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Effect of antioxidants on push-out bond strength of hydrogen peroxide treated glass fiber posts bonded with two types of resin cement

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Objectives: Hydrogen peroxide (H_2O_2) surface treatment of fiber posts has been reported to increase bond strength of fiber posts to resin cements. However, residual oxygen radicals might jeopardize the bonding procedure. This study examined the effect of three antioxidant agents on the bond strength of fiber posts to conventional and self-adhesive resin cements. ***Materials and Methods:*** Post spaces were prepared in forty human maxillary second premolars. Posts were divided into five groups of 8 each: G1 (control), no pre-treatment; G2, 10% H_2O_2 pre-treatment; G3, G4 and G5. After H_2O_2 application, Hesperidin (HES), Sodium Ascorbate (SA) or Rosmarinic acid (RA) was applied on each group respectively. In each group four posts were cemented with Duo-Link conventional resin cement and the others with self-adhesive BisCem cement. Push-out test was performed and data were analyzed using 2-way ANOVA and tukey's *post-hoc* test ($\alpha = 0.05$). ***Results:*** There was a statistically significant interaction between the cement type and post surface treatment on push-out bond strength of fiber posts ($p < 0.001$, F = 16). Also it was shown that different posts' surface treatments significantly affect the push-out bond strength of fiber posts ($p = 0.001$). H_2O_2 treated posts (G2) and control posts (G1) cemented with Duo-link showed the highest (15.96 ± 5.07MPa) and lowest bond strengths (6.79 ± 3.94) respectively. ***Conclusions:*** It was concluded that H_2O_2 surface treatment might enhance the bond strength of fiber posts cemented with conventional resin cements. The effect of antioxidants as post's surface treatment agents depends on the characteristics of resin cements used for bonding procedure. (*Restor Dent Endod* 2014;39(4):303-309)

Key words: Antioxidant; Fiber post; Hydrogen peroxide; Resin cement; Surface treatment

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Introduction

Root-filled teeth with extensive loss of tooth structure require intracanal posts for adequate retention of restoration.[1] Mismatch of the modulus of elasticity between metal or stainless steel posts (200 GPa) and dentin (20 GPa) often leads to unfavorable root fractures.[2] Therefore, drawbacks of metal posts prompted the development of fiber reinforced posts (FRPs) with similar elastic modulus to dentin.[3,4]

The rate of irreversible root fractures significantly decreased following the introduction of FRPs.[5] However, adhesive failures became a main concern.[6] Various luting agents including glass ionomers and resin cements were introduced to improve the bond strength of FRPs. It was shown that similar elastic modulus of resin cements to fiber posts and dentin could increase the fracture resistance and retention of fiber

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posts compared to conventional cements.[7,8]

Durable bond between resin cements and fiber posts relies on micromechanical and chemical interactions.[9-12] Highly cross-linked epoxy resins with high degree of conversion constitute the main component of the fiber posts.[13] However, epoxy resins are unable to react with the functional monomers of resin cements.[13] Therefore various posts' surface treatments were suggested to expose the posts' fibers to enhance the micromechanical interactions.[10] Oxidant agents like potassium permanganate, sodium ethoxide and hydrogen peroxide (H_2O_2) can dissolve the epoxy matrix and expose the fibers without compromising the integrity of the posts.[12,14] However, it was hypothesized that the presence of oxygen residuals in dentinal tubules following application of oxidant agents might impede the resin infiltration and compromise the post cementation.[15-17]

Several studies speculated that antioxidant agents could negate the negative effects of residual oxygen through a scavenging mechanism.[18,19] The effects of Rosmarinic acid (RA) and hesperidin (HES) as antioxidants for post surface treatment have been investigated in different studies.[20,21] RA contains p-toluenesulfinic acid sodium salt and it was shown that application of RA for 30 seconds can improve the bond strength of resin adhesives to NaOCl-treated dentin.[22]

To the best of our knowledge, no previous studies have investigated the effect of common antioxidant agents on the bond strengths of H_2O_2 treated fiber posts to resin cements. Therefore, this study aimed to evaluate the effect of HES, sodium ascorbate (SA) and RA antioxidants on bond strength of H_2O_2 treated fiber posts bonded with conventional and self-adhesive resin cements. The null hypotheses tested were that H_2O_2 application would not affect the bond strength of fiber posts, and antioxidant agents have no significant effect on the push-out bond strength of H_2O_2 treated posts cemented by conventional or self-adhesive resin cements.

Materials and Methods

Specimen preparation

Forty human maxillary second premolars with similar root lengths, fully formed apices, and without caries, cracks, or previous endodontic treatments were selected. Teeth were stored in 0.2% thymol solution at room temperature for 6 months and external debris was removed using a hand scaler. The coronal part of each tooth was sectioned 1 mm below the cementoenamel junction with diamond disks (Ref. 070, D&Z, Berlin, Germany) to achieve a uniform length of 15 mm. Root canals were prepared to a working length of 1 mm from the apex by using the crown-down technique with rotary instruments (size S1 to F3, Pro-Taper, Dentsply-Maillefer, Ballaigues, Switzerland) to a size #30 (0.09 taper). Canals were irrigated using 2.5% sodium hypochlorite (AriaDent, Tehran, Iran) and normal saline during the instrumentation. Then canals were dried by absorbent paper points (AriaDent) and filled using a cold lateral condensation technique with the same size Protaper gutta-percha points (Suredent, Seongnam, South Korea) and resin-based endodontic sealer AH26 (Dentsply DeTrey GmbH, Konstanz, Germany). The endodontically treated teeth were sealed with glass ionomer restorative material (Vitremer, 3M ESPE, St Paul, MN, USA) and stored in water at 37℃ for 1 week to allow the endodontic sealer to set.

Cementation procedure

The gutta-percha was removed from the coronal portion of each root with a Gates Glidden drill #3 (Dentsply-Maillefer), leaving a 4 mm of gutta percha in the apices for adequate apical seal. 10 mm length post spaces were prepared to receive prefabricated FRPs (Svenska Dentorama, Stockholm, Sweden) by using appropriate drills (Fibio, Anthogyr, Sallanches, France). A new drill was used for every five specimens. Specimens were irrigated after post space preparation with normal saline. Prepared roots were randomly divided into 5 groups (n = 8) based on the posts' surface treatment protocol:

Group 1 (G1, control group): Posts received no surface treatment.

Group 2 (G2): posts were treated with 10% H_2O_2 for 20 minutes, then rinsed with distilled water and air-dried.

Groups 3, 4 and 5: posts' surfaces were treated with 10% H_2O_2 for 20 minutes and then posts were immersed in 6.5% HES (G3), 100 μM RA (G4), and 10% SA (G5) for 20 minutes, respectively, and then rinsed with distilled water and air-dried.

Table 1 shows the materials used in the study and their chemical compositions.

G2, G3, G4 and G5 posts were coated with Silane (Bis-silane, Bisco Inc., Schaumburg, IL, USA) using a micro brush (Multi-brush Multi-colors, Denbur Inc., Oak Brook, IL, USA) for 60 seconds and then washed with warm water and air dried.

Then the root-specimens in each group were divided into 2 sub-groups of Duo-Link and BisCem cements according to the type of resin cement used for post cementation (n = 4):

Duo-Link subgroup: Duo-link conventional resin cement (Bisco Inc.) was used. One layer of bonding agent (All-Bond 3, Bisco Inc.) was applied on the post surface with a microbrush according to the manufacturer's instructions and light cured for 10 seconds (600 mw/cm², Demi-LED light curing system, Kerr Corp, Orange, CA, USA). For the roots supposed to receive the posts coated with the bonding agent and Duo-Link cement, canals were etched using 37% phosphoric acid (Uni-Etch, Bisco Inc.) and then washed and dried with paper points. Then one layer of

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Table 1. Materials used in this study and their chemical compositions and instructions of use

Product	Company	Composition	Instructions
Glass fiber post (Dentorama)-No3: 1.25 mm diameter	Dentorama, Svenska, Sweden	Glass fiber + epoxy resin matrix	
BisCem self adhesive resin cement (Paste-paste dual syringe, automix)	Bisco Inc, Schaumburg, IL, USA	TEGDMA, HEMA, phosphate, dental glass	Inject into air dried canal without pretreatment, apply finger pressure for 5 - 10 sec, remove excess cement with microbrush light cure for 30 sec
Duo-Link resin cement (Paste-paste dual syringe, automix)	Bisco Inc, Schaumburg, IL, USA	Base: Bis-GMA, TEGDMA, urethane dimethacrylate, glass filler Catalyst: Bis-GMA, TEGDMA, glass filler	Etch and bond intracanal dentin and inject into canal, apply finger pressure for 5 - 10 sec, remove excess cement with microbrush, light cure for 40 sec
H_2O_2	Kimia, Tehran, Iran	Hydrogen peroxide	20 min immersion, washing and drying
Hesperidin 6.5%	Sigma Aldrich, Spain		20 min immersion, washing and drying
Rosmarinic acid 100 µM	Baridgessence, Kashan, Iran		20 min immersion, washing and drying
Sodium ascorbate 10%	Chemfil, Germany		20 min immersion, washing and drying
Bis-silane	Bisco Inc, Schaumburg, IL, USA	MPS, ethanol, water	Apply with micobrush for 60 sec and air dry
All bond 3 adhesive (Etchant, Uni-Etch Part A & B)	Bisco Inc, Schaumburg, IL, USA	32% Phosphoric acid Part A: Ethanol, Na-N-tolylglycine glycidyl methacrylate Part B: Bisphenyldimethacrylate, Bis-GMA, 2-HEMA	Etch the canal using Uni-Etch for 15 sec, rinse thoroughly and remove excess water with a brief burst of air and paper point. Dispense an equal number of drops of All-Bond3 Parts A and B (1 : 1), mix well for 5 sec Remove excess pooling, thoroughly air dry. Apply one layer of All-Bond 3 to the post and air dry. Light cure for 10 sec

TEGDMA, triethylene glycol dimethacrylate; HEMA, hydroxyethyl methacrylate; Bis-GMA, bisphenol A glycidyl methacrylate; MPS, methacryl-oxypropyltrimethoxysilane.

multi step etch-and-rinse bonding agent (All-Bond3) was applied into the canal surfaces using micro brushes, dried with paper points to remove the excess and then gently air dried and cured for 10 seconds.

BisCem sub-group: Posts were cemented with BisCem self-adhesive cement (Bisco Inc.). The treatment of the root canal with etching agent was omitted. Instead, the root canals were washed by distilled water and dried. The cement was inserted with Centrix syringe and needle headed (Centrix, DFL Ind. e Com. S.A., Rio de Janeiro, RJ, Brazil).

After post insertion (No3, 1.25 mm diameter, Svenska Dentorama), finger pressure was applied for 30 seconds to assure complete seating of the posts. Posts were light cured for 40 seconds after excess cement was removed. A 2 mm composite resin (Z100, 3M ESPE) layer was placed over the root access and light polymerized for 40 seconds. All roots were stored at 37℃ and 100% humidity for 24 hours for final adaptations (all specimens underwent 1,000 thermocycles, 5 - 55℃, 30 seconds dwell time).

Push-out Test

The tooth was sectioned perpendicular to its long axis into 1.1 ± 0.1 mm thick coronal, middle and apical sections using a diamond disc (Ref. 070, D&Z) mounted in a cutting machine (TL-3000, Vafaei Industrial, Tehran, Iran) at low speed under constant distilled water irrigation. The coronal and apical sections were discarded using a band saw (IsoMet, BUEHLER LTD., Lake Bluff, IL, USA). Four sections were obtained from the middle root and push-out test (Universal Testing Machine, LFM-L, PCS1000, Walter+bai AG, Switzerland) was performed for 16 test specimens (4 root-specimens x 4 sections) in each subgroup at a crosshead speed of 0.5 mm/min.

Since the posts were parallel in the middle third of the roots, the exact bonding surface can be calculated with the following equation:

$$A = 2\pi RH$$

where $\pi = 3.14$, A = surface area (mm), R = radius (mm) of the post and H = thickness (mm) of each section. The peak force (N) required to extrude the fiber post from each section was recorded for all specimens. Results were expressed in MPa by dividing the resultant force in Newton by the bonded area of the specimen. Failure mode was determined with a stereomicroscope (x50, Vanox, Olympus, Tokyo, Japan) and classified as adhesive between post/cement or cement/dentin, cohesive within post, and mixed.[23]

The push-out data were compared with a 2-way analysis of variance (ANOVA) (cement versus post surface treatment protocol), and *post-hoc* Tukey test was used to determine significant differences among the groups ($\alpha = 0.05$).

Results

A. Push-out bond strength

Mean bond strength values in control and experimental groups are presented in Table 2. H_2O_2 treated posts (G2) and control posts (G1) cemented with Duo-link showed the highest (15.96 ± 5.07MPa) and the lowest bond strengths (6.79 ± 3.94), respectively. A two-way ANOVA was conducted to examine the effects of cement type and post surface treatment on bond strength of fiber posts. There was a statistically significant interaction between the cement type and post surface treatment on push-out bond strength of fiber posts ($p < 0.001$, $F = 16$). Also it was shown that the bond strengths were significantly influenced by different posts' surface treatment protocols and the different luting cements ($p = 0.001$).

Duo-link cement

Among the post cemented with Duo-Link, G2 (15.96 ± 5.07MPa) and control (6.79 ± 3.94) groups showed the highest and lowest bond strengths, respectively. ANOVA and tukey's *post-hoc* test were used to determine the difference in the bond strength of the posts cemented with Duo-Link. There was a significant difference between control and all experimental groups (G2, G3 and G4) except G5. Also bond strength of G2 group was significantly higher than control and study groups, cemented with Duo-link ($p = 0.001$).

BisCem cement

Among the posts cemented with BisCem, RA treated posts (G4) and control group showed the highest (15.28 ± 4.61MPa) and lowest (7.2 ± 3.1) bond strength, respectively. Bond strength of all groups, treated with antioxidant agents (C3, C4 and C5) was significantly higher than the control group ($p < 0.05$). Also bond strength of G4 and G5 posts were significantly higher than G2 posts ($p = 0.023$). However there was no significant difference between control and G2 groups ($p = 0.65$).

In this study, it was shown that different types of cement could significantly affect the bond strength of fiber posts. In G2 ($p = 0.001$) and G4 ($p = 0.032$) groups Duo-Link and BisCem cement showed higher bond strength, respectively. However bond strength of fiber posts in control, G3 and G5 groups were not significantly affected by the type of cement ($p > 0.05$).

Table 2. Descriptive statistics of the post push-out strengths in each experimental group

Luting agent	Post surface treatment				
	No treatment-control group (G1)	H_2O_2 (G2)	H_2O_2 + HES (G3)	H_2O_2 + RA (G4)	H_2O_2 + SA (G5)
Self-adhesive BisCem cement	7.2 ± 3.1^{aA}	7.9 ± 4.3^{abA}	10.79 ± 5.26^{bcA}	15.28 ± 4.61^{cA}	11.37 ± 3.04^{bcA}
Conventional dual-cure Duo-link cement	6.79 ± 3.94^{aA}	15.96 ± 5.07^{cB}	10.28 ± 3.4^{bA}	10.62 ± 2.41^{bB}	8.35 ± 3.99^{abA}

Different lowercase letters within rows and uppercase letters within columns indicate significantly different means at the 5% level.

HES, hesperidin; RA, Rosmarinic acid; SA, sodium ascorbate.

Table 3. Percentage (%) of failures recorded in each experimental group

Group	Cement	Type of bond failure				
		AC	AD	CP	Mixed	Premature failure
G1	Duo-Link	50.0	16.6	-	33.4	-
	BisCem	16.6	8.4	-	75.0	-
G2	Duo-Link	41.6	16.6	-	41.6	-
	BisCem	33.4	-	-	58.2	8.4
G3	Duo-Link	41.6	16.6	8.4	33.4	-
	BisCem	16.6	-	8.4	75.0	-
G4	Duo-Link	16.6	8.4	-	75.0	-
	BisCem	8.4	8.4	16.6	66.6	
G5	Duo-Link	50.0	16.6	-	33.4	-
	BisCem	12.0	-	8.4	66.6	-

G1, Control; G2, H_2O_2; G3, H_2O_2 + HES; G4, H_2O_2 + RA; G5, H_2O_2 + SA.
AC, Adhesive at post-cement interface; AD, Adhesive at cement-dentin interface; CP, Cohesive within post.

B. Failure modes

The results of the failure modes are summarized in Table 3. In all the groups, the majority of failure modes were mixed and adhesive cement/post failures. There were no significant differences in failure modes between the experimental groups (p = 0.86).

Discussion

The first null hypothesis tested (H_2O_2 application would not affect the bond strength of fiber posts) was rejected. H_2O_2 treated fiber posts, cemented with Duo-link showed significantly higher bond strength than control and study groups cemented with the same cement. Also surface treatment of fiber posts with H_2O_2 did not adversely affect the bond strength of fiber posts cemented with BisCem compared to the control group. These findings are in agreement with previous studies[16,23] indicating that fiber post treatment using H_2O_2 could enhance the bond strength. Epoxy resins, with a high degree of conversion and few reactive sites cover most of the fiber posts.[13] It seems that bond strength durability of fiber posts luted with conventional resin cements depends on micromechanical interlocking of resin tags.[24] However, self-adhesive cements contain multifunctional phosphoric acid methacrylates that rely on chemical reactions with the hydroxyapatite of hard tooth tissue to ensure an adequate bond.[24] Oxidant application can create spaces between the fibers, which are favorable for the micromechanical interlocking between fiber posts and resin cements.[12] These statements may explain the higher bond strength of H_2O_2 treated posts (G2) cemented with conventional resin cements compared to G2 group cemented with Biscem. Also it might be suggested that the effect of H_2O_2 differs in combination with different types of resin cements. From the results of this study we may speculate that self-adhesive resin cements are more likely to be adversely affected by oxygen residuals following oxidant application. However this statement needs to be investigated in further studies.

In the present study, the bond strength of fiber posts was significantly affected by the different post surface treatment protocols. Therefore, the second null hypothesis indicating that different antioxidant agents have no effect on the bond strength of H_2O_2 treated posts was rejected. There are contradictory results regarding the most effective fiber posts surface treatments.[7,20] In our study, among posts cemented with self-adhesive BisCem cement, posts treated with RA antioxidant showed the highest bond strength compared to other experimental groups. Also SA treated posts showed significantly higher bond strength than H_2O_2 treated posts with no application of antioxidant (G2).

It has been suggested that antioxidants use different mechanisms like free radical chain breaking, metal chelating and free radical quenching to negate the negative effects of free radicals following application of oxidant agents.[20] In the case of the free radical quenching mechanism, antioxidants can react with oxidants to neutralize unpaired electrons and form a stable product, which limits the activity of oxidant agents.[25] Some studies indicated that the SA could improve the bond strength of fiber posts to dentin.[26,27] However, Prasansuttiporn *et* *al*. stated that SA did not significantly increase the bond strength of adhesives to dentin.[20] This inconsistency might be attributed to the different application time and concentration of SA. Therefore, further researches are required to confirm the exact application protocol of SA. RA (a-o caffeoyl- 3,4-dihydroxyphenylacetic acid) is a diphenolic compound that contains two catechol (1,2-dihydroxybenzene) rings.[28] The antioxidant activity of RA can be explained by the ability of catechol to form an intermolecular hydrogen bond between the free hydrogen of its hydroxyl and phenoxyl radicals.[20] It was reported that application of 100 µM RA for 5 or 10 seconds could significantly increase the bond strength of adhesive systems to NaOCl treated dentin.[20] The antioxidant activities of reducing agents depend on their total antioxidant capacity value. Polyphenols like RA have a higher total antioxidant capacity than SA.[29] This might explain the higher bond strength of RA treated groups than SA treated groups in the present study.

In our study, all antioxidant agents significantly deteriorated the bond strength of fiber posts cemented with conventional resin cements compared to H_2O_2 treated group (G2). From these results we can speculate that antioxidant agents are not compatible with conventional resin cements and they may impede the resin infiltration during the cementation procedure. However this statement has to be confirmed in further researches.

In the present study, the type of cement significantly affected the bond strength of fiber posts in G2 and G4 groups. While in other groups, bond strength was not affected by the type of cement. In a recently published study, the highest bond strength of fiber post was obtained with the conventional resin cements when compared with self-adhesive cements.[30] Conversely, in another study, the dual-polymerized conventional resin cements showed significantly lower mean push-out bond strength than the other cements.[31] These statements along with the results of our study may indicate that the efficacy of resin cements could be modified by different post surface treatments. Also various results in different study groups of our study may be explained by the fact that application and handling techniques could influence the bond strength of different self-adhesive cements when used for post cementation.[32]

Although in this study, root canal therapy was performed and then posts were cemented to simulate the clinical conditions, our study is an *in vitro* investigation and does not fully replicate oral conditions. In addition, factors like thermal, load cycling, and water storage aging methods that challenge the adhesive interface should be investigated in further studies. Finally, all proposed luting cements and all post surface treatments during FRP cementation merit further clinical trials to assess the results of our study.

Conclusions

From the results of the present study, it can be concluded that H_2O_2 surface treatment can enhance the bond strength of fiber posts cemented with conventional resin cements. Also it was shown that the effect of antioxidant agents depends on the characteristics of resin cements used for bonding procedure. It seems that antioxidant agents, especially rosmarinic acid are more compatible with self-adhesive resin cements.

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