

Original Article

Effect of different post-space pretreatments on fiber post bonding to root dentine

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ABSTRACT

Background: Before the resin bond endodontic post-cementation, various solutions are used to improve bond strength of these posts to root dentine. The purpose of this study was to evaluate the effect of the different pre-treatments (ethanol, ethylene de amine tetra acetic acid [EDTA] and EDTA + ethanol) on the bond strength of adhesively luted tooth colored fiber post.

Materials and Methods: Forty-eight human anterior teeth (incisor and canine) were selected in this experimental study. The species were endodontically treated, and the post-space was prepared. The species were randomly divided into four groups of 12 teeth in each and were treated as follows: (i) 0.1 M EDTA, (ii) 99.6% ethanol, (iii) 0.1 M EDTA followed by 99.6% ethanol and (iv) without any treatment (control). A push-out test was carried out after 24 h. Each tooth was cut into three 1 mm-thick sections. Fracture type was assessed using stereomicroscope. The data were analyzed by one way-ANOVA followed by Scheffe *post-hoc* test at $P = 0.05$.

Results: Almost, in EDTA and (EDTA + ethanol) every sample failed at the interface between post and luting cement. Predominant failure mode in ethanol and control group was mixed failure. The surface treatment methods did not have any significant effect ($P > 0.05$) on any group but EDTA group had the highest bond strength.

Conclusion: Surface treatment with (ethanol, EDTA, and EDTA + ethanol) does not significantly appear to be effective in improving the bond strength of fiber posts into root dentine. The highest bond strength was observed in EDTA group and the lowest in the control group.

Key Words: Bond strength, ethanol, ethylene de amine tetra acetic acid, fiber post

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INTRODUCTION

Endodontically treated teeth were more prone to fracture than vital teeth because of loss of tooth structure and structural integrity as a consequence of decays, change in dentine, and access preparation.^[1,2] The use of intraradicular post is often essential for

rebuilding these teeth.^[3] Fibers reinforced post have been supposed to restore root-filled teeth, mainly regarding favorable mechanical properties.^[3-5]

The recent trend has been towards the self-etch primer (SEP) system^[4,6] because of easily manageable clinical procedure related to them.^[6] SEP modified the smear layer that contributed to forming complex hybrid layer.

Crown smear layer is a reflection of the composition of dentine matrix. However, root treatment smear layer contains organic and inorganic materials, microorganism, and necrotic material.^[7] The preparation of post-space produces new smear layer in root filled teeth. The penetration and chemical reaction of adhesive

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system that uses in cementation of fiber post may be compromised by the cumulative effect of these two layers.^[3] The effective infiltration of self-etch system in this smear layer still remains a concern.^[8]

Most tooth failures with restored fiber post are associated with root/cement/resin debonding^[4,5] due to failure of the weak point in interface (resin-dentine).^[3,5] In order to decrease, the risk of debonding, several dentine surface treatments for example, chlorhexidin, ethanol, ethylene de amine tetra acetic acid (EDTA), sodium hypochlorite (NaOCl), and ethylene acetate were suggested for optimizing bond to root dentine and increasing the retention of the fiber post.^[3,4,9-15]

EDTA is a di-valant cationic material used to chelat calcium ions as part of an endodontic therapy procedure and in the removal of smear layer in restorative bonding dental procedure.^[16] EDTA conditioning of crown dentine before application of SEP increases bond strength and forms homogenous hybrid layer.^[16-19] EDTA conditioning has much more ca remains compare to acid phosphoric conditioning so the functional monomer of SEP may have better chemical reaction.^[20]

Several studies have been performed on the efficacy of wet ethanol bonding system in root. However, their consequences were different.^[3,4,10-15] The time of ethanol application is reported to be 3 min or 4 min.^[11,14] These application times are not empirical in clinic. Sauro *et al.*, (2011) reported 1 min application time for ethanol in crown dentine. They conditioned dentine prior wet ethanol bonding by EDTA.^[20] Bear in mind that the bonding durability of adhesive cement is critical for the endurance posts used as a retainer for indirect restoration. Thus, increasing the durability of dentine-resin bond is a hot topic in restorative dentistry. The application of matrix metalloproteases (chlorhexidin)^[4,9] or ethanol wet bonding^[4,21] and EDTA increased the durability of dentine-resin bond.^[17,20] To authors' knowledge, low concentration of EDTA and the combination therapy of wet ethanol system and EDTA have not been tested in root dentine to regain a better bond strength. The aim of this study was to evaluate the effect of the different pre-treatments (EDTA, ethanol, and EDTA followed by ethanol) on bond strength of fiber post to root dentine. The null was that the pre-treatment of post-space do not significantly affect the bond strength.

MATERIALS AND METHODS

Specimen preparation

Forty-eighth upper central incisors and canine teeth, extracted for periodontal disease from subjects between 30 years and 60 years old were entered in this experimental study. The inclusion criteria were: Straight root, absence of root caries and/or root crack and/or previous restoration and endodontic treatment, root length at least 13 mm. Teeth were handscaled and store in 0.5% chloramin T solution at 4°C to pervert bacterial growth and used them within 3 months following extraction. Each tooth was decrowned 1 mm incisally from the cement-enamel junction, using the diamond disk (Degussa Dental, Hanau, Germany) in a slow speed hand piece, under water cooling.

The preparation of all root canals was carried out by one trained operator. The root canals of the teeth were instrumented by a working length of 1 mm from the apex. Cleaning and shaping were carried out using nickel-titanium rotary instrumentation (size S1, S2, and F3, Protaper; Dentsply Mailfer, Ballaiguse, Switzerland). The root canal was irrigated between instrumentation with 1 ml 0.2% chlorhexidine and finally dried using absorbent paper (Ariadent, Iran). The final irrigation was distilled water. Each root canal was obturated using lateral compaction with gutta-percha points (Ariadent, Iran) and AH Pus sealer (Dentsply Mailfer, Ballaiguse, Switzerland). The access cavities of the teeth were temporized with Cavite (Ariadent, Iran). The roots were stored in 100% moisture at 37°C for 24 h.

After storage, the temporary restorations were removed and also a part of sealing material was removed with size of two Pisso reamer (Mani Inc, Tochigi, Japan). The post space was readied with no. 2 DT light drill from DT light-post system (Bisco, Schaumburg, IL) to 9 mm deep, as measured from the buccal surface of root, leaving behind at least 4 mm gutta-percha. The post that was selected in this study was no. 2 DT light-post system (Bisco, Schaumburg, IL). The root was rinse with 5 ml distilled water to remove the debris and dried it with paper point (Ariadent, Iran). Any remaining gutta-percha in the walls of the root was assessed by radiography. The specimens were randomly distributed into four groups: (G1) 0.1 M EDTA (Na₂EDTA, Panreac, Spain), (G2) 99.6% Ethanol (Merck, Germany, 99.6% D), (G3) 0.1 M EDTA that followed by 99.6% Ethanol, (G4) without any pre-treatment regime [Table1].

Table 1: Experimental groups and mode of application

Group	Mode/steps of application
EDTA (G1) (Panreac)	Root was filled with 1 ml of 0.1 M EDTA for 1 min, irrigate by distilled water, dried the space with absorbent paper until the last paper point was dry
Ethanol (G2) (Merck)	Root was filled with 99.6% ethanol for 1 min, the excess ethanol was gently removed
EDTA + ethanol (G3)	Root was filled with 1 ml of 0.1 M EDTA for 1 min, irrigate by distilled water, the dried space with absorbent paper until the last paper point was dry, root was filled with 99.6% ethanol for 1 min, the excess ethanol was gently removed
Control (G4)	Root did not receive any pretreatment solution, dried the with absorbent paper until the last paper point was dry

EDTA: Ethylene de amine tetra acetic acid

Table 2: Mean and standard deviation (MPa) of bond strength following push out testing

Group	nV*	nF**	nR***	MPa	SD
G1	3	1	32	41.56	13.86
G2	0	1	34	35.95	17.11
G3	4	4	28	35.55	21.94
G4	0	4	32	35.29	20.25

*nV: Specimen had a large void in the fiber post and root dentine interfaces;

nF: Premature failures during the cutting or the testing procedures; *nR:

Remained specimen; MPa: Mean of bond strength following push out testing; SD: Standard deviation

The root surface in G1 and G2 was completely dried in G2 and G3 contained ethanol (Wet-ethanol bonding system).^[13] The surface of the post was freshened with ethanol 99.6% for 30 s and dried for 10 s. They were cemented with dual-cured resin system (Panavia F 2.0; Kuraray Co Ltd.) according to the manufacturer's instructions.^[3] Subsequently, the fiber post was covered with cement and was applied in the canal using lentulo drill (Mani Inc, Tochogi, Japan), seated under figure pressure, and the excess cement was removed with a brush. The cement was light cured for 40 s (20 s from buccal and 20 s for lingual aspects) with LAVA LED unit (Ultradent Products, Inc. Ultradent Products Inc., South Jordan, UT, USA) (output 1000 Mw/cm²). The light tip was placed in direct contact with buccal and lingual aspects of the post. The teeth were stored in 100% at 37°C for 24 h.^[3,15]

Push-out test

Each root was cut with a cutting device and diamond covered disk (Mecatome, Presi, France) to make

four 1 mm-thick slices. The first slice was not used. Finally, we had one piece for each root area (coronal, middle, apical). The apical face of each slice was marked with permanent color ink. The thickness of each slice was checked by digital caliper.

The customized push out device^[3] was used to carry out push-out test. The acrylic resin mold had a 2.5 mm space in the center root section attached holes during the push-out test. A comprehensive load was applied on the apical aspect of the slice via universal testing machine (Zwick, Ulm, Germany) equipped with a 1-mm diameter cylindrical plunger. Loading was performed at a 0.5 mm/min until failure. The conversion of the bond strength was done by dividing the load of failure in Newton (F) by the interface area of the root slice (A). The interface area was calculated by AutoCAD software 2006 (Autodesk Inc., San Rafael, CA, USA) based on scan photos of all slices before doing push out test.

Mode of failure

The fractured slices were assessed at $\times 30$ magnification under stereomicroscope (Nikon type 102, Nikon Crop, Tokyo, Japan) to classify: (1) Adhesive failure between post & resin cement(no cement around the post); (2) Adhesive failure between dentine & resin cement (post enveloped by resin cement); (3) Mixed, with resin cement covering 0% to 50% of the post diameter; (4) Mixed, with resin cement covering 50% to 100% of the post diameter; (5) Cohesive in post; (6) Cohesive in dentine. The randomly selected slices were reexamined using scanning electron microscopy (SEM).

Statistical analysis

The mean and standard deviations of bond strength were calculated and the data were analyzed using the One-way ANOVA and Scheffe *post-hoc* test. The failure mode data were analyzed using the Chi-square test. The accepted level of errors was set at $P < 0.05$.

RESULTS

The mean bond strength values of the fiber post to root dentine for the experimental and control groups are summarized in Table 2. The highest and the lowest bond strength mean values were found respectively in G1 and G4 (41.56 + 13.8 vs. 35.29 + 20.25). However, no significant differences in bond strength were found between the groups ($P > 0.05$). The mean and SD excluded premature failure and voided specimen.

Table 3: Failure modes distribution in the groups (%)

Group	Failure modes					
	Adhesive (post-cement)	Adhesive (cement dentine)	Mixed (0-50%)	Mixed (50-100%)	Cohesive (in post)	Cohesive (in dentine)
G1	46.9	6.3	9.4	37.5	0	0
G2	35.3	5.9	5.9	47.1	0	5.9
G3	39.3	7.1	17.9	32.1	0	3.6
G4	13	9.7	6.5	38.7	0	3.2

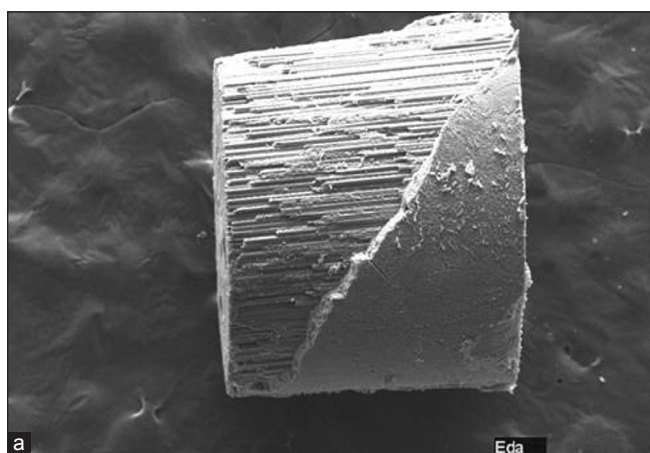
**Figure1:** Scanning electron microscopy images from push-out tested specimens. Cohesive failure in post in G3 (a) and G4 (b)

Table 3 shows the failure modes observed in each group based on stereomicroscope assay. Failure modes of G1 and G3 groups demonstrated that the majority of the bond failure was in an adhesive between post and resin cement. Predominant failure type in G2 and G4 was mixed failure with resin cement covering from 50% to 100% of post surface. The comparison of the failure modes after the use of each surface treatment using the chi-square test indicated that no statistical difference between the groups.

The SEM examination showed two cohesive failures in post [Figure 1]. The majority of failure was an adhesive between the post and resin cement and presented main residual resin cement on root dentine.

DISCUSSION

This present study investigated the effect of post-space pre-treatments on bond strength of fiber post dentine through push-out test. In light of the results of this study, no significant differences in bond strength were found between the different pretreatments ($P > 0.05$). Thus, the null hypothesis was confirmed.

The bond strength between fiber post and teeth has been evaluated using the different methods. These tests

consist of pull-out, push-out, and microtensile tests. The push-out test is more reliable and reproducible than microtensile test^[21,22] and is similar to the clinical condition.^[23] The vibrations of specimen during the bur trimming on microtensile test are transmitted to the interfaces, which are traumatized in uncontrolled ways. The need to a huge number of post increased price and reduced its popularity of pull-out test.^[24] Hence, based on these considerations we selected push out the test for the evaluation of bond strength.

The application of posts in restoring root filled teeth is necessary to provide the retention for the core of a definitive restoration.^[12] The ideal post transfer loads in the planned mode to the tooth in order not to reason undue susceptibility to root fracture.^[25] Corrosion resistance^[2] and elastic modulus of fiber post may influence the fracture resistance of restored teeth.^[2,26] Consequently, fiber post with low elastic modulus and proper corrosion resistance may suggest restoring root filled teeth.

In order to distribute stress over the entire bonded surface and increasing retention, using adhesive resin system was recommended for the cementation of post.^[23,27,28] The elimination of problem related to the moist use technique in root canal can be reduced when

using the self-etch system because this system can be applied on wet or dry methods.^[8] SEP adhesive system provides a better bond to any area of the root dentine than total-etching adhesive system.^[3] These resin systems are etched and penetrated into the dentine simultaneously. Some problems such as thicker smear layer,^[8] poor control of moisture,^[29] low acidity,^[19] and neutralization of functional acidic monomer of SEP^[30] exists in root dentine, therefore, the quality of penetration of the system in root dentine is doubtful.

The present study evaluated the effect of post-space pre-treatment with EDTA, ethanol, and (EDTA + ethanol) prior to the application of self-etching system (Panavia F2.0) on bond strength of fiber post showed no significant difference between the experimental and control groups.

Based on our review of literature, only five laboratory studies assessed the application of EDTA for improving bond strength to dentine. Rasimick *et al.*,^[15] Tori *et al.*,^[19] and Osori *et al.*,^[17] results agreed with our study. However, Blomof reported that 24% EDTA conditioning prior to the application of the two-component adhesive system compared one bottle adhesive system significantly and provided better shear bond strength.^[31] Tori *et al.*,^[19] and Osori *et al.*,^[17] used the 0.1 M concentration of EDTA.

Cheng *et al.*,^[32] compared the effect of 0.5 M EDTA and phosphoric acid pre-treatment prior to the application of one-bottle and multi-step bonding agents. Microtensile bond strength of one-bottle system as compared to the multi-step system was decreased in EDTA conditioning group.

One of these studies assessed bond strength in root dentine. Rasimick *et al.*, irrigated post space before the application of fiber post with either NaOCl or NaOCl followed by 17% EDTA. Post-space was prepared by post drill without any root canal therapy. Only the post system which when applied with mild self-etch system (pH = 4) showed improvement on bond strength after final irrigation with EDTA.^[15] In this work, the application of 0.1 M EDTA in root filled teeth was evaluated.

EDTA conditioning provides thinner hybrid layer with no denaturation of collagen, which penetrates more in resin than phosphoric acid.^[20] It displays a mild decalcifying capacity and minor influence on the dentine.^[19] The avoidance of denaturation of collagen due in part to the presence of more residual apatite crystals left in collagen matrix may

be one consequence application of EDTA.^[33,17] In addition, it was considered that the residual had more mineral content in the dentine substrata, which was incorporated in a chemical reaction with functional monomer of SEP. An increase in the bond strength value of EDTA group results from providing lower thickness smear layer.

There are ongoing problems related to dentine bonding systems such as poor durability and poor wetness with hydrophobic resin on wet dentine, however, the use of more hydrophilic resin causes more hydrolytic degradation.^[21,34] For the latter problem, Pashley *et al.*, introduced the wet ethanol bonding. This method leads to increase the bond strength of both hydrophilic and hydrophobic resin.^[35] Smaller diameter of collagen fibril in ethanol-wet versus water-wet dentine^[13] may cause high bond strength. The reduction of water sorption and/or the better sealing of collagen fibrils in hydrophobic resins and increment of durability in hydrolytic resins with the result of the ethanol-wet method were used.^[21]

This study revealed that ethanol pre-treatment of post space did not have any significant effect on bond strength ($P > 0.05$). This finding is similar to the other studies.^[4,10,12,36] Carvalho *et al.*, reported that the application of ethanol-wet bonding in three-steps bonding system (All bond 2) increased the bond strength of fiber post and root dentine.^[13] Controversy, Sauro *et al.*,^[20] and Castangalo *et al.*,^[14] confirmed that ethanol-wet bonding significantly improved the bond strength of fiber post to root dentine. In these two studies, saturation time in ethanol was 3 min. and 4 min. respectively. Only these two studies showed the significant effect of ethanol-wet bonding on bond strength and other studies with shorter saturation time (1 min)^[4,10] did not report any improvement. Reaching to proper wetness in deep and narrow post space is very difficult. Remittent water in root space and water solvent of bonding system that were used in this study dilute the ethanol, so ethanol saturation time should be increased to regain a better result which may be the cause in this study why the bond strength of fiber post and dentine were not affected by the ethanol pre-treatment of post space.

More concerns have increased that more ethanol saturation time of dentine, may be impractical in clinics. 0.1 M EDTA conditioning using ethanol-wet bonding suggested by Sauro *et al.*, (2011). When EDTA was used in combination with

ethanol-wet bonding method, resin infiltration into dentine improved due to the creation of thinner of demineralized layer. In this modified ethanol-wet bonding method, ethanol saturation time was 1 min.^[20] The treatment of water saturated dentine with ethanol was formed and collagen matrix was expanded due to the making of matrix interpeptids H-bonds.^[35,37] The creation of these H-bonds may mainly manifest in EDTA treated surface 15% shrinkage in the collagen matrix occurs by conditioning acid etched dentine with 100% ethanol as a result of removing water from the collagen fibrils. Stiff collagen matrix with high content of hydroxiapetite is provided by EDTA conditioning, which prevents the shrinkage of matrix during resin infiltration.^[20] It may also reduce the shrinkage of ethanol saturated dentine matrix that its results are more interfibrillar space for resin infiltration.

Previously, it has been mentioned that the durability of resin-dentine interface is poor because of (i) water sorption, (ii) activation of matrix metalloproteinases (MMPs)^[34,37] and (iii) the denuded collagen fibrils represented in partly infiltrated hybrid layer.^[38]

Interestingly, both EDTA and ethanol have a preventive effect on the degradation of resin-dentine. The prevention of nanoleakage,^[39] antimicrobial effect^[40] decreased micropermeability^[20,41] and the properties of EDTA may have highlight effect on bond durability in root dentine.

Several possible mechanisms may be supposed for the improvement of durability of resin-dentine interface made to ethanol-wet bonding method such as better resin infiltration and removing excessive water from the collagen matrix. The latter mechanism provides durable bond with hydrophilic resin because of a similarity between infiltrations of MMPs bind to collagen to collagen itself, thereby, inactivating MMPs.^[34] The cumulative effect of EDTA and ethanol will be evaluated in future studies.

About the fracture investigation, it should be emphasized that the pre-dominant failure types in the (G1 and G3) groups and (G2 and G4) groups respectively were adhesive failure between post and cement and mixed failure (resin cement covering from 50% to 100% of the post diameter). The finding of Perdigão *et al.*, de Durão Mauricio *et al.*, and Balbosh and Kern is also in accordance with our finding in G1 and G3 group.^[42-44] However, Rasimick *et al.*'s finding is different from ours. They reported that the

adhesive failure between dentine and adhesive resin was a pre-dominant failure in EDTA group.^[15] The findings in G2 and G3 groups were in agreement with the findings from previous studies.^[4,13,45] The value achieved from bond strength in this work is higher than those available in earlier studies.^[3,4,13,15] This might be due to the fact that none of these studies used chlorhexidin irrigation between changing instruments in root canal therapy. Cement was applied to root canal by lentullo spiral and the post-coated with cement and then inserted to the canal in this study. Most studies^[3,13] only used a lentullo spiral or syringe^[4] to apply cement to root canal.

In this study, the experiment samples were not entirely restored and neither thermal cycling and nor mechanical cycling was applied. A final rinsing agent in post preparation was distilled water that was rarely used in clinics. These factors may limit the expressed use of study consequences to clinical states.

We speculated that higher and facilitated infiltration of SEP adhesive system observed in modified wet-ethanol bonding method (EDTA conditioning prior to ethanol-wet bonding) would convert ethanol-wet bonding method to practical and proper clinical approaches. Long-term follow-up studies can test the cumulative effect of EDTA and ethanol pretreatment of post space on durability of post/cement/dentine interfaces.

CONCLUSION

According to the result of the present study, it was possible to conclude that the application of the different pretreatment solutions examined did not improve the bond strength of the adhesive system used in this study to cement fiber post to root dentine.

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