments. Roots were longitudinally cleaved into 2 hemisections by using a chisel, leaving the entire length of the root canal exposed. Specimens (hemisections) were then dehydrated, sputter coated with gold, and examined using scanning electron microscopy (SEM) (VEGA3; TESCAN). Three arbitrary images (original magnification  $\times 5000$ )<sup>28</sup> of the middle third were obtained from each hemisection.

For the remaining roots, the universal adhesive systems were used either in the etch-and-rinse or in the self-etch mode. In the etch-and-rinse mode, the root dentin conditioning was carried out with 37% phosphoric acid (Condac 37; FGM) for 15 seconds, washed for 15 seconds, and dried for 5 seconds with an air stream and absorbent paper points. For the self-etch mode, no preliminary etch-and-rinse step was performed. The adhesive was applied, as described earlier, after the post spaces had been irrigated with water, and dried, trying to reach an ideal moisture<sup>29,30</sup> as described for the etch-and-rinse groups.

For all groups, a glass fiber post (Whitepost DC#2; FGM) was used. The luting procedure and the composition of the cementation systems are described in Table 1. Resin cements were handled according to the manufacturer's recommendations and inserted into the root canal using a syringe (Centrix; DFL), and the post was positioned. All light-polymerization procedures were performed by using a light-emitting diode polymerization device (Radii Plus; SDI Ltd). After the post luting procedures, the roots were stored in 100% relative humidity (Eppendorf tubes supported by gauze soaked in distilled water) at 37°C for 1 week.

Eight teeth per group were randomly selected by lottery for push-out bond strength testing, and 4 teeth were selected for the evaluation of nanoleakage within the hybrid layer. To prepare the specimen for push out and nanoleakage analyses, a saw (Isomet 1000; Buehler) was used to create 2 cervical, 2 middle, and 2 apical slices from each root; their thicknesses were measured with digital calipers (Digimatic Caliper; Mitutoyo).

**Table 1.** Cementation and adhesive systems used

Material/ Manufacturer	Etching Mode Etch-and-Rinse (ER) and Self-etch (SE)	Application Mode	Batch No(s).
Single Bond Universal/3M ESPE	<b>ER:</b> Apply phosphoric acid gel 37% inside endodontic canal for 15 s, rinse thoroughly for 15 s, dry dentin for 5 s, and apply 2 absorbent paper points #40. <b>SE:</b> Not etched; Irrigation with distilled water for 15 s, dry dentin for 5 s, and apply 2 absorbent paper points #40.	Apply adhesive with disposable applicator inside canal vigorously for 20 s, dry with a slight jet of air for 5 s to evaporate solvent, apply 1 absorbent paper point #40, and light polymerize for 10 s.	523652
Prime & Bond elect/ Dentsply Sirona	<b>ER:</b> Apply phosphoric acid gel 37% inside endodontic canal for 15 s, rinse thoroughly for 15 s, dry dentin for 5 s, and apply 2 absorbent paper points #40. <b>SE:</b> Not etched; Irrigation with distilled water for 15 s, dry dentin for 5 s, and apply 2 absorbent paper points #40.	Apply adhesive with disposable applicator inside canal vigorously for 20 s, dry with a slight jet of air for 5 s to evaporate solvent, and light polymerize for 10 s.	130811
RelyX ARC/3M ESPE	–	Dispense appropriate amount of cement onto a mixing pad and mix for 10 s, apply cement around and inside endodontic with specific syringe, seat post within post space, removing excess cement, and light polymerize for 40 s through remainder of post.	1426900809 1435200564 1505700641 1425200564
Enforce/Dentsply Sirona	–	Dispense appropriate amount of cement onto a mixing pad and mix for 20 s, apply cement around and inside endodontic canal with specific syringe, seat post within post space, removing excess cement, and light polymerize for 40 s through remainder of post.	052813G 908380F 946206G 946205G 962368G

For the push-out test, slices were photographed on both sides, and an optical microscope was used to measure the internal coronal and apical diameters of the root canal walls in order to calculate their individual bonding area, based on the lateral surface of a truncated cone.<sup>31</sup> This measurement was made using software (ImageJ k1.45; National Institutes of Health). Then, each slice was subjected to a push-out test, using a universal loading device (EMIC 23-10; Instron Corp) with the load applied until the post was dislodged. The maximum failure load was recorded in N and converted to MPa by dividing the load by the bonded area.

After the push-out test, the fracture pattern of all specimens was evaluated using an optical microscope (BX51; Olympus Corp) at magnification  $\times 40$  and categorized as adhesive fracture between resin cement and root dentin; adhesive fracture between cement and post; cohesive fracture of cement; cohesive fracture post; cohesive fracture of root dentin; or mixed fracture.

For nanoleakage evaluation, the slices from 4 roots of each group were used. Specimens were immersed in a tracer solution of 50% silver nitrate ammonium for 24 hours at 37°C and then washed in distilled water for 2 minutes.<sup>5</sup> Specimens were then immersed in a developing solution and exposed to direct fluorescent light for 8 hours. Specimens were then rinsed, placed in metallic stubs, and polished sequentially with silicon carbide papers (#600 to #3000) in a polishing machine (AROPOL E Polisher; Arotec S/A Industry & Commerce) under cool water and with felt disks with diamond paste (FGM). Then, specimens were dehydrated in colloidal silica for 48 hours.

The specimens were sputtered with a mixture of gold and palladium for SEM evaluation (VEGA3; TESCAN) at 15 kV in backscattered electron mode.

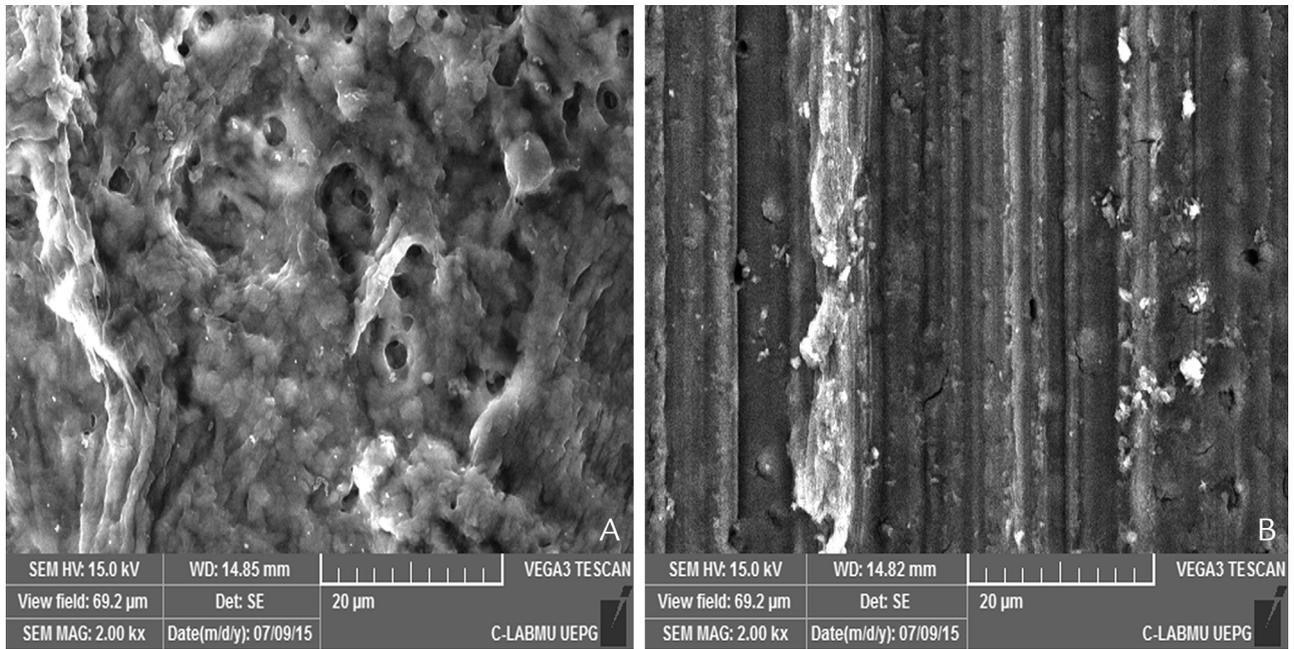
The percentage of nanoleakage was analyzed using ImageJ software. Five images per slice from each tooth were obtained, 1 at a lower magnification ( $\times 60$ ) and 4 of arbitrary areas at higher magnification ( $\times 1000$ ). For each high-magnification image, the value of the total area of the hybrid layer and silver nitrate-infiltrated area was calculated, and these 2 values obtained were divided and multiplied by 100, deriving the percentage of nanoleakage. Then, for each slice, an average of the values of the 4 measurements (higher magnification) was calculated to obtain an average value of nanoleakage for each slice.

The experimental unit for all properties was the root; therefore, an average of the values of all slices from each root was calculated for statistical purposes. Data regarding the bond strength and nanoleakage of each cementation system were statistically analyzed by 2-way ANOVA (rotary device versus bonding strategy) and by the Tukey honest significant differences post hoc test to determine the effect of these variables on the push-out and nanoleakage values for extracted premolars ( $\alpha=.05$ ). The analysis was performed using statistical software (SigmaPlot v11.0; Systat Software).

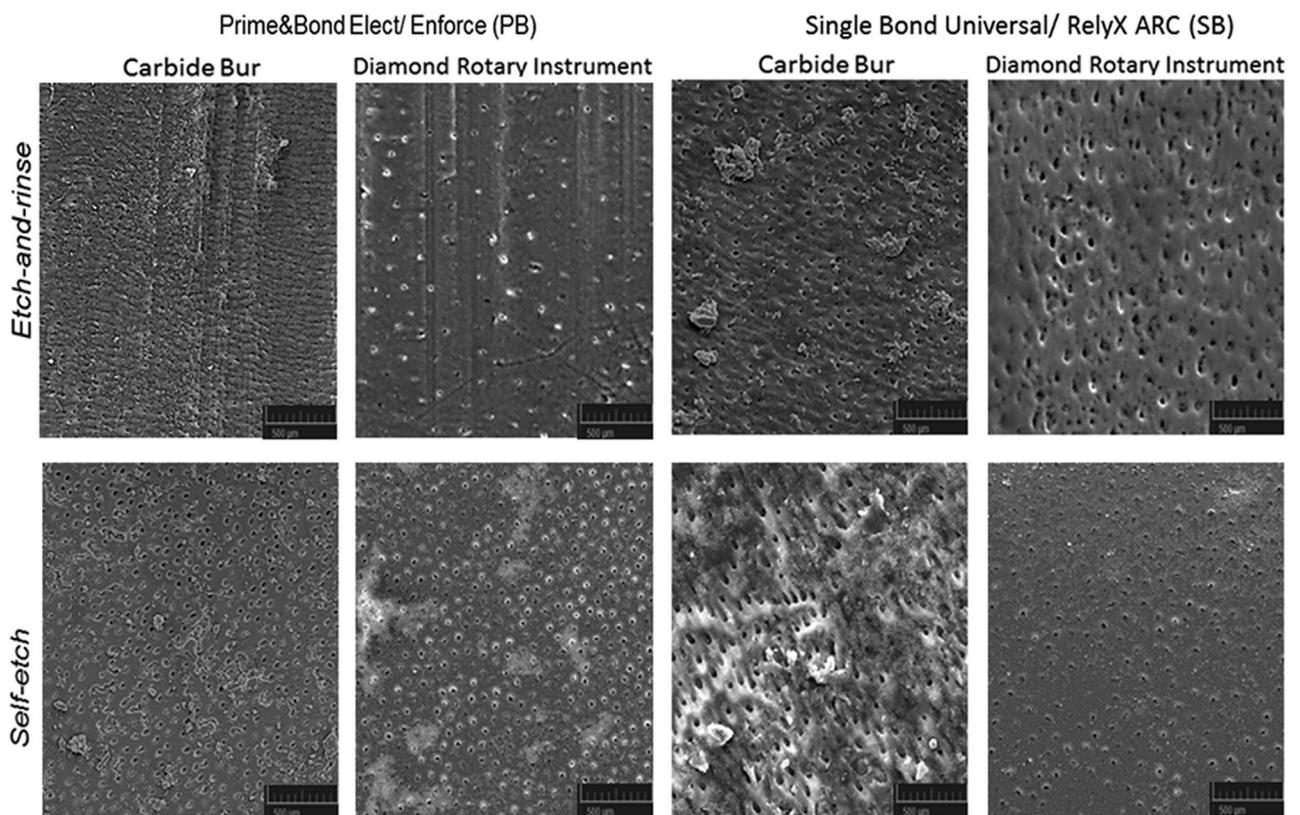
## RESULTS

Differences between the rotary instruments were observed in the resulting smear pattern layer (Fig. 1). When the preparation was made using the tungsten carbide bur, an irregular and disorganized smear layer was seen. When the preparation was made using the diamond rotary instrument, the smear layer surface appeared flat and smooth with uniform grooves. In both images, some open dentin tubules were observed.

SEM analysis of the surface after preparation with the respective rotary instrument and the use of the adhesives in the different bonding strategies (Figs. 2, 3) showed a



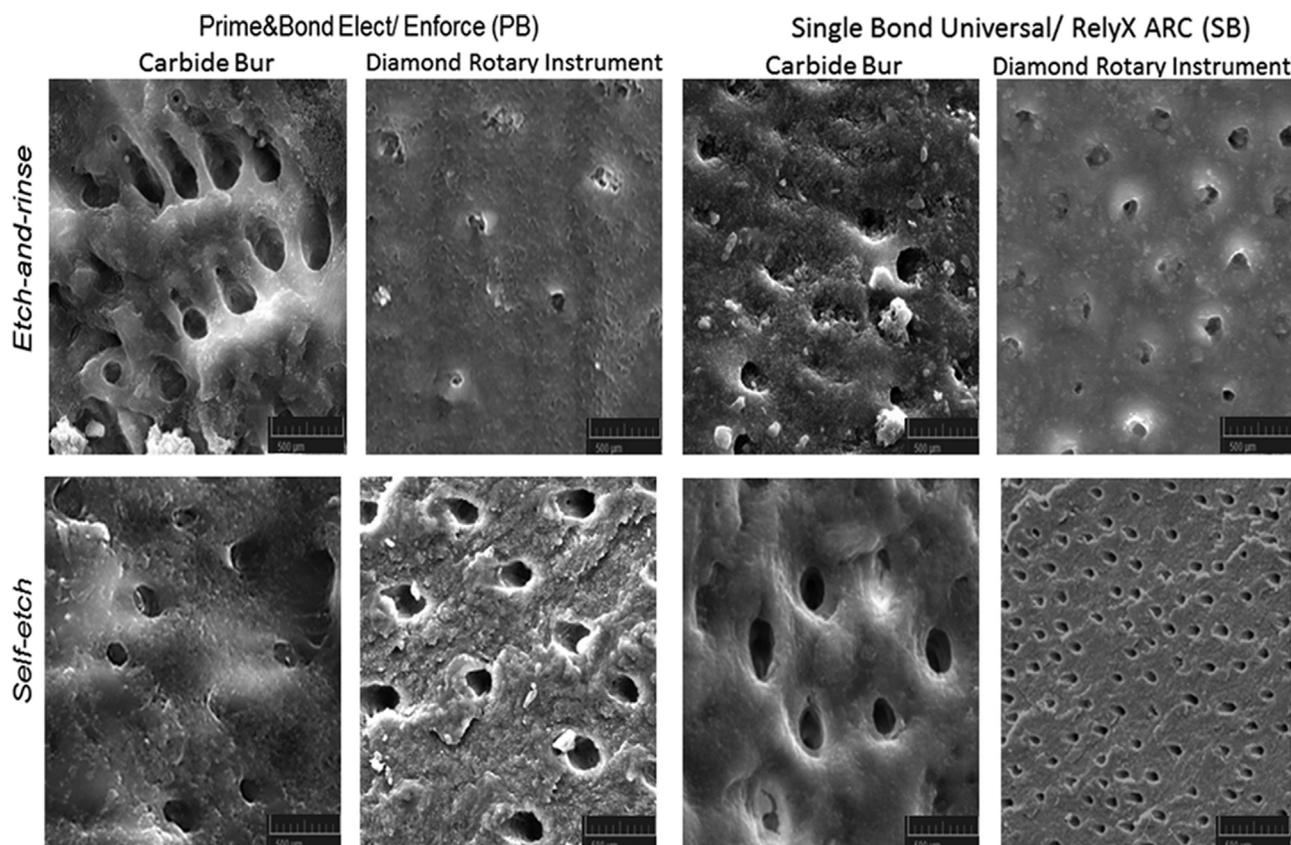
**Figure 1.** Representative scanning electron photomicrographs (original magnification  $\times 2000$ ) of preparations showing differences in smear layer patterns formed. A, Tungsten carbide bur. B, Diamond rotary instrument.



**Figure 2.** Representative scanning electron photomicrographs (original magnification  $\times 1000$ ) of the middle region of root canal and preparation performed according to different experimental groups.

greater number of unobliterated tubules in the self-etch mode and a more regular surface when prepared with a diamond rotary instrument.

For Prime & Bond elect/Enforce, higher bond strength values ( $P=.009$ ) were observed for root preparation using a diamond rotary instrument (overall mean,  $7.5 \pm 3.1$  MPa)



**Figure 3.** Representative scanning electron photomicrographs (original magnification  $\times 5000$ ) of middle region of root canal and preparation performed according to different experimental groups.

**Table 2.** Means  $\pm$ SD values for different experimental groups using the Prime & Bond elect/Enforce cementation system

Etching Mode	Bond Strength (MPa) <sup>a</sup>		Nano-leakage (%) <sup>a</sup>		Fracture Pattern <sup>b</sup>	
	Tungsten Carbide Bur	Diamond Rotary Instrument	Tungsten Carbide Bur	Diamond Rotary Instrument	Tungsten Carbide Bur	Diamond Rotary Instrument
Etch-and-rinse	4.6 $\pm$ 1.1 <sup>Bb</sup>	5.8 $\pm$ 0.8 <sup>Ab</sup>	30.8 $\pm$ 6.0 <sup>Bb</sup>	21.8 $\pm$ 7.1 <sup>Ab</sup>	41/11/1	34/14/2
Self-etch	7.8 $\pm$ 1.8 <sup>Ba</sup>	9.1 $\pm$ 1.8 <sup>Aa</sup>	23.2 $\pm$ 7.2 <sup>Ba</sup>	19.4 $\pm$ 5.8 <sup>Aa</sup>	44/9/1	37/11/0

Comparisons only valid within same property. <sup>a</sup>Different superscript letters (uppercase, comparison of instruments; lowercase, comparison between etching mode) indicate significant differences ( $P < .05$ ). <sup>b</sup>Adhesive-dentin cement/mixed/cohesive post or dentin.

**Table 3.** Means  $\pm$ SD values of different experimental groups for Single Bond Universal/RelyX ARC cementation system

Etching Mode	Bond Strength (MPa) <sup>a</sup>		Nano-leakage (%) <sup>a</sup>		Fracture Pattern <sup>b</sup>	
	Tungsten Carbide Bur	Diamond Rotary Instrument	Tungsten Carbide Bur	Diamond Rotary Instrument	Tungsten Carbide Bur	Diamond Rotary Instrument
Etch-and-rinse	7.0 $\pm$ 1.8 <sup>C</sup>	9.7 $\pm$ 1.9 <sup>B</sup>	31.3 $\pm$ 7.6 <sup>Ba</sup>	23.9 $\pm$ 6.7 <sup>Aa</sup>	37/11/0	29/15/4
Self-etch	6.5 $\pm$ 1.4 <sup>C</sup>	13.9 $\pm$ 2.4 <sup>A</sup>	30.5 $\pm$ 6.5 <sup>Ba</sup>	23.7 $\pm$ 7.2 <sup>Aa</sup>	33/15/0	26/21/6

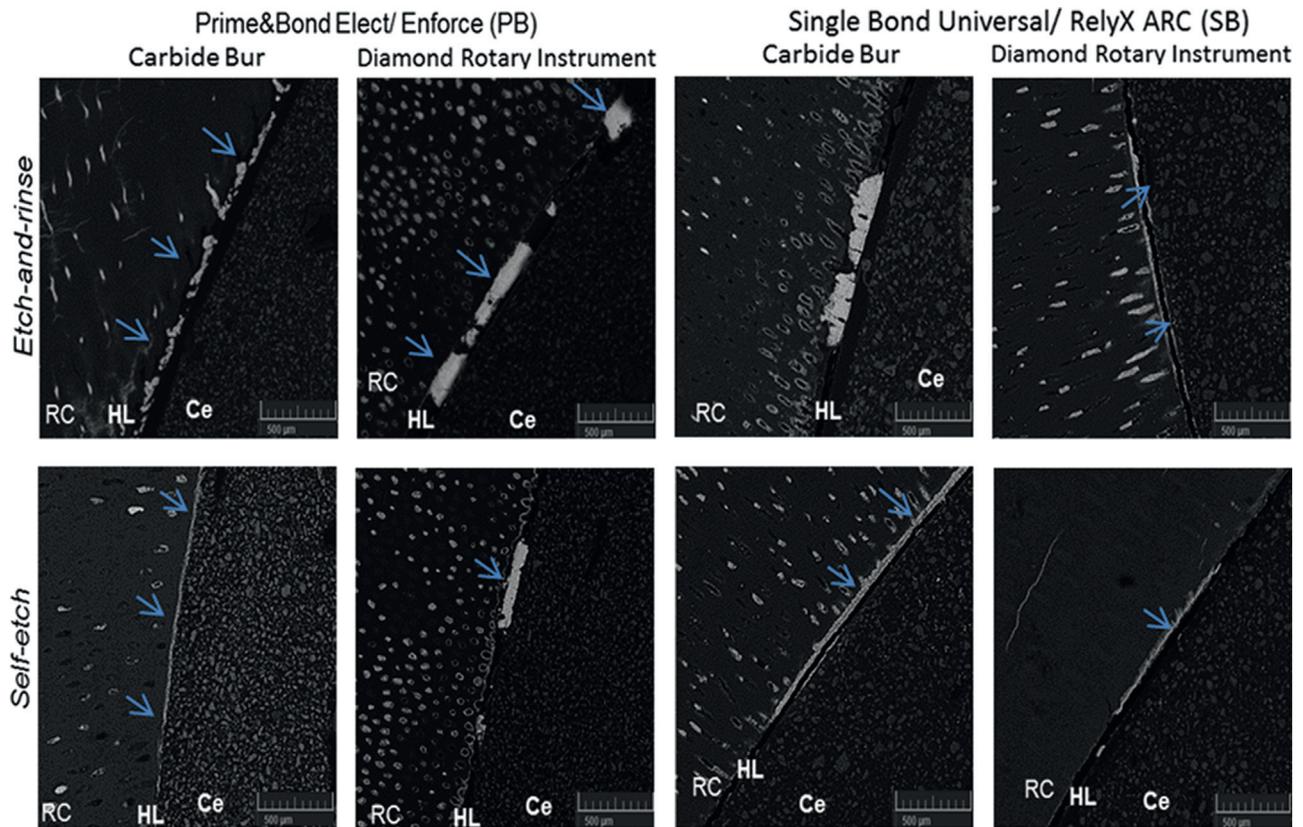
Comparisons only valid within same property. <sup>a</sup>Different superscript letters (uppercase, comparison of instruments; lowercase, comparison between etching mode) indicate significant differences ( $P < .05$ ). <sup>b</sup>Adhesive-dentin cement/mixed/cohesive post or dentin.

than by using the tungsten carbide bur (overall mean, 5.9  $\pm$ 2.9 MPa). The self-etch mode produced a statistically higher ( $P < .001$ ) bond strength (overall mean, 8.5  $\pm$ 3.0 MPa) than the etch-and-rinse mode (overall mean, 5.1  $\pm$ 2.2 MPa) (Table 2).

For the cementation system, Single Bond Universal/RelyX ARC, the cross-product interaction (rotary device versus bonding strategy) was significant ( $P < .001$ ). The highest bond strength value was observed for root preparation using a diamond rotary instrument

( $P < .002$ ) (Table 3). For the bonding strategy, both strategies were similar when used after the tungsten carbide bur, but the self-etch was better than the etch-and-rinse mode after the use of a diamond rotary instrument (Table 3).

Concerning the fracture pattern, some cohesive fractures (3.5%) were observed in dentin. The predominant adhesive failure occurred between the dentin and the cement (70.6%), followed by mixed failure (26.9%) (Tables 2, 3).



**Figure 4.** Representative scanning electron photomicrographs (original magnification  $\times 1000$ ) of post cementation interfaces for different cementation systems and adhesion strategies on middle third of root canal. Arrows indicate silver nitrate infiltrated in hybrid layer. Nano-infiltration was significantly reduced in PBSE group compared with PBER group, as well as diamond rotary instrument, compared with tungsten carbide bur for same PB group. In SB group, no lower nano-infiltration in groups of diamond rotary instrument compared with tungsten carbide bur groups. CB, tungsten carbide bur; Ce, resin cement; DR, diamond rotary instrument; HL, hybrid layer; RC, root canal.

For the Prime & Bond cementation system, lower nanoleakage percentage values ( $P=.001$ ) were observed for root preparation using a diamond rotary instrument (overall mean,  $20.0 \pm 9.3$ ) than using tungsten carbide burs (overall mean,  $26.9 \pm 9.2$ ). The self-etch (overall mean,  $20.9 \pm 9.5$ ) showed significantly lower ( $P=.037$ ) nanoleakage than the etch-and-rinse (overall mean,  $25.5 \pm 9.9$ ) (Table 2). In SEM images (Fig. 4), silver accumulation was significantly lower in the Prime & Bond self-etch group and in the diamond rotary instrument group.

For the Single Bond cementation system, the diamond instrument (overall mean:  $23.8 \pm 8.7$ ) resulted in lower percentage of nanoleakage ( $P=.046$ ) than the tungsten carbide bur (overall mean:  $31.0 \pm 13.9$ ) (Table 3, Fig. 4).

## DISCUSSION

The null hypothesis of this study was rejected because the different rotary instruments and the bonding strategies of the universal adhesive systems influenced the properties evaluated. The push-out test was selected because it has been used to evaluate the bond strength of endodontic posts to root dentin<sup>31</sup> and because it

simulates the shear stress at the interface between the resin cement and root dentin. Higher bond strength values were found for the groups that had the root canal spaces prepared with a diamond rotary instrument, regardless of the bonding strategy, which is probably related to the morphology of the smear layer produced by these different instruments. These findings are consistent with those from studies that showed that bond strength between coronal bonding substrates and adhesives can be significantly affected by the smear layer pattern produced with different rotary instruments.<sup>14,15,18</sup>

Although a diamond rotary instrument removes dentin by using an abrasion process, tungsten carbide burs cut the surface by using blade action. The diamond rotary instrument formed a thinner, smoother, more uniform smear layer surface, with the presence of superficial and deep grooves. This contrasted with the smear layer produced by the tungsten carbide bur, which was irregular, thicker, and more disorganized. These findings are consistent with those in studies conducted using coronal dentin.<sup>13,14,19</sup>

Studies in coronal dentin<sup>20</sup> showed that an extra fine-grit diamond rotary instrument produces a smear layer with a thickness of  $1.0 \pm 0.3 \mu\text{m}$ .<sup>20</sup> This facilitates its

dissolution and may enable better infiltration of adhesive systems, ultimately resulting in higher bond strength values.<sup>26</sup> The thicker smear layer produced by tungsten carbide burs<sup>21</sup> may not be easily dissolved by acidic monomers and phosphoric acid. However, additional micromorphological studies are needed on root dentin bonded with adhesives after instrumentation with different rotary devices.

The instrument alone used for root canal preparation did not affect bond strength. For both cementation systems tested, the self-etch strategy of universal adhesive systems showed higher bond strength values for glass fiber posts to root dentin. The root dentin conditioning with phosphoric acid promotes demineralization and assists in removing the smear layer, which results in the exposure of collagen fibrils.<sup>22</sup> However, the etch-and-rinse strategy in root canals is more challenging. Because of limited visual access, the complete removal of phosphoric acid inside the root canal and the achievement of an ideal degree of moisture of the root dentin after acid washing<sup>29,30</sup> are difficult, making the procedure sensitive and subject to the operator's skill and experience.<sup>32</sup> This probably explains why the etch-and-rinse strategy performed less well than Prime & Bond or equal to Single Bond the self-etch strategy in the root canal; in no case was it better than the self-etch strategy.

The nanoleakage test also identified the same trend. Nanoleakage was recognized by silver ion deposition-free nanometric spaces within the hybrid layer where adhesives were unable to penetrate.<sup>33</sup> Lack of monomer penetration may be attributed to the presence of water, solvent entrapment, or inadequate polymerization.<sup>34</sup> When conditioning is not followed by resin impregnation, as with inadequate etchant removal, unprotected collagen fibrils form a porous collagen layer,<sup>9</sup> which is susceptible to degradation and identified by nanoleakage. For both cementation systems, higher nanoleakage was found in root canals prepared with tungsten carbide burs. Studies have shown that higher nanoleakage is correlated with reduced bond strength.<sup>35</sup> This fact agrees with the results of this study, where a greater amount of silver nitrate infiltration was found in the groups prepared with the tungsten carbide burs, the ones with lower bond strength values.

Based on the results obtained in this study, we suggest the use of a diamond rotary instrument for root canal preparation before cementation of glass fiber posts. The use of universal adhesive systems using the self-etch strategy is also more beneficial due to the challenging protocol of the etch-and-rinse mode.

## CONCLUSIONS

Based on the findings of this *in vitro* study, the following conclusions were drawn:

1. Better adhesion of fiber posts to root canals was achieved by using a diamond rotary instrument to prepare the root canal.
2. Better adhesion of fiber posts to root canals was achieved by applying a universal adhesive in the self-etch mode.

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