



Effect of Chlorhexidine and Ethanol Application on Long-term Push-out Bond Strength of Fiber Posts to Dentin

¹Fabiana Mantovani Gomes França, ²Rafaela Crystyan Vaneli, ³Carolina de Melo Conti
⁴Roberta Tarkany Basting, ⁵Flávia Lucisano Botelho do Amaral, ⁶Cecília Pedroso Turssi

ABSTRACT

Aim: The aim of this study was to evaluate the effect of chlorhexidine and ethanol application on the push-out bond strength and bond durability of fiber posts cemented with an etch-and-rinse adhesive system/resin cement to intraradicular dentin.

Materials and methods: Fifty-four bovine roots were shaped for the cementation of a fiberglass post and received the application of 37% phosphoric acid. They were then randomly divided into three groups, according to the type of dentin treatment (n = 18) performed: no treatment (control group), 100% ethanol, or 2% chlorhexidine. Next, the adhesive system (Adper Scotch Bond Multipurpose Plus, 3M ESPE) was applied to the dentin, according to the manufacturer's instructions. Glass fiber posts were cemented with dual resin cement (Rely X ARC, 3M ESPE). After 48 hours, the specimens were serially sectioned for push-out test analysis, providing two slices from each root third (cervical, medium and apical), one of which was tested immediately and the other stored in distilled water for 180 days. The data were analyzed with three-way analysis of variance (ANOVA) for repeated measures and Tukey's test at a 5% significance level.

Results: Intraradicular treatment with chlorhexidine yielded the highest bond strength means, followed by ethanol treatment. The control group presented the lowest bond strength means. Water storage exerted no effect on bond strength values.

Conclusion: Both chlorhexidine and ethanol improved push-out bond strength to intraradicular dentin, with the former providing the best results, regardless of the storage time.

Clinical significance: The application of 2% chlorhexidine or 100% ethanol may be an important step that can be taken to enhance bond strength of fiber posts to intraradicular dentin, when dual resin cements are used.

Keywords: Laboratory research, MMP inhibitors, Resin cements, Surface treatment.

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INTRODUCTION

Fiber reinforced posts are indicated for the restoration of endodontically treated teeth with excessive loss of coronal tooth structure.¹⁻³ These posts have a modulus of elasticity similar to that of dentin.^{2,4} Their advantages include—performing cementations without causing friction on the root canal walls, reducing vertical fractures in the root and distributing occlusal forces more uniformly. As a consequence, fractures occur less frequently and in a more favorable manner, meaning that fractures are repairable in most of the cases.²

However, many factors may affect the bond strength between the fiber post, the cement and the root canal, such as dentin hydration, type of conditioning agent and the cement used as well as length, conformation and diameter of intraradicular retainers, and the different anatomical features, density and orientation of dentinal tubules in different depths of root canal.^{2,3,5,6} Additional factors may also represent a significant challenge in the cementation procedures, such as the difficulty of light transmission to the apical portion of the root, the limited direct observation inside the root canal, the difficulty in controlling moisture and the application of adhesive

¹⁻⁶Department of Restorative Dentistry, São Leopoldo Mandic Institute and Dental Research Center, Campinas, São Paulo Brazil

Corresponding Author: Fabiana Mantovani Gomes França Professor, São Leopoldo Mandic Institute and Dental Research Center, José Rocha Junqueira, 13, Swift, Campinas-SP 13045-755 Brazil, e-mail: biagomes@yahoo.com

systems.⁷ These factors may impair immediate bond strength and durability over time.²

The use of ethanol as an auxiliary method of conditioning intraradicular dentin has been proposed for controlling dentinal moisture and enhancing bond durability.⁸ This technique consists of applying ethanol before applying a two-step self-etching or a three-step etch-and-rinse adhesive system, so that the remaining water may be replaced with ethanol.^{3,8-10} Monomers are hydrophobic, and may penetrate more deeply in ethanol-saturated than in water-saturated coronal dentin.^{3,9,10}

The application of ethanol to root walls has already been described in endodontic therapies for dehydrating root canals before filling them with sealer material.^{8,11,12} Nonetheless, little evidence has described the relation between ethanol and bond strength to intraradicular dentin.⁸

Furthermore, bond durability in dentin has been related to collagen matrix hydrolysis in hybrid layers.¹³ Enzymes of the matrix metalloproteinase (MMP) family are involved in the breakdown of extracellular matrix components.^{14,15} This is important in the context of bond durability, because these MMPs can be activated in low-pH environments.¹³ Adhesive systems (resulting from their etch-and-rinse and self-etch approaches) probably contribute to the activation process due to their acidity.¹³ Thus, exposed collagen fibrils (eventually demineralized and not subsequently protected by resin monomers) are susceptible to hydrolytic and MMP-mediated degradation.¹⁶

In this context, techniques and protocols have been developed to optimize hybrid layer maintenance and minimize MMP action, as exemplified by the use of chlorhexidine.¹⁷ Because of the MMP-inhibitory potential of chlorhexidine, its application to dentin after acid-conditioning and before application of adhesives may impair or retard the degradation of exposed collagen fibrils at the base of the hybrid layer.^{18,19}

Bearing in mind the importance of improving bonding durability either by enhancing monomer penetration or by inhibiting MMP activity, the aim of this study was to evaluate the influence of ethanol and chlorhexidine application on the bond strength of an etch-and-rinse adhesive to intraradicular dentin. The null hypothesis was that the application of neither ethanol nor chlorhexidine would influence bond strength to intraradicular dentin.

MATERIALS AND METHODS

Experimental Design

The factors under evaluation were:

- *Intraradicular dentin treatment, on three levels:* (1) phosphoric acid associated with an etch-and-rinse

adhesive system (Scotchbond Multiuso 3M ESPE); (2) phosphoric acid and ethanol associated with an etch-and-rinse adhesive (Scotchbond Multiuso 3M ESPE); (3) phosphoric acid and chlorhexidine associated with an etch-and-rinse adhesive system (Scotchbond Multiuso 3M ESPE).

- *Storage time, on two levels:* Forty-eight hours and 180 days.
- *Thirds:* Cervical, medium and apical (obtained from the same experimental unit).

Each experimental group consisted of 18 bovine roots restored with glass fiber posts (each considered as an experimental unit). The response variable was push-out bond strength testing, expressed in MPa.

Specimen Preparation

Fifty-four inferior bovine incisors stored in 0.1% thymol solution were used. Teeth were submitted to debriding with scalpel blades and periodontal curettes. The crowns were then removed at the cemento-enamel junction, using a diamond saw (Microdont Ltda., São Paulo, SP, Brazil). Roots had to have a length of at least 17 mm to be included otherwise, they were discarded. The diameters of the mesiodistal and buccolingual canals were then measured with a digital caliper (Mitutoyo Sul Americana, Suzano, SP, Brazil), so that only canals with similar dimensions were used. Roots were randomly divided into three groups (n = 18): Group 1—application of an adhesive system according to the manufacturers' instructions; group 2—application of 37% phosphoric acid, followed by ethanol and an adhesive system; group 3—application of 2% chlorhexidine, followed by ethanol and an adhesive system (Table 1).

The first 9 mm of the canal was shaped with Largo Peeso Reamers (Dentsply, Milford DE, USA) at increasing diameters until reaching #4. The canal conducts were then rinsed and aspirated through an aspiration cannula, keeping dentin moist. The conducts were conditioned with 37% phosphoric acid (Condac 37, FGM Produtos Odontológicos, Joinville, SC, Brazil) for 15 seconds, and rinsed for 1 minute. The conducts were then dried gently with absorbent paper points #80 (Dentsply/Maillefer, Tulsa, Oklahoma, USA). At this time, the conducts of group 2 received an application of 100% ethanol (Chemco Indústria e Comércio Ltda., Hortolândia, SP, Brazil), and the conducts of group 3 received the application of a 2% chlorhexidine solution (FGM Joinville, SC Brazil), according to Table 1.

Next, the etch-and-rinse adhesive system was applied (Adper Scotch Bond Multi-Use Plus 3M ESPE, St Paul, MN, USA). The excess was removed with paper points

Table 1: Commercial name, manufacturer, composition, application steps of materials used

<i>Commercial name, manufacturer (batch number)</i>	<i>Composition</i>	<i>Application steps</i>
Chlorhexidine 2% FGM Joinville, SC, Brazil (240811)	2% digluconate chlorhexidine	After acid conditioning, 2% chlorhexidine solution was applied to root canal for 60 seconds with a disposable syringe. Next, the excess was removed with absorbent paper points, keeping dentin moist.
Ethanol CHEMCO (24631)	100% ethylic alcohol	After acid conditioning, ethanol was applied to the root canal for 60 seconds with a disposable syringe (Carvalho et al, 2009). Next, the excess was removed with absorbent paper points, keeping dentin moist.
Adper Scotch Bond Multipurpose Plus 3M ESPE (1109000164)	Primer—HEMA, polyalkenoic acid polymer, water Adhesive—BisGMA, HEMA, photoinitiator.	Primer was applied to etched dentin with a disposable brush. The excess was removed with absorbent paper points. The adhesive was applied with a disposable brush. The excess was removed with absorbent paper points. Light-curing was performed for 60 seconds (20 seconds on each surface).
Rely X ARC 3M ESPE (1101800550; 1101800501)	Paste A: BisGMA, TEGDMA, silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol. Paste B: Silane treated ceramic, TEGDMA, BisGMA, silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide	Cement was mixed for 10 seconds and applied to the root canal with a Centrix syringe. Light-curing was performed for 40 seconds on each of the four sides of the root.
Reforpost no. 2 (Angelus, Londrina, PR, Brazil) (18826, 18990, 19586, 18224, 19469)	Glass fiber, Epoxy resin with a 1.25 mm and cone apex	Posts (1.25 diameter, parallel shaped/ conical apex) received the application of 37% phosphoric acid for 30 seconds and were rinsed for 1 minute. Posts were air-dried and a silane agent was applied for 1 minute. A layer of adhesive was applied on the posts and light-curing was performed for 20 seconds. During cementation, the post was placed inside the root canal and pressed for 10 seconds; the excess of cement removed with a spatula.
Phosphoric acid Condac (FGM)	37% phosphoric acid	Acid was applied to dentin for 15 seconds, and rinsed abundantly for 60 seconds. Dentin was dried with absorbent paper points, keeping dentin moist.
Silano Angelus (15939)	Silane and ethanol	Applied on post for 1 minute.

BisGMA: Bisphenol A glycidyl methacrylate; TEGDMA: Triethylene glycol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate

(Table 1), and light polymerization was performed with a halogen light-curing device (Demetron Kerr, Orange, CA, USA).

Glass fiber posts (Reforpost no. 2/ Angelus, Londrina, PR, Brazil) were cleaned by applying 37% phosphoric acid for 30 seconds, and then rinsed for 1 minute. Posts were air-dried, and a silane-coupling agent (Silano/ Angelus, Londrina, PR, Brazil) was applied for 1 minute. Afterwards, a layer of adhesive (Adper Scotch Bond

Multi-Use Plus 3M ESPE, St. Paul, MN, USA) was applied on posts, and light-curing was performed for 20 seconds (Table 1).

The dual resin cement (Rely X ARC 3M ESPE, St Paul, MN, USA) was inserted into the root canal with a Centrix syringe (DFL, Jacarepaguá, RJ, Brazil). A glass fiber post was then placed inside the root canal and pressed for 10 seconds, the excess cement was removed. Finally, light-curing was performed for 20 seconds on each of the

four sides of the root, totaling 160 seconds. The output of the light-curing unit was measured periodically by a radiometer (Newdent Equipamentos Ltda, Ribeirão Preto, SP, Brazil), considering a minimum irradiance of 450 mW/cm².

Roots with cemented posts were stored for 48 hours in distilled water at 37°C. After this, the portions of the roots corresponding to the bonded fiber post were transversely sectioned into 1 mm thick serial slices, using a slow speed diamond saw (Extec Corp), under water irrigation. Two slices of each root third (cervical, medium, apical) were obtained, one of which was tested immediately and the other of which was stored in distilled water, for 180 days. Water was replaced once a week.

Push-out Test

After 48 hours or 180 days of water storage, sections were fixed to a stainless steel device attached to a universal testing machine (EMIC, São José dos Pinhais—PR, Brazil). A 1.0 mm diameter punch tip was fixed to the load cell (50 KN) and then positioned to the center of the post. The apical surface of each slice was placed facing the punch tip, ensuring that loading forces were distributed in an apical to coronal direction. Push-out bond tests were performed with a crosshead speed of 0.5 mm/min. Push-out strength data were converted to MPa by dividing the load in Newton by the bonded surface area (BSA) in mm². Surface area was calculated with the surface area of the cylinder formula ($BSA = 2\pi R * h$, being $\pi = 3.1416$; R = radius; h = slice height).

Failure Mode

After the end of push-out tests, each slice was observed under a stereomicroscope (EK3ST, CQA, São Paulo, Brazil) at 40× magnification, to assess the failure modes, which were classified as: (1) adhesive between post and cement; (2) adhesive between dentin and cement; (3) mixed failure; (4) cohesive failure (failure in dentin, cement or post material).

STATISTICAL ANALYSIS

The level of significance was set at 0.05. Since the data did not meet the assumption of homogeneity of variance (Levene test) or normal distribution (Shapiro Wilk test),

square root transformation was conducted. The data were analyzed with three-way analysis of variance (ANOVA) for repeated measures and Tukey’s test.

RESULTS

The three-way ANOVA for repeated measures revealed that there were significant differences between the intraradicular treatments ($p < 0.001$), and that the p-value was borderline for the effect of storage time ($p = 0.06$). There was no significant effect among root thirds ($p = 0.88$) or treatment x time ($p = 0.266$), treatment x root third ($p = 0.771$), time x third ($p = 0.213$) and treatment x time x third ($p = 0.531$).

Intraradicular treatment with chlorhexidine yielded the highest bond strength means, followed by ethanol treatment. The control group presented the lowest bond strength means ($p < 0.001$). There was a trend of diminishing bond strength after 180 days of water storage ($p = 0.06$) (Table 2).

Figure 1 presents the failure mode percentages. The control group had a high frequency of adhesive failures between dentin and cement (type 2). Failures in the chlorhexidine-treated group were predominantly adhesive, between post and cement (type 1), regardless of water storage time. After 48 hours, the group treated with ethanol presented failures between post and cement (type 1), dentin and cement (type 2), and mixed failures

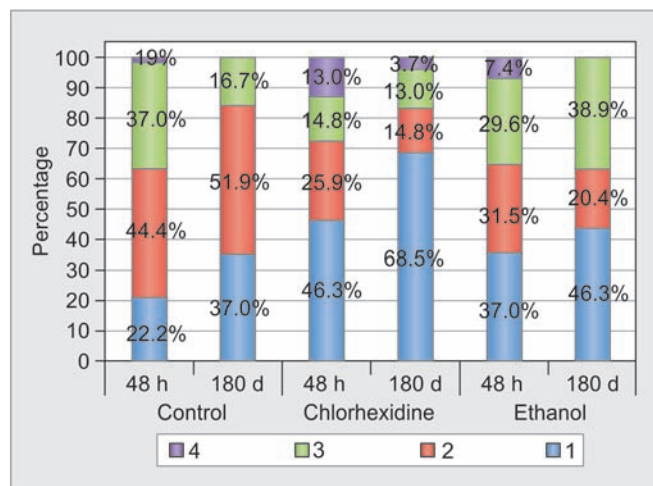


Fig. 1: Frequency (in percentage) of failure modes, according to treatments and water storage time. Type 1: Adhesive between post and cement; Type 2: Adhesive between dentin and cement; Type 3: Mixed failure; Type 4: Cohesive failure

Table 2: Bond strength means, grand mean (mpa) and standard deviations according to treatments and root thirds

	48 hours			180 days			Grand mean
	Coronal	Middle	Apical	Coronal	Middle	Apical	
Control	15.17 (9.46)	14.79 (9.82)	15.35 (10.75)	11.08 (6.83)	8.96 (10.73)	11.79 (20.26)	12.86 (12.03) C
Chlorhexidine	32.16 (21.73)	33.1 (24.84)	35.38 (16.4)	22.07 (13.16)	25.98 (14.82)	29.33 (30.90)	29.67 (21.22) A
Ethanol	23.06 (13.12)	22.5 (16.44)	29.5 (13.05)	15.86 (10.14)	12.53 (8.58)	9.23 (7.73)	18.78 (13.53) B
Grand mean		24.56 (17.35)			16.32 (16.72)		



(type 3). After 180 days of water storage, most of the failures occurred between post and cement (type 1) and or were mixed (type 3).

DISCUSSION

One factor that is crucial for adequate clinical performance of glass fiber posts is the maintenance of the bond between post, cement and intraradicular dentin.⁶ In this context, ethanol and chlorhexidine applications associated with etch-and-rinse adhesives, have been studied with the aim of improving the bond durability of adhesive systems to coronal dentin.¹⁹⁻²³

Ethanol application is performed to diminish dentin permeability and to produce a more hydrophobic hybrid layer.⁸⁻¹⁰ Chlorhexidine has been applied because of its metalloproteinase inhibitory potential to retard the degradation process that occurs in adhesive-based restorations.^{24,25}

The results of the present study showed that ethanol application enhanced bond strength as compared to the control group. Thus, the null hypothesis was rejected. In this group, the fracture pattern occurred predominantly between the post and the cement (type 1); whereas in the control group, most of the fractures were adhesive, between the dentin and the cement (type 2). This indicates that ethanol application may have benefited bond strength, in so far as the adhesive interface was maintained in most of the cases. Ethanol is miscible in water, thus allowing the residual water molecule remaining in dentin tubules to be substituted by ethanol. Since hydrophobic monomers have more affinity with ethanol, they can penetrate more deeply.²⁶ Moreover, the collagen fibril diameter in ethanol-saturated dentin is smaller than in water-saturated dentin, leaving more free space for the adhesive agent and for cement impregnation.⁸ As a consequence, bond strength may be enhanced.

Furthermore, chlorhexidine application to intraradicular dentin was found to improve bond strength, as compared to ethanol application and with the control group. The failure mode in the chlorhexidine-treated group confirms this result, considering that most of the failures were adhesive, between the post and the cement (type 1), even after 180 days of water storage. Therefore, it may be posited that the hybrid layer formed in intraradicular dentin was maintained as a result of chlorhexidine application.

Other studies also demonstrated that chlorhexidine application did not interfere in the immediate bond strength of etch-and-rinse adhesives.^{19,22} Nonetheless, chlorhexidine application has not been commonly

reported as a procedure that improves immediate bond strength to intraradicular or to coronal dentin.^{22,25,26} In the present study, however, chlorhexidine application improved immediate bond strength, in agreement with the results of Lindblad et al.²⁷ The mechanism by which chlorhexidine may enhance initial bond strength to dentin has yet not been elucidated, but previous researches have demonstrated that chlorhexidine pretreated dentin exhibited normal structural integrity of the collagen network.^{19,22}

Although there was no statistical difference in the results according to water storage time (48 hours × 180 days), bond strength values tended to decrease over time ($p = 0.06$). This may be attributed to several factors that may affect adhesion to intraradicular dentin, such as difficulties in light penetration and a high configuration factor inside the root canal.^{28,29} These factors may interfere in the stress generated at the adhesive interface during resin polymerization shrinkage.^{30,31} Furthermore, degradation of the post/cement bond may occur as a consequence of the cementing agent/adhesive system type used, post surface pretreatment, or suboptimal bonding between the cementing agent and the post surface. It is known that prefabricated posts have a high-reticulated polymeric matrix between their fibrils. This may be responsible for the reduction in adhesion between posts and cement. It could be that the adhesive monomers do not penetrate into such a polymeric matrix, thus hampering additional polymerization.³²

Hydrolytic degradation of the resinous cement may be confirmed in the fracture patterns observed. The failures observed in the ethanol and the chlorhexidine groups were mostly adhesive, between post and cement (type 1), or mixed (type 3). This indicates that the adhesive interface between cement and dentin was preserved, at least in part.^{8,10,25,33} In regard to the control group, most of the fractures were seen to have occurred between the cement and the dentin (type 2).

Since, no statistical differences were observed between the root thirds, one could posit that the increase in dentin surface area may be responsible for the reinforcement of bond strength after acid-conditioning.³⁴ Furthermore, the use of a centrix appliance allows the resinous cement to be inserted more homogeneously, thus reducing the formation of air bubbles.³⁵ In addition, tooth crowns were removed, and the extremity of the light-curing unit tip was positioned close to the cervical region of the root during the procedure. These factors may explain the similarity in bond strength among the root thirds.

According to the results of this study, the application of 2% chlorhexidine or 100% ethanol may be an important

clinical step that may be taken to enhance bond strength of fiber posts to intraradicular dentin, when dual resin cements are used.

CONCLUSION

Both chlorhexidine and ethanol improved push-out bond strength to intraradicular dentin, with the former providing the best results, regardless of the storage time.

CLINICAL SIGNIFICANCE

The application of 2% chlorhexidine or 100% ethanol may be an important step that can be taken to enhance bond strength of fiber posts to intraradicular dentin, when dual resin cements are used.

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