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Resin Bonding to Root Canal Dentin: Effect of the Application of an Experimental Hydrophobic Resin Coating after an All-in-one Adhesive

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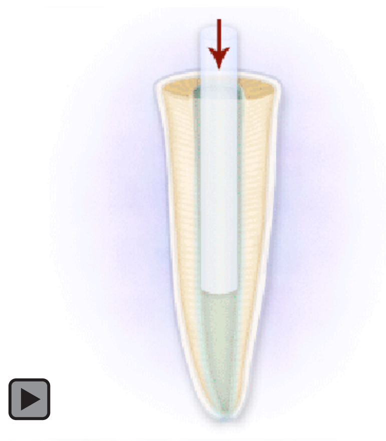

Abstract

Aim: Based on the hypothesis the application of a low-viscosity hydrophobic resin coating improves the bond of all-in-one adhesive, the purpose of the study was to evaluate the bond strength of four adhesive systems to bovine root dentin using the push-out test method.

Methods and Materials: The root canals of 32 bovine roots (16 mm) were prepared to a length of 12 mm using a FRC Postec Plus preparation drill. The specimens were allocated into four groups according to the adhesive system used: (Group 1) All-in-one Xeno III; (Group 2) All-in-one Xeno III+ScotchBond Multi-Purpose Plus Adhesive; (Group 3) Simplified Etch & Rinse One Step Plus; and (Group 4) Multi-Bottle Etch & Rinse All-Bond 2. A fiber-reinforced composite retention post was reproduced using an additional silicon impression and fabricated with DuoLink resin cement. The root specimens were treated with the selected adhesive systems, and the resin posts were luted in the canals with DuoLink resin cement. Each root specimen was cross sectioned into four samples (±1.8 mm in thickness), and the post sections were pushed-out to determine the bond strength to dentin.

Results: Group 2 (2.9±1.2) was statistically higher than Group 1 (1.1±0.5) and Group 3 (1.1±0.5). Groups 1 and 3 showed no statistically significant difference while Group 4 (2.0±0.7) presented similar values ($p>0.05$) to Groups 1, 2, and 3 [(one-way analysis of variance (ANOVA)] and Tukey test, $\alpha=0.05$).

Conclusion: The hypothesis was accepted since the application of the additional layer of a low-viscosity bonding resin improved the bond of the all-in-one adhesive. Further studies must be conducted to evaluate the long-term bond.

Keywords: Bond strength, adhesive systems, root dentin, simplified adhesive, push-out test

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Introduction

Dental adhesives have been used in several clinical conditions especially to improve the retention strength of restorations and to prevent microleakage. Initially multi-bottle, total-etch adhesive systems were used. Now simplified adhesive systems, such as total-etch and self-etch systems, have been introduced.[1-5]

The adhesive luting of prefabricated fiber-reinforced composite (FRC) posts has been considered mandatory in order to optimize the pull-out strength of these posts. The clinical performance of a prosthetic restoration with a fiber post depends on several factors[6] such as: the type of the post material, shape, dimension, and length;[7-10] the quality and quantity of remaining dentin;[11-12] the type of adhesive and cement used; and the adaptation of the post inside the root canal.[13-14]

However, the resin bond to root dentin is problematic as the bond strength can be affected by some factors as follows:

1. The dentin substrate provides a lower hybridization potential.[15,16]
2. There is a higher configuration factor in root canals (high polymerization stress of resin cements),[17,18] and the bond to root canal dentin appears to be attributed to friction resistance.[19]
3. It is difficult to perform light curing inside the root canal.[14,20]
4. There may be chemical incompatibility between the adhesive system and resin cement.[21-27]

Some studies have indicated a probable chemical incompatibility between adhesive systems with low pH and resinous materials of chemical- and dual-polymerization.[21-27] A correlation was observed between the decline in microtensile bond strengths of chemical-cured composites coupled to bonded dentin and the acidity of these adhesives.[23] Another study[21] evaluated the experimental application of an additional hydrophobic adhesive layer on the crown dentin after a self-etching primer was used for adhesive luting. The authors noted this additional resin layer significantly improved the bond strength by 35%, mainly due to the decrease in the pH of the surface contacting the resin cement.

Thus, the aim of this study was to evaluate the push-out bond strengths of a multi-bottle and three simplified adhesive systems to bovine root dentin. The hypothesis was the experimental application of an additional layer of a non-acidic low-viscosity hydrophobic bonding resin improves the bond strength of the all-in-one adhesive.

Methods and Materials

Specimen Preparation

Thirty-two single-rooted bovine teeth (mandibular incisors) were used in the present study. The teeth were cleaned with periodontal curettes and stored in distilled water. The coronal and cervical portions of the root were sectioned to standardize the length of the roots at 16 mm. The coronal diameters of the canals were measured with a digital caliper (Starrett® 727, Starrett, Itu, Brazil), and specimens presenting diameters much larger than the diameter of a retention post (1.5 mm) were discarded and replaced by other specimens meeting this requirement.

To create the test specimens the root canals were prepared at 12 mm, using the #3 preparation bur of a taper glass fiber-reinforced-resin post system (FRC Postec Plus, Ivoclar-Vivadent AG, Schaan, Liechtenstein), at low-speed under a water coolant.

Each specimen was embedded in a cylinder-shaped silicon mold filled with chemically cured acrylic resin (Dencrilay®, Dencril, Caieiras, SP, Brazil). The following procedure was performed to appropriately place the specimen in the mold:

1. The preparation bur of the post system was placed inside the prepared root canal.
2. This bur-root assembly was attached to an adapted surveyor with the long axis of the bur, specimen, and cylinder parallel to each other and to the y axis.
3. The acrylic resin was prepared and poured inside the cylinder, covering half of the root length.[3]

Adhesive Luting

Initially, one FRC was molded in polyvinylsiloxan impression material (Elite H-D Putty, Zhermack, Rovigo, Italy, Batch #19915) that was then used to fabricate 32 posts using DuoLink resin cement (Bisco, Schaumburg, IL, USA, Batch #0300014555). Each resin post was prepared immediately before its cementation.

The adhesive systems were applied using the manufacturer's instructions as described below:

Group 1 (X-III)

Xeno III Single-step self-etching adhesive (Dentsply, Milford, DE, USA; Batch #0411001721) was used as follows:

1. The all-in-one adhesive was applied on the root dentin with microbrushes (Cavi-Tip®, Svenska Dental Instrument AB, Upplands Värby, Sweden).
2. Excess adhesive was removed with No. 80 absorbent paper points.
3. Curing was performed with an XL 3000 (3M/Espe, St. Paul, MN, USA) light curing unit (LCU) for ten seconds at a light intensity of 600mW/cm^2.

Group 2 (X-III+Adh)

Xeno III and an additional application of a non-acidic low-viscosity hydrophobic bonding resin (ScotchBond Multi Purpose Plus Adhesive®, 3M/Espe, St. Paul, MN, USA; Batch #12010) was used as follows:

1. The self-etching adhesive Xeno III was applied as in Group 1.
2. An additional resin coat (ScotchBond Multi Purpose Plus Adhesive® Adhesive) was applied with a microbrush (Cavi-Tip®).
3. Excess adhesive was removed with No. 80 absorbent paper points.
4. The adhesive was light cured for ten seconds using the XL 3000 LCU at a light intensity of 600mW/cm^2.

Group 3 (OS)

One-Step Plus® (Bisco, Schaumburg, IL, USA; Batch #0400001016), a simplified etch and rinse adhesive, was used as follows:

1. The root dentin was etched with 32% phosphoric acid (Uni-etch, Bisco, Schaumburg, IL, USA) and rinsed with 10 mL of water using a disposable syringe.
2. Excessive water was removed with No. 80 absorbent paper points.
3. Two coats of the One-Step Plus were applied Cavi-Tip® microbrushes for ten seconds each followed by removing the excess with No. 80 absorbent paper points.
4. The adhesive was light cured for ten seconds using the XL 3000 LCU at a light intensity of 600mW/cm^2.

Group 4 (AB2)

All Bond 2® (Bisco, Schaumburg, IL, USA; Batch #0400000860), a multiple-bottle, etch and rinse adhesive, was used as follows:

1. Etching with 32% phosphoric acid for 30 seconds.
2. Washing with 10 mL of water using a disposable syringe.
3. Removal of excess water with No. 80 absorbent paper points.
4. Mixing Primer A and Primer B (All Bond 2 System),applying the mixture, and removing excess material with a Cavi-Tip® microbrush.
5. Application of pre-Bond resin (All Bond 2 system) and removal of excess material with a microbrush.

The base and catalyst pastes of the dual resin cement DuoLink (Batch #0300014555) were mixed and inserted into the root canal with a Lentulo #40 (Dentsply/Maillefer, Ballaigues, Switzerland) and the resin post was placed inside the root canal. Light curing was performed with an XL 3000 LCU (3M/Espe, St. Paul, MN, USA) at a light intensity of 600mW/cm^2 on the coronal aspect for 40 seconds. No surface conditioning of the resin post was accomplished before it was cemented. The specimens were stored in distilled water at 37ºC for 24 hours.

Sectioning and Push-out Testing

Each specimen was fixed on the metallic base of a sectioning machine (LabCut 1010, Extec Corp., Enfield, CT, USA), allowing perpendicular sectioning along the root axis (Y axis) with a diamond disc under cooling spray to create approximately 1.8 mm thick slices of the filled roots. The first cervical slice (approx. 1 mm) was discarded because the excess of cement in that region could influence the result of the adhesive resistance. Four to five other slices per specimen were prepared.

Each specimen was positioned on a metallic device with a central opening (Ø = 3 mm) larger than the root canal diameter with the most coronal portion of the specimen placed downwards. For push-out testing, a metallic cylinder ($Ø_{extremity}$ = 0.85 mm) induced a load from the apical to coronal direction on the resin post. As the embedding of the specimens into the acrylic resin was performed parallel to the Y axis and the specimens were sectioned perpendicular to that axis, the resin post was submitted to parallel pressure to the highest possible extent in relation to the root axis (Y axis).

The test was performed in a universal testing machine (EMIC, São José dos Pinhais, Brazil) at a speed of 1 mm/min.

The bond strength (σ) in MPa was obtained by the formula:

$$\sigma = F/A$$

Where, **F** = load for specimen failure (N) and **A** = bonded area (mm^2).

To calculate the bonded area, a formula was applied to calculate the lateral area of the conical geometric figure of the sectioned retention post in the root canal that formed a circular straight cone trunk of parallel bases. The formula used for the area calculation was:

$$A = \pi \times g \times (R_1 + R_2)$$

Where, **π** = 3.14, **g** = trunk generatrix, R_1 = smaller base radius, R_2 = larger base radius. (Figure 1A)

For the conical trunk generatrix (g) calculation the Pythagorean theorem (the square on the hypotenuse is equal to the sum of the squares on the other two sides) was used as expressed in the following formula:

$$g^2=h^2+[R_2-R_1]^2$$

Where **g** = conical trunk generatrix, **h** = section height. R_1 and R_2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, corresponding to the internal diameter between the root canal walls. These diameters and h were measured with a digital caliper (Starrett® 727, Starrett, Itu, Brazil) (Figure 1B).

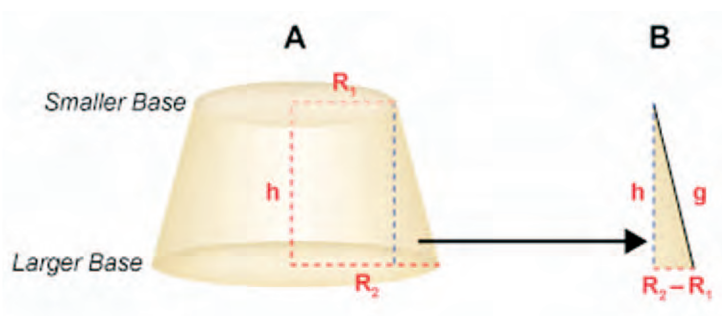

Figure 1. A. Schematic drawing that corresponds to the internal section of the canal walls, a geometric figure of a circular straight cone trunk of parallel bases; **B.** Geometric figure for calculation of the cone trunk.

Statistical Analysis

The mean bond strength values from each specimen were initially calculated from their respective repetitions. Considering each group was composed of eight specimens then eight bond strength values of each group (n=8) were employed for statistical analysis using a one-way analysis of variance (ANOVA) and a post-hoc Tukey test, $\alpha=0.05$.

Analysis of the Failure Modes

The tested specimens were analyzed under an Optical Microscope (Zeiss MC 80 DX, Zeiss, Jena, Germany) at a magnification of x50 in order to evaluate the type of fracture in the samples. The failure classification used was as follows:

1. Cohesive failure: fracture of the resin post
2. Adhesive failure: fracture between the resin cement and root dentin (pull-out of the resin post-cement)
3. Cohesive fracture of the root dentin: all samples were analyzed by three calibrated observers

Results

The ANOVA test revealed there was a statistically significant difference among the groups (*P*=.0002) (Table 1).

The post hoc Tukey test showed the bond strength (MPa) of Group 2 (X-III+Adh) (2.8±1.9[a]) to be significantly higher than Group 1 (X-III) (1.1±0.5[b]) (*P*=0.000771) and Group 3 (OS) (1.1±0.5[b]) (*P*=0.000615) bond strengths. Group 4 (AB2) (2.0±0.8[ab]) bond strength had an intermediary bond strength, being similar to Group 2 (*P*=0.147697), Group 1 (*P*=0.129451) and Group 3 (P=0.104906). Note different superscript letters indicate a significant difference while equal superscript letters indicate no significant difference (Figure 2).

The analysis of the samples tested revealed all of the fractures occurred between the resin cement and the root dentin (Type 2, adhesive type). The pattern of failure was very similar in all the groups. There was no cohesive fracture of the resin post or dentin.

Table 1. One-way ANOVA of the bond strength data.

Source	*df*	SS	MS	F	*P*
Between	3	17.745	5.915	9.44	0.0002
Within	28	17.5458	0.6266		
Total	31	35.2911			

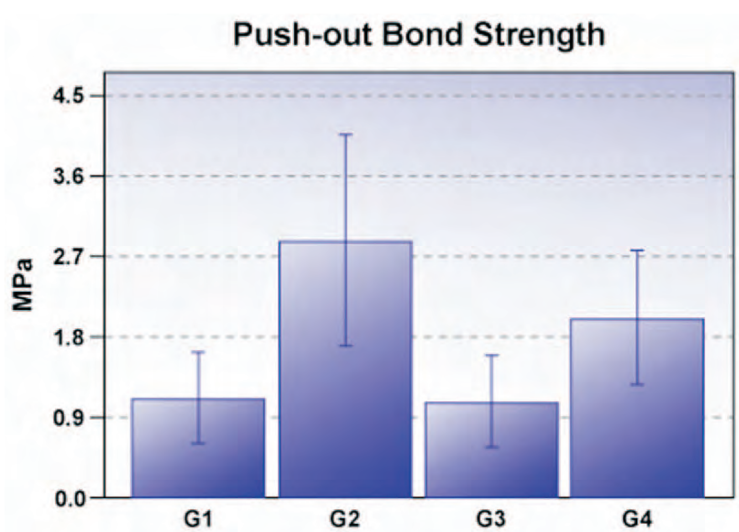

Figure 2. Mean and standard deviation of the push-out bond strength data.

Discussion

Resin posts were bonded into the root canal as a “substitute for a fiber post.”[17] Resin posts were cemented in this study to evaluate the influence of different adhesive strategies on the bond of resin to root dentin. This was done to prevent the possibility of failures occurring between the resin cement and a fiber post. At the same time, a higher configuration factor of root canals was reproduced.[17,18] It is obvious the cementation of the resin post is not clinically indicated.

The current study used bovine teeth to evaluate the push-out bond strength. The use of bovine teeth as a substitute for human teeth in bond strength- or microleakage-tests is controversial. While some studies have shown similarities of bovine teeth to human teeth,[28-31] other studies have found discrepancies between the two and the investigators have criticized the use of bovine teeth as a substitute-substrate.[32-34] It is suggested bovine teeth can be used initially to evaluate an adhesive-material or technique before implementing it clinically.[35-37] Tests using bovine teeth can be the first parameter in the evaluation process.

The results of the present study demonstrated the experimental application of an additional layer of a non-acidic hydrophobic low-viscosity resin after application of Xeno III (an all-in-one self-etching adhesive system) improved the bond strength significantly (Group 2: 2.9±1.2 MPa) when compared to only the application of the self etching adhesive all-in-one as recommended by the manufacturer.

These findings agree with the existing literature.[22-27] The self-etching adhesive system employed in Group 1 exhibited a low pH value, producing a chemical match with the polymerization chemical process of the dual-cure resin cement. Some studies[22-27] have indicated a possible chemical incompatibility between adhesive systems with low pH and resinous materials of chemical- and dual-polymerization. There is a correlation between the decline in microtensile bond strengths of chemical-cured composites coupled to bonded dentin and the acidity of these adhesives. The coupling of chemical/dual-cured composites with hydrated dentin bonded with these single-step self etch adhesives was also found to be inferior to the coupling achieved with light-cured composites since they contain a higher concentration of acidic resin monomers. It is known acidic resin monomers retard the polymerization of chemical/dual-cured composites initiated via peroxide-amine type binary redox catalysts.

Interaction between acidic adhesive resin monomers and the basic composite tertiary amines results in the consumption of the latter in acid-base reactions, depriving their capacity to generate free radicals in subsequent redox reactions.[24-25]

When an additional coat of a non-acidic hydrophobic resin was experimentally applied after the self-etch adhesive, the resin-coating more likely “neutralized” the acidic monomers in the self-etch adhesive. This phenomenon could have prevented the contact of dual resin cement with the acidic layer thereby reducing or avoiding the “attack” on the tertiary amines from the resin cement. Carvalho et al.[21] also noted the application of an additional hydrophobic adhesive layer, after a self-etching primer was used for adhesive luting, significantly improved bond strength by 35%.

King et al.[2] stated an “apparent incompatibility” to auto-cured composites resulted from the inherent permeability of one-step self-etching primers. Conversely, “true incompatibility” to auto-cured composites was caused by an adverse acid-base interaction masking the inherent permeability of this adhesive. According to the authors, “true-” and “apparent incompatibility” were eliminated upon their conversion to two-step self-etch adhesives.

However, “apparent incompatibility” develops in the long-term and not just after 24 hours. The aging of specimens using load masticatory simulation (mechanical cycling) can also be carried out. Thus, the low bond strength values to root canal dentin can be related to the combination of chemically incompatible materials rather than just the permeability of the simplified adhesive systems.

As noted by Goracci et al.,[19] the bond between the root post and root dentin is related to the friction resistance of the post to dentin rather than the bond of the adhesive system to dentin. This could explain the improved push-out bond strength of experimental Group 2 is the increased friction resistance. The application of an adhesive layer polymerized prior to the resin post cementation might have reduced the resin cement space between the resin post and dentin walls increasing the friction resistance.

Even though this experimental application of a hydrophobic adhesive layer increased the bond strength to root dentin, this procedure is not recommended. This finding is important to strengthening the chemical incompatibility between the all-in-one self-etch adhesives and self-cured/dual cured composites.

However, the application of this hydrophobic coat is common for multi-bottle etch and rinse adhesive systems. Although these adhesive systems are older than simplified adhesives, they have demonstrated good bond performance to dentin.[1-5] In the current study the bond strength of the multi-bottle etch-&-rinse adhesive system was similar to the “experimental group” Group 2. The multi-bottle adhesives use a hydrophobic resin coat which optimizes the chemical compatibility of self-cured and dual-cured composites.

Conclusion

1. The application of an additional layer of a low-viscosity bonding resin improved the bond strength of all-in-one Xeno III adhesive. The hypothesis was accepted.
2. The chemical incompatibility between dual-cured resin cement and all-in-one self-etch was observed through the bond strength test.

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