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# Effect of ethanol application on post-luting to intraradicular dentine

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## Abstract

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**Aim** To examine the effect of the application of an ethanol rinse before luting fibre posts to intraradicular dentine with etch-and-rinse adhesive systems by means of push-out bond strength evaluation and scanning electron microscopic (SEM) analysis.

**Methodology** Fibre posts were luted to single-canal premolars using Dual Link as a luting agent in combination with a three-step (All Bond 2) or a two-step (One Step Plus) etch-and-rinse adhesive system, which were applied as per manufacturers' instructions (control) or with the additional rinse of ethanol on acid-etched dentine prior to the bonding procedures (experimental). Bonded specimens were sectioned into 1-mm-thick slabs and subjected to push-out bond strength testing. In addition, specimens from each group were processed for SEM analysis. Data were analysed by Kruskal–Wallis followed by Dunn's *post hoc* test at $P = 0.05$.

**Results** Push-out bond strength of All Bond 2 was significantly increased if the adhesive was applied on ethanol-saturated dentine ($P < 0.05$), whilst no significant difference ($P > 0.05$) was detected amongst experimental and control groups for One Step Plus. Irrespective of the adhesive, the SEM analysis revealed good impregnation patterns when both bonding techniques were employed.

**Conclusions** The use of the additional ethanol rinse on acid-etched dentine revealed higher bond strength of All Bond 2 when compared with the control application procedure, used in combination with a resin-based cement to lute fibre posts into the dowel space. However, no bond strength improvements were detected using One Step Plus. Further investigations are needed to develop a clinically applicable ethanol/bonding/luting technique.

**Keywords:** dentine-bonding agents, ethanol application, post-luting, resin-based materials.

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## Introduction

The advent of dentine-bonding systems greatly modified the restorative procedures that could be applied to endodontically treated teeth (Schwartz & Fransman 2005), and various combinations of luting materials and dental adhesives have been proposed to bond fibre posts to intraradicular dentine (Foxton *et al.* 2003, 2005, Schwartz & Robbins 2004, Cury *et al.* 2006).

Dental adhesives are blends of hydrophilic and hydrophobic resin monomers, thus producing an amphiphilic compound that can permit bonding between tooth tissue (with hydrophilic properties) and the restorative resin-based materials (typically hydrophobic) (Nunes *et al.* 2001, Tay & Pashley 2003). Adhesive monomers are dissolved in solvents, such as water, acetone and ethanol that are needed to displace residual water from the demineralized dentine (Perdigão *et al.* 2000). Previous studies investigated the

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ability of the water-wet bonding technique to infiltrate dentine collagen matrices with hydrophilic resins (Kanca 1996, Tay *et al.* 1996, Van Meerbeek *et al.* 1998) and clarified that the process of impregnation is a fundamental pre-requisite for current dentine-bonding systems.

As hydrophobic resins have higher stiffness and greater stability in an aqueous environment, thus improving longevity of the adhesive interface when compared with hydrophilic ones (Ito *et al.* 2005, Malacarne *et al.* 2006, Breschi *et al.* 2008), it was recently proposed to replace residual water prior to the application of bonding agents with ethanol to coax hydrophobic monomers into the ethanol-saturated etched dentine (Nishitani *et al.* 2006, Pashley *et al.* 2007, Sadek *et al.* 2007, Tay *et al.* 2007). The goal of this technique is to dilute and displace all water present in the acid-etched dentine with ethanol, leaving the unsupported collagen fibrils in an ethanol-moist rather than water-filled environment to allow relatively hydrophobic resins to impregnate the substrate (Pashley *et al.* 2007). Despite the promising results of the ethanol-wet bonding technique when used on coronal dentine (Nishitani *et al.* 2006, Pashley *et al.* 2007, Sadek *et al.* 2007, Tay *et al.* 2007), little is known regarding this technique when applied to lute posts to intraradicular dentine.

Clinically, the ethanol-wet bonding technique may be considered more complicated because of the additional step or difficulty if applied into root canals. To simplify the application in the present investigation, the ethanol-wet bonding technique was not tested in combination with experimental resins as previously reported (Pashley *et al.* 2007), but with commercially available etch-and-rinse adhesive systems to replace residual water with the bonding and to fix the exposed collagen fibrils. The additional ethanol application step was previously proposed in endodontics to dehydrate the root canal before filling with gutta-percha and to completely dry the root canal to evaluate the sealer coverage (Wilcox & Wiemann 1995, Stevens *et al.* 2006).

Therefore, the purpose of this study was to investigate the effectiveness of two etch-and-rinse adhesive systems applied with or without the previous application of ethanol to intraradicular dentine in association with a resin-based cement by means of push-out bond strength test and micromorphological scanning electron microscopy (SEM) analysis. The hypothesis tested was that there is no difference in push-out bond strength and adhesive impregnation patterns to intraradicular dentine, compared with the ethanol wet-bonding application versus the water-wet bonding technique.

## Material and methods

### Specimen preparation

Twenty single-rooted and single-canal premolars extracted for orthodontic reasons were selected, after informed consent was obtained under a protocol approved by the University of Siena. Teeth were hand-scaled and stored in 1% chloramine T solution at 4 °C until use. Cleaning and shaping was performed using a crown-down preparation technique employing nickel-titanium rotary instruments (size S1, S2 and F3, Protaper; Dentsply Maillefer, Ballaigues, Switzerland) to size 30, 0.09 taper. Irrigation was performed with 5% sodium hypochlorite (NICLOR 5; Ogna, Maggio, Italy) after every change of file size. Root canals were dried using an air stream and absorbent paper points (Dentsply DeTrey, Konstanz, Germany) and filled with gutta-percha and a resin-based endodontic cement (AH-26, Dentsply DeTrey) using the lateral condensation technique. The coronal portion of filled roots was temporarily sealed with a glass–ionomer cement (Fuji VII; GC Corporation, Tokyo, Japan) and stored in 100% humidity in labelled containers for 24 h at 37 °C, awaiting testing.

After removing the temporary coronal seal, roots were prepared for the post-placement by removing the coronal gutta-percha using a low-speed universal drill recommended for RelyX Fiber Post size 2 (3M ESPE, St. Paul, MN, USA). A standardized 7 mm post-space was drilled in each root canal, and not less than 4 mm of apical seal was maintained. The specimens were randomly divided into four groups ($n = 5$). The tested adhesives were: All Bond 2 (ALL2; Bisco Inc, Itasca, IL, USA) and One Step Plus (OSP, Bisco Inc) and they were applied either in accordance with the conventional water-wet bonding technique (i.e. as per manufactures instructions, groups 1 and 3 respectively; control groups) or following the experimental ethanol-wet bonding technique in group 2 (ALL2) and 4 (OSP).

In detail the root canal walls were etched with 32% phosphoric acid (Bisco Inc) for 15 s using intracanal tips followed by water rinses with endodontic needles. Excess water was removed from the post-space using adsorbent paper points. In the experimental groups (i.e. group 2 and 4), the root canals were completely filled with 99.6% ethanol (Sigma-Aldrich, St. Louis, MO,

 

USA) for 1 min, then excess ethanol was gently removed and the adhesive systems were applied on ethanol-saturated dentine. Aside from ethanol application in the above-mentioned experimental specimens, the adhesive systems were applied following the manufacturers' instructions as in control groups (i.e. group 1 and 3).

The post-surfaces were cleaned with ethanol for 30 s and treated with a silane solution (Porcelain Primer; Bisco Inc) for 60 s using a disposable brush and air-dried for 5 s. A dual-cure composite luting cement (Dual Link; Bisco Inc) was mixed and placed into the post-cavity with a lentulo drill. Cement was placed on the post-surface and the post was seated with a slight finger pressure whilst excess of the resin cement was removed, maintaining a seal of the exposed dentine along the coronal part of the root.

Light-curing was performed using a conventional quartz–tungsten–halogen light (600 mW $cm^{-2}$ output; VIP; Bisco Inc) by placing the light tip perpendicularly through the post for 40 s. The bonded roots were then placed in individually labelled containers in 100% humidity for 24 h at 37 °C.

### Specimens preparation for push-out strength test

After 24 h, the root portions corresponding to the bonded-fibre posts were sectioned transversely into five to six 1-mm-thick serial slices using a low-speed Isomet (Isomet, Buehler, Lake Bluff, IL,USA) saw under water-cooling. The apical surfaces of the slices were marked with a dot using permanent black ink. The push-out test was performed on these slabs using a universal testing machine (Controls S.P.A., Milan, Italy) connected to a load cell, operating at a cross-head speed of 0.5 mm $min^{-1}$. The apical surface displaying the ink dot was placed facing the punch tip, ensuring that loading forces were introduced from an apical to coronal direction. Bond failure was manifested by the dislodgment of the fibre post from the root section. Push-out strength data were converted to Mega Pascal (MPa) by dividing the load in Newton by the bonded surface area ($S_L$) in $mm^2$, and $S_L$ was calculated as the lateral surface area of a truncated cone using the formula: $S_L = \pi(R + r)\left[(h^2 + (R - r))^2\right]^{0.5}$ where $R$ is the coronal post-radius, $r$ the apical post-radius and $h$ the thickness of the slice. The wider and the narrowest diameters of the post and the thickness of the slice were individually measured using a digital calliper with 0.01-mm accuracy. Failure modes were assessed with a stereomicroscope (Nikon type 102; Nikon Corp, Tokyo, Japan) at 30× magnification and classified as (A) adhesive between dentine and cementing agent, (M) mixed, (PA) adhesive between post and cementing agent or (C) cohesive in cementing agent failures.

### SEM sample preparation

Representative fractured slabs from each group were randomly selected for SEM analysis. Slices were smoothed with wet silicon-carbide papers of decreasing abrasiveness (up to 1200 grit). The specimens were demineralized with silica-free 32% phosphoric acid (Bisco Inc) for 20 s and subsequently immersed for 2 min in 2.5% NaOCl to remove the organic and mineral components of the dentine, then thoroughly rinsed with water and dehydrated with absolute ethanol to selectively analyse the hybrid layer and resin tag formation. The specimens were then mounted on aluminium stubs, sputter-coated with gold (Polaron Range SC7620; Quorum Technologies, Newhaven, UK) and observed under SEM (JSM 6060 LV; JEOL, Tokyo, Japan). Micrographs were taken at different magnifications to provide an overview of each area and to evaluate the type of micromorphological pattern of the representative specimens.

### Statistical analysis

The level of significance was set at $P < 0.05$. The normally distributed data (Kolmogorov–Smirnov test) with no homogeneous group variances (Levene test) were analysed with Kruskal–Wallis one-way analysis with push-out strength in MPa as dependant variable followed by Dunn's test for *post hoc* comparisons.

## Results

Means and SDs of push-out bond strength expressed in MPa and failure modes (%) are summarized in Table 1. Groups 3 and 4 (OSP) had a significantly higher bond strength than that observed in group 1 (ALL2 water-wet bonding technique); in addition, a statistically significant difference was detected between group 1 and 2 ($P < 0.05$), i.e. the ethanol-wet bonding technique increased the bond strength of ALL2. On the other hand, OSP showed no difference from experimental versus control group. No premature failures were reported either during the cutting procedure or during the testing procedure. SEM observations of the interfacial morphology (Fig. 1a–d) revealed a good interaction between the luting/bonding materials and the root

**Table 1** Mean push-out bond strength values* (SD) expressed in MPa and percentage of failure mode distribution recorded in the experimental groups

| Groups | Bond strength (SD) | Failure mode (%) A/M/PA/C |
|---|---|---|
| 1. Dual Link + All Bond 2 | 3.91 (2.0)[a] | 30/48/8/13 |
| 2. Dual Link + EtOH + All Bond 2 | 5.86 (3.07)[b] | 20/45/15/20 |
| 3. Dual Link + One Step Plus | 6.25 (4.37)[b] | 29/53/14/4 |
| 4. Dual Link + EtOH + One Step Plus | 6.25 (2.93)[b] | 15/55/14/15 |

*Values identified by same superscript letter are not significantly different ($P > 0.05$) by the Dunn's test.
Failure mode (in percentage): A = adhesive between dentin and cementing agent; M = mixed; PA = adhesive between post and cementing agent; C = cohesive in cementing agent.

canal dentine. Hybrid layer and resin tags formation was evident in all groups. Failure modes of tested adhesive interfaces showed that the majority of the bonds failed in a mixed mode.

## Discussion

Applying the ethanol rinse on intraradicular acid etched dentine before the bonding application, a significant difference between groups 1 and 2 (i.e. ALL2 control vs. experimental; $P < 0.05$) was found, whilst no difference between groups 3 and 4 was detected (i.e. OSP control vs. experimental). Thus, the study hypothesis can be partially rejected as differences in push-out bond strength to intraradicular dentine between ethanol application versus water-wet bonding techniques were detected only for ALL2. The SEM analysis revealed excellent impregnation and proper resin tags formation for both adhesives used following both strategies of impregnation.

It is well known that the ability of current etch-and-rinse adhesive systems to bond to the dentine substrate relies on micromechanical retention because of proper impregnation of the exposed dentine matrix, whilst no chemical bond is present (Van Meerbeek *et al.* 2003). The dentine matrix is mainly composed of type I collagen and noncollagenous proteins, generating an intricate three-dimensional network of highly cross-linked fibrils (Marshall *et al.* 1997, Breschi *et al.* 2008). As these fibrils are intrinsically wet because of their high affinity to water, their full impregnation with hydrophobic adhesive monomers represents an ideal goal almost impossible to achieve completely. For this reason, manufacturers recently blended hydrophilic monomers within the adhesive formulations so as to promote bonding impregnation in a reasonable clinical time. The inclusion of different solvents (i.e. water, ethanol or acetone) into the adhesive formulation additionally facilitates water substitution within the demineralized dentine matrix during adhesive infiltration. However, as more solvents and hydrophilic monomers are blended in the bonding agent, more resin blend is prone to degradation over time because of water sorption, resin leaching and other water-mediated ageing phenomena that weaken the polymer structure of the adhesive leading to the failure of the

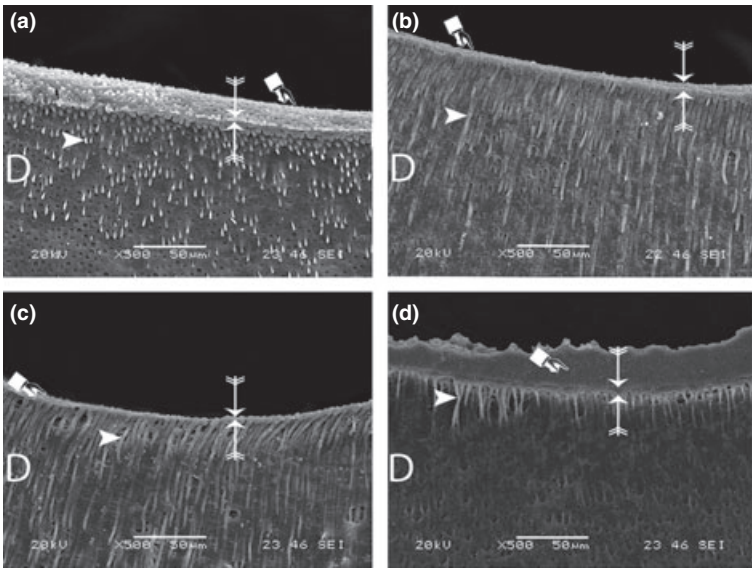


**Figure 1** Scanning electron microscopy images from push-out tested specimens. All groups demonstrated an evident hybrid layer (between arrows), and a high tendency to failure in a mixed mode. (a) Dual Link/All Bond 2 and (b) Dual Link/Ethanol/All Bond 2 using ethanol technique, (c) Dual Link/One Step Plus and (d) Dual Link/Ethanol/One Step Plus resulted in the formation of long resin tags (arrow heads) into the dentin (D) and a deep penetration of the bonding and showed a majority of mixed failures. Bonding interface with adhesive failures (groups 1, 2 and 3) and a cohesive failure (group 4) within the luting material (pointers) are shown (magnification 500×, bar 50 μm).

adhesive interface (Tay & Pashley 2003, Malacarne *et al.* 2006). For this reason, recent findings indicate that the more the adhesive blends are hydrophobic, the more the bonds are stable over time (Malacarne *et al.* 2006, Pashley *et al.* 2007). As hydrophobic monomers are immiscible with water, their ability to impregnate water-filled dentine matrices is minimal. On the other hand, as ethanol can replace water from dentine and dissolve hydrophobic monomers, the application of hydrophobic adhesives on ethanol-impregnated dentine matrices has been shown to be effective in improving the coronal dentine infiltration of hydrophobic adhesives (Nishitani *et al.*2006, Pashley *et al.* 2007, Sadek *et al.* 2007, Tay *et al.* 2007).

This study revealed that ethanol application can also be used to improve dentine bonds to intraradicular dentine using a commercially available adhesive system such as ALL2. The reasonable explanation of the improved bonding ability of ALL2 to ethanol-saturated intraradicular dentine is related to the ability of ethanol to accelerate the dentine water substitution rate thus reducing the intrinsic wetness of the root canal at the same time. Conversely, if OSP was employed no difference between ethanol- and wet-bonding technique was evident. A possible explanation of the lack of an affect of OSP to the ethanol pre-treatment could be related to the difference in adhesive composition as OSP contains acetone solvent in the formulation instead of ethanol (Table 2).

Interestingly, in ethanol-saturated dentine, the diameter of collagen fibrils is smaller than those in water-saturated dentine matrices, leaving larger interfibrillar spaces available for resin impregnation (Tay *et al.* 2007). As higher bond strength and wider interfibrillar spaces are correlated (Carvalho *et al.* 2003), the use of ethanol can increase bond strength (Eddleston *et al.* 2003). Thus, the type of solvent in the primer significantly affects resin/dentine bond strengths as ethanol could dehydrate and stiffen the matrix without allowing interpeptide H-bonds to collapse the dentine matrix (Carvalho *et al.* 2003). This may additionally explain why ALL2 performed better when applied to ethanol-saturated dentine. Proper resin infiltration provides a second important aspect to be discussed based on the results of the present study. Presumably, the positive benefits of ethanol pre-treatment could not be achieved with OSP because of the fact that the resin monomer in the

**Table 2** Chemical composition and application mode of the materials used in the study

| Dentin treatment | Groups | Bonding system | Luting cement | Application procedure |
|---|---|---|---|---|
| 32% phosphoric acid etching; rinse after 15 s; air-dry and paper points | 1 | All Bond 2<br>Primer A: acetone; ethanol; water na-n-tolylglycine glycidylmethacrylate<br>Primer B: acetone; ethanol; biphenyl dimethacrylate; photoinitiator<br>D/E Resin: bisphenol A diglycidylmethacrylate; urethane dimetyhacrylate; hydroxyethylmethacrylate; photoinitiator; amine activator | Dual Link<br>Bisphenol A diglycidyl methacrylate; triethyleneglycol dimethacrylate; glass filler; urethane dimethacrylate*<br>*Base only | All Bond 2 procedure: Mix Primer A + B (1 : 1). Apply in five coats. Air-dry after 5 s. Light-cure for 10 s<br>Dual Link procedure: Dispense cement into the root canal. Light-cure for 40 s |
| | 2 | All Bond 2/Ethanol technique | Dual Link | All Bond 2 as in group 1<br>Ethanol procedure: Apply 99.8% ethanol for 1 min<br>Dual Link as in group 1 |
| | 3 | One Step Plus<br>Biphenyl dimethacrylate; 2-hydroxyethyl methacrylate; Acetone; amine; photoinitiator; dental glass | Dual Link | One Step Plus procedure: Apply adhesive in two coats with agitating movements for 10 s. Air-dry after 10 s. Light-cure for 10 s<br>Dual Link as in group 1 |
| | 4 | One Step Plus/Ethanol technique | Dual Link | Apply One Step Plus as in group 3<br>Apply Ethanol as in group 1<br>Dual Link as in group 1 |

primer/adhesive mixture is dissolved in acetone rather than in ethanol.

It should also be considered that impregnation of the intraradicular dentine matrix is clinically challenging because of the limited access within the root canal space. The control of substrate humidity during the water-wet bonding technique and the proper application of the primer/adhesive agent on the demineralized intraradicular dentine are usually performed without appropriate visual control, thus both steps represent a critical clinical aspect. The use of ethanol application, although it increases the application time by adding an additional step, may significantly reduce the technique sensitivity related to operator's ability when ALL2 is used.

Recent studies also revealed that the adhesive blends containing water-based solvents could jeopardize the adhesive interface as a result of phase separation and/or inadequate solvent evaporation (Van Landuyt *et al.* 2005). Several studies have shown the formation of blisters/droplets along the intraradicular dentine (Chersoni *et al.* 2005, Bonfante *et al.* 2007). Nevertheless, the outcome of these studies may be misleading and substantiates the clinical relevance of adequate storage media maintenance and the proper solvent/water evaporation (Van Landuyt *et al.* 2007), this also raises the question regarding the clinical relevance of dental hydromechanics evaluation *ex vivo*. Recently, Ferrari *et al.* (2007) demonstrated in a laboratory study that the residual unevaporated water/solvent mixture entrapped within the adhesive interface might be the major factor in blisters/droplets formation, as there is a general consensus that the dowel space must be adequately dried prior to sealing endodontic posts (Hosoya *et al.* 2000). In addition, the use of ethanol may increase dentine fracture resistance as recently reported in coronal dentine (Nalla *et al.* 2006).

In addition, the effects of replacement of ethanol instead of water should be further investigated in long-term studies, as the ethanol application may improve bond stability over time. The removal of all possible water from the hybrid layer in a root filled tooth should, in fact, clearly reduce the presence of water over time thus reducing ageing phenomena of the adhesive interface occurring in the polymeric structure and to the collagen fibrils [*via* endogenous dentine matrix metalloproteinases (MMPs) collagenolytic activity], finally increasing bond stability over time (Pashley *et al.* 2004, Breschi *et al.* 2008). In particular, recent studies demonstrated that dentine endogenous MMPs are present within the dentine matrices and they can be activated in the presence of water after the application of dental adhesives as they are incompletely inactivated by phosphoric acid etching (Pashley *et al.* 2004, Mazzoni *et al.* 2006). Thus, the use of the ethanol may have potential benefits also for all adhesives in terms of bond strength stability over time.

## Conclusion

These preliminary results reveal that the ethanol-wet intraradicular dentine-bonding technique can improve the bond strength compared with traditional water-wet bonding technique, when a three-step ethanol-based bonding system is used. The importance of ethanol application to properly replace water from intraradicular dentine still requires further investigations, especially to clarify if this technique may reduce the effect of ageing and improve the stability of the bond, when used to lute fibre posts into the root canal.

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