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Regional Bond Strengths to Root Canal Dentin of Fiber Posts Luted with Three Cementation Systems

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This study evaluated the influence of the cementation system on the regional push-out bond strength and failure pattern of fiber posts to radicular dentin. The roots of 48 extracted human incisors were prepared and divided into 3 groups (n=16), according to the cementation system: AdperScotchbond Multi-Purpose + resin cement RelyX ARC (SBMP+ARC); Adper SingleBond 2 + RelyX ARC (SB+ARC) and; RelyX U100 self-adhesive resin cement (U100). The posts were cemented as *per* manufacturer's instructions for each cementation system. After 1 week, the roots were sectioned transversely into 6 discs. Two discs were obtained from the cervical, middle and apical thirds and the push-out test was carried out. The failure pattern was examined on all debonded specimens. The data were analyzed by two-way repeated measures ANOVA and Tukey's test. When U100 was used, no statistically significant difference ($p>0.05$) was observed among the different root regions. Statistically higher push-out bond strength values were detected in the cervical third for SBMP+ARC and SB+ARC ($p<0.05$). The U100 showed significantly more mixed failures than SBMP+ARC in the apical third ($p<0.05$). In conclusion, the self-adhesive cement RelyX U100 was the only cement not sensitive to the root canal region.

Key Words: Bond strength, fiber posts, resin cements, root dentin, post retention.

INTRODUCTION

Endodontically treated teeth may require extensive coronal reconstruction and, depending on the severity of the coronal tissue loss, intracanal post placement may be necessary to achieve retention to the core and restoration (1). Posts made of metal alloys were reported to have less retention, cause serious types of root fractures, compromise esthetics and are susceptible to corrosion (2).

As alternatives, fiber-reinforced composite (FRC) posts were developed with intensive research interest. There has been rapidly increasing development and use of these FRC root canal posts over the last 10 years. Many investigators have suggested that these materials have the advantage of reducing the risk of root fracture thanks to their modulus of elasticity (16-40 GPa) being comparable with that of composite resins (5.7-25 GPa) and dentin (18.6 GPa) (1). Despite these advantages, bonding to radicular dentin offers less favorable conditions than coronal dentin and it is still considered the weakest link of the restoration (3). The success of the root dentin adhesive restorative technique is directly associated with the hybridization quality produced by the adhesive system infiltration into the demineralized dentin substrate (4).

Several factors affect the retention of FRC post within root canals, such as time of post space preparation and cementation, type of post and its adaptation to the post space, type of endodontic cement, adhesive and cementation system, and operative procedures (5,6). Furthermore, the unfavorable cavity configuration factors found within post spaces in addition to the high wall-to-wall shrinkage experienced in bonding posts are even a greater challenge to the bonding protocol in root canal walls (7).

In addition, resin cement distribution in the post space during the luting procedure and the anatomical

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and histological characteristics of the root dentin play a significant role in bond strength between the resin luting agent and root canal regions (3). Adequate luting agent polymerization is necessary to achieve high mechanical properties of the resin cement and consequently, an adequate bond to the root canal walls. However, as light intensity for light polymerization systems is reduced with increasing distance from the light source tip (8), the apical areas of post preparation in the root canal continue to represent a challenge in terms of the bonding protocol. As a result, there are additional difficulties with regard to the insertion and light-curing of adhesive restorative systems. Thus, one may hypothesize that materials that do not rely solely on light activation may achieve a better retention in the apical thirds of root canals (9).

Various luting agents and corresponding adhesive systems have been proposed for bonding FRC posts to root canal dentin. These materials can be light polymerized or dual cured. Recently, a self-adhesive resin cement that requires no dentin pretreatment and has a dual-cure mechanism was introduced on the dental market (10). Unfortunately, there is controversy about the regional bond strength of fiber posts to root canal dentin luted with self-adhesive cement in comparison with conventional dual-cure cement.

The purpose of this study was to evaluate the influence of the cementation system on the regional push-out bond strength of fiber post to root canal dentin.

MATERIAL AND METHODS

The research project was approved by the Ethics Committee of the Dental School of the State University of Ponta Grossa. Forty-eight extracted human maxillary central incisors were stored in distilled water at 4^{o}C and used within 6 months after extraction. The inclusion criteria were absence of restoration, caries or root cracks, absence of previous endodontic treatments, posts or crowns, absence of severe root curvatures and a root length of 14 ± 1 mm, measured from the cementoenamel junction (CEJ).

Endodontic Treatment

Teeth were sectioned transversally immediately below the CEJ using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Endodontic access was made using a tapered fissure bur with a high-speed handpiece and water spray. Working length was established by inserting a #10 Flexofile into each canal until it was visible at the apical foramen. One millimeter was subtracted from this length to establish the working length. A crown-down technique was used for instrumentation with Gates Glidden drills #2 to #4. Apical enlargement was performed to size 40 and .06 taper. After every change of instrument, irrigation was performed with 1 mL of 1% NaOCl solution and 17% EDTA solution alternately. Roots were dried with absorbent paper points (Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil), filled with AH Plus (DeTrey, Dentsply, Konstanz, Germany) and tapered gutta-percha points using the vertical warm condensation technique. The root access was temporarily filled with a glass ionomer cement (Vitro Fil; DFL, Rio de Janeiro, RJ, Brazil). The roots were stored at 37^{o}C in 100% humidity for 1 week.

Post-Space Preparation

After 1 week, gutta-percha was removed using #2, 3 and 4 Gates Glidden burs, leaving 4 mm of the apical seal. The post space was then prepared with a low-speed bur provided by the post manufacturer (Tenax Fiber Trans Drills, Coltène/Whaledent, Cuyahoga Falls, OH, USA) up to a fixed depth of 10 mm from the CEJ. The diameter of the post space preparation was constant for all teeth. One bur was used for only five preparations. All specimens were prepared by a single operator in a standardized procedure.

After preparing the post spaces, the canals were irrigated with 10 mL of distilled water and dried with paper points. The post space walls were checked by radiographic examination, for the presence of any residual gutta-percha.

Experimental Groups

At this point, the specimens were randomly divided into 3 groups (n=16), according to the cementation system: Group SBMP + ARC - adhesive system Adper Scotchbond Multi-Purpose + resin cement RelyX ARC, Group SB + ARC - adhesive system Adper Single Bond2 + RelyX ARC and Group U100 - self-adhesive resin cement RelyX U100 (Table 1). The composition of the materials used for the cementation procedure is described in Table 2. In group SBMP + ARC, a dual-cure three-step adhesive system was used, while in group SB + ARC, a light-cure two-step etch-

Table 1. Bonding procedures.

Cementation system/ Manufacturer	Mode of application (Batch number)
Adper Scotchbond Multi-Purpose/ 3M ESPE + RelyX ARC/ 3M ESPE (SBMP plus ARC)	35% phosphoric acid etching (lot: 7KU) for 15 s; Rinse with water for 15 s and air dry for 2 s; Remove excess moisture with a paper point; Apply activator (lot: 7KY) of the adhesive system in canal and remove excess with air drying (5 s); Apply primer (lot: 7BJ) of the adhesive system in canal and remove excess with air drying (5 s); Apply catalyst (lot: 7BA) of the adhesive system in canal; Dispense cement (lot: GN8JA) onto a mixing pad and mix for 10 s; Apply cement in and around canal; Place a thin layer of mixed cement on post and seat the post; Remove excess cement while holding post in place; Light cure for 40 s from an occlusal direction.
Adper Single Bond 2/ 3M ESPE + RelyX ARC/ 3M ESPE (SB plus ARC)	35% phosphoric acid etching (lot: 7KU) for 15 s; Rinse with water for 15 s and air dry for 2 s; Remove excess moisture with a paper point; Apply two consecutive coats of adhesive (9WH) in the canal and remove excess with air jet (5 s); Remove excess (if any) with a dry paper point; Light-polymerize for 10 s; Dispense cement (lot: GN8JA) onto a mixing pad and mix for 10 s; Apply cement in and around canal; Place a thin layer of mixed cement on post and seat the post; Remove excess cement while holding post in place; Light cure for 40 s from an occlusal direction.
RelyX U100/ 3M ESPE (U100)	Irrigate the canals with 2.5% NaOCl and with distilled water; Remove excess moisture with a paper point; Dispense cement (338618) onto a mixing pad and mix for 20 s; Apply cement in and around canal; Place a thin layer of mixed cement on post and seat the post; Remove excess cement while holding post in place; Light cure for 20 s from an occlusal direction.

Table 2. Composition of the materials.

Material	Composition
Adper Scotchbond Multi-Purpose (SBMP)	Activator: ethanol based solution of a sulfinic acid salt and a photo-initiator component. Primer: aqueous solution of HEMA (hydroxy ethyl methacrylate) and polyalkenoic acid co-polymer. Catalyt: HEMA and Bis-GMA (bisphenol-A-glycidyl methacrylate).
Adper Single Bond 2 (SB)	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system, methacrylate functional copolymer of polyacrylic and polyitaconic acids.
RelyX ARC (ARC)	Paste A: Bis-GMA, triethyleneglycol dimethacrylate, zircon/silica filler, photoinitiators, amine, pigments. Paste B: Bis-GMA, triethyleneglycol dimethacrylate, benzoic peroxide, zircon/silica filler.
RelyX U100 (U100)	Paste Base: glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated silica, sodium persulfate. Paste Catalyst: glass fiber, dimethacrylates, silanated silica, p-toluene sodium sulfate, calcium hydroxide.

and-rinse adhesive was selected. U100 does not require the use of an adhesive system and presents a dual-cure mechanism to bond to the root walls.

Post-Luting Procedures

Before cementation procedures, each fiber glass post (cylindrical with a tapered end [Tenax Fiber Trans Esthetic Post System, Coltène/Whaledent]) was marked at a distance of 13 mm from the apical end and was horizontally sectioned at this point, using a water-cooled diamond rotary cutting instrument. Ten mm of the post length was cemented inside the root canal while the other cervical 3 mm served as a guide to standardize the distance of the light curing device from the cervical root region.

The posts were tried in, cleaned with alcohol and cemented in accordance with the manufacturer's instructions for each cementation system described in Table 1. The adhesive systems were applied inside the root canals by means of micro-brushes (Vigodent, Rio de Janeiro, RJ, Brazil) and the resin cements were applied with a Centrix syringe (DFL, São Paulo, SP, Brazil). A LED light curing device (L.E.Demetron I/Kerr Corporation, Orange, CA, USA) with a power density of 800 mW/cm^2 was used for activation purposes. After the post luting procedures, all samples were stored in water at 37°C for 1 week.

Preparation of Sections for the Push-Out Test

After this, the roots were embedded in polyvinyl chloride (PVC) tubes using acrylic resin (Duralay; Reliance, Dental Mfg. Co., Worth, IL, USA) and the portion of each root containing the bonded fiber post was sectioned perpendicular to the long axis into six 1- mm-thick serial slices, using the Isomet 1000 (Buehler) saw under water cooling, being two slices from the cervical, middle and apical thirds.

Subsequently, all specimens were observed with a light stereomicroscope at ×10 magnification to detect any artifacts caused by the sectioning procedure. The coronal side of each slice was identified and its thickness measured with a digital caliper (Mitutoyo Corporation, Tokyo, Japan) accurate to the nearest 0.01 mm. The slices were also photographed on both sides, with an optical microscope (model BX 51; Olympus, Tokyo, Japan) at a ×40 magnification in order to measure the coronal and apical diameters of the posts, with the purpose of calculating their individual bonding area. This measurement was performed with UTHSCSA Image Tool 3.0 software (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA).

Each slice was subjected to a push-out test using a universal loading device (AG-I, Shimadzu Autograph, Tokyo, Japan) at a crosshead speed of 0.5 mm/min with the load applied in the apical-coronal direction until the post was dislodged. Care was taken to centralize the push-out pin at the center of the post surface, without stressing the surrounding post space walls. With regard to the tapered post design, different sizes of punch pins were used, which matched the diameter of the post region being tested.

The maximum failure load was recorded in Newton (N) and converted into MPa by dividing the applied load by the bonded area (S_L). The latter, being the lateral surface of a truncated cone, was calculated by the formula: $S_L=\pi(R + r)[(h^2 + (R - r)^2]^{0.5}$

Where $\pi = 3.14$, R = coronal post radius, r = apical post radius, and h = root slice thickness.

Failure Mode Analysis

After push-out bond strength evaluation, the failure mode of all specimens was evaluated under a stereomicroscope (×40 magnification). However as this method does not provide detailed information, approximately 35% of the specimens from each group were randomly selected and processed for scanning electron microscopy (SEM) evaluation. The slices were rinsed in a 95% alcohol solution for 1 min and air-dried. Each slice was mounted on a metal stub and sputter-coated with 200 µm of gold-palladium in a Polaron SC7620 "Mini" sputter Coater (Quorum Technologies Ltd., East Sussex, UK) for 5 min at a current of 10 mA. After this, each slice was examined by SEM (JSM 6360LV; Jeol Ltd., Tokyo, Japan) using 15-kV accelerating voltage at different magnifications (×40, ×100 and ×200) and SEM micrographs were taken for further analysis.

Each debonded specimen was analyzed by two independent operators and the failure modes were classified according to the following criteria: (1) adhesive failure between dentin and luting cement; (2) adhesive failure between luting cement and post; (3) cohesive failure within luting cement; (4) cohesive failure within the post;(5) cohesive failure within dentin and (6) mixed failure.

Statistical Analysis

The fracture pattern of the specimens was submitted to two different statistical approaches: 1) specimens from the same root at each third were averaged for statistical purpose. Differences among cements in each root third was compared with Fisher's exact test (α = 0.05) and; 2) specimens from the same root for each cement were averaged for statistical purpose. Differences among thirds for each cement was compared with Fisher exact test (α = 0.05)

The push-out bond strength of specimens from the same root at each third was averaged for statistical purposes. The data (in MPa) were subjected to a two-way analysis of variance (cement *vs*. root region) and Tukey's test (α=0.05) for pair-wise comparisons.

RESULTS

The analysis of failure modes is shown in Table 3. No cohesive failures within the luting cement, post or dentin were observed in this investigation. No statistically significant difference ($p>0.05$) in the fracture pattern was observed among root regions for each cement. As regards the different root canal thirds, the only significant difference ($p<0.05$) was found between RelyX U100 and Adper Scotchbond Multi Purpose + RelyX ARC cement. RelyX U100 showed more mixed failures than the SBMP + ARC in the apical third ($p<0.05$). Most mixed failures (79%) occurred between resin cement and dentin with cohesive failure of the cement. A representative image of the most prevalent failure mode can be seen in Figure 1.

The overall push-out bond strength means are shown in Table 4. The cross-product interaction cement vs. root region was statistically significant ($p<0.0001$). From Table 4, it can be observed that when RelyX U100 was used no significant difference ($p>0.05$) was observed for the different root regions. On the other hand, statistically significant higher push-out bond strength values ($p<0.05$) were detected in the cervical area for Adper Single Bond + RelyX ARC cement and Adper Scotchbond Multi Purpose + RelyX ARC cement.

Table 4. Means and standard deviations (MPa) of push-out bond strength means in the groups.

Cementation system	Cervical third	Middle third	Apical third
SBMP plus ARC	16.5 ± 8.6 a	9.9 ± 6.4 b,c	8.6 ± 4.9 c,d
SB plus ARC	14.7 ± 8.3 a,b	8.4 ± 4.7 c,d	6.2 ± 4.4 d
U100	13.7 ± 6.8 a,b	12.0 ± 7.0 a,b	16.1 ± 7.5 a

Different lowercase letters indicate statistically significant difference at 5%.

DISCUSSION

The good immediate performance of adhesive systems when bonded to enamel and coronal dentin has been well documented (11). However, some aspects related to intraradicular dentin remain uncertain, as some failures have been clinically observed. Different root regions show different distributions and densities of dentin tubules. The densities and the number of dentin tubules decreased significantly from the coronal to apical root regions (12). The findings of the present

Table 3. Failure mode distribution in the groups.

Cementation system	Cervical third		Middle third		Apical third	
	Adhesive failures (dentin-cement)	Mixed failures	Adhesive failures (dentin-cement)	Mixed failures	Adhesive failures (dentin-cement)	Mixed failures
SBMP plus ARC	5	5	3	7	7	3
SB plus ARC	3	7	6	4	3	7
U100	1	9	5	5	1	9

SBMP = Adper Scotchbond Multi-Purpose; ARC = RelyX ARC; SB = Adper Single Bond 2; U100 = RelyX U100. No cohesive failure in dentin, post or adhesive failure between cement and the post was observed. Significantly more mixed failures occurred for U100 in the apical group in comparison with SBMP + ARC group ($p<0.05$), Fisher's exact test).

study demonstrated that SBMP+ARC and SB+ARC provided lower bond strength values in the apical than in the cervical region. These findings are in agreement with previous findings in the literature (3,13).

One of the factors responsible for the different bond strength values at the various depths of the same root canal is acid etching. A preliminary study has shown that different areas of the same root canal did not respond equally to the acid etching protocol (3). Apical root dentin is a less favorable bonding substrate because of areas devoid of tubules, irregular secondary dentin, cementum-like tissue on the root canal wall and numerous accessory canals (14).

Another factor that influences the adhesive-dentin bond is the formation of resin tags, because previous SEM investigations have demonstrated that the mechanism for adhesive bonding to root dentin is based on resin tag formation (4). According to Gwinnett (15), these tags contribute about 30% to the total bond strength. These findings seem to suggest that if there are fewer tubules *per* square millimeter in the apical region, the bond strength will be lower since there will be less resin tag formation. The better performance in the coronal region is also attributed to the fact that this is the most accessible part of the canal space (3), making it easier to etch and more thoroughly apply the adhesive agents (13).

In addition, the apical areas of post preparation in the root canal pose additional difficulties with regard to the insertion and photo-activation of adhesive restorative systems. The difficulty in obtaining direct light irradiation in apical regions is likely to be the main

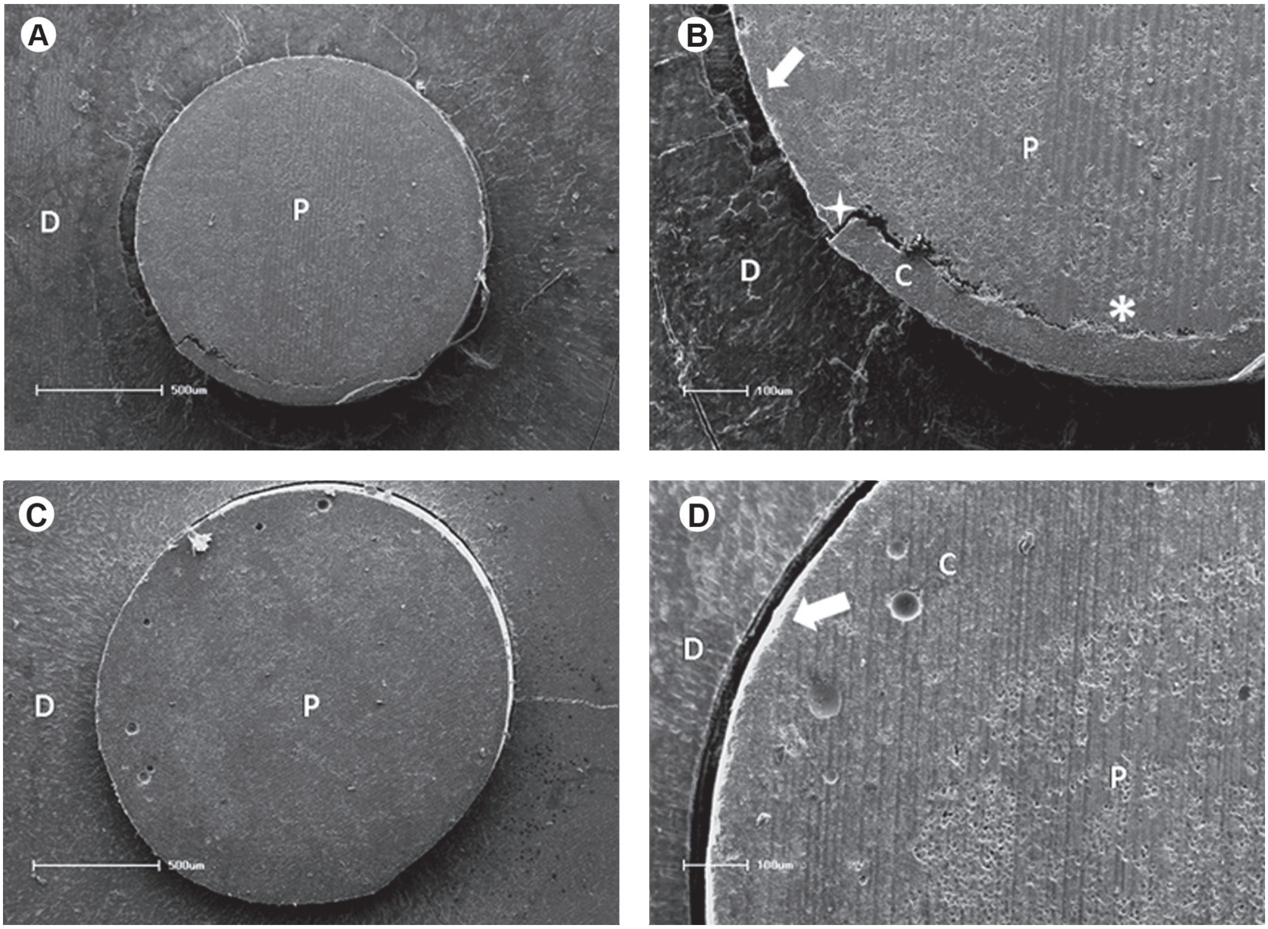

Figure 1. Scanning electron micrographs of representative fracture patterns. A mixed failure mode in the apical third for RelyX U100 can be seen in low (A) and high magnification (B). In panel B, one can observe the adhesive failure between the cement and the dentin interface (arrow) along with a cohesive failure within cement (star) and an adhesive failure between the cement and the post (asterisk). An adhesive failure mode in the apical third for SBMP + ARC can be seen at low (C) and high magnification (D). Observe in panel D that the failure occurred between the cement and dentin (arrow). D = dentin; P = post; C = cement.

reason for the lower bond effectiveness in this region (9). There is a significant reduction in the quantity of light transmitted into the root canal as the depth increases, and this has been shown to reach levels insufficient for achieving polymerization, especially in the apical third (16).

Furthermore, the two adhesives (SBMP+ARC and SB+ARC) rely on the same bonding strategy, namely the etch-and-rinse approach. This technique requires the dentin substrate to be kept moist for optimal bonding after phosphoric acid etching (17). However, because of limited access, the moisture control within the root canal is quite difficult (3), and may also result in low bond strength values in the apical third, in the same way as occurred with the conventional (etch-and-rinse) resin cement in the present investigation.

In an attempt to reduce the clinical steps involved in post cementation to root walls, a new type of luting material that requires no pretreatment of the tooth surface has been developed, and is called a self-adhesive cement. This cement does not require rinsing, decreasing the problem of substrate moisture control, thus simplifying the clinical procedure. No dentin pretreatment is indicated in this one-step technique (10).

Despite this, Bitter et al. (18) have affirmed that RelyX U100 showed a significantly lower number of penetrated dentinal tubules, lower hybrid layer thickness and the penetration of this cement into the dentinal tubules was found in only a few specimens in comparison with conventional dual-cure cements. However, the manufacturer of RelyX U100 claims that the bonding mechanism of this self-adhesive cement is based on micromechanical retention and chemical adhesion to hydroxyapatite (19). Furthermore, a recent investigation documented an intense chemical interaction of RelyX U100 with hydroxyapatite (20). This is probably the factor responsible for the homogeneous bond strength values of RelyX U100 in all root dentin regions.

In addition, the formation of water during the neutralization reaction of the phosphoric acid methacrylate, basic fillers and hydroxyapatite may also be responsible for the higher tolerance to moisture (19). This helps to explain the best results of this material in bonding to the apical root region. Future studies should be conducted to evaluate this hypothesis. However, it is worth mentioning that there is no unanimity in the literature with regard to the homogeneous bond strength of RelyX U100 in the different thirds of the root canal (18). The reason for this controversy could be methodological differences, but this needs to be further investigated.

It should be pointed out that this study possesses some limitations. The test specimens have not had their crowns completely restored and neither thermal cycling nor mechanical stressing was applied. These factors may limit the direct application of the study results to clinical conditions.

Within the limitations of this *in vitro* study, it may be concluded that the bond strengths were significantly affected by the root canal region for the conventional (etch-and-rinse) resin cement, but not for the self-adhesive resin cement.

RESUMO

Esse estudo avaliou a influência do sistema de cimentação na resistência de união regional e os padrões de fratura de pinos de fibra à dentina radicular. As raízes de 48 incisivos humanos extraídos foram preparadas e divididas em 3 grupos (n=16) de acordo com o sistema de cimentação: AdperScotchbond Multi-Purpose + cimento resinoso RelyX ARC (SBMP+ARC); AdperSingle Bond 2 + RelyX ARC (SB+ARC) e cimento resinoso autoadesivo RelyX U100 (U100). Os pinos foram cimentados conforme as recomendações dos fabricantes para cada sistema de cimentação. Após uma semana, as raízes foram seccionadas transversalmente em 6 discos. Dois discos foram obtidos para os terços coronário, médio e apical e o teste de push-out foi realizado. O padrão de fratura foi avaliado em todos os espécimes. Os dados obtidos foram analisados através dos testes ANOVA dois fatores e Tukey. Quando o cimento U100 foi testado, não foram observadas diferenças significativas (p>0,05) entre as diferentes regiões radiculares. Valores de resistência de união significativamente superiores foram encontrados no terço coronário para SBMP+ARC e SB+ARC (p<0,05). O U100 apresentou significativamente mais fraturas mistas que o SBMP+ARC no terço apical (p<0,05). Conclui-se que o cimento resinoso autoadesivo RelyX U100 foi o único cimento não afetado pela região do canal radicular.

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