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Effect of Surface Treatment of Fiberglass Posts on Bond Strength to Root Dentin

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This study evaluated the influence of the surface treatments of fiberglass posts on bond strength to root dentin using push-out test. Forty bovine incisor roots were endodontically treated. The surface of the fiberglass posts (Exacto #2, Angelus) were treated using 4 different protocols (n=10): Control - 70% ethanol for 1 min; 37% phosphoric acid for 1 min; 10% hydrofluoric acid for 1 min; and 24% hydrogen peroxide for 1 min. After a silane coupling agent was applied for 1 min and all posts were cemented using self-adhesive resin cement (RelyX Unicem, 3M-ESPE). The roots were sectioned and two 1-mm-thick slices were obtained from each third: cervical, middle and apical. The specimens were subjected to the push-out test with a crosshead speed of 0.5 mm/min. Data were analyzed by repeated measures ANOVA followed by Tukey's HSD tests (α=0.05). The surface treatment ($p<0.001$) and root third region ($p=0.007$) factors were significant. The retention to root canal was affected by surface treatment type. The post surface treatment with 24% hydrogen peroxide for 1 min yielded significantly higher bond strength when the fiberglass posts were cemented with RelyX Unicem.

Key words: surface treatments, fiberglass posts, push-out test, self-adhesive resin cement.

Introduction

Fiberglass posts are a viable alternative for restoration of endodontically treated teeth (1-3) and their retention with composite restorations depends on the quality of the bond established at different interfaces (4). The most frequent cause of failure is debonding of a post restoration to the root canal (5,6). Therefore, in order to improve the bond strength between the post and the resin cement, surface pre-treatment procedures for posts have been investigated (2,7).

Surface treatment is a common method for improving the adhesion properties of a material, by facilitating chemical and micro-mechanical retention between different constituents (8). These procedures fall into three categories: 1) chemical bonding between a composite and post (silane coating); 2) surface roughening (sandblasting and etching); or 3) combination of micromechanical and chemical components by using the two above-mentioned methods (4).

Silane coupling agent is a hybrid organic-inorganic compound that can mediate adhesion between inorganic and organic matrices through intrinsic dual reactivity capability to increase surface wettability, creating a chemical bridge with OH-covered substrates, such as glass (4,8). A chemical bond may be achieved between the core resin matrix and the exposed glass fibers of the post at the interface level (9,10). However, the interfacial strength is still relatively low because of the absence of chemical union between the methacrylate-based resin composites and the epoxy resin matrix of fiber posts (11).

Hydrofluoric acid in combination with a silane-coupling agent is often employed to enhance the bond strength between composite resins and feldspathic ceramics (12,13). Because silica and quartz present in fiber posts are comparable in chemical structure with ceramic materials, hydrofluoric acid has recently been proposed for etching fiberglass posts (2). It is intended to create a rough pattern on the surface, which allows for micromechanical interlocking with the resin cement and composite (4). On the other hand, the treatment using hydrogen peroxide (HP) dissolves the epoxy resin matrix, exposing the surface of fibers to silanization (2,7,14,15). The spaces between fibers provide additional sites for micromechanical retention of the resin composites (10,11). Etching with phosphoric acid is also employed as a fiberglass surface treatment indicated by manufacturers; however, they recommend this substance as a cleaning agent (16).

The aim of this study was to evaluate the influence of different surface treatments of fiberglass post on their bond strength to root canal, using a push-out test. A delayed photo-activation protocol was used for the self-adhesive resin cement to allow for analyzing the adhesion of the posts to the root canal walls, excluding interferences of the cement-dentin interface and enabling a better chemical and photo-polymerization of resin cement. The morphological aspects of the fibers and the post-surface characteristics following the different pretreatments were also observed using scanning electron microscopy (SEM).

The hypothesis generated is that push-out bond strength of fiberglass post to root dentine is not affected by different post surface treatments.

Material and Methods

Forty bovine incisors with similar dimensions had their coronal portion removed to obtain 15-mm-long roots. Canals were instrumented with Gates Glidden drills (Dentsply Maillefer, Ballaigues, Switzerland), irrigated with 1.0% NaOCl and saline and filled with gutta-percha cones (Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil) and calcium hydroxide-based endodontic sealer (Sealer 26 Dentsply Ind. e Com. Ltda.). Post spaces were prepared to a depth of 10 mm using a heated instrument (GP heater; Dentsply Maillefer) to remove gutta-percha, and a specific drill system (Exacto #2, Angelus, Londrina, PR, Brazil).

The roots were randomly divided in 4 groups according to the surface treatment (n=10) of the opaque and cylindrical/conical fiberglass posts (Exacto; Angelus, size 2, with 1.5 mm in diameter and 17.0 mm in length): Control - the post surfaces were cleaned with 70% ethanol according to the manufacturer's recommendation over the post surface using a microbrush for 1 min, dried for 1 min and the silane coupling agent (Silano, Angelus) was applied and allowed to evaporate for 1 min; PA37% group, the post surface was etched with 37% phosphoric acid (Condac 37, FGM, Joinville, SC, Brazil) for 1 min, followed by rising with water for 1 min and drying. Next, a silane coupling agent was applied for 1 min; HF group, the post surface was etched with 10% hydrofluoric acid gel (Condac Porcelana, FGM) applied over the post surface for 1 min followed by rising and drying. The silane agent was then applied on the post surface for 1 min allowing solvent evaporation; HP group, the fiber post was immersed in 24% HP solution placed in Eppendorf tubes for 1 min followed by rising and drying. Afterwards, the silane was applied for 1 min.

All posts were cemented to the root canal using self-adhesive resin cement (RelyX Unicem, 3M ESPE, St Paul, MN, USA) according to the manufacturers' instructions by hand mixing the predisposed cement portions in a mix-pad for 20 s until reaching material homogeneity and inserted into the root canal with size 40 stainless steel K-files (Dentsply Maillefer). The posts were coated with the resin cement and seated under finger pressure. Five minutes after insertion, photo-activation was performed through the coronal portion of the root at the buccal, lingual, and coronal faces for 40 s, a total of 120 s light exposure. The photo-activation procedures were performed using a light-curing unit with 800 mW/Cm^2 intensity (XL3000, 3M-ESPE).

Push-Out Test

After storing for 24 h in distilled water at 37 °C, the roots were sectioned transversally into six slices with a low-speed diamond blade (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling. Two 1 mm thick slices were obtained from the cervical, middle and apical third regions. After measuring the thickness with a digital caliper (Mitutoyo, São Paulo, SP, Brazil), each specimen was marked on its coronal surface with an indelible marker, and the diameter of top and bottom surfaces of post was measured using an optical microscope under 40× magnification (Mitutoyo TM-500). The specimen was positioned on a universal testing machine (EMIC DL 2000I; São José dos Pinhais, Paraná, Brazil) in such a way that the load applicator tip coincided with the metal base orifice, and then submitted to compression loading in on apex to crown direction at a speed of 0.5 mm/min, until failure by displacement of the post. The bonding strength values were calculated using the equation (17):

$$\text{Area} = \pi\,(R_1 + R_2)\,\sqrt{R_1 - R_2)^2 + h^2}$$

where π is the constant 3.14, R_1 is the top post radius, R_2 is the bottom post radius, h is the specimen thickness in mm.

To determine the failure mode, all specimens tested were air dried and analyzed under a confocal laser scanning microscope (LSM510, Carl Zeiss Laser Scanning Systems, Oberkochen, Germany). The failure modes were evaluated using a classification system modified from Castellan et al. (18): (I) Adhesive failure between post and luting cement; (II) Adhesive failure between dentin and luting cement; (III) Cohesive failure in cement; (IV) Cohesive failure in dentine; and (V) Mixed failure.

Specimens tested were mounted on aluminum stubs, sputter coated with gold (Bal-Tec SCD 050; Balzers, Liechtenstein) and examined with a scanning electron microscope (SEM; LEO 435 VP; LEO Electron Microscopy Ltd., Cambridge, UK). SEM images were obtained at different magnifications to illustrate the failure modes and the effect of the different surface treatments on the morphological aspects of the fiberglass post surface.

Statistical Analysis

The bond strength data were tested for normal distribution (Shapiro-Wilk, $p<0.05$) and equality of variances (Levene's test, $p<0.05$). Data were analyzed by repeated-measured ANOVA to evaluate the effect of the study factor (surface treatment) with the repetition defined as the third regions; followed by the Tukey's Honestly Significant Difference (HSD) test at a 5% level of significance.

Results

The mean and standard deviations of the bond strengths for the post surface treatments measured in each root region are shown in Table 1. The results of the repeated measures ANOVA revealed that the surface treatment ($p<0.001$) and root third region ($p=0.007$) factors were significant. The Tukey's HSD test showed that the HP group had a significantly higher bond strength than the other surface treatments. The post-surface treatment with ethanol 70% (control), PA37% and HF had similar bond strength. The push-out bond strength of the fiberglass cemented with RelyX Unicem was significantly higher in the cervical than in middle and apical regions.

Confocal observation demonstrated the distribution of the failure modes for each experimental group in Table 2. All samples were classified according to the failure pattern. The cohesive failures in cement (Fig. 1C) were more prevalent in the control and HF groups and the adhesive failures between dentin and luting cement (Fig. 1B) were more prevalent in the PA37% and HP groups. Adhesive failures between post and luting cement (Fig. 1A) were more prevalent in PA37% group when compared to the others groups. Cohesive failures in dentin (Fig. 1D) were not seen in HP group and mixed failures (Fig. 1E) were observed in the PA37% and HP groups.

Representative specimens of the investigated pretreatment procedures are shown in Figure 2. SEM observation revealed that the post surface etched with ethanol (Fig. 2A) and phosphoric acid (Fig. 2B) was not modified. An aggressive attack on the epoxy matrix and fiberglass was observed by SEM evaluation on the surfaces etched with hydrofluoric acid (Fig. 2C). This technique produced substantial damage to the fiberglass and affected the integrity of the post. Treatment with HP exposed more superficial fibers and caused surface dissolution of the epoxy resin matrix to a greater depth (Fig. 2D). It also was observed that fibers were not damaged by the etching treatment.

Table 1. Mean push-out bond strength values (S.D.) in MPa

Surface treatment	Region			
	Cervical	Middle	Apical	Pooled average
24% hydrogen peroxide	19.9 (4.8)	16.7 (4.7)	15.8 (4.4)	17.5 (2.2)[A]
10% hydrofluoric acid	14.0 (5.2)	11.4 (5.5)	10.8 (2.8)	12.1 (1.7)[B]
37% phosphoric acid	11.6 (6.3)	10.2 (6.5)	10.1 (4.5)	10.6 (0.8)[B]
70% ethanol (control)	11.8 (3.8)	9.9 (4.9)	9.8 (6.2)	10.5 (1.1)[B]
Pooled average	14.3 (3.9)[a]	12.1 (3.2)[b]	11.6 (2.8)[b]	

Different letters indicate statistically significant difference verified by the Tukey's test ($p<0.05$). Uppercase letter is used to compare surface treatment (in columns), and lowercase letters is used to compare root region (in rows).

Table 2. Failure mode distribution in the groups

Surface treatment	Failure modes				
	I	II	III	IV	V
70% ethanol (control)	7 (12%)	20 (33%)	30 (50%)	3 (5%)	-
37% phosphoric acid	12 (20%)	24 (40%)	19 (32%)	3 (5%)	2 (3%)
10% hydrofluoric acid	6 (10%)	18 (30%)	34 (57%)	2 (3%)	-
24% hydrogen peroxide	6 (10%)	30 (50%)	20 (33%)	-	4 (7%)
Total	31 (13%)	92 (38%)	103 (43%)	8 (4%)	6 (2%)

I, Adhesive failure between post and luting cement; II, Adhesive failure between dentin and luting cement; III, Cohesive failure in cement; IV, Cohesive failure in dentin; and V, Mixed failure.

Discussion

The tested hypothesis was rejected; the bond strength of glass fiber posts to root canal was affected by the different post surface treatments. The bond strength of fiberglass post to root canal was significantly higher when the 24% HP surface treatment was used compared with the other surface treatments, 70% ethanol, 37% phosphoric acid and 10% hydrofluoric acid.

The push-out test has been considered the most appropriate method to assess the adhesion of luted posts to root canal dentin (19). The push-out test involves the use of an indenter to push a small fiber diameter into a specimen with a thickness of approximately 1 mm, which allows a more uniform distribution of the load applied throughout the bonded interface (19). Nevertheless, the pin diameter, specimen thickness, and elastic modulus of the filling material all have effects on the push-out bond strength results (20).

Resin-based adhesive luting materials are widely used for the fixation of posts, and currently all the resin cements are based upon the use of either an etch-and-rinse or of a self-etch adhesive, along with a low-viscosity resin composite. This multistep application procedure is complex and somewhat technique-sensitive, which could compromise the bonding effectiveness. Self-adhesive resin cements were introduced in 2001 to simplify luting procedures and are have been a good option to fix fiberglass post due to their bonding performance and low viscosity eliminating the need for pretreatment of tooth. The manufacturer to be based upon acid monomers that demineralize and infiltrate the tooth substrate, resulting in micromechanical retention, claims the adhesive proprieties. Secondary reactions have been suggested to provide chemical adhesion to hydroxyapatite. A recent systematic review on the role of resin cement on bond strength of glass-fiber posts seems to suggest that the use of self-adhesive resin cement, especially with RelyX Unicem, could improve the retention of fiberglass posts in root canals (21).

The results obtained in this study for bond strength showed significantly higher values in the cervical region than in the middle and apical regions for all groups. It could attributed to the presence of smear layer, generated during endodontic treatment and post space preparation, which are deposited on the root canal walls. The presence of such a layer impairs a proper contact between the acidic methacrylates of self-adhesive resin cements and the underlying dentin during adhesive procedures, interfering with its bond strength to dentin (22). A previous study reported that self-adhesive cements presented a limited decalcification/infiltration for into the underlying dentin and that no hybrid layer and/or resin tag formation was detectable at the interfaces bonded with self-adhesive cements (23). Moreover, light curing provides higher bond strengths to dentin than self curing mainly when the light source is closer to the composite material (24).

The use of ethanol has been recommended by the manufacturers to clean the post surface to remove any kind of organic contamination and/or particles adhered to the post, which could impair the bond strength to luting agent improving the potential of the interaction with resin-based

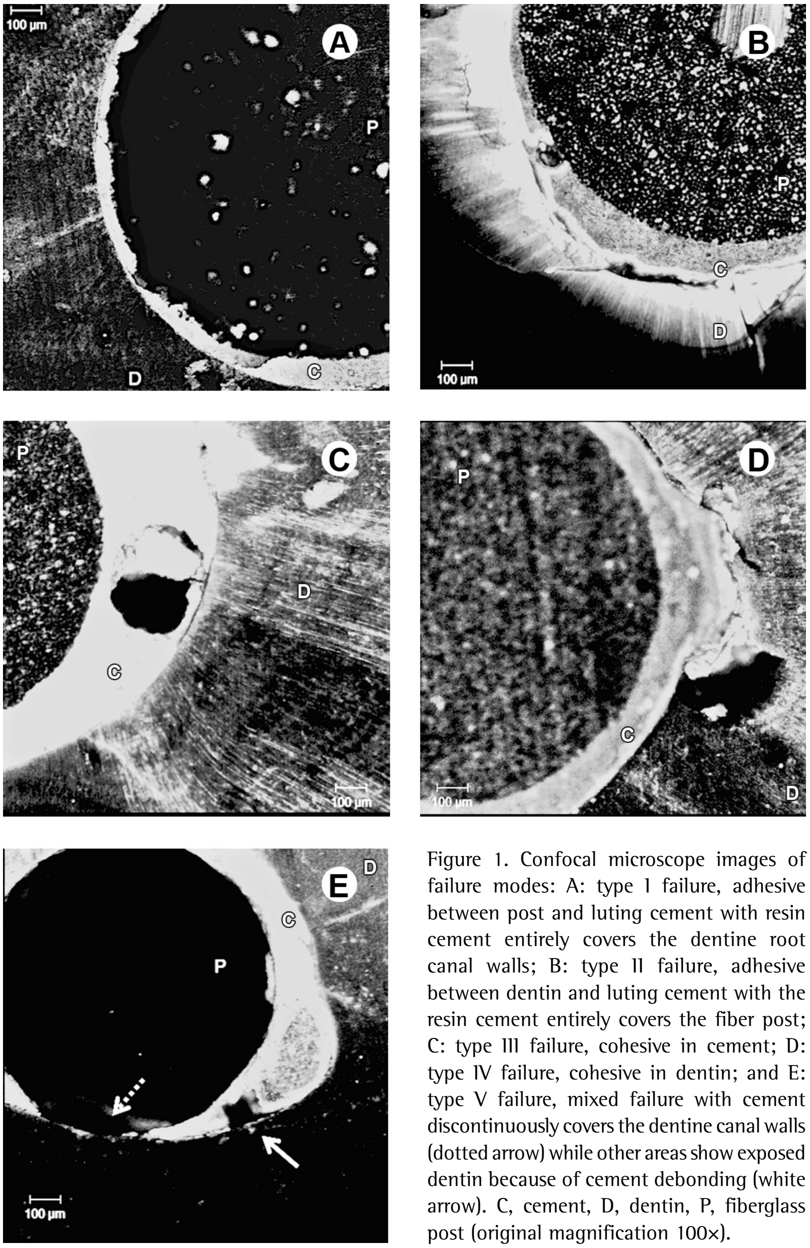

Figure 1. Confocal microscope images of failure modes: A: type I failure, adhesive between post and luting cement with resin cement entirely covers the dentine root canal walls; B: type II failure, adhesive between dentin and luting cement with the resin cement entirely covers the fiber post; C: type III failure, cohesive in cement; D: type IV failure, cohesive in dentin; and E: type V failure, mixed failure with cement discontinuously covers the dentine canal walls (dotted arrow) while other areas show exposed dentin because of cement debonding (white arrow). C, cement, D, dentin, P, fiberglass post (original magnification 100×).

materials. In this research, ethanol was used as a control group. The results of this study showed that this treatment resulted in similar bond strength of the phosphoric acid and hydrofluoric acid groups. SEM observation revealed that the post surface etched phosphoric acid was not modified by maintenance the same pattern of ethanol group. The roughness of the post surface produced by the phosphoric acid may have been insufficient to attain strong mechanical interlocking between the cement and the post surface.

On the other hand, the SEM analysis revealed cracking of the fibers, as well as on underlining treated epoxy resin, when hydrofluoric acid was used (Fig. 2C). This technique produced substantial damage to the glass fibers, which affected the integrity of the post, probably due to the extremely corrosive effect of hydrofluoric acid on the glass phase (1,12).

Using HP over the post surface has also demonstrated mechanical-chemical bonding of resin-based material to the fiber post (7,11). The etching effect of HP is based on oxidation of the post surface, thus breaking epoxy resin bonds. A recent study (15) comparing 24% and 35% HP showed that higher concentration results in higher oxidizing effect, and improved the bond strength for 35% regardless of application or immersion mode; however immersion for 24% resulted in higher values on bond strength. In this study, 24% HP was able to remove a superficial layer of epoxy resin, exposing the larger surface area of fibers to silanization (Fig. 2D) with no damaging generated on the fiber network when this product is applied at 1 min (2,7,11). This explains the results in which higher values were found when coupling this treatment protocol was applied. When the surface of the post was etched with 24% HP, probably a more reactive surface was generated for better retention of the post and resin cement.

Silane coupling agents mainly exert their function by bonding chemically to the posts and core material and improving surface wettability (1,10). With the removal of the superficial layer of epoxy resin via surface treatment as previously showed in this study, more exposed fibers in terms of surface area are available for reacting with the silane molecules (1,2,9,11). In this study, silane was applied in all samples after the specific surface treatment of each group. It is likely that their application improved the bond strength, mainly when the 24% HP was used, showing both chemical and micromechanical interaction.

Confocal microscopy was used and appears to be a noteworthy alternative for the evaluation of the bond failure pattern in loaded specimens, since it is less time-consuming and does not require any preparation of samples. In the present study, the failure modes of tested samples showed a wider variation. Failure modes were primarily cohesive failures in cement (43%). When mechanical

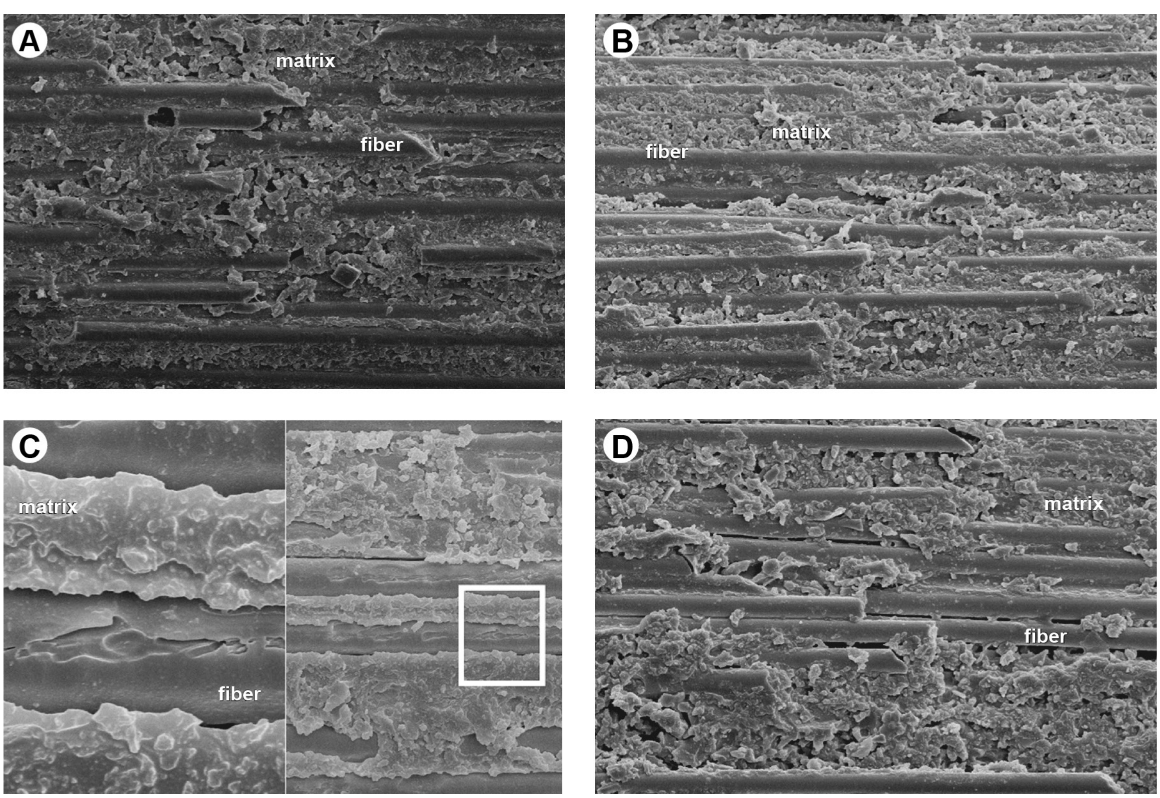

Figure 2. SEM micrograph of fiberglass post surface etched with 70% ethanol (A), 37% phosphoric acid (B), 10% hydrofluoric acid (C) and 24% hydrogen peroxide (D). The white square in image "C" shows areas with damaged fibers caused by the hydrofluoric acid action (original magnification 250×).

treatment is not effective, failure tends to occur along the interfaces, but when chemical bonding is involved, the failure often occurs cohesively through the cement itself (25). The second most frequent type was adhesive failure between dentin and luting cement (38%), which could be explained by smear layer presence that can affect the bond strength. The higher adhesive failure mode at the post-resin cement interface was observed in the ethanol (12%) and phosphoric acid groups (20%), in which lower bond strength values were found. This failure mode (I) was 13% compared within the groups, which could be attributed to the fact that this study used a delayed photo-activation protocol after 5 min, which could result in a lower shrinkage of resin cement and a higher bond strength between the structures.

This study presents some limitations such as lack of mechanical or thermal cycling and limited number of surface treatment protocols. Clinical trials are necessary to validate the results of this investigation, as well as the evaluation of the stress distribution using finite element analysis. However, fiberglass post is now the best option to restore root treated teeth that need additional retention, and the use of the adequate post surface treatment to improve the post retention should be a clinical challenge. The use of 24% HP appeared as a very promising post surface treatment for this strategy.

Considering the methodology applied and the limitations of this *in vitro* study, it can be concluded that the use of treatment surface on fiber post is important to improve the post-cement-dentin interaction. The application of 24% HP enhanced significantly the interfacial bond strength between fiber posts and root dentin. Additionally, bond strength to root was significantly higher in the cervical region than in middle and apical regions, irrespective of post surface treatment with the use of a self-adhesive resin cement.

Resumo

Este estudo avaliou a influência de tratamentos de superfície de pinos de fibra de vidro na resistência de união à dentina radicular por meio do teste de push-out. Quarenta raízes de incisivos bovinos foram submetidas a tratamento endodôntico. A superfície dos pinos de fibra de vidro (Exacto #2, Angelus) foram tratadas com 4 protocolos diferentes (n=10): Controle - 70 % de etanol durante 1 min; 37 % de ácido fosfórico durante 1 min, 10% de ácido fluorídrico durante 1 min e 24 % de peróxido de hidrogênio durante 1 min. Depois foi aplicado agente de união silano por 1 min e todos os pinos foram cimentados com cimento resinoso auto-adesivo (RelyX Unicem, 3M- ESPE). As raízes foram seccionadas e foram obtidas duas fatias de 1 mm de espessura em cada terço: cervical, médio e apical. Os espécimes foram submetidos ao teste de push-out com uma velocidade de 0.5 mm/min. Os dados foram analisados pelo teste ANOVA com medidas repetidas, seguido pelo teste de Tukey HSD (α=0,05). Os fatores tratamento de superfície (p<0,001) e região do terço radicular (p=0,007) foram significantes; no entanto, a interação entre os dois fatores não foi significante (p=0,827). A retenção ao canal radicular foi afetada pelo tipo de tratamento de superfície. O tratamento de superfície com 24% de peróxido de hidrogênio por 1 min rendeu significativamente maior resistência de união quando os pinos de fibra de vidro foram cimentados com RelyX Unicem.

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