

Introduction

Fiber-reinforced composite (FRC) posts were introduced in the early 1990s as an alternative to cast post and cores, or metal dowels, to restore endodontically treated teeth. The preference for, and popularity of, FRC posts can be chiefly ascribed to their having an elastic modulus closer to that of dentin than metal posts/dowels, thus reducing the risk of vertical root fractures. Other advantages of FRC posts include biocompatibility, no potential for corrosion or allergic hypersensitivity, mechanical strength, improved light transmission, and better optical effects in esthetic restorations.¹ Quartz or glass fiber posts can be used where esthetics is of prime importance.² Several in vitro studies have shown that FRC posts distributed occlusal stress more evenly in the root dentin, resulting in more favorable root fractures, which were often reparable.³⁻⁵

Loss of retention of posts has been found to be the most frequent mode of failure. Other modes of failure include risk of caries or caries development in the root canal and fracture of the root, the post, or the core.^{4,6,7} Post retention is influenced by several factors including post type, cement type, and the interaction between the cement and dentin and between the cement and the post. Regarding the post, parameters such as length, diameter, design, and surface structure of the post affect post retention.⁸ One possible reason for loss of retention is the absence of chemical bonding between resin composites (methacrylate-based) and the matrix of fiber posts, which are often composed of epoxy resin. The lack of chemical bonding is likely due to the fact that epoxy polymers exhibit a high degree of conversion and high cross-linked structures. As silane coupling agents and the methacrylate-based resin composite do not bond well with the epoxy matrix. A silane chemical bond can only occur between the resin composite and exposed glass fibers of the post.^{9,10} Several post surface treatments to enhance bonding have recently been suggested. These procedures fall into three categories: 1) treatments resulting in chemical bonding between composite and post, 2) treatments which roughen the surface (sand-blasting or etching), and 3) combined micromechanical and chemical components using the former methods.⁹

Hydrogen peroxide is commonly employed in immunological electron microscopy to partially dissolve the resin surface of epoxy resin embedded tissue sections by breaking epoxy resin bonds through a mechanism of substrate oxidation.^{9,11} It is reported that etching the fiber post surface with 24% hydrogen peroxide for 10 minutes enhanced the interfacial bond strength between posts and resin composites.^{11,12} Partially dissolving the resin matrix by this treatment allowed for the modification of the post surface morphology, resulting in exposure of the superficial fibers, which did not appear to be damaged in the process.^{9,11-13}

This study evaluated the effect of various concentrations and durations of hydrogen peroxide post surface treatments on the push-out bond strength and evaluated the effect of root region on push-out bond strength of fiber posts in endodontically treated teeth.

Materials and Methods

All experimental procedures were approved by the Ethics Committee of the Faculty of Dentistry, Chulalongkorn University. Thirty five single-rooted human mandibular first premolars with fully developed apices, extracted for orthodontic

treatment, were selected for this study. The selection criteria for inclusion in the study were absence of: caries, root cracks, endodontic treatment, posts, or crowns. The selected teeth were immediately immersed in 5.25% NaOCl for 5 minutes and then stored in 0.9% saline solution at room temperature.¹⁴

The teeth were decoronated below the cementoamel junction (CEJ) using a low-speed diamond saw (Isomet 1000, Buehler, IL, USA) under copious water cooling. All roots were endodontically instrumented to a working length of 1 mm from the apex up to a #40 master apical file. A step-back technique was used with stainless steel K-files. Throughout the shaping process, the root canals were irrigated with a 5.25% sodium hypochlorite solution after each file, rinsed with distilled water, and dried with paper points. To obturate the canals, gutta percha points were coated with root canal sealer (AH-Plus[®]; Dentsply DeTrey GmbH, Konstanz, Germany) and laterally condensed. Root canal access openings were filled with a provisional restorative material (Cavit[™] G, 3M ESPE AG, Seefeld, Germany), and the teeth were stored in humidified container for 7 days at 37° C.¹⁵

Post spaces were created with a warm plugger and DT light Post drill #1, nine mm deep from the CEJ. The prepared roots were randomly divided into seven groups according to post surface treatment (Table 1) and posts were immersed in hydrogen peroxide at a concentration and length of time dependant on the group for study group 2-7.

Table 1 Chemical surface treatment groups

group	chemical surface treatment
1	No chemical surface treatment
2	24% H ₂ O ₂ /5 minutes
3	24% H ₂ O ₂ /10 minutes
4	30% H ₂ O ₂ /5 minutes
5	30% H ₂ O ₂ /10 minutes
6	35% H ₂ O ₂ /5 minutes
7	35% H ₂ O ₂ /10 minutes

H₂O₂: hydrogen peroxide

Prior to luting in the prepared canals, the fiber posts were cleaned with distilled water in an ultrasonic cleaner for 2 minutes and gently air-dried for 1 minute. Silane was applied to the posts with a brush, left undisturbed for 60 seconds,

and gently air-dried for 5 minutes to promote adhesion between the fiber posts and resin cement. All posts were marked at a distance of 9 mm. from the apical end corresponding to the length of the prepared post space.

In preparation for post cementation, the root canals were etched for 15 seconds using 37% phosphoric acid (Total Etch, Ivoclar Vivadent, USA) and rinsed thoroughly with distilled water using endodontic syringes. Excess water was removed from the post space with 4 paper points. Dentin bonding agent (Excite DSC[®], Ivoclar Vivadent, USA) was applied for 10 seconds to the canal according to the manufacturer's directions, and dried with 4 paper points. The surface treated posts were cemented with Multicore Flow (Ivoclar Vivadent, Liechtenstein) and excess cement was removed. The Multicore Flow[®] cement was cured for 60 seconds with a halogen light (Elipar[®] TriLight, USA). All roots were stored in a humidified container at 37° C for 24 hours. Subsequently, each root was embedded in clear acrylic resin (15 mm in diameter and 30 mm in height) along the root long axis using a surveyor, at the center of a silicone mold.

A thin-slice push-out test was used in this study to evaluate bond strength. Each root was sectioned horizontally into six 1-mm-thick specimens with a saw microtome (Leica SP 1600) under water cooling to represent the cervical, middle, and apical regions of the root (Fig. 1). Each specimen was marked on its coronal surface with an indelible marker, and the exact thickness of each slice was measured using a digital caliper (0.01 mm accuracy; Mitutoyo, Tokyo, Japan). The cervical and apical canal radii were measured using a stereomicroscope.

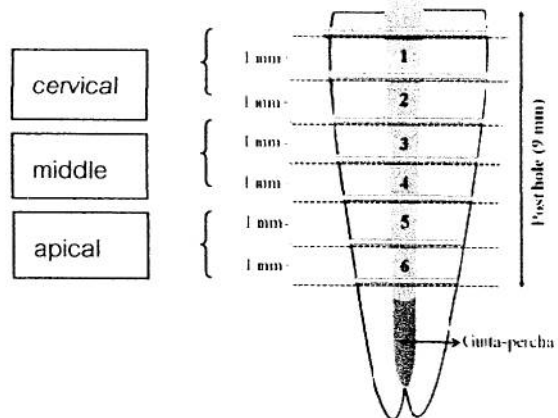


Fig.1 Root sectioned into six 1 mm thick specimens

The post segment was loaded with a cylindrical plunger (0.8 mm in diameter) centered on the post segment, having no contact with the surrounding dentin surface. Load was applied with a universal testing machine (Instron[®] 8872, Instron, Fareham, UK), in an apical-to-cervical direction, at a crosshead speed of 0.5 mm/min until failure occurred. Push-out bond strength was calculated for each specimen by using the following formula:

$$\text{Debond stress (MPa)} = \text{Debonding force (N)} / A \text{ (mm}^2\text{)}$$

where A=area of the post-dentin interface. The latter was determined using the formula for the surface area of a frustum (radii of the top and bottom surfaces of the post along with the height of the slice) as follows:

$$A = \pi(R_1 + R_2) \sqrt{(R_1 - R_2)^2 + h^2}$$

After push-out strength evaluation, the failure mode of each debonded specimen was analyzed under a stereomicroscope (MEIJI ML 9300) at $\times 15$ magnification. The failure modes were classified according to the following criteria: (1) Adhesive failure between dentin and luting cement; (2) Adhesive failure between luting cement and post; (3) Cohesive failure within luting cement; (4) Cohesive failure within the post; and (5) Mixed failure.

Statistical analysis

Push-out bond strength data were first verified for the normality of data distribution using the Komogorov-Smirnov test and by Levene's test for homogeneity of variances. Three-way analysis of variances at a 95% confidence level was subsequently performed on the push-out bond strength data with the concentration of hydrogen peroxide, duration of post surface treatments, and root region as the dependent variables, and push-out bond strength as the fixed factor. Post hoc tests were carried out using the Tukey HSD multiple comparison test, with a probability level set at $\alpha=0.05$ for statistical significance.

Results

Three-way analysis of variances revealed significant differences between concentrations and durations of hydrogen peroxide immersion assayed ($p<0.05$). Tukey HSD post hoc analysis showed significant differences in push-out bond strength between groups (Table 2) ($P<0.05$).

Table 2 Mean Push out bond strength and standard deviation: MPa \pm SD

Group/root region	cervical	middle	apical	mean
1. No treatment	15.6795 \pm 4.28	13.0238 \pm 4.18	10.2613 \pm 4.48	12.9882 \pm 4.73 ^{a,b}
2. 24% H ₂ O ₂ 5 minutes	12.3846 \pm 2.24	12.0799 \pm 4.33	12.1302 \pm 4.27	12.1982 \pm 3.61 ^a
3. 24% H ₂ O ₂ 10 minutes	15.9790 \pm 4.40	16.3878 \pm 3.19	15.7547 \pm 2.80	16.0405 \pm 3.41 ^{c,d}
4. 30% H ₂ O ₂ 5 minutes	15.9112 \pm 3.34	15.8363 \pm 3.80	15.5684 \pm 4.02	15.7720 \pm 3.60 ^{b,c}
5. 30% H ₂ O ₂ 10 minutes	19.2422 \pm 4.50	19.1609 \pm 3.03	18.5913 \pm 5.32	18.9981 \pm 4.24 ^d
6. 35% H ₂ O ₂ 5 minutes	16.3612 \pm 3.17	16.2786 \pm 4.24	16.3517 \pm 4.85	16.3305 \pm 4.00 ^{c,d}
7. 35% H ₂ O ₂ 10 minutes	13.3032 \pm 3.01	14.6000 \pm 4.20	13.7374 \pm 4.89	13.9335 \pm 4.01 ^{a,b,c}

Groups with same superscript letters were not significantly different ($p>0.05$)

The highest push out bond strength was recorded for the 30% H₂O₂ for 10 min group at 18.9981 \pm 4.24 MPa, followed by the 35% H₂O₂ for 5 min group at 16.3305 \pm 4.00 MPa and 24% H₂O₂ for 10 min group at 16.0405 \pm 3.41 MPa. There was, however, no significant difference between these groups. The 30 % H₂O₂ for 5 min, 35% H₂O₂ for 10 min groups

and no hydrogen peroxide post surface treatment group were next with push out bond strengths of 15.7720 ± 3.60 MPa, 13.9335 ± 4.01 MPa and 12.9882 ± 4.73 , respectively. These groups were not significantly different than each other.

The lowest push out bond strength was recorded for the 24% H₂O₂ for 5 min at 12.1982 ± 3.61 MPa, which was not significantly different from that of the 35% H₂O₂ for 10 min group and no hydrogen peroxide post surface treatment group. There were no significant differences observed between root regions ($p > 0.05$) on push-out bond strength of fiber posts in endodontically treated teeth.

Failure mode analysis, Table 3 shows the distribution of failure modes in this study. Adhesive failure between dentin and luting cement was the most frequently occurring failure mode in the experimental group 1. Adhesive failure between luting cement and fiber post was the most frequently occurring failure mode in the experimental group 3,4 and 6. Mixed failure was the most frequently occurring failure mode in the experimental group 2,5 and 7. No cohesive failures within the post were observed in this study.

Table 3 Distribution of failure modes according to the experimental groups (%)

Experimental groups	Failure modes				
	1	2	3	4	5
1. No treatment	18 (60)	2 (6.7)	1 (3.3)	-	9 (30)
2. 24% H ₂ O ₂ 5 minutes	3 (10)	11 (36.7)	1 (3.3)	-	15 (50)
3. 24% H ₂ O ₂ 10 minutes	-	23 (76.7)	-	-	7 (23.3)
4. 30% H ₂ O ₂ 5 minutes	4 (13.3)	19 (63.3)	1 (3.3)	-	6 (20)
5. 30% H ₂ O ₂ 10 minutes	7 (23.3)	9 (30)	2 (6.7)	-	12 (40)
6. 35% H ₂ O ₂ 5 minutes	5 (16.7)	13 (43.3)	2 (6.7)	-	10 (33.3)
7. 35% H ₂ O ₂ 10 minutes	6 (20)	8 (26.7)	-	-	16 (53.3)

1: Adhesive failure between dentin and luting cement; 2: Adhesive failure between luting cement and fiber post; 3: Cohesive failure within the luting cement; 4: Cohesive failure within the post; 5: Mixed failure

Discussion

To determine the retention of luted posts or selectively the strength at the post-cement-dentin interfaces, studies have been performed using microtensile, pull-out, or push-out tests. The small specimens used in microtensile and thin-slice push-out tests allow for uniform stress distribution, discrimination of regional differences, and reduce the number of teeth needed for data collection.¹¹ However, the reliability and applicability of the microtensile technique is limited by the frequent occurrence of pre-test failures, affecting data distribution.^{16,17}

The present study investigated bond strength using a push-out model. Push-out tests result in shear stress at the interface between post and cement which is comparable to the stress occurring under clinical conditions. Furthermore, the push-out design used allows polymerization stresses comparable to the clinical situation.¹⁸

Previous studies have shown that immersing fiber posts in 10% hydrogen peroxide for 20 minutes or 24% hydrogen peroxide for 10 minutes dissolved the epoxy resin matrix, broke epoxy resin bonds, exposed the surface of post fibers to silanization, modified the surface morphology of fiber posts, significantly enhanced the interfacial strength between fiber posts and resin composite, and significantly improved retention compared to silanization alone.^{9,12,13,19} With the dissolution of the outer layer of epoxy resin, an increased surface area of undamaged fibers is exposed and becomes available for reacting with silane. The coupling agent bridges between the silica in the fibers and the methacrylate-based resin.^{11,20}

The present study revealed that post surface treatment with 30% hydrogen peroxide for 10 minutes showed the highest push-out bond strength while the push out bond strength for 30% hydrogen peroxide at 10 minutes was the highest value seen it was not statistically significant different from 35% hydrogen peroxide for 5 minutes and 24% hydrogen peroxide for 10 minutes. There were no statistical differences between post surface treatments with 24% hydrogen peroxide for 10 minutes, 30% hydrogen peroxide for 5 minutes, 35% hydrogen peroxide for 5 minutes, and 35% hydrogen peroxide for 10 minutes.

Our study showed that both concentration and duration of post surface treatments had a negative correlation with push-out bond strength. At the same concentration, the push out bond strength from post surface treatment with 35% hydrogen peroxide for 10 minutes was less than that for 35% hydrogen peroxide for 5 minutes. At the same duration, the push out bond strength from post surface treatment with 35% hydrogen peroxide for 10 minutes was less than that for 24% hydrogen peroxide for 10 minutes with a similar relationship shown by post surface treatment with 35% hydrogen peroxide for 5 minutes and 30% hydrogen peroxide for 5 minutes. Post surface treatment with 35% hydrogen peroxide for 10 minutes did not improve the push-out bond strength of fiber posts when compared with that of 24% hydrogen peroxide for 10 minutes, 30% hydrogen peroxide for 5 minutes, 30% hydrogen peroxide for 10 minutes, and 35% hydrogen peroxide for 5 minutes. The epoxy resin matrix was extensively dissolved with 35% hydrogen peroxide for 10 minutes such that the superficial fibers were removed and the resin cement could not sufficiently infiltrate to fill all the space. This allowed gaps, resulting in a decrease in push out strength.²¹ Post surface treatment with 24% hydrogen peroxide for 5 minutes showed the lowest push-out bond strength. This low concentration and duration of post surface treatment may have been unable to dissolve the epoxy resin matrix sufficiently for improved retention. Typically, 30% hydrogen peroxide and 35% hydrogen peroxide are used in dental clinics for teeth whitening²². The present study used 35% hydrogen peroxide bleaching agent in solution, which was diluted to prepare the other H₂O₂ solutions. As 35% hydrogen peroxide is readily available, post surface treatment with 35% hydrogen peroxide for 5 minutes may an alternative method for post surface treatment, not requiring on-hand solution dilution, saving clinical time.

In the present study, the root region did not affect the push-out bond strength supporting the results of Boillagüe S. et al.²³ Previous studies have found that root region significantly affected push-out bond strength, with the highest bond strength values achieved in the cervical region, and the lowest values obtained in the apical region. These results may be due to more difficult access to the apical region and a possible limitation of cement flow. Additionally, at the middle and apical regions, a reduction in curing light transmission could result in decreased polymerization of the luting cements.^{24,25}

In contrast, some authors^{23,26} obtained the best results in the apical region. Such discrepancies in bond strength results might be attributed to differences in the distribution and density of dentinal tubules at different root regions. It has been reported that the density of dentinal tubules in the cervical region is higher than that in the apical region, and the diameter of tubules decreased in the apical direction.²⁷

In the present study, root dentin was etched with 37% phosphoric acid. The gel was introduced into the root canal with a needle, rinsed thoroughly with distilled water using an endodontic syringe, and dried with 4 paper points to remove excess water and control moisture. Excite DSC[®], a hydrophilic dual-cure bonding system allows for complete polymerization in cavity areas inaccessible to light. The brushing motion used to apply the bonding agent allows the adhesive solution to penetrate into open tubules along the cavity walls. In examining the density and length of resin tags in the dentin specimens treated with Excite DSC[®], Dagostin and Ferrari²⁸ showed a layer of hybridized dentin was present. The hybrid layer thickness was between 4-6 micrometers, consisting a hydrophilic resin monomer exposed collagen network and a demineralized dentin surface forming a resin-dentin interdiffusion zone. The uniform high density of resin tags and hybridized dentin increased the possibility of achieving higher bond strength. Multicore Flow[®], is a dual-activated composite used for cementation. Studies using Multicore Flow[®] recorded relatively high bond strengths confirming that high filler load and flowable consistency are a favorable combination of properties for adhesion to the post surface.²⁹ The properties of Multicore Flow[®], including low viscosity, when used in conjunction with the unit-dose adhesive agent Excite DSC[®], allows for cementation of endodontic posts. In our study, Multicore Flow[®] was delivered from a cartridge with auto-mix and intra-oral mixing tips. An automix delivery system simplifies placement of this flowable cement and its dual-curing capability assures adequate polymerization. These easy handling properties reduce technique sensitivity, allowing better retention.

Analysis of the failure modes in the present study revealed that most failures were predominantly adhesive failures between luting cement and fiber post, which was in accordance with the results of a recently published investigation.³⁰ When the canal is flared because of carious extension, trauma, pulpal pathosis, iatrogenic causes, a thicker layer of resin cement is produced between dentin and post. A finite-element analysis study demonstrated that if cement has a lower mechanical strength than the two materials it joins, a zone of highly concentrated loads and stress will be created. Therefore, photoirradiation of dual-curing resin cement may be preferred to obtain optimal mechanical properties of resin cements to reinforce the root.³¹

Under the limitations of this study, post surface treatments improved retention. However long-term clinical studies are needed prior to making a general recommendation.

Conclusion

Chemical surface treatment is a method to improve bond strength between fiber posts and resin cements in endodontically treated teeth. The present study showed that post surface treatment with 30% hydrogen peroxide for 10 minutes provided the highest push-out bond strength. However, post surface treatments with 35% hydrogen peroxide for 5 minutes may be an alternative method to improve the retention and reduce the clinical time.

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