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# Effect of Different Adhesive Strategies on Bond Quality of Fiber Posts Cemented in Endodontically Treated Teeth

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**ABSTRACT**

**Introduction:** The purpose of this *in vitro* study was to assess the effects of anatomic root levels, different adhesive strategies and cementation system on bond strength (BS), nano leakage (NL) and degree of conversion (DC) after fiber posts cementation. **Methods and Materials**: Sixty-six roots of human premolars were endodontically prepared and divided according to the combination of adhesive application technique (manual passive, manual active and active vibratory) and the cementation system (Adper Single Bond 2/RelyX ARC [SBAR] and Single Bond Universal/RelyX Ultimate [SBUL]). Specimens were transversally sectioned into six 1-mm-thick serial slices, which were subjected to BS testing ($n$=7), to NL analysis using scanning electron microscopy (SEM) after slice immersion in silver nitrate ($n$=2), and micro-Raman spectroscopy for DC ($n$=2). Data were analyzed using two-way ANOVA and Bonferroni post-hoc test ($\alpha$=5%). **Results**: Bond strength values significantly increased with active vibratory application technique ($P$<0.001), no significant difference was noted between adhesive systems. The cementation system Single Bond Universal/RelyX Ultimate demonstrated the lowest nano leakage values ($P$<0.05). The manual active and vibratory application techniques showed statistically higher degree of conversion values than the manual passive technique at the coronal and medium thirds ($P$<0.05); and the lowest results were observed with manual passive application to Single Bond Universal/RelyX Ultimate ($P$=0.016). **Conclusion:** The study concluded that techniques with active application (vibratory and manual) resulted in higher bond strength values. The mode of adhesive application influence the results.

**Keywords:** Bond Strength; Dental Adhesive; Fiber post; Scanning Electron Microscopy

## Introduction

Fiber glass posts are used to provide support and adequate retention for restoration after endodontic treatment. Its elastic modulus is similar to dentin [1], which promotes better stress distribution, reduces the possibility of catastrophic failures [2] and provides resiliency and satisfactory adhesion between dentin and resin cement [3].

Adhesion between resin luting agent and dentin is still considered weaker than the cement/fiber glass post interface [4]. This may be attributed by products resulting from endodontic treatment before post cementation [5], type of the endodontic sealer [6, 7], smear layer removal, the high C-factor within the root canal, morphology of root dentin, incompatibility between some adhesive systems and resin-based cements, residual water and/or solvent [5, 8] and compromised micromechanical retention between the demineralized dentin matrix and the polymerized adhesive system [8].

Considering these factors that hinder fiber glass posts adhesion, several strategies have been suggested in the literature to reduce the technical failures, in order to improve the adhesive systems performance. Beyond the root canal cleaning and preparation [9] studies have shown better results for manual active application compared to manual passive application [10], as the first facilitates solvent evaporation and monomer infiltration [9, 11]. Another alternative of active application would be the use of sonic frequency, which increased bond strength values [12], once the adhesive is agitated into the root canal.

The use of an ultrasonic tip would be an interesting choice; it may be a viable and low-cost alternative, and does not need previous operator calibration. However, no studies in the literature have evaluated its use to increase the adhesive/resin cement adhesion in fiber glass post cementation. Therefore, the objective of this study was to evaluate the bond strength, nano leakage and degree of conversion of the adhesives applied in the root dentin, with passive and active manual application and vibratory application using an ultrasonic tip prototype, designed for this purpose.

## Materials and Methods

Sixty-six single rooted premolars were used in this *in vitro* study. The inclusion criteria were absence of clinical signs of caries, radicular dilacerations, previous endodontic treatment, and at least 14 mm of radicular length, measured from the cement-enamel junction. Teeth were transversally sectioned immediately above the cement-enamel junction using a low-speed diamond saw (ISOMET 1000, Buehler, Lake Bluff, IL, USA) under constant refrigeration. Canal exploration was performed by #10 K-files (Dentsply Maillefer, Ballaigues, Switzerland), up to #40 and the crown-down technique was made until #55 K-file. The canals were irrigated with 1% sodium hypochlorite between each file.

The canals were neutralized with saline solution and dried with paper points (Dentsply Maillefer, Ballaigues, Switzerland), and only 4 mm of the apical portion were sealed, by the warm vertical condensation technique, with gutta-percha points (Tanari, Manacapuru, AM, Brazil) and resin-based sealer (AH-Plus, Dentsply Maillefer, Ballaigues, Switzerland). The root access was temporarily filled with glass ionomer (Vitro Fill, DFL, Rio de Janeiro, RJ, Brazil), and the specimens were stored in distilled water (37±1 °C) for one week.

Afterwards, the post-spaces were prepared with low-speed burs Gates Glidden drills #2 to #4 (Dentsply Maillefer; Ballaigues, Switzerland) with apical enlargement to size 40/0.06, indicated by the post manufacturer (White Post DC #2, FGM, Joinville, Brazil), and leaving the 4 mm of apical seal. The canals were irrigated with 10 mL of distilled water and dried with paper points (Dentsply Maillefer, Ballaigues, Switzerland). The fiber glass posts were cleaned with 70% alcohol for 30 sec.

At this moment, the specimens were randomly divided in 2 groups (*n*=33), according to the cementation system: Adper Single Bond 2/RelyX ARC (SBAR) and Single Bond Universal/RelyX Ultimate (SBUL). Each group was then subdivided in 3 subgroups (*n*=11), according to the adhesive application technique: manual passive (MP); manual active (MA) and vibratory application (VA) using an ultrasonic tip prototype.

In manual passive (MP): the adhesive system was just left on the root walls with a microbrush (Vigodent, Rio de Janeiro, Brazil), without digital pressure, neither agitation. Manual active (MA): the adhesive system was applied vigorously according to the manufacturer's instructions in the root walls with a micro brush (Vigodent, Rio de Janeiro, Brazil), with a pressure of 34.5±6.9 gr (the operator was calibrated in an analytical balance). For vibratory application (VA), the adhesive system was applied in the root walls according to application protocol suggested by the manufacturer's instructions and the micro brush (Vigodent, Rio de Janeiro, Brazil) was adapted to an ultrasonic tip prototype, connected to the ultrasonic equipment (DabiAtlant, Ribeirão Preto, SP, Brazil) adjusted at frequency of 24.000-30.000 Hz.

In SBAR group the root canal walls were etched with 37% phosphoric acid gel (Condac 37, FGM, Joinville, SC, Brazil) for 15 sec and then washed for 10 sec. The SBUL group was just washed for 10 sec, then the root canals were dried with compressed air for 10 sec at 2 cm and dried with two #40 paper points, the adhesive systems were applied according to the different application techniques previously described and light curing with a LED light-curing device (Radii Plus, SDI; Victoria, Australia) using a power density of 1200 mW/cm$^2$, positioned at of the root for 20 sec.

The fiber posts were cleaned with 70% alcohol for 5 sec. The resin cement was inserted into the root canal with a #0 insulin syringe (BD, São Paulo, SP, Brazil) with #40 needle (Injex, Ourinhos, SP, Brazil). Using a LED light-curing device (Radii Plus, SDI, Victoria, Australia) using a power density of 1200 mW/cm$^2$, ten millimeters of the post lengths were cemented inside the root canal, while the remaining cervical 3 mm served as a guide to standardize the distance of the light-curing device from the cervical root, for 20 sec.

After cementation, all specimens were stored in distilled water at 37±1° C for one week, and then embedded in acrylic resin (Jet®, Art. Clássico; São Paulo, SP, Brazil) inside polyvinyl chloride tubes. The specimens were transversally sectioned with a low-speed diamond saw (ISOMET 1000, Buehler, Lake Bluff, IL, USA) under constant refrigeration. Slices of 1.0±0.1 mm were obtained, measured by a digital caliper (Mitutoyo, Digimatic Caliper, Tokyo, Japan), with accuracy of 0.01 mm. The first slices of all roots were discarded, as the excessive resin cement in this region would influence the results. For each root, 6 slices were obtained, 2 for each third: coronal, medium and apical. In each group (*n*=11), seven teeth were randomly evaluated by push-out bond strength test, two teeth for nano leakage and two teeth for degree of conversion, by micro-Raman spectroscopy.

For the push-out bond strength (BS) test, each slice was identified, and its thickness was measured with digital calipers (Mitutoyo; accuracy of 0.01 mm). The slices were photographed on both sides with an optical microscope under 40× magnification (Olympus, model BX 51, Olympus, Tokyo, Japan) to measure the coronal and apical diameters of the posts with the purpose of calculating their individual bonding areas. This measurement was made with software (Software Image J 1.48 version, NIH, USA).

The push-out test was performed in a universal loading machine (Instron, Canton, MA, USA) at a load of 50 kg and a crosshead speed of 0.5 mm/min. The load was applied in the apical-coronal direction until the post was dislodged. Care was taken to center the push-out pin on the center of the post surface without stressing the surrounding post space walls. The failure load was registered in Newtons (N), and the bond strength value, in MPa, was calculated with the surface equation, side of a truncated cone.

For nano leakage (NL) at the bonding interface, all the slices of the 2 teeth were immersed in a tracer agent (aquous silver nitrate solution), for 24 h. The specimens were cleaned with distilled water for 2 min, and then developed for 8 h (developing solution, Kodak). After this period, the specimens were ultrasonically cleaned for 10 min and under tap water.

The specimens (*n*=2) were mounted in stubs and polished with a 1000, 1500, 2000 and 2500-grit SiC papers and 1 and 0.25 µm diamond paste (Buehler Ltd, Illinois, USA) under constant refrigeration. Afterwards, the slices were gold-sputtered (Balzers SCD 050 Sputter Coater) and analyzed by Scanning Electron Microscopy (SSX-550; Shimadzu, Tokyo, Japan) operated in the backscattered electron mode at 12 kVp. Three pictures were taken of each slice (1000× and 60× of magnification), where the silver nitrate amount at the bonding interface would be quantify. The relative percentage of silver nitrate uptake within the adhesive layer areas was measured in all pictures using the Image J software.

***Table 1.*** Mean (SD) of bond strength values (MPa) of the different experimental groups

| Zone | Application Technique | | | Cementation System | Application Technique | | |
|---|---|---|---|---|---|---|---|
| | Manual Passive | Manual Active | Vibratory Active | | Manual Passive | Manual Active | Vibratory Active |
| **Coronal** | 11.3 (1.4) $^{Cd}$ | 17.4 (3.7) $^{B}$ | 23.8 (0.8) $^{A}$ | Adper Single Bond/Relyx Arc | 09.2 (01.7) $^{C}$ | 17.2 (04.0) $^{Ab}$ | 17.4 (08.2) $^{Ab}$ |
| **Middle** | 08.1 (0.2) $^{Cd}$ | 13.2 (1.6) $^{Bc}$ | 15.0 (4.8) $^{Bc}$ | Single Bond Universal/Relyx Ultimate | 10.3 (02.9) $^{C}$ | 17.8 (12.7) $^{B}$ | 21.3 (04.3) $^{A}$ |
| **Apical** | 07.5 (0.8) $^{Cd}$ | 13.5 (0.5) $^{Bc}$ | 10.6 (1.4) $^{Cd}$ | | | | |

** Distinct letters show statistically significant differences (P≤0.05)*

***Table 2.*** Mean (SD) values of the different experimental groups

| Zone | Cementation System | | Cementation System | Application Technique | | |
|---|---|---|---|---|---|---|
| | Adper Single Bond/ Relyx Arc | Single Bond Universal/Relyx Ultimate | | Manual Passive | Manual Active | Vibratory Active |
| **Coronal** | 25.7±09.5 C | 15.5±05.9 Ab | Adper Single Bond/ Relyx Arc | 39.1±08.2 D | 24.8±10.4 C | 19.6±05.7 B |
| **Middle** | 27.6±08.8 C | 18.1±02.5 B | Single Bond Universal/Relyx Ultimate | 16.2±07.6 B | 17.5±08.3 B | 11.4±05.3 A |
| **Apical** | 30.2±12.0 C | 11.5±01.7 A | | | | |

** Distinct Letters Show Statistically Significant Differences (P≤0.05)*

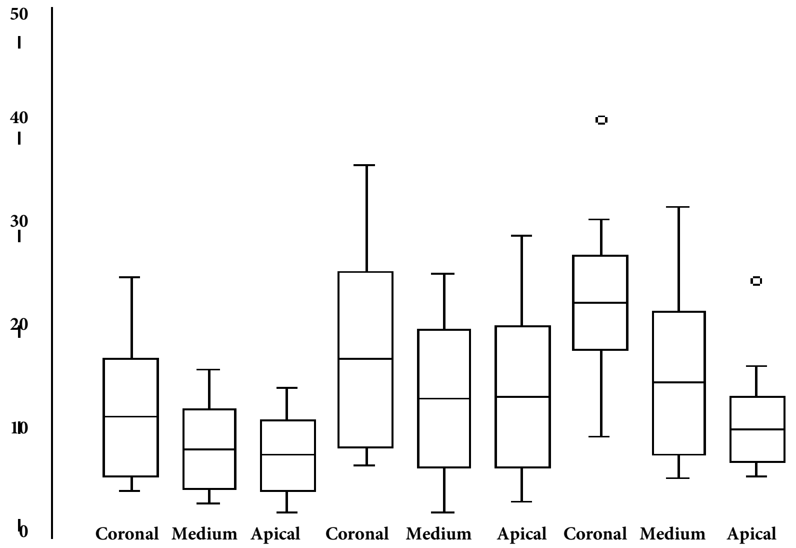


***Figure 1.*** Box-Plox, Mean bond strength values (MPa) and standard deviation of the different experimental groups. 2-way ANOVA between application technique/cementation system ($P$=0.045) were significant *

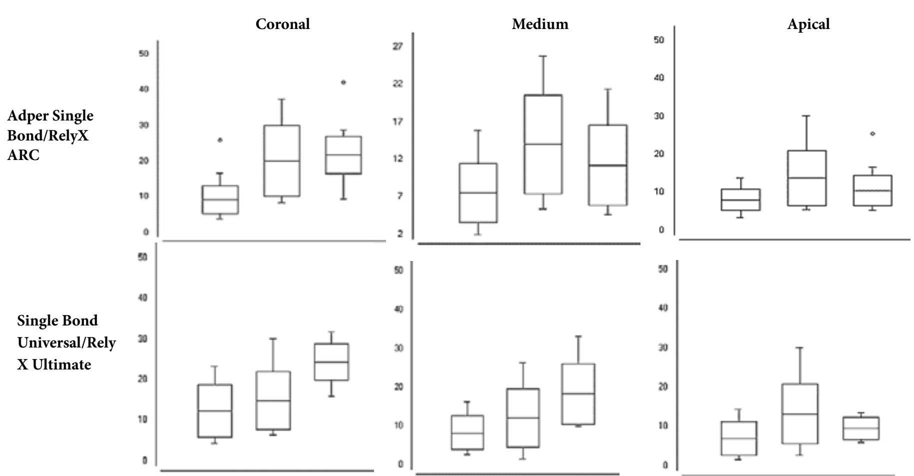


***Figure 2.*** Box-Plox, Mean bond strength values (MPa) and standard deviation of the different experimental groups. 2-way ANOVA between application technique/root region ($P$<0.001)

To evaluate the degree of conversion (DC) of the adhesive system at the hybrid layer, the micro-Raman spectroscopy was performed according to a previous study [13]. Each slice was polished with 1500 and 2000-grit SiC papers (Buehler Ltd, Illinois, USA) under constant refrigeration, ultrasonically cleaned for 30 min and then stored in water for 24 h at 37˚C before taking the DC readings. The micro-Raman equipment (Senterra, Bruker Optik GmbH, Karlsruhe, Germany) was first calibrated for zero and then for coefficient values using a silicon sample. The spectrometer consisted of a 20 mW Neon laser with 785 nm wavelength, spatial resolution of ≈3 µm, spectral resolution ≈5 $cm^{-1}$. The slit width of the spectrograph was set at 25 µm. Five co-additions were performed with an accumulation time of 30 sec, at 20× magnification (Olympus BX 51 model, Olympus; Tokyo, Japan)) to a ≈1 µm beam diameter. Spectra were taken at three sites per each slice, in the hybrid layer. The spectral region analyzed was 1785-875 $cm^{-1}$ [14]. The DC of the adhesive system was calculated using the specific equation.

The results of bond strength, nano leakage and degree of conversion were submitted to Image J software (National Institutes of Health; Bethesda, MD, USA) and analyzed by two-way ANOVA (comparing application technique, cementation system and root third) and Tukey's post-hoc ($\alpha$=0.05).

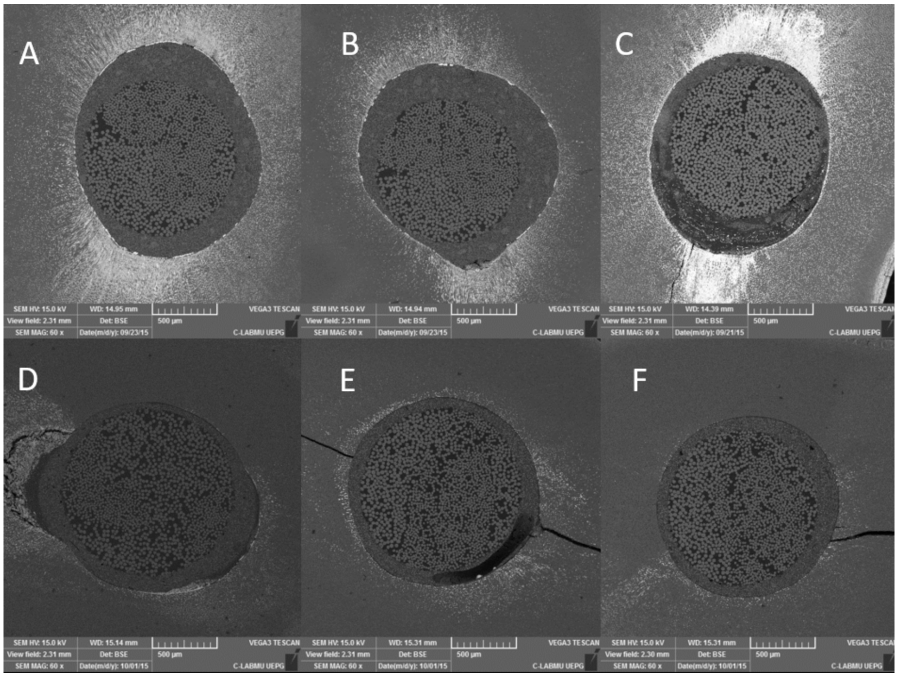


***Figure 3*** Images of nanoleakage obtained by the SEM (60×), of representative root region and cementation systems. The letters A and D represents coronal thirds, B and E medium thirds and C and F apical thirds. The letters A, B and C have used Adper Single Bond 2/RelyX ARC cementation systems and letters D, E and F have used Single Bond Universal/RelyX Ultimate cementation systems

## Results

### *Bond strength (BS)*

Two-way ANOVA between application technique/root region ($P<0.001$) (Figure 1) and application technique/cementation system ($P=0.045$) (Figure 2), were significant (Table 1).

For the interaction between application technique/root region, a significant increase in BS values was observed with the vibratory application using the ultrasonic tip in the cervical third ($P<0.001$), followed by manual active application in the same third. In relation to the interaction between application technique/cementation, for both cementation systems, the lowest results were observed with manual passive application.

### *Nano leakage (NL)*

Three-way ANOVA showed that the triple interaction was not statistically significant ($P=0.18$), but the double interaction between cementation system/root region ($P<0.001$) and cementation system/application technique ($P=0.045$) were statistically significant (Table 2).

For the interaction cementation system/root region, for Adper Single Bond/RelyX ARC (SBAR), no significant differences were observed among the different root regions, being all values statistically higher than Single Bond Universal/ RelyX Ultimate (SBUL) (Figure 3).

In relation to the interaction between cementation system/application technique the manual passive application technique for SBAR showed the highest NL values; and the lowest values were observed with vibratory application technique for SBUL (Figure 4).

### *Degree of conversion (DC)*

For the interaction between application technique/root region, to coronal and medium thirds of the root canal, the manual active and vibratory application techniques showed statistically higher DC values than the manual passive technique. However, for the apical third, no differences were observed among the different application techniques. In relation to the interaction between cementation system/ application technique, the lowest results were observed with manual passive application for SBUL.

## Discussion

Several factors may account for the lower adhesion performance at the root canals, such as: the effect of operator experience [15] the brand of the material [16]. The variables of commercial adhesives occurs within a narrow range of moisture levels and depends on the solvent composition of the adhesive [17], it is most improbable that solvent evaporation occurs adequately in this region [16].

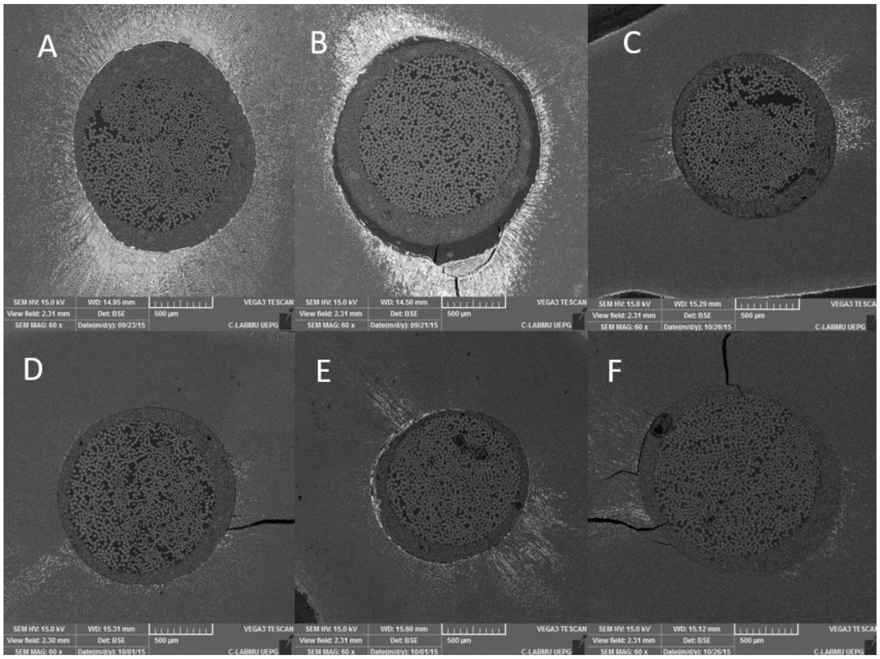


***Figure 4.*** Images of nanoleakage obtained by the SEM (60×), of representative cementation systems and application technique. The letters A and D represents manual passive, B and E manual active and C and F vibratory application technique. The letters A, B and C have used Adper Single Bond RelyX ARC and letters D, E and F have used Single Bond Universal/RelyX Ultimate cementation systems

Considering all these variables, an active mode application which reduces the sensitivity of the technique, dispensing the operator calibration, was developed at this study with the ultrasonic tip prototype. An appropriate design was necessary to be suitable to root dentin, with corresponding frequencies to provide an ideal vibration for the clinical application of this study [18]. Currently, the ultrasonic frequencies used in Dentistry operate in the 25-40 kHz range [7]. The prototype used in this study was adjusted in the 24-30 kHz range, according to the ultrasonic device used.

The results of present study showed that any use of active application, both with ultrasonic or active manual application improve the BS and NL comparing passive application, these results are in agreement with [8, 19]. The effects of sonic application on BS and NL values were similar to those obtained with active manual application as recommended by the manufacturers. A plausible explanation for the better BS results with the active and vibratory techniques would be that adhesive agitation into the root dentin promotes faster solvent evaporation. At the same time facilitating the penetration of resin monomers in substrate [19] and reaches better micromechanical retention with the subjacent substrate [20].

The universal adhesive system (Single Bond Universal) demonstrated better BS and NL values associated with the ultrasonic application. All the NL values for this self-etch universal adhesive system were lower than those for the (Adper Single Bond), regardless of the root area. SBU contains the acidic monomer 10-methacryloxidimethyl phosphate (MDP), a hydrophilic monomer that can form stable MDP-Ca salts through strong chemical bonding with hydroxyapatite and is also resistant to hydrolysis, contrary to the ASB adhesive (Feitosa Gruber). Also, the lower NL results are attributed to the vibratory technique could be explained because vibratory tip does not need previous operator calibration, reducing the sensibility of the technique (14). All these above mentioned facts may contribute to better NL results obtained by the self-etch adhesive (Single Bond Universal) in root dentin, compared to conventional etch-and-rinse adhesive.

In general, DC values did not show significant differences between the groups. The lowest values were attributed to the passive application technique with the etch-and-rinse strategy. Although few studies have investigated *in situ* DC of the hybrid layer in root dentin, these results are similar to that reported in other studies [18], showing that root-dentin adhesion is still a highly complex procedure.

It is worth to mention that, as an *in vitro* study, active application was standardized which did not reproduce the clinical routine. Further clinical studies are required to better evaluate the long-term performance of the ultrasonic technique protocol in a clinical routine, with other cementation systems and application modes.

With the limitation of this study, it may be concluded that the active application techniques (manual or vibratory) of the adhesive systems should be the first choice for clinicians. The ultrasonic tip may be an interesting choice, once it is a low-cost alternative and enhances the adhesion between fiber glass posts and root dentin.

## Conclusion

The highest bond strength value was observed for the active vibratory application technique in the cervical third, followed by the manual active technique at the same root region.

The cementation system Single Bond Universal/RelyX Ultimate demonstrated the lowest nano leakage values, obtaining better results using vibratory application with the ultrasonic tip, compared to manual active and passive application.

The manual active and vibratory application techniques showed statistically higher degree of conversion values than the manual passive technique at the coronal and medium thirds; and the lowest results were observed with manual passive application to Single Bond Universal/RelyX Ultimate.

Conflict of Interest: 'None declared'.

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