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## **Active application of liquid etching agent improves adhesion of fibre posts to intraradicular dentine**

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**Running Title:** Active application of liquid phosphoric acid in post spaces

**Key words:** fibre post, phosphoric acid, post space, scanning electron microscopy

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## **Abstract**

**Aim** To assess the effectiveness of an active application of liquid etching, compared with the standard gel formulation on smear layer removal from post space walls and push-out bond strength of luted fibre posts.

**Methodology** Human extracted teeth were collected and root filled. After post space preparation and cleaning with 10% ethylenediaminetetraacetic acid for 30 s, teeth were assigned to four groups (n=11) according to etching procedure: 1) 37% phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) gel; 2) 37% H<sub>3</sub>PO<sub>4</sub> liquid applied with an endodontic needle; 3) 37% H<sub>3</sub>PO<sub>4</sub> liquid applied with an Endovac; 4) no etching procedure (control group). Three teeth per group were sectioned longitudinally and prepared for SEM examination to evaluate the presence of smear layer, debris, sealer/gutta-percha remnants, and the number of open tubules. Eight teeth per group were bonded with an etch-and-rinse adhesive, and fibre posts were luted with a resin-based cement. After cutting, specimens were prepared for a push-out test. Data were analysed by ANOVA and post hoc tests (p<0.05).

**Results** Improved smear layer removal was obtained in Group 2, followed by Group 1, Group 3, and the control group (p<0.05). The mean values for the bond strength of the push-out test were: Group 1, 8.3 ± 2.9 MPa (coronal); 7.7 ± 3.0 (middle); 3.3 ± 1.9 MPa (apical); Group 2, 7.8 ± 2.1 MPa (coronal); 6.9 ± 3.9 MPa (middle); 3.7 ± 1.3 MPa (apical); Group 3, 9.7 ± 2.8 MPa (coronal); 8.6 ± 2.1 MPa (middle); 6.9 ± 2.3 MPa (apical); and Group 4, 2.9 ± 3.0 MPa (coronal); 2.6 ± 2.0 MPa (middle); 1.1 ± 2.0 MPa (apical).

**Conclusions** Liquid phosphoric acid applied with an endodontic needle yielded better canal wall smear layer removal and higher bond strength values when an etch-and-rinse system is used.

## Introduction

Root filled teeth have been reported to have shorter survival times than teeth with vital pulp (Tang *et al.* 2010). This difference is believed to reflect the fact that root filled teeth have lost a large amount of coronal tooth substance and are exposed to shearing chewing forces. For these reasons, the placement of a post inside the root canal is frequently required to increase the retention of the restoration and improve their fracture resistance (Mangold *et al.* 2011). Traditional metallic cast posts have been gradually replaced by fibre posts (Bitter & Kielbassa 2007) due to several advantages, such as a greater elastic modulus (Plotino *et al.* 2007), a better biocompatibility, aesthetic, corrosion resistance (Boschian Pest *et al.* 2002, Maccari *et al.* 2003) and a decreased number of irreparable root fractures (Naumann *et al.* 2008, Salameh *et al.* 2008).

Durability and effective bonding between the fibre post, root dentine, and adhesive resin cement are essential for the longevity of restorations (Bonfante *et al.* 2008). Various luting approaches are available for bonding to intraradicular dentine (Mazzoni *et al.* 2009), however, several *in vivo* studies have shown that the debonding of fibre posts is the most common clinical failure in fibre post-retained restorations (Rathke *et al.* 2009).

Etch-and-rinse adhesive systems have shown predictable results in fibre-post adhesive cementation to radicular dentine (El Guindy & Fouda 2010). The bonding mechanism of etch-and-rinse adhesive systems to the dentine walls of canals is essentially micromechanical (Pashley *et al.* 2011, based on hybridisation of the demineralised surface and on resin tag and adhesive lateral branch formation (Mannocci *et al.* 1998, Ferrari & Mannocci 2000). To achieve a satisfactory hybrid layer and increase micromechanical retention, removal of the smear layer and endodontic debris from dentine canal walls and the initial parts of dentinal tubules has been assumed to be necessary (Morris *et al.* 2001).

Previous research has suggested that the efficacy of dentine adhesives depends mostly on smear layer removal and the formation of the resin-dentine interdiffusion zone (Bonfante *et al.* 2008, Gu *et al.* 2009). However, a critical aspect with all etch-and-rinse adhesives is achieving

effective removal of the thick secondary smear layer created by the calibrated burs for post-space creation on dentine (Breschi *et al.* 2009), which differs in the various regions of the root and consequently influences the bond strength of fibre posts (Kurtz *et al.* 2003, Mallmann *et al.* 2005).

In view of the difficulties experienced regarding access of instruments and chemical solutions to the apical region of the post space, there is a continuing need for new techniques focused on dentinal wall debridement. The physical state of the solution affects etching, and a recent study (Salas *et al.* 2011) showed that liquid phosphoric acid enhanced endodontic smear layer removal, and thus fibre-post bond strength. While gels can be applied in a more controlled manner than liquids, liquid solutions achieve a deeper penetration (Salas *et al.* 2011).

Root canal irrigation with several liquid solutions is needed to eliminate organic and inorganic debris and bacteria from the root canal system. Several irrigation devices and techniques have been proposed to control the flow liquid solutions and enhance their efficacy. EndoVac (SybronEndo, Orange, CA, USA) is an apical negative pressure irrigation device developed to irrigate and remove debris in the apical third without forcing irrigants into the periapical tissues (Nielsen *et al.* 2007). EndoVac generates an apical current liquid flow due to an apical negative pressure, when cannula tips are placed at various depth within the root canal (Nielsen *et al.* 2007).

No reported study has assessed the ideal application method to obtain better debridement of post space walls with liquid phosphoric acid. Thus, the aim of this laboratory study was to evaluate the effects of different phosphoric acid formulations and application techniques on the removal of the smear layer and the bond strength of fibre posts luted with a three-step etch-and-rinse dual-curing adhesive system in combination with a resin-based cement. The null hypotheses tested were that the etching agent formulation and the method of liquid phosphoric acid application would affect (1) the efficacy of endodontic smear layer removal or (2) fibre-post bond strength.

## **Materials and Methods**

In total, 44 extracted, caries-free, human single-rooted teeth were selected. After debriding the root surface, specimens were stored in 0.5% chloramine at 4°C. Each tooth was sectioned at the cementum-enamel junction, perpendicular to the long axis of the tooth. The mean length of roots was 15 mm ± 1. Root canals were instrumented to the working length using Pathfiles (1-2-3) and ProTaper files (S1-S2-F1-F2-F3; Dentsply Maillefer, Ballaigues, Switzerland). The working length was established when the tip of the file became visible at the apical foramen. The endodontic procedure and post space preparation were performed using 3.2x loupes (Orasoptic, Middleton, Wisconsin, USA)

Irrigation was performed with 10 mL 5% sodium hypochlorite (NaOCl; Niclor 5; Ognà, Muggiò, Italy) alternated with 2 mL 10% ethylenediaminetetraacetic acid (EDTA; Tubuliclean; Ognà), using a 2-mL syringe and 22-gauge needle. The canals were dried with paper points (size medium; Mynol; Curaden Healthcare, Milano, Italy). According to the continuous wave technique, specimens were filled with endodontic cement (Pulp Canal Sealer EWT; Kerr, Sybron, Romulus, MI, USA) and medium gutta-percha points (Inline; B.M. Dentale S.a.S., Torino, Italy) with DownPack (Hu-Friedy,) and Obtura II (Analytic Technologies, Redmond, WA, USA). After 1 week, the post space was prepared using size 1 and 2 Largo drills for removing the gutta-percha, and dedicated drills were used subsequently (Torpan; Dentsply Maillefer) in the first 10 mm of the root canal.

Post space cleaning was performed with 10% EDTA (Tubuliclean; 2.5 mL for 60 s) with a continuous brushing technique (Microbrush, Grafton, WI, USA). Then, specimens were randomly assigned to four groups according to the etching protocol. Group 1 was treated with 36% phosphoric acid gel for 30 s (Conditioner 36; Dentsply Maillefer); Group 2 was treated with 5 mL water solution of 36% phosphoric acid (Sigma Chemical, St. Louis, MO, USA) for 30 s (the first 15 s with continuous rinsing with an endodontic needle); Group 3 was treated with 5 mL water solution of 36% phosphoric acid (Sigma Chemical) applied for 30 s using an Endovac

(SybronEndo, Orange, CA, USA) with a macrocannula tip; Group 4 (control group) received no etching procedure.

Finally, the post spaces were irrigated with an air-water syringe spray for 30 s and with 5 mL physiological solution applied with an endodontic needle.

#### *Scanning electron microscopic evaluation*

Three teeth per group were split longitudinally and one half of each tooth was randomly chosen for scanning electron microscopic (SEM) evaluation. The halves were immersed in ascending ethanol concentration solutions, air-dried, mounted on metallic stubs (Ted Pella, Redding, CA, USA) with adhesive carbon disks (Ted Pella), and painted with colloidal quick-drying silver paint (Ted Pella). The specimens were finally sputter coated with gold-palladium in an E-5100 sputter coater (Polaron, Watford, UK), and observed under SEM (EVO MA10; Carl Zeiss SMP, NTD, Cambridge, UK) at 1000× and 5000× magnifications.

In total, 144 serial 1000× and 5000× images were numbered with a coding system to allow evaluation by two blinded independent investigators, according to the following criteria. The amount of debris observed was coded as 0, no debris particles; 1, few debris particles with maximum diameters < 20 µm; or 2, a large number of debris particles and/or presence of debris with diameters > 20 µm in any direction. The number of open dentinal tubules was coded as 0, all dentinal tubules open and lacking debris, smear layer, or sealer/gutta-percha remnants; 1, some dentinal tubules open, with a thin smear layer and a small amount of debris or sealer/gutta-percha remnants covering these openings; or 2, all dentinal tubules blocked by a thick smear layer with debris and sealer/gutta-percha remnants (Serafino *et al.* 2006).

For each tooth, the mean debris and dentinal tubule opening scores were calculated separately for three parts of the radicular dentine (coronal, middle, and apical thirds). Before beginning the evaluation, the two evaluators were trained in the use of the assessment criteria in a blinded manner. Intra-examiner reliability was assessed using the kappa (κ) test.



### *Push-out test*

Eight samples per group were prepared for the push-out test. Fibre posts (3M ESPE, St. Paul, MN, USA) were luted with a three-step etch-and-rinse dual-curing adhesive system (All Bond 3; Bisco, Schaumburg, IL, USA) and a dual-curing cement (Core-X Flow; Dentsply Italy, Rome, Italy), and light curing was performed for 40 s using a light-emitting diode lamp (Translux Power Blue; Heraeus Kulzer, Hanau, Germany) directly in contact with the posts. After cementation, samples were stored in physiological solution for 7 days at 37°C. Each sample was sectioned perpendicular to the post axis using a low-speed diamond saw (Micromet; Remet, Bologna, Italy) under water cooling to obtain six 1-mm-thick root slices, two each representing the coronal (C), middle (M), and apical (A) regions of the post space. The push-out test was performed by applying an axial load to the post at a crosshead speed of 0.5 mm/min using an instrom Machine I (model 10/D; Sintech, MTS, Canton, MA, USA). The most coronal region was turned downward (load direction, apical–coronal). The maximum failure load was recorded in Newtons (N) and converted into megapascals (MPa) by dividing it by the interfacial area of the post fragment, which corresponds to the bonded area (in millimetres squared), in accordance with Ferrari *et al.* (2009).

### *Statistical analysis*

The final debris and open tubule scores were compared statistically using the Kruskal–Wallis analysis of variance (ANOVA) by ranks, with a post hoc Dunn's test for multiple comparisons as needed.

Bond strength values were analysed for normal distribution using the Kolmogorov–Smirnov test and homogeneity of variance. A one-way ANOVA was used, with Tukey's test for post hoc comparisons as needed.

In all tests, the level of significance was set at  $p < 0.05$ . The data were analysed with the SPSS software (ver. 19.0 for Windows; SPSS Inc, Chicago, IL, USA).

## Results

Means, medians, and standard deviations (SDs) of open tubule and debris scores in each group are reported in Tables 1 and 2. The kappa test confirmed intra-examiner reliability ( $\kappa = 0.92$ ).

A multiple comparison test revealed that the etching protocol significantly influenced post space cleaning. Open tubule and debris scores were significantly lower in Group 2 (i.e. liquid water solution of phosphoric acid applied for 30 s) than in the other groups ( $p < 0.001$ ).

Mean bond strength values ( $\pm$ SD) obtained in the different groups are listed in Table 3. Within each group, bond strength varied significantly between apical and middle/coronal regions ( $p < 0.001$ ), except for Group 4. Push-out bond strength varied significantly according to the etching formulation, and was significantly higher in Group 2 than in the other groups ( $p = 0.021$ ).

## Discussion

Restoring a root filled tooth with a fibre post and a resin luting system is a common clinical procedure. The bonding of an etch-and-rinse adhesive to dentine is based on the micromechanical retention created by hybrid layer formation within the demineralised substrate and resin tag creation (Pashley *et al.* 2011). Consequently, post space cleaning and smear layer removal are critical procedures that can affect the bond strength of fibre posts (Boone *et al.* 2001, Breschi *et al.* 2009).

Within etch-and-rinse adhesive techniques, the most common etching agent is 32–37% phosphoric acid gel, however no agreement has been reached about the optimal etching time for radicular dentine (Albashaireh *et al.* 2009). In the present study, endodontic smear layer removal and fibre-post bond strength were significantly influenced by the phosphoric acid formulation and application method, so both hypotheses tested were accepted. The liquid phosphoric acid applied with either the conventional method through endodontic needle irrigation (Group 2) or a negative pressure technique (Group 3) using the EndoVac system yielded improved push-out strength compared to the gel formulation.

The findings showed that the application of 36% phosphoric acid gel for 30 s (Group 1) produced an irregular etching pattern, with an over-etched substrate in coronal post space portions, where the demineralisation zone became too deep for subsequently placed primers to penetrate completely (Fig. 1, Group 1, Coronal). Endodontic debris and remnants of the smear layer remained in the deeper portions of the post space (Fig. 1, Group 1, Middle and Apical), probably because of the high viscosity of the gel formulation. These findings suggest that effective contact times differ along the post space dentinal surface, thus the reduction of etching times is suggested (Albashaireh *et al.* 2009) and continuous movement of the phosphoric acid gel with a microbrush after application to renew it and improve contact time.

Moreover, rinsing of the gel formulation from the apical parts of the post space is difficult, resulting in greater amounts of small debris (silica remnants within the dentinal tubules, Fig 1, Group 1), which can influence bonding effectiveness over time (Potesta *et al.* 2008) impairing bonding infiltration. In contrast, the liquid formulation tested in this study was silica free and, thus, no silica remnants were found (Fig. 1, Group 2, 3).

Liquid phosphoric acid may have better wettability and lower surface energy compared with a gel formulation. The high viscosity of the gel does not allow the etchant to flow along the post space walls, thereby creating areas where the smear layer is not removed. Consequently, the capacity of the liquid formulation to reach the most difficult regions of the post space, due to its improved flow properties, could explain the higher bond strength values in comparison with those achieved using gel formulations.

Baharav *et al.* (1988) showed that liquid and gel phosphoric acid formulations had similar penetrations when they were combined with mechanical agitation, even when etchant application was tested on flat, visible enamel surfaces. Salas *et al.* (2011) showed that a liquid acid formulation yielded higher bond strength values to root dentine only in the apical regions of the samples. In the present study, improved bond strength with a liquid formulation was found in all post space regions.

These inconsistencies may be attributed to the different methods of applying the liquid phosphoric acid.

Negative pressure mediated through the Endovac vacuum could be clinically useful in controlling the overflow of liquid phosphoric acid during its application within the post space. However, the application of liquid phosphoric acid through the Endovac did not achieve proper post space cleaning or smear layer removal (Fig. 1, Group 3). These results are probably due to the dimensions of the macrocannula, which stays in contact with the radicular walls, preventing them from being properly etched by the liquid acid. Moreover, the liquid vacuum from the apical portion of the post space and its return through the macrocannula effectively reduced the contact time between the dentinal walls and etching agent. In contrast, the application of 36% liquid phosphoric acid with a 30s continuous rinse by endodontic needle created a different liquid pressure, improving the flow on the post space walls and achieving a ‘rinsing effect’ that facilitated debris removal. The significantly better results in Group 2 (i.e. no EndoVac was used and liquid phosphoric acid was applied with a syringe) in terms of smear layer removal and dentinal tubule opening, even in deeper areas of the post space, were probably due to two factors: more efficient penetration of the liquid formulation into the dentinal tubules than achieved with the more viscous gel formulation (Group 1), and the augmented effective contact time of the liquid in comparison with the Endovac system (Group 3).

This study also confirmed that obtaining a properly clean dentine surface is difficult in deeper areas with open dentinal tubules when no etching is performed (Fig. 1, Group 4, Apical). Consequently, because of insufficient smear layer removal, the control group (Group 4) had significantly lower bond strength values when compared with the other groups (Group 1,2,3). Several previous studies (Sen *et al.* 1995, Teixeira *et al.* 2005) have reported that the use of an EDTA/NaOCl combination obtained clean dentinal surfaces along the entire post space, but NaOCl causes the liberation of oxygen, which may inhibit the polymerisation of resinous bonding materials and induce collagen denaturation (Morris *et al.* 2001, Erdemir *et al.* 2004). In the light of these

findings, the acid conditioning of root dentine must be considered an essential step in achieving clean root canal walls and, consequently, reliable fibre-post bond strength (Zhang *et al.* 2008).

In summary, post space irrigation with liquid phosphoric acid effectively removed the smear layer and open dentinal tubules after post space preparation. This conditioning significantly benefitted fibre-post bond strength created by an etch-and-rinse adhesive system, as shown by the push-out test results. However, further studies of the effects of liquid phosphoric acid on coronal hard tissues must be performed to validate its clinical use.

## **Conclusions**

Within the limitations of this laboratory study, 36% liquid phosphoric acid applied with an endodontic needle yielded better endodontic smear layer removal and higher bond strength values when an etch-and-rinse adhesive system is used.

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