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Influence of endodontic sealer cement on fibreglass post bond strength to root dentine

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Abstract

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Aim To test the hypothesis that the composition of endodontic sealer cements and the time elapsed between root filling and fibreglass post fixation interferes with adhesion to root canal dentine.

Methodology Sixty bovine incisor roots were divided into five groups ($n = 12$): CI, unfilled; SI, filled with a calcium hydroxide-based cement-Sealer 26, and immediate post fixation; S7, Sealer 26 and post fixation after 7 days; EI, filled with a zinc oxide and eugenol-based cement-Endofill and immediate fixation; and E7 Endofill and post fixation after 7 days. The posts were cemented with adhesive system and dual resin cement. Ten roots were cross-sectioned to obtain two 1-mm-thick discs for each cervical (TC), middle (TM) and apical (TA) third of the prepared root portion. The posts were submitted to a micropush-out test. The other two teeth were evaluated using scanning electron microscopy to analyse the bond interface. Data were analysed using ANOVA, Tukey and Dunnett tests ($P < 0.05$).

Results Group EI was associated with a significant reduction in bond strength values irrespective of the root region; TC = 3.50 MPa ($P = 0.0001$); TM = 2.22 MPa ($P = 0.0043$) and TA = 1.45 MPa ($P = 0.003$). Region of canal had an influence on the values for the cement used in group E7, in which only the TA presented differences from the CI.

Conclusions Endofill interfered negatively with the bond to root dentine along its full length and in the TA when post fixation was delayed for 7 days. Bond strength decreased from crown to apex in all groups.

Keywords: bond strength, endodontic cement, eugenol, intra-radicular dentine, root post.

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Introduction

The restoration of root filled teeth is a challenge, because they often have insufficient coronal structure to retain the restorative material. Thus, the use of the root canal is necessary to make coronal reconstruction feasible (Morgano & Brackett 1999, Boone *et al.* 2001).

Fibreglass posts are considered appropriate for retention inside root canals because their insertion technique is simple and rapid (Malferrari *et al.* 2003), as they can be prepared and moulded in a single session. Furthermore, in combination with an adhesive system and a resinous cement, they present biomechanical characteristics similar to that of dentine (Ferrari *et al.* 2000a, Malferrari *et al.* 2003), and favour the distribution of stresses from occlusal forces on the root structure.

Root reinforcement resulting from a fibre post cemented by means of an adhesive system and resin-type materials (Ferrari *et al.* 2000a, Grandini *et al.* 2004) is dependent on effective bonding between the adhesive components and the tooth substrate (Rosenstiel *et al.* 1998). The effectiveness

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of the bond may be compromised by the difficulty in obtaining direct light irradiation in apical regions, and it is usually necessary to use resin-type cements with dual or chemical polymerization (Foxton *et al.* 2003). Furthermore, phenolic eugenol-based materials or residual calcium hydroxide (Paul & Scharer 1997) may negatively influence the bonding process (Hansen & Asmussen 1987), and reduce the bond strength.

In this study two hypotheses were tested: (i) that the chemical composition of the endodontic sealer cement and (ii) the period of time elapsed between canal filling and fibreglass post fixation had a negative influence on the retention of posts inside the root canal. Thus, the aim was to analyse, by means of a mechanical micropush-out test, the bond strength of adhesively cemented fibreglass posts in the cervical, middle and apical thirds of root canals of bovine incisor teeth. The posts were either cemented immediately or 7 days after root filling with gutta-percha and one or other of a zinc oxide and eugenol-based or calcium hydroxide-based sealer.

Materials and methods

From among 780 extracted bovine mandibular incisor teeth, 60 were selected and stored in 0.2% buffered aqueous thymol solution. The selection criterion used was the similarity of the external and internal anatomical morphology and a canal diameter smaller than 1 mm. Roots 15 mm in length were produced after removing the coronal portion. The canals were instrumented 1 mm from the apex by the stepback technique using endodontic files and Gates-Glidden burs (Lot 1498110; Dentsply Maillefer, Ballaigues, Switzerland) numbers 2, 3 and 4 with diameters of 0.7, 0.9 and 1.1 mm, respectively, leaving the canals with a diameter of 0.9 mm in the middle and apical thirds, and 1.1 mm in the cervical third. Throughout instrumentation the canals were irrigated profusely with 1% sodium hypochlorite solution. After final irrigation with physiological solution, the canals were dried with absorbent paper tips (Lot 3386; Dentsply Maillefer, Petrópolis, Brazil) and the samples were randomly divided into five groups (n = 12 by means of Minitab Statistical Software; Minitab Corporation, Chicago, IL, USA). Group 1 (CI) canals were instrumented, but not filled (control). The other samples were filled with gutta-percha (Lot 262739; Dentsply Maillefer) and the specific cement for each group, using the lateral compaction technique. Group 2- (SI) calcium hydroxide-based cement (Sealer 26, Lot 236880; Dentsply Maillefer) and immediate post fixation; Group 3- (S7) Sealer 26 and post fixation after 7 days; Group 4- (EI) zinc oxide and eugenol-based cement (Endofill, Lot 288028; Dentsply Maillefer) and immediate post fixation; and Group 5- (E7) Endofill and post fixation after 7 days.

Post spaces were prepared immediately after filling to a depth of 10 mm, using heated instrument (GP heater; Dentsply Maillefer) to remove gutta-percha; post preparations were completed with a bur 5 (Lot 0459; GS Brasil, São Paulo, Brazil), 1.5 mm in diameter.

In the groups with post cemented 7 days after filling, access to the canals of the specimens was immediately sealed with glass–ionomer cement (Vidrion R, Lot 008; SS White, Rio de Janeiro, Brazil) and specimens were then stored in distilled water at 37 °C. After 7 days, the canals were prepared with the number 5 bur as described above.

Cylindrical fibreglass posts, with conical apical ends and circumferential mechanical retainers (Reforpost No. 3, Lot 1980; Ângelus, Londrina, Brazil), 1.5 mm in diameter were used in all groups. The external surfaces of the roots were covered with utility wax and the canals etched with 37% phosphoric acid (Lot 242437; Dentsply Maillefer) for 15 s, washed and dried with absorbent paper points (Lot 242437; Dentsply Maillefer). The adhesive system (Adper Scotchbond Multi Purpose, Lot 4NY; 3M-ESPE, St Paul, MN, USA) was used in accordance with Dong *et al.* (2003). Two consecutive layers of primer were applied using a microbrush, gently air dried after 20 s, followed by the application of the adhesive. Excess adhesive was removed from the canal using a clean microbrush, to ensure post adaptation (Grandini *et al.* 2004). Photo-activation was performed for 20 s on the cervical root face, parallel to the long axis of the root, using a halogen light unit with intensity of 750 mW cm^{-2} (XL 3000; 3M-ESPE). The post was cleaned with 70% alcohol in a single application using a microbrush, and after drying a silane agent was applied (ceramic Primer, Lot 4WC; 3M-ESPE). The dual cement (RelyX ARC, Lot ENEW; 3M-ESPE) was manipulated in accordance with the manufacturer's instructions, introduced into the canal using a low speed lentulo spiral filler (Lot 5042170600; Dentsply Maillefer), and after being coated, the post was inserted in the canal. Excess cement was removed after 1 min. Three minutes after insertion, photo-activation was performed for 40 s each on the cervical face of the root, in the direction of the long axis of the root, and obliquely to the buccal and palatal surfaces, totalling 120 s. After polymerization, the samples were stored in distilled water at 37 °C for 24 h.

After removing approximately 0.5 mm of the external portion of the post, 10 roots from each group were cross-sectioned into six slices, with a double face diamond disc (4″ × 0.12 × 0.12, Extec, Enfield, CT, USA) at low speed under water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA). Thus, two 1 mm thick discs were obtained from the cervical (TC), middle (TM) and apical (TA) third regions. The slices were obtained with a single cut to guarantee flat surfaces.

To perform the micropush-out test, a device specially developed for this test was used. It consisted of a stainless steel metal base, 3 cm in diameter, with a 2 mm orifice in the central region and a load applicator tip 1 mm in diameter and 3 mm long. After the specimen was positioned on the test machine (EMIC DL 2000l; São José dos Pinhais, Brazil) containing the 50 kgf load cell, the tooth discs were positioned so 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^{-1}, until failure by displacement of the post. The values were obtained in Newton, and subsequently expressed in MPa, they were divided by the area of the adhesive interface, calculated by the following formula:

$$A = 2\pi r \times h,$$

where π is the constant 3.14, r is the post radius and h is the specimen thickness in mm, using a 0.001 mm scale (digital calliper, S235; Sylvac, Neuchâtel, Switzerland) (Goracci *et al.* 2004).

Exploratory analysis indicated that square root transformation of the data was required to meet with the presuppositions of a parametric test. After data transformation, analysis of variance (ANOVA) in a split plot arrangement was performed, with the plot represented by the factors material, time and interaction among them and the sub-plot represented by the canal regions. The control was considered as additional treatment. The comparison among the control and the other treatments was made in each canal region, using the Dunnett test ($\alpha = 0.05$).

Two roots from each group, which were not submitted to the micropush-out test, were selected for adhesive interface analysis by scanning electron microscopy (SEM). One root was cross-sectioned into three slices of 3 mm thickness, in order to obtain one disc per third of the root. The other root specimens were longitudinally sectioned, polished with 1000, 1200, 1500 grain abrasive papers and with diamond pastes (Arotec, São Paulo, Brazil) with granulations of 6, 3 1 and 0.25 μm and ultrasonically cleaned in distilled water for 30 min. They were then fixed in 2.0% glutaraldehyde for 15 h at 4 °C, washed three times in distilled water, dehydrated in alcohol at the following concentrations: 50%, 70% and 95% for 10 min each and at the concentration of 100% for 30 min. They were then stored in an oven inside a receptacle containing silica for 8 h to remove moisture. The specimens were metal coated (MED 010; Balzer, Leichtenstein) and the bond interface evaluated by analysing the hybrid layer and presence of a gap by SEM (LEO; Carl-Zeiss, Deutschland, Germany) with standardized magnification of 100, 200, 500 and 1000× in three observation regions: cervical third (from 0 to 3 mm from the cervical surface); middle third (between 4 and 6 mm distant from the cervical surface) and apical third (between 7 and 9 mm distant from the cervical surface of the root).

Results

The bond strength values in MPa (mean and standard deviation) in terms of the chemical composition of the filling cement, time and region for the experimental groups, are shown in Table 1 and Fig. 1. ANOVA indicated a statistically significant difference among the groups, and the Tukey ($P < 0.05$) and Dunnett ($\alpha = 0.05$) tests demonstrated that the bond strength to root dentine in the middle and cervical thirds for the control group (CI), the groups filled with Sealer 26 immediately (SI) and after 7 days (S7) and the group filled with Endofill with post cementation after 7 days (E7) were not significantly different. The group filled with Endofill with immediate post cementation (EI) had a significantly reduced bond strength, irrespective of the root region; for cementation after 7 days (E7) significant differences were observed only in the apical third of the root.

For the groups filled with Sealer 26, values from the cervical third were similar to those of the middle but different from the apical third, which were similar between groups. The groups filled with Endofill had a significantly different result between all regions of the canal: cervical, middle and apical. Depth potentiated the effect of the cement for the group filled with eugenol-based cement with post cementation after 7 days (E7), in which only the apical third was different from the control group.

Table 1 Bond strength in MPa, (mean and standard deviation) as a result of composition, time and root region

		Region					
		Cervical		Middle		Apical	
Time	Composition	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
Immediate	Sealer 26	5.19 A	1.65	4.96 AB	2.04	4.12 B	2.28
Immediate	Endofill	3.50[a] A	0.76	2.22[b] B	0.50	1.45[c] C	0.70
7 days	Sealer 26	6.92 A	1.99	5.40 AB	1.77	4.50 B	1.64
7 days	Endofill	6.59 A	1.75	4.39 B	1.40	2.45[c] C	0.68
Control		6.76 A	1.51	5.46 AB	1.92	4.45 B	1.83

Mean values followed by different upper-case letters in the horizontal differ among them by the Tukey test ($P < 0.05$).
[a]Differ from the control in the cervical region by the Dunnett test ($P < 0.05$).
[b]Differ from the control in the middle region by the Dunnett test ($P < 0.05$).
[c]Differ from the control in the apical region by the Dunnett test ($P < 0.05$).

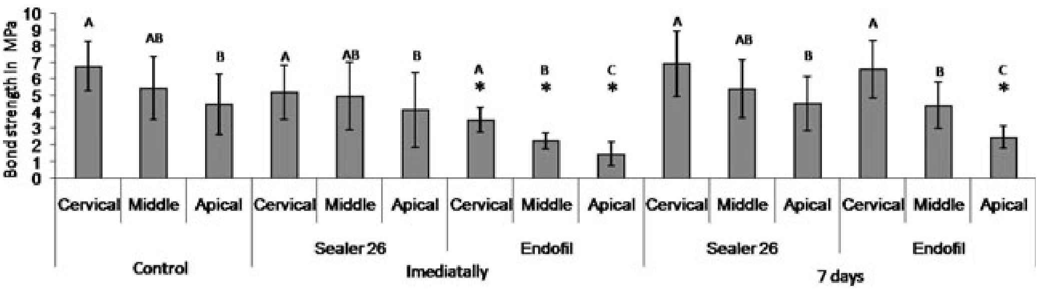

Figure 1 Bond strength in MPa (mean and standard deviation) as a result of composition, time and root region.

The Dunnett test revealed that in relation to the time factor, when the post was cemented 7 days following root filling the bond strength was greater only for the group filled with Endofill in all the regions ($P < 0.05$). For the group filled with Sealer 26, there was no difference between the times ($P > 0.05$). Furthermore, bond strength was greater for the groups filled with Sealer 26 in comparison with groups filled with Endofill, irrespective of time and region ($P < 0.05$).

A substantial decrease in bond strength was noted with increase in depth, with lower values moving from the cervical to apical direction. As regards the control group, statistical differences were observed in the cervical, middle and apical thirds. For group EI a difference was also noted among the three regions, and for E7 only in the apical third.

The results obtained by SEM complemented the mechanical micropush-out test. The bond interface in the middle and apical regions of the groups filled with calcium hydroxide-based cement, for both the immediate and the test after 7-day time, was homogeneous, presenting gaps at the dentine–cement interface only in the apical region, which was also observed for the control group. The presence of blisters could also be observed in the fixation cement in the cervical, middle and apical regions of all the experimental groups. In the apical region of group EI, the presence of gaps was also observed along the entire surface.

Discussion

The hypotheses of this study were confirmed. The results suggested that the chemical composition of the sealer cement and the time interval between root filling and post fixation had an influence on post bond strength. The results from groups with eugenol-containing sealer cement, in which the bond strength decreased significantly in the cervical, middle and apical thirds in the immediate period and only in the apical third after 7 days, are in agreement with previous studies, in which the presence of eugenol interfered with the polymerization of dentine adhesive systems (Hansen & Asmussen 1987, Cohen *et al.* 2002).

Bond strength to root dentine has been measured through conventional tensile (Tjan & Nemetz 1992, Boone *et al.* 2001), pull-out (Drummond 2000), push-out (Boschian Pest *et al.* 2002, Kurtz *et al.* 2003), microtensile (Ngoh *et al.* 2001) and micropush-out tests (Goracci *et al.* 2004). High, nonuniform stress concentrations may, however, be developed at the bond interface when thick specimens are used (Ngoh *et al.* 2001). The use of samples with reduced thickness in microtensile tests (Pashley *et al.* 1999) allowed more uniform stress distribution at the bond interface, in addition to making it possible to obtain real measurements of small areas inside the canal, and to analyse regional differences at the three levels of the root canal (Bouillaguet *et al.* 2003). However, premature failures in microtensile tests have been observed when beam shape specimens obtained by sectioning of teeth (Foxton *et al.* 2003) and hourglass shapes (Bouillaguet *et al.* 2003) were used in the post microtensile tests. Thus, in bond strength studies with the use of posts, the micropush-out laboratory test would seem to be safer than the microtensile test, as there is no premature loss of samples during the specimen manufacture phase (Goracci *et al.* 2004).

Although human teeth are preferable and have been used for many years, due to bioethical reasons, bovine teeth have increasingly been used to assess properties of restorative materials (Nakamichi *et al.* 1983, Schilke *et al.* 2000, Bouillaguet *et al.* 2001, Miyazaki *et al.* 2002, Dong *et al.* 2003) and accepted as possible substitutes for human teeth in either dentine or enamel bond strength tests (Reis *et al.* 2004). Bovine teeth are more easily collected, enable age standardization and reduce the risk of transmitting infectious-contagious diseases.

The mean bond strength values for all the groups, including the control, were higher in the cervical and lower in the apical thirds. This was possibly because of poor resinous cement activation in the regions distant from the activating light source (Foxton *et al.* 2003). Hence, a homogeneous bond interface was observed in the cervical and middle thirds of the control group, with gaps only in the apical region (Fig. 2).

The use of double activation for adhesive systems and resin-based cements has been proposed to prevent polymerization failures in deep regions (Rueggeberg & Caughman 1993). However, Dong *et al.* (2003) suggested that dual-activated ScotchBond Multipurpose Plus leaves an acid surface which negatively affects the degree of conversion of resin-based cement. In the present study a physically and chemically activated resin-based cement (RelyX ARC) in association with the adhesive system ScotchBond Multipurpose Plus activated only by light was used.

Calcium hydroxide cement is difficult to remove (Fonseca *et al.* 2005) and residues may remain on the root canal walls, interfering with the bonding process (Paul & Scharer 1997). In this study, however, the groups filled with calcium hydroxide-based cement, irrespective of the time factor, presented no significant difference in comparison with the control group. This is in agreement with the results of Hagge *et al.* (2002), in which microscopic images suggestive of similar adhesive interfaces were observed (Figs 3 and 4). This is probably justifiable because the remaining sealer cement was removed from the root canal walls during preparation for post fixation, performed with a wide bur No. 5, after canal filling.

The samples of group EI had values statistically different from the control, SI and S7 groups in all canal

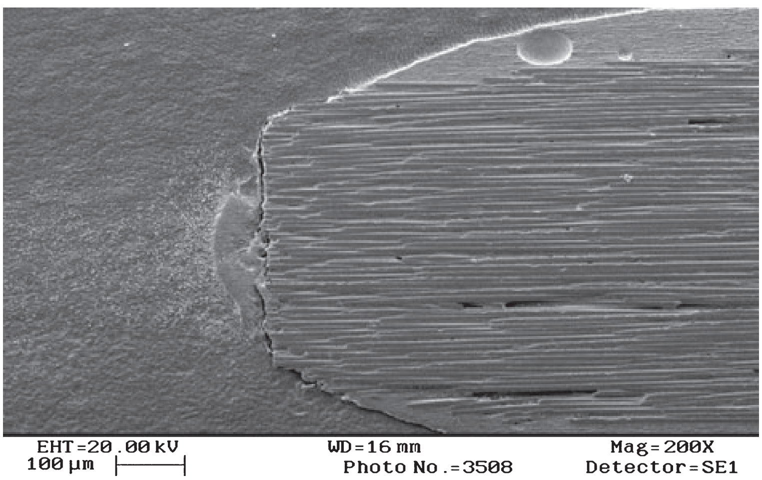

Figure 2 CI (control): Bond interface in the apical region.

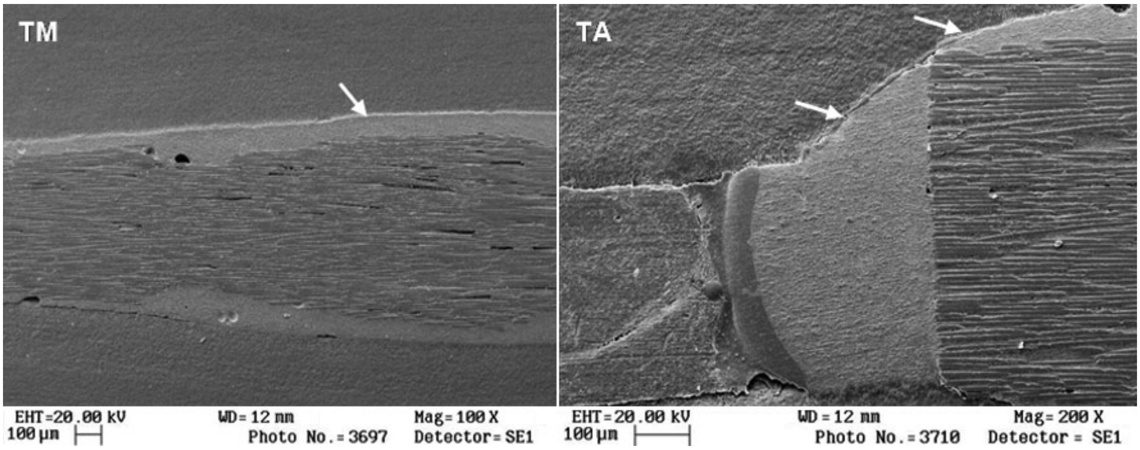

Figure 3 SI (immediate sealer): Bond interface in the middle (TM) and apical (TA) thirds.

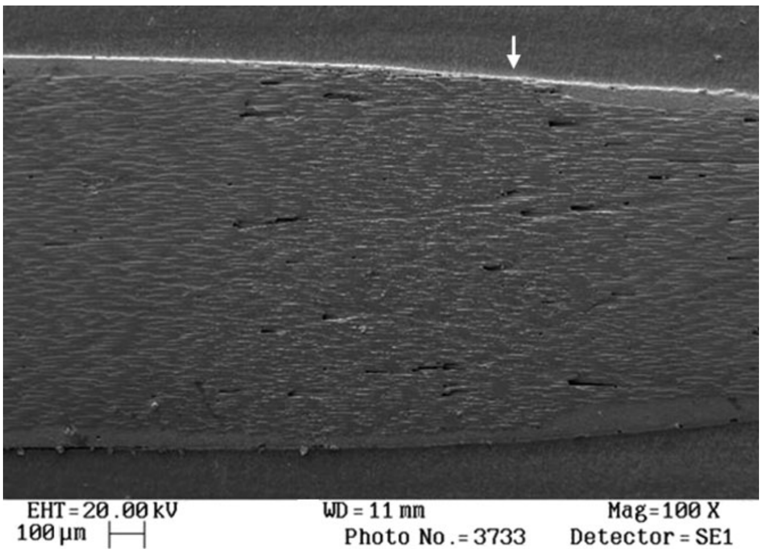

Figure 4 S7: Representative image of the bond interface in the middle third.

regions. The lowest bond strength values were found in the apical third of all groups. Gaps in the bond interface of group EI (Fig. 5) were observed by means of SEM, possibly because the polymerization reaction of composite resins and dentine adhesive agents was inhibited by the hydroxy group present in the eugenol-containing filling cement, which tends to protonate and block the reactivity of radicals responsible for polymerization (Paul & Scharer 1997). Markowitz *et al.* (1992) reported that a chelating reaction occurs when zinc oxide is mixed with eugenol, resulting in grains of zinc oxide imbibed in a zinc eugenolate matrix, which makes it impossible for eugenol to be released. However, due to the presence of fluids inside the dentinal tubules, this reaction becomes reversible; the eugenol released penetrates the dentine and tends to concentrate at the tooth–adhesive interface (Ganss & Jung 1998). The possible explanation for the similarity of values between E7 and control groups in the cervical and middle thirds may be that the effect of eugenol disappears after 7 days, as reported by Fonseca *et al.* (2005) in the indirect restoration cementing process. The significantly lower values from the apical third of group E7 in comparison with the control group may be the result of poor polymerization associated with the effect of the large amount of residual eugenol (Yap *et al.*

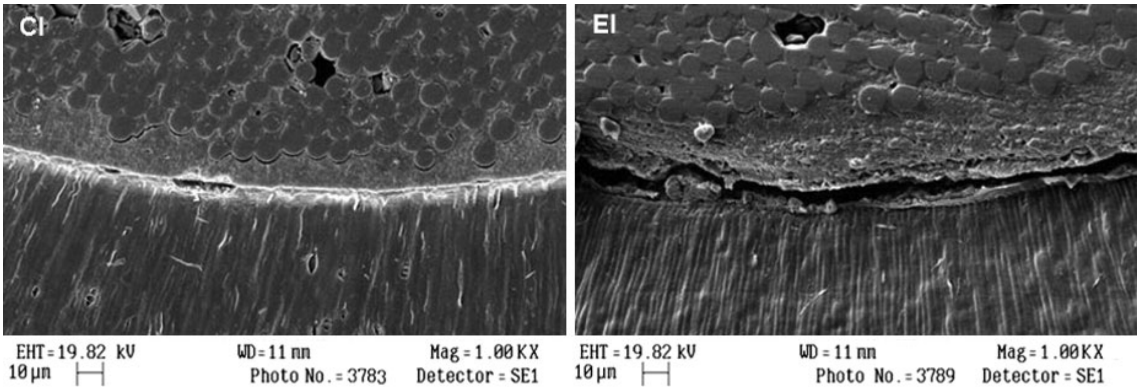

Figure 5 Representative image of the bond interface in the apical third of groups CI and EI.

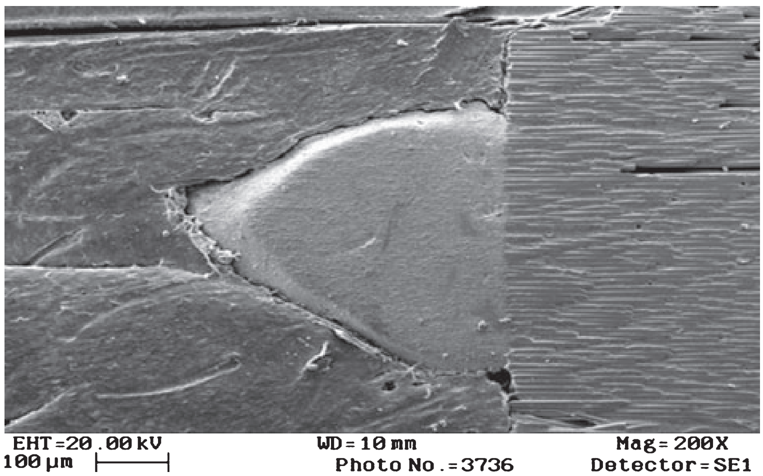

Figure 6 Group E7 apical third: image suggestive of the remaining filling in contact with the fixation cement and the post.

2001) remaining in this region, as the apical extremity of the post is in close contact with the remaining sealer cement (Fig. 6).

Different results obtained in other studies indicate that eugenol-containing endodontic sealer cements had no influence on bonding to dentine (Schwartz *et al.* 1998, Boone *et al.* 2001, Hagge *et al.* 2002). This may be related to the probability that these studies did not consider the clinical situation in which the root canal is prepared for post cementation after the root canal is filled (Boone *et al.* 2001). Thus, the results may be masked due to the presence of remaining filling cement on the canal walls, acting as a barrier and preventing the action of the dentinal adhesive agents. Another possibility would be the time following post space preparation, a factor not taken into account in the majority of studies (Schwartz *et al.* 1998, Hagge *et al.* 2002), in which the eugenol-based provisional material was replaced after a period of 7 days, when its inhibitory effect on the polymerization process was reduced.

Considering that further canal preparation is necessary to provide a post (Prado *et al.* 2006), and that materials used for temporary coronal sealing do not inhibit, but only decrease bacterial infiltration, it could be beneficial to cement the post immediately after root canal preparation. This procedure might reduce the risk of coronal and apical leakage, as there would be no empty space inside the root canal, and the filling and restorative procedure could be performed in a single session.

Considering the negative influence of eugenol-containing cement on the bonding mechanism and that in clinical practice posts are often cemented immediately after root filling, the use of a calcium hydroxide-based sealer cement may be a feasible alternative. For cementation after 7 days, the result of this investigation demonstrated drawbacks with the eugenol-based filling cement, as lower mean bond strength values were observed in the apical third, than those of the control group.

Some limitations in the present study should be considered: samples were not submitted to thermal and mechanical cycling, in order to simulate intra-oral situations more precisely. There is, however, need for further studies with regard to the effect of eugenol-based materials at different time periods. Other materials, such as resin-based endodontic cements, as an alternative to eugenol-based cement, should be investigated.

Conclusion

The following conclusions were drawn.

1. The calcium hydroxide-based endodontic sealer (Sealer 26) did not influence the pattern of bonding to root dentine irrespective of depth and time evaluated.

2. The eugenol-based endodontic sealer (Endofill) had a negative influence on bonding in all regions of the canal when placed immediately following root filling. For the 7-day period, this negative influence was noted in the apical third only.

3. The influence of canal depth, because of poor polymerization, was observed as the bond strength decreased from the cervical to apical third in all the groups.

4. When posts are to be cemented immediately after canal filling, calcium hydroxide-based sealer cements may be preferable to eugenol-containing sealers.

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