Effect of Adhesive System Type and Tooth Region on the Bond Strength to Dentin

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Purpose: This study evaluated the bond strength of two total-etch adhesive systems (two- and three-step) and a self-etching system to coronal and root canal dentin.

Materials and Methods: The root canals of 30 human incisors and canines were instrumented and prepared with burs. The posts used for luting were duplicated with dual resin cement (Duo-link) inside Aestheti Plus #2 molds. Thus, three groups were formed (n = 10) according to the adhesive system employed: All-Bond 2 (TE3) + resin cement post (rcp) + Duo-link (Dl); One-Step Plus (TE2) + rcp + Dl; Tyrian/One-Step Plus (SE) + rcp + Dl. Afterwards, 8 transverse sections (1.5 mm) were cut from 4 mm above the CEJ up to 4 mm short of the root canal apex, comprising coronal and root canal dentin. The sections were submitted to push-out testing in a universal testing machine EMIC (1 mm/min). Bond strength data were analyzed with two-way repeated measures ANOVA and Tukey's test ($p < 0.05$).

Results: The relationship between the adhesives was not the same in the different regions ($p < 0.05$). Comparison of the means achieved with the adhesives in each region (Tukey; $p < 0.05$) revealed that TE3 (mean ± standard deviation: 5.22 ± 1.70) was higher than TE2 (2.60 ± 1.74) and SE (1.68 ± 1.85).

Conclusion: Under the experimental conditions, better bonding to dentin was achieved using the three-step total-etch-system, especially in the coronal region. Therefore, the traditional total-etch three-step adhesive system seems to be the best choice for teeth needing adhesive endodontic restorations.

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In 1990, both dentists and dental materials companies noticed that metallic posts were too stiff when compared to dentin, leading to a critical transmission of loads to teeth previously weakened by instrumentation, dental caries, and extensive restorations.[11] This led to the development of fiber posts, which have been replacing the older methods for restoring endodontically treated teeth – that required large coronal destruction (metallic posts, crowns, and nonadhesive cements) – by techniques that preserve the dental tissue by utilizing adhesives and resin cements.[11,28]

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Currently, several types of dentinal adhesives are available:9 total dentin etching, with previous acid etching followed by primer and adhesive applied separately (three-step) or combined (two-step), and self-etching systems, which contain a self-etching primer and an adhesive, separately (two-step) or in one solution (all-in-one).

The total-etch systems require rinsing the acid etchant, which may overetch the dentin or obstruct the spaces around the collagen fibers if the echant is not well removed. Those with the primer and adhesive in a single bottle, and which contain acetone as a solvent, are very susceptible to moisture conditions of the dentin. When the dentin is excessively dried, the interfibrillar space is reduced and the diffusion of resin monomers is incomplete.9 On the other hand, excess moisture leads to dissolution of acetone in water, with the accumulation of resin, resulting in bubbles and incomplete dentin sealing.[1] Within this context, self-etching primers seem to be more advantageous, since they contain a high concentration of acidic monomers that demineralize the substrate, do not need to be removed with water, and simultaneously bond to the dentin.[31,34]

The dentin structure is another important factor that should be taken into account in terms of bonding. Dentinal

Table 1 Chemical composition and application procedures of the adhesives

All-Bond 2	Primer A: acetone, ethanol, Na-N-tolylglycine glycidyl methacrylate; Primer B: acetone, ethanol, biphenyl dimethacrylate; Pre-Bond resin: bisphenol A diglycidyl methacrylate, triethylene glycol dimethacrylate, benzoyl peroxide	1. Etching with 32% phosphoric acid (Uni-Etch, Bisco; Schaumburg, IL, USA) for 15 s 2. Water rinsing 3. Drying with absorbent paper points # 80 4. Application of Primer A + B with a microbrush (SDI Brasil Industria e Comercio; São Paulo, SP, Brazil) 5. Application of "Prebond Resin"
One-Step Plus	Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, glass	1. Etching with 32% phosphoric acid (Uni-Etch, Bisco) for 15 s 2. Water rinsing 3. Drying with absorbent paper points # 80 4. Application of two coats of One-Step Plus on the dentin with a microbrush 5. Air drying for 10 s 6. Light curing for 10 s with the tip of the light curing unit at the root canal opening and parallel to the root long axis
Tyrian SPE	Part A: Ethanol; Part B: 2-acrylamido-2-methylpropane suflonic acid, bis (2-(methacryloyloxy)ethyl) phosphate, ethanol	1. Application of solution primer Tyrian A + Tyrian B in the root canal with a microbrush and removal of excess with a microbrush after 20 s 2. Application of two coats of adhesive One-Step Plus on the dentin with a microbrush 3. Air drying for 10 s 4. Light curing for 10 s with the tip of the light curing unit at the root canal opening and parallel to the root's long axis

tubules in the root are straighter, less divergent,[19] and not as numerous as in the crown.[20] The cavity design is another critical variable in the development of stresses in root canal restorations. Even though the C-factor (the proportion of composite resin surfaces bonded to the cavity walls) varies from 1 to 5 in coronal restorations, it might be higher than 200 when posts are luted in the three-dimensional environment of the root canal.[7]

Regarding adhesively luted posts, authors agree that most failures, which are caused by several factors, occur at the interface between dentin and resin cement.[21,23] Thus, the objective of this study was to evaluate the bonding of three types of adhesive systems to the coronal (pulp chamber) and root canal dentin (cervical, middle, and apical levels). The following null hypotheses were established: a) the interaction effect on bond strength between the type of adhesive system and the tooth region does not exist, ie, the action of the adhesive system is the same for all tooth regions; b) the bond strength to dentin is not influenced by the type of adhesive system (μ All Bond = μ One-Step Plus = μ Tyrian+ One-Step Plus); c) the bond strength to dentin is not influenced by the tooth region (μ coronal = μ cervical = μ middle = μ apical).

MATERIALS AND METHODS

This study was conducted on 30 human maxillary incisors and canines, extracted for periodontal reasons. Soon after extraction, the teeth were placed in saline solution, cleaned with periodontal curettes, and frozen in distilled water (-18°C) for 15 days at most until use in the study. Selection of specimens was based on teeth with straight root canals and without caries or root resorptions.

The teeth were randomly divided into three groups (n = 10): a) Group 1: treated with the three-step total dentin etching adhesive system (TE3), All-Bond 2 (Bisco; Schaumburg, IL, USA); b) Group 2: treated with the two-step total-etch dentin adhesive system (TE2), One-Step Plus (Bisco); c) Group 3: treated with the two-step self-etching adhesive system (SE), Tyrian SPE/One-Step Plus (Bisco). The chemical composition of the adhesive systems is presented in Table 1.

Part of the crown of each tooth was removed 4 mm coronal to the CEJ, perpendicular to the tooth long axis on the buccal aspect, by means of a water-cooled diamond saw at low speed (Microdont; São Paulo, SP, Brazil). Following crown removal, the pulp was removed with a no. 15 K file (Dentsply Maillefer; Ballaigues, Switzerland). The root canal was widened up to 4 mm short of the apex with no. 15, 20,

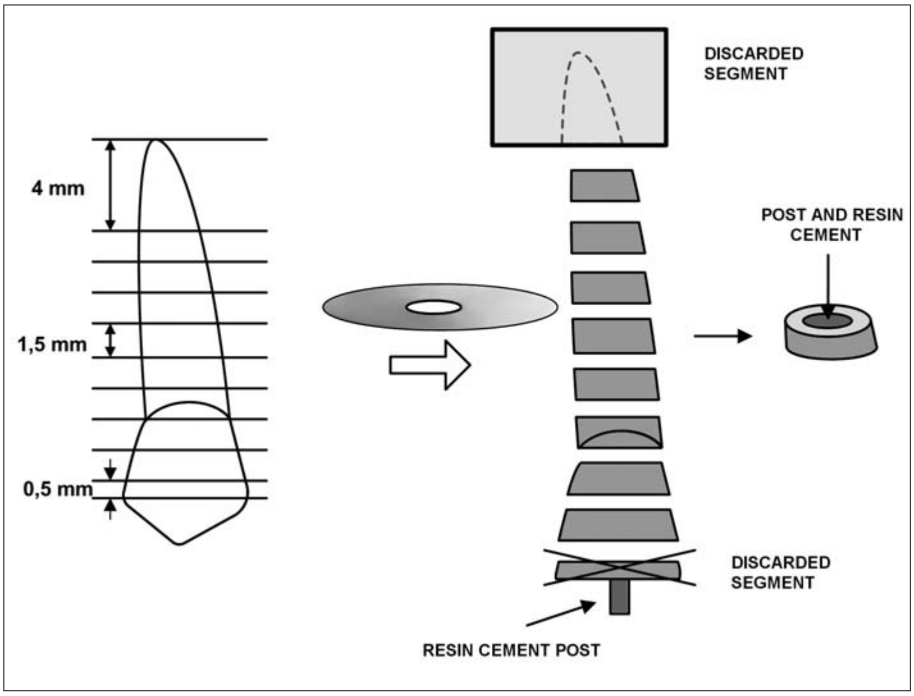


Fig 1 Schematic drawing of the tooth regions evaluated in the study and the preparation of transverse segments.

25, and 30 files (Dentsply Maillefer) followed by a no. 2 Largo bur (Dentsply Maillefer). At each change of instrument, the root canal was thoroughly irrigated with 0.5% NaOCl and suction was performed. To receive the posts, the roots were prepared with a preshaping bur followed by a no. 2 bur of the Aestheti Plus post system (Bisco).

Each root was positioned in the center of a cubic silicone mold (3 x 3 x 3 mm) and the surrounding space was filled with clear, chemically cured acrylic resin (Jet, Artigos Odontológicos Clássico; SP, Brazil). To allow the tooth long axis to be as perpendicular as possible to the ground, embedding was performed with the no. 2 bur of the Aestheti Plus post system inside the root canal, with its upper part connected to a surveyor (Bio Art Equipamentos Odontológicos; São Carlos, SP, Brazil). The posts were also attached to the surveyor for cementation. All of these procedures allowed the specimens to be cut in transverse segments where the adhesive interface format was approximately that of a circular right cylinder.

One fiber post (Aestheti Plus) was duplicated in dual-curing resin cement (Duo-link, Bisco) using a mold made out of silicon impression material. Then, 30 cement posts were produced.

Before cementation, the external lateral walls of the teeth received a coat of black nail varnish to allow passage of light only through the most coronal portion, since the root is clinically covered by periodontal tissues.

The materials were applied following manufacturers' instructions (Table 1). The posts were luted with Duo-link cement, prepared by mixing equal parts of base and catalyst for 10 s until a homogeneous color was achieved. Afterwards, the cement was inserted in the root canal with a no. 40 Lentulo bur (Dentsply Maillefer) and the cement post was placed in position. Each tooth was light cured for 40 s (Optilight Plus – Gnatus Equipamentos Médico-Odontológicos; Ribeirão Preto, SP, Brazil) at a light intensity of 450 mW/cm^2.

The embedded teeth were bonded to a metallic base with cyanoacrylate adhesive gel (Super Bonder gel, Loctite-Henkel; Itapevi, SP, Brazil). The metallic base was connected to a sectioning machine and the teeth were sectioned perpendicular to their long axis with a diamond saw (Microdont; São Paulo, SP, Brazil) under water irrigation. The first 0.5-mm section was discarded because the excess cement could lead to overestimation of the bond strength values in this segment. Overall, 8 segments, measuring nearly 1.5 mm, were achieved, with two from each study region (coronal; cervical, middle, and apical regions of the root) (Fig 1).

Each segment was positioned on a metallic device with a central opening larger than the root canal diameter. The most coronal portion was always placed facing downwards in relation to the load tip (apical-coronal load). The tip, a metallic cylinder with a diameter of 0.85 mm diameter at the end, was pressed onto the post center in an attempt to not touch the dentin. The test was performed with a universal testing machine (EMIC DL-1000, EMIC; São José dos Pinhais, PR, Brazil) at a crosshead speed of 1 mm/min with a load cell of 10 kgf. It should be noted that the calculation of the interface area (A) was performed with the formula for calculating the lateral area of a cylinder (Fig 2A).

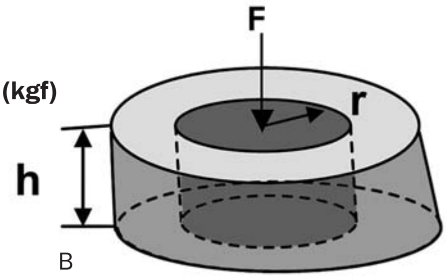
The radius (r) was obtained by measuring the internal diameters of the bases, corresponding to the internal diameter of the root canal walls in the segment. Both the diameter

R = F/A, where

A = 2Pr x h (mm^2)

F = load for rupture of the interface (kgf)

A



B

Fig 2 Formulas for calculating the interface area “A” and bond strength “R” (A) and a schematic drawing of the root canal walls of segment (B).

Table 2 Mean bond strengths (MPa) ± standard deviations and coefficient of variation (%) of the adhesive systems in four different regions

	Adhesives			
Region	All Bond 2	One Step Plus	Tyrian+One Step Plus	Row
Coronal	6.20 ± 1.99; (32.09)	5.18 ± 0.97; (18.72)	4.28 ± 1.50; (35.04)	5.22 ± 1.70; (32.56)
Cervical	3.87 ± 1.68; (43.41)	1.44 ± 0.92; (63.88)	2.50 ± 1.68; (67.20)	2.60 ± 1.74; (66.92)
Middle	3.35 ± 2.14; (63.88)	0.87 ± 1.10; (126.44)	0.80 ± 0.76; (95.00)	1.68 ± 1.85; (110.12)
Apical	3.56 ± 1.95; (54.77)	1.18 ± 1.01; (85.59)	0.64 ± 0.99; (154.69)	1.79 ± 1.86; (103.91)
Column	4.25 ± 2.20; (51.76)	2.17 ± 2.02; (93.08)	2.06 ± 1.94; (94.17)	

n =10

Table 3 Repeated measures ANOVA for bond strength data (MPa) after logarithmic transformation

Source of variation	df	SQ	QM	F	p
Adhesive	2	1.97799	0.98900	24.91	0.0001*
Residue I	27	1.07189	0.03970		
Region	3	3.61087	1.20362	41.54	0.0001*
Adhesive vs Region	6	0.55663	0.09277	3.20	0.0072*
Residue II	81	2.34687	0.02897		
Total	119	9.56425			

*p < 0.05

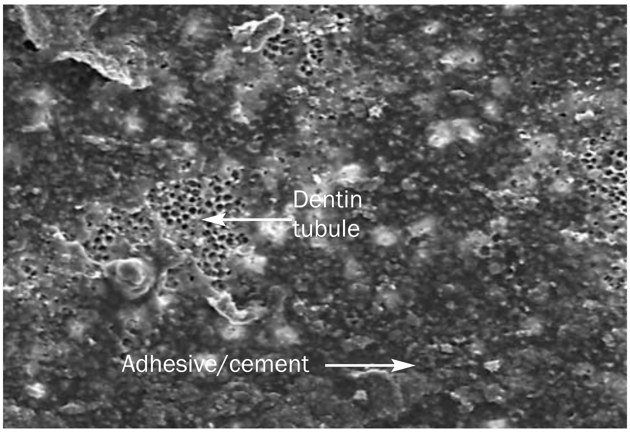


Fig 3 TE2; cervical region. The arrows show (a) a dentin tubule and (b) adhesive/cement remnants.

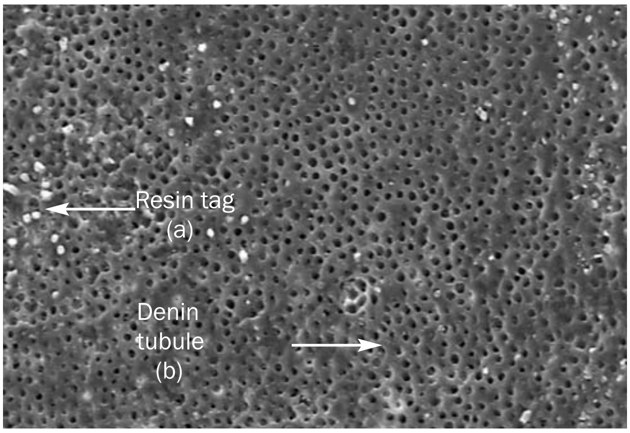


Fig 4 TE3; cervical region. The arrows show (a) a dentin tubule and (b) a resin tag.

and height (h) were measured after testing with a digital caliper (Fig 2B).

The bond strength values, initially in kgf/mm^2, were then converted into MPa by multiplying by a conversion factor of 9.807. The mean bond strength was calculated for the two sections of each tooth region.

Some specimens of each group were submitted to SEM analysis (JEOL-JSM-T330A, JEOL; Tokyo, Japan) to observe the fractured interfaces.

RESULTS

Due to different experimental conditions, dispersion (different standard deviation values), high coefficients of variation, and higher bond strength values in the coronal region, logarithmic transformation of the data was performed. Descriptive statistics of these data are presented in Table 2.

Application of the two-way repeated measures ANOVA test ($p < 0.05$) to investigate the influence of "type of adhesive" and "region" on the bond strength revealed that the interaction effect of these two variables was statistically significant (Table 3). This indicates that the relationship between the adhesives was not the same for the different regions.

When the means achieved in each region were compared by Tukey's test ($p < 0.05$), the following was observed:

a) For the coronal region: SE was lower than TE3, and TE2 was not different from the others.
b) For the cervical region: TE2 was lower than TE3, and SE was not different from the others.
c) For the middle region: SE and TE2 were lower than TE3 and were not different from each other.
d) For the apical region: SE and TE2 were lower than TE3 and were not different from each other.

The images representing the fractures in three segments, one for each type of adhesive, are displayed in Figs 3 to 5. The fracture patterns are discussed below.

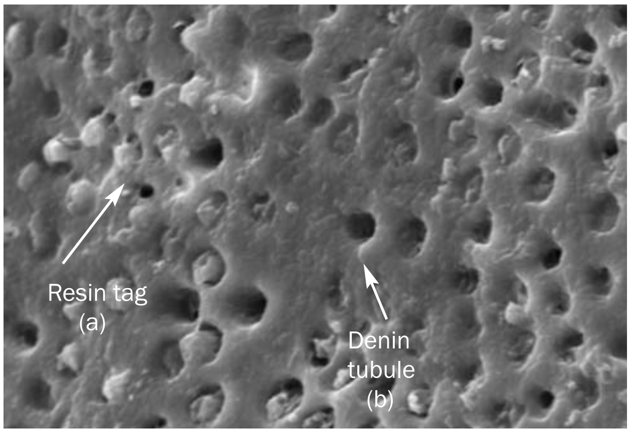


Fig 5 SE; coronal region. The arrows show (a) a dentin tubule

DISCUSSION

The great advantage of push-out testing is the possibility of evaluating regional differences in bonding with the same tooth,[15, 32] and thus it has been widely employed for evaluating adhesive materials in the root canal.[15,18,25,26] However, when compared to conventional shear and tensile methods, there is a reduction in bond strength in push-out specimens, since the resin polymerization stresses could pull the restoration from the dentinal walls, generating stresses at the interface with the tooth.[18] While discussing the validity of the single push-out test for bone fixed implants, An and Draughn[3] stated that it is known that most push-out tests do not measure pure shear strength, but report a value that is a combination of various fixation principles, such as friction, mechanical interlocking and chemical bonding. The authors say that researchers should use strictly designed and detailed protocols that allow comparisons within a single study, although the procedures do not allow studies to be comparable. Comparing the performance of specimens for the microtensile test (machined or not) with the push-out test, Goracci et al[15] observed a large number of early failures in the former case, supporting use of the push-out test to determine bond strengths to root canal dentin.

Many studies have already been conducted concerning the effect of obturation materials, or their components, on adhesives and resin cement polymerization and bond strengths to dentin.[4,6,16,18,24] A clean dentinal surface after removing the obturation material is a critical variable for retention of fiber posts luted with resin.6 Since the objective of this study was to investigate the bond strength between adhesive and dentin, any other material present at this interface could lead to misinterpretation of the results. Thus, root canal obturation was not performed.

Depending on the region, hybridization is also hampered by the low number of tubules per mm^2, dropping from 40,000 in the coronal dentin to 14,400 in the apical portion of the root.[12] That is, fewer tubules are available for resin penetration into the apical portion of the root canal.[22] Another problem is sclerotic root dentin, which is less soluble in acids when compared to nonsclerotic dentin. Thus, the acidic monomers of self-etching systems may not solubilize enough mineral to achieve a long-lasting bond between resin and sclerotic dentin.[34] These features might lead to great variability in bond strength data, making it imperative to consider the variability before reaching any conclusions.

With the exposure of the collagen fiber network after acid etching and infiltration with resin, a resin-dentin interdiffusion zone forms, with resin tags and lateral branches of adhesive, creating micromechanical retention to the demineralized dentin. After microscopic analysis, Ferrari et al[13] suggested two explanations for the reduced formation of the resin-dentin interdiffusion zone and tags in the apical third of post preparation: 1) the pressure applied by the microbrush with the solution is maximal in the cervical third and minimal in the apical third, leading to little penetration of the adhesive; 2) the number of tubules in the apical third is lower. In a similar study, Vichi et al[30] observed that the formation and morphology of resin tags were more evident at the cervical and middle root thirds. In addition, they observed no statistical difference between groups for the coronal region, even though the two-step total-etch systems achieved fewer tags in the middle and apical regions than did the the three-step groups.

Therefore, bond strength variability due to dentin bonding locations can be more than a structure-related problem. In the present study, Tyrian/One-Step Plus bond strengths to dentin were probably greatly influenced by the thick smear layer in the most apical areas. It is believed that the acids of self-etching materials, such as Tyrian SPE, are weak and less effective than phosphoric acid for dissolution of the thick smear layer observed after preparation with burs.[5,29] It is also assumed that the minerals present in the smear layer are able to neutralize the acidity of self-etching primers.[27] One advantage of self-etching systems is that they demineralize and infiltrate the monomer into the dentin simultaneously. However, a recent study by Wang and Spencer[33] showed that microvoids might exist in the self-etching adhesive interfaces with dentin, mainly because collagen fibrils were not totally enveloped by the resin.

Furthermore, both the extrinsic and intrinsic moisture of the dentinal substrate should be taken into account to achieve successful bonding. Some moisture in dentin is important, since the dried substrate presents a collapsed collagen network with up to 65% shrinkage of the dentin matrix, impairing the infiltration of resin.[1] Despite that, even in endodontically treated root canals in which a very moist substrate is not expected, adhesives comprising a lower number of steps have a problem related to permeability. According to Chersoni et al,[10] this explains the appearance of large bubbles on the surface of a dentin model treated with the two-step total-etch system, One-Step Plus. Even the self-etching primer Tyrian, which is expected to not entirely remove the smear layer, did not present a reduction in permeability. In the same work,[10] the number of fluid droplets followed the pattern All-Bond 2 < One-Step Plus < Tyrian SPE/One-Step Plus. The authors emphasized that One-Step Plus is a fairly permeable adhesive, suggesting that the low permeability of Pre-Bond Resin might have accounted for fewer fluid droplets in the All-Bond 2 group.

In addition to the problem of permeability of adhesives, it is known that composite resins containing tertiary amines as catalysts do not present good bonding to most two-step total-etch adhesives and single-step self-etching systems. In the current study, the One-Step Plus group showed failures primarily along the cement-adhesive interface (Fig 3).[27] This reveals not only the poor polymerization of the cement layer at these regions due to the distance from the light source,[2] which was a problem for all three groups in the current study, but also the effect that residual uncured acidic resin monomers from simplified adhesives have on the polymerization kinetics of dual-curing resin cements. Other specimens analyzed by SEM revealed a pattern of fracture along the resin-dentin interface, with some tubules still obliterated by resin tags (Figs 4 and 5).

King et al[17] have wisely called the incompatibility to auto-cured composites that resulted from the inherent permeability of one-step self-etching primers "apparent incompatibility". Conversely, "true incompatibility" to auto-cured composites is caused by an adverse acid-base interaction, masking the inherent permeability of this adhesive. According to the authors of that study, "true and apparent" incompatibilities were eliminated upon their conversion to two-step self-etching adhesives by the application of a hydrophobic adhesive coating over the self-etching system. Another practical way to minimize the effects of simplified adhesive systems was proposed by Cadenaro et al,[8] who employed longer curing times than those recommended by the manufacturers. Therefore, it is assumed that the low light irradiance used in the present study (450 mW/cm^2) affected the resin conversion in the root canal, reducing the bond strengths towards the apical areas.

The results of the present study demonstrated that bonding between dentin and resin cements is not as strong in root canals as it is in the crown. An important barrier to good dentinal bonding is the high C- factor (proportion of composite resin surfaces bonded to the cavity). In three-dimensional and confined cavities, as in the root canal, the competition between polymerization stress of composite resins and their bonding to the cavity walls may compromise the adhesive interface.[7,14] According to the results achieved in the present study, the null hypotheses were rejected, as the three-step total-etch dentin adhesive system presented the best bonding results for the regions analyzed, especially at the crown. Clinically, this might be translated into good retention of the final restoration and satisfactory endodontic sealing.

Due to the low bond strength values found for all systems investigated, other factors should be considered in future studies. A detailed characterization of the substrate and hybrid layer by histological and chemical analysis, in concert with clinical studies, are necessary to predict the long-term durability obtained with different adhesive systems.

CONCLUSIONS

In the present study, better bonding to dentin was achieved with the three-step total-etch system. The traditional total-etch three-step adhesive system is the best choice for teeth needing adhesive endodontic restorations, especially in the presence of a coronal remnant.

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