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Can viscosity of acid etchant influence the adhesion of fibre posts to root canal dentine?

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Abstract

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Aim To evaluate the influence of acid viscosity, endodontic sealer and root canal region on the push-out bond strength of a glass fibre post.

Methodology Seventy-eight single-rooted human teeth were selected (60 for push-out and 18 for scanning electron microscopy characterization, SEM, $n = 13$ per group). The root canals were prepared with a step-back technique and then filled with Endofill or AH Plus sealer. Teeth without root fillings were used as controls. The preparation of the post-space was to a length of 11 mm using standardized rotary instruments. The root dentine was treated with 37% phosphoric acid (gel or liquid). The fibre posts (Reforpost) were silanized, and resin cement (Enforce) was used for luting procedures. Each root was cross-sectioned, and samples from the cervical and apical regions were subjected to a push-out bond strength test. Specimens from each group were sectioned longitudinally and subjected to SEM characterization for the dentine/cement/post interface. Statistical analysis for push-out tests was carried out using factorial ANOVA followed by Tukey's test ($P < 0.05$).

Results The three factors under evaluation (acid, endodontic sealer and region) and their interaction significantly influenced bond strength values ($P < 0.05$). In general, liquid phosphoric acid had significantly higher bond strength values in the apical region ($P < 0.05$), with hybrid layer formation, while endodontic sealers reduced bond strength values compared to the control ($P < 0.05$).

Conclusion The use of a liquid acid etchant created higher bond strength values in the apical region.

Keywords: bond strength, endodontic posts, luting cements, push-out, root dentine, total-etching.

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Introduction

Several types of post have been proposed to improve the mechanical and aesthetic properties of restorations. In this context, the use of pre-fabricated glass fibre posts is becoming increasingly popular (Giachetti *et al.* 2009). The stiffness (or modulus of elasticity) of these posts is similar to that of dentine, so as to distribute the functional forces evenly along the length of the root (Assif *et al.* 1993, Asmussen *et al.* 1999, Watzke *et al.* 2008).

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The clinical performance of a restoration with a fibre post depends on several factors namely: post-material, shape, dimension, and length (Mannocci *et al.* 1999, Akkayan & Gülmez 2002, Maccari *et al.* 2003, Lassila *et al.* 2004), the quality and quantity of remaining dentine (Malferrari *et al.* 2003, Monticelli *et al.* 2003), the type of adhesive and cement used, and how the post is adapted inside the root canal (Ferrari *et al.* 2002, Giachetti *et al.* 2003, Goracci *et al.* 2005). Although bonding between different types of posts and resin cements has been demonstrated, light curing is difficult inside the root canal (Giachetti *et al.* 2003), dentine substrate provides a lower hybridization potential (Mannocci *et al.* 2004) and there is a higher configuration factor in root canals (Bouillaguet *et al.* 2003).

The apical third of the root canal is the location where most smear layer, debris and sealer/gutta-percha residues are found after post-space preparation and acid etching (Serafino *et al.* 2004). The bond strength between resin cements and root dentine is generally low (Ferrari *et al.* 2000, Bouillaguet *et al.* 2003). This low bond strength may not be capable of overcoming the shrinkage stresses generated during polymerization of the resin luting agent, as a thin layer of curing resin with limited free-surface for stress relief creates an undesirable scenario when C-factor is of concern (Tay *et al.* 2005).

While adhesives and resin cements are being studied in relation to adhesive luting of pre-fabricated posts, no studies have addressed the role of acid viscosity on etching and on the bond strength of posts. Thus, the aim of this study was to evaluate the push-out bond strength of pre-fabricated glass fibre posts cemented in human root canals, investigating the effect of two endodontic sealers, two types of phosphoric acid and two root regions. The null hypothesis assumed no differences for the factors acid viscosity (gel or liquid), endodontic sealer (control, Endofill or AH Plus), region of evaluation (cervical or apical) or interactions, considering the response variable bond strength.

Materials and methods

Experimental design

The study was approved by the Research and Ethics Committee of the Federal University of Pelotas (29/04). It involved a randomized and examiner-blinded design with 78 teeth selected and prepared for post-placement. After preparation and post-cementation, specimens were cross-sectioned to obtain cervical and apical root slices to perform the push-out test. Tested specimens from each group were subjected to SEM preparation to observe the dentine/cement/post interface. All endodontic procedures were carried out by a single operator and all the measurements by two blinded and trained examiners. Teeth were considered as experimental units for statistical analysis. The materials used in this study are listed in Table 1.

Specimen preparation

Seventy-eight single-rooted teeth with at least 14 mm of straight root canal extracted for periodontal reasons were selected. The teeth were cleaned and stored in 0.5% chloramine T for 1 week. The teeth were randomly allocated to six groups (n = 13), depending on the type of etching, liquid or gel, and the sealer, Endofill or AH Plus (Dentsply Caulk, Milford, DE, USA). Sixty teeth were used for the push-out bond strength test (n = 10 per group), and 18 additional teeth were used for SEM characterization (n = 3 per group). Teeth with no endodontic treatment but with either liquid or gel etching application served as controls.

The crowns were removed from the tooth at 1.5–2 mm from the cemento-enamel junction using a diamond saw, and the pulp tissue removed. The root canals were prepared by hand instrumentation using Flexofile instruments (Dentsply Maillefer, Ballaigues, Switzerland). Instruments sizes 15–40 were used to create an apical stop 1 mm short of the canal terminus. A step-back preparation with 1 mm increments was performed up to instrument size 70. Debris generated after each instrument was rinsed with 2 mL of freshly

Table 1 Composition of materials

Material (Batch number)	Manufacturer	Composition
Enforce (253795)	Dentsply Caulk	Bisphenol A glycol dimethacrylate (Bis-GMA), butylated hydroxytoluene (BHT), ethyl dimethylamino-benzoate (EDAB), triethyleneglycol dimethacrylate (TEGDMA), fumed, silica, silanized barium, aluminium, borosilicate glass (66 wt.%)
Prime & Bond 2.1 (32010)	Dentsply Caulk	Acetone, elastomeric resin, PENTA, cetylamine hydrofluorid
Gel 37% Phosphoric acid (176675)	Dentsply Caulk	Phosphoric acid, water, silica, inorganic pigments
Liquid 37% Phosphoric acid (–)	–	Phosphoric acid, MilliQ water
Silane (209071)	Dentsply Caulk	Silane, ethanol, acetic acid
AH Plus (0408000142)	Dentsply Caulk	Paste A: Bisphenol-A epoxy resin, Bisphenol-F epoxy resin, Calcium tungstate, Zirconium oxide, Silica, Iron oxide pigments; Paste B: Dibenzyldiamine, Aminoadamantane Tricyclodecane-diamine, Calcium tungstate Silica, Silicone oil
Endofill (3163)	Dentsply Caulk	Powder: zinc oxide, staybelite resin, bismuth subcarbonate, barium sulphate, sodium borate; Liquid: eugenol

prepared 2% sodium hypochlorite. Root canals were dried for 30 s with paper points (Dentsply Maillefer) and filled with AH Plus or Endofill sealer and gutta-percha using lateral condensation.

After 2 weeks storage in distilled water at 37 °C, gutta-percha was removed from the coronal and middle thirds of each root canal with Gates Glidden drills (Dentsply Maillefer) sizes 1 and 2 (ISO sizes 050 and 070, respectively). Finally, Peeso reamers (Dentsply Maillefer) sizes 2 and 3 (ISO sizes 070 and 090, respectively) were used to refine the post-space. No lateral pressure was applied against the root canal walls when Gates Glidden drills and Peeso reamers were used. Each drill was measured with silicon stops to ensure that the post-space was 11 mm long. Finally, the Largo drill corresponding to the size that came with the post was used to standardize post-spaces. Two millilitre of sodium hypochlorite was again used after each instrument. After preparation, root canals were dried with paper points.

Post-cementation

Gel or liquid 37% phosphoric acid (Dentsply) was injected into the canal with a syringe until overflow and allowed to rest for 15 s, rinsed with 15 mL of water for 15 s with another syringe and dried with paper points. The needle was measured with silicon stops to ensure that the full length of the root canal was etched. Prime & Bond 2.1 (Dentsply) was applied with a microbrush, the excesses were removed with paper points, gently air dried and polymerized for 20 s (500 mW cm^{-2}; XL3000, 3M ESPE). The post was once again tested inside the root canal to guarantee it fitted.

The glass fibre posts (No. 2; Reforpost, Angelus, Londrina, PR, Brazil) were cleaned with 95% ethanol for 5 min. Silane was applied with a micro-brush for 60 s (Silano, Angelus, Brazil). Fibre posts were cemented with dual-cured resin cement (Enforce; Dentsply). Cement was placed into the canal with a lentulo spiral filler, starting at the apical end and withdrawing back towards the canal entrance. The posts were seated into the canal with firm pressure, and excess cement was removed. The roots were placed in a pressure device under 5 kg loading for 10 min (Ottl *et al.* 2002), and light curing was performed for 40 s using a XL 3000 light curing unit (3M ESPE, St Paul, MN, USA), with energy higher than 450 mW cm^{-2} and the light unit directly in contact with the posts. After cementation specimens were stored in distilled water at 37 °C for 24 h.

Push-out test specimen preparation

Three sections were prepared from each tooth to create coronal, middle and apical sections; the coronal and apical sections were subjected to bond strength testing. The roots were sectioned at right angles to their long axis with a low-speed diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA), under water cooling. Two 1.5-mm-thick slices for each region were produced and polished with 600-grit silicon carbide paper. The slices were mounted in a universal testing machine (INSTRON 4411; High Wycombe, UK) with the apical surface facing up and loaded with 100 N until fracture at a speed of 0.5 mm min^{-1}. Results were reported in MPa and calculated according to the surface adhesion area.

SEM characterization

To evaluate the interface between dentine–cement–post, 18 additional specimens (three per group) were prepared and tested for push-out as previously described. The slices after fracture were dehydrated, mounted on a stub, air-dried, sputter-coated with gold and the adhesive interface examined with a scanning electronic microscope (JEOL JXA 6400, Tokyo, Japan) under an accelerating voltage of 20.0 kV with a working distance of 13–25 mm. Adhesive failures were observed and recorded for these samples (Fig. 1).

Statistical analysis

The assumptions of equality of variances and normal distribution of errors were checked, and to fit these assumptions, MPa data were transformed in ranks and analysed by three-way ANOVA and the Tukey's test ($\alpha = 0.05$, Table 2). Data were analysed using the SAS software v. 9.0 (SAS Institute Inc., Cary, NC, USA).

Results

Push-out bond strength was found to vary significantly according to the interaction of type of sealer, acid viscosity and root region ($P < 0.05$). The liquid acid had a significant effect on the push-out bond strength values in the apical region for the control and Endofill groups, while it decreased the bond strength values for Endofill in the cervical region ($P < 0.05$, Table 3). The bond strength values were affected by the type of endodontic sealer, and this was dependent on the root region and acid viscosity (Table 3). Overall, no endodontic sealer use (control) was associated with bond strength values

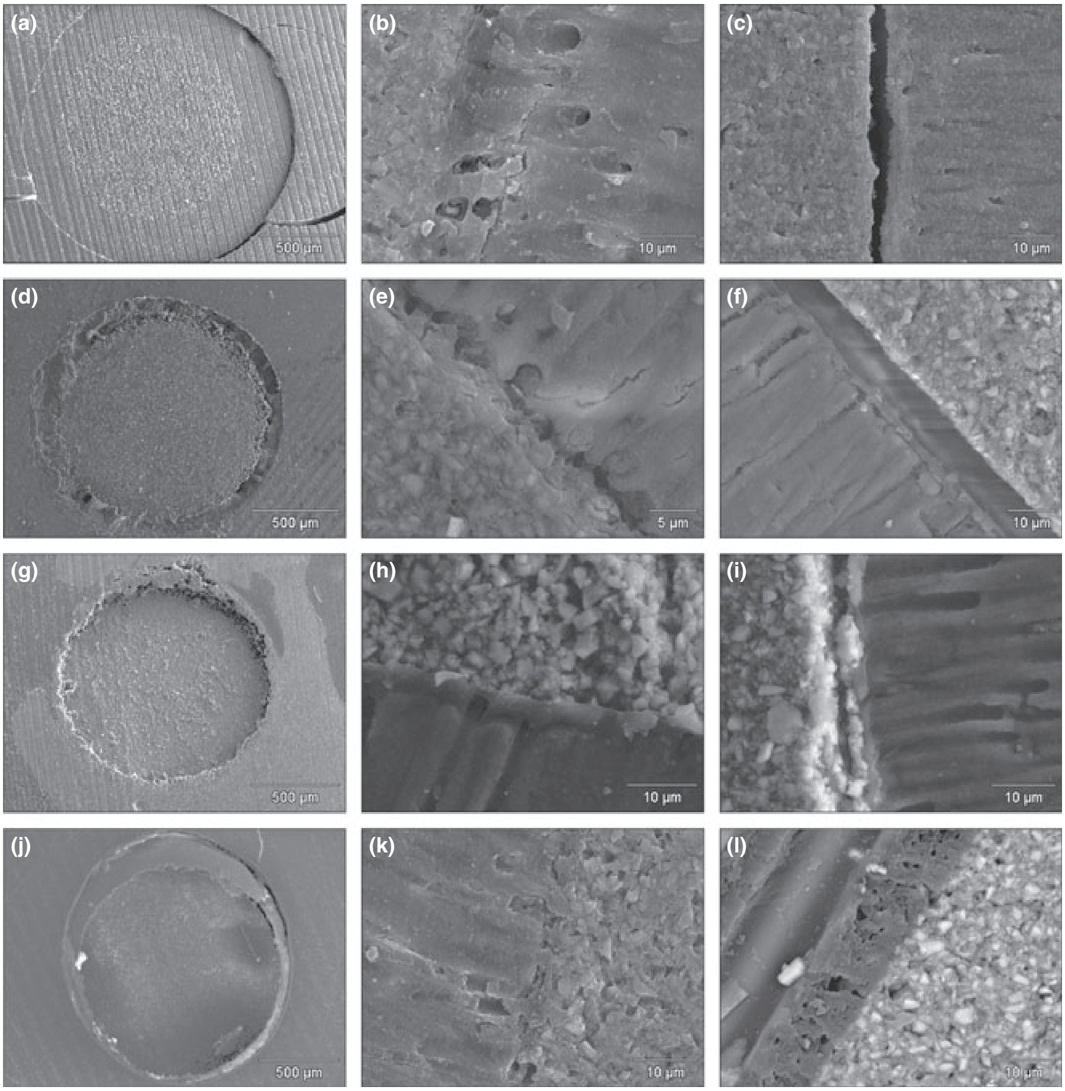

Figure 1 (a–c) Specimens filled with Endofill and etched with phosphoric acid gel. Push-out test with failure was observed at the cement–dentine interface (a) and a defined hybrid layer formed at the coronal region (b), while in the apical region a gap is observed (c); (d–f) specimens filled with Endofill and etched with liquid phosphoric acid. Debonding at the cement–dentine interface is seen (d) and a well delimited and continuous hybrid layer is observed in both coronal (e) and apical regions (f); (g–i) specimens filled with AH Plus sealer and etched with acid gel. The push-out specimen reveals the failure at the cement–dentine interface (g), with the formation of a continuous hybrid layer in the coronal third (h), while such features were not observed in the apical region (i); (j–l) specimens filled with AH Plus sealer and etched with liquid acid, with debonding occurring at the cement–dentine interface (j), with a continuous hybrid layer formed in coronal (k) and also in the apical region (l).

higher than use of Endofill or AH Plus ($P < 0.05$, Table 3). Apical push-out bond strength was significantly lower than coronal push-out ($P < 0.05$).

Scanning electron microscopy illustrative analysis revealed that the largest number of failures was observed between the cement–dentine interface (adhesive failures), irrespective of the sealer, region or acid, and no spaces or gaps were observed in the cement–post interface (Fig. 1a,d,g,j). When the acid was used in gel form, regardless of the endodontic sealer, hybrid layer formation and tag formation were evident in the cervical region (Fig. 1b,h), but in the apical root region, hybrid layer was rarely observed or was discontinuous (Fig. 1c,i). However, with the use of liquid acid, tags

Table 2 ANOVA results for bond strength

Source	df	Type III sum of squares	Mean square	*F*	*P*
Sealer	2	35400.133	17700.067	40.918	<0.001
Acid	1	22609.719	22609.719	52.268	<0.001
Region	1	110676.386	110676.386	255.857	<0.001
Sealer × Acid	2	31483.367	15741.683	36.391	<0.001
Sealer × Region	2	1700.908	850.454	1.966	0.144
Acid × Region	1	59573.186	59573.186	137.719	<0.001
Sealer × Acid × Region	2	7590.375	3795.188	8.774	<0.001
Residual	148	64020.667	432.572		
Total	159	341320.000	2146.667		

Table 3 Push-out bond strength values in MPa (Mean ± SD)

	Liquid acid		Gel acid	
Sealer	Cervical region	Apical region	Cervical region	Apical region
Control	13.6 ± 2.3B	13.6 ± 3.5C*	13.2 ± 2.3B	7.2 ± 1.3B
AH Plus	12.6 ± 2.3AB	10 ± 1.3B*	10.8 ± 1.0A	3.8 ± 1.5A
Endofill	8.8 ± 1.6A*	8.6 ± 1.4A*	14.1 ± 3.0B	4.4 ± 0.9A

Upper case letters represents differences among sealers within each level for the factors root region and acid viscosity ($P < 0.05$).
*Represents differences between acid viscosities within each level for the factors root region and the type of endodontic sealer ($P < 0.05$).

and hybrid layer formation extended up to the apical third being constant along the whole root length (Fig. 1e,f,k,l).

Discussion

The biomechanical properties of fibre posts have been reported to be close to those of dentine (Piovesan *et al.* 2007, Plotino *et al.* 2007), and clinical prospective and retrospective studies have reported convincing results (Fredriksson *et al.* 1998, Malferrari *et al.* 2003, Piovesan *et al.* 2007). In addition, for aesthetic considerations, dentine-coloured post-and-core materials are now frequently used for all ceramic crown restorations (Quintas *et al.* 2000, Michalakis *et al.* 2004). Despite these advantages, several difficulties related to the use of glass fibre post remain. Bonding to intraradicular dentine presents challenges because of its complexity and technique sensitivity (Ferrari *et al.* 2000, Goracci *et al.* 2005), thus resulting in the most common cause of failure: debonding. The loss of adhesion is critical in apical regions because of a nonhomogeneous application of the etching and bonding procedures, incomplete cement polymerization in deeper areas in root canal because of lack of light penetration, even with dual cure cements (Foxton *et al.* 2003).

The peculiar histologic characteristics of the intraradicular dentine and the presence of a endodontic smear layer have prompted some to recommend a preliminary etching step of the dowel space before bonding (Zhang *et al.* 2008). The present study has shown that acid viscosity directly influenced push-out bond strength of glass fibre posts in the apical region, leading to a better adhesive performance. It is also important to highlight that one limitation of the study was the absence of artificial ageing. Further studies are needed to elucidate whether storage ageing will still result in the same findings for all groups.

The luting agent used in the present study does not contain any other surface treatment except for the etching (liquid or gel) and priming described, and no additional etching procedures were undertaken. Notwithstanding this, additional etching of dentine with phosphoric acid could, in principle, create an over etched situation where the demineralization zone becomes too deep for subsequently placed primers to completely penetrate.

In addition, the different endodontic sealers used affected the results. Endofill has eugenol in its composition and resulted in lower bond strength values in the apical region when liquid acid was used. However, it was noted that in general, both endodontic sealers reduced the push-out bond strength values, regardless of eugenol presence, compared to the control groups. This corroborates other studies where eugenol-based sealers have shown the same results (Ganss & Jung

1998, Leirskar & Nordbø 2000, Baldissara *et al.* 2006, Demiryürek *et al.* 2010). This reduction is probably associated with the presence of endodontic sealer debris during the post-space preparation, which would impair the acid etching and adhesive procedures in the root canals. In the present study, no significant difference was found between the eugenol and resin-based sealer groups when acid gel was used in the apical region. A possible explanation for this finding is that as in other studies where acid gel was used (Sevimay & Kalaycı 2005, Demiryürek *et al.* 2010), eugenol residues remaining on the dentine may interfere with the polymerization of adhesive resin. Resin-based sealers are compatible with dentine and penetrate deeply into dentinal tubules; therefore, sealer remnants in the tubules may have decreased the bond strength of resin cement.

The bond strength between the resin luting agent and post-space dentine is influenced by the distribution of resin cement in the coronal, middle and apical third of the root during the luting procedure and by the anatomic and histologic characteristics of the root canal, including the orientation of dentine tubules (Ferrari *et al.* 2000, Mannocci *et al.* 2004). Reasonable explanations for differences between root thirds include the high cavity configuration factor, quantity, orientation and volume of tubules toward the apical portion (Onay *et al.* 2010). This is probably because of the limited ability of the light to diffuse across the entire length of the resin cement, thus compromising the polymerization of the cement in the most apical regions (Roberts *et al.* 2004). Additionally, it is difficult to control for moisture and adhesive application towards the apical region of the canal (Bonfante *et al.* 2008).

Bonding to root canal dentine is hampered by limited visibility, anatomic features (Mjor *et al.* 2001) and a comparably high configuration factor inside the root canal (Tay *et al.* 2005) and was found to be less effective than bonding to coronal dentine. Bond integrity inside the root canal is challenged by the limited capacity to dissipate polymerization shrinkage stresses in long narrow post-spaces exhibiting a highly unfavourable configuration factor (Tay *et al.* 2005). An important issue when dealing with debonding of posts is how these data relate to the clinical scenario. For instance, the acid gel groups resulted in a bond strength approximately 40–50% (Endofill and AH Plus) lower than the control in the apical region. In the liquid form, the bond strength was 35–40% (Endofill and AH Plus) lower when compared to control. Thus, the significant differences between sealers may not be clinically relevant. On the other hand, when the liquid and gel acids were compared, the results may be interpreted differently when it comes to clinical practice. In the apical region, the use of liquid acid led to 3 and 2 $\times$ higher bond strength for AH Plus and Endofill, respectively, when compared to the acid gel.

Thus, liquid acid could be a feasible alternative to guarantee better etching in the apical region because of its decreased viscosity. It is hypothesized that liquid acid would have better wettability and lower surface energy compared to the gel acid, improving the capability of reaching the most difficult regions into the root canal.

Conclusion

The use of liquid acid yielded higher bond strength values for the fibre post to root dentine in the apical region.

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