Effects of matrix composition and surface treatment on the bond strength of glass fiber-reinforced composite posts to root dentin

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Abstract

Objective: This study evaluated the effects of resin matrix composition and surface treatment on the bond strength of glass fiber-reinforced composite (FRC) posts to intraradicular dentin.

Methods: Fifty-six extracted premolars were obtained. Post spaces were prepared for either epoxy resin-based posts (White Post DC) or Bis-GMA-based posts (Postec Plus) (n=28). The posts received different surface treatments (n=7): 70% ethanol (control), air abrasion and silanization, 35% H3PO4, and 24% H2O2. Posts were cemented and the samples were sectioned into cervical, middle, and apical thirds. The push-out bond strength of samples was compared between the groups using two-way ANOVA (α =0.05).

Results: There was a significant difference in bond strength between the different treatment methods in all root sections (P < 0.05). The highest push-out bond strength in the cervical, middle, and apical sections was observed in subgroups treated with air abrasion and silanization, with a significant difference from other groups in most comparisons (P < 0.05). Epoxy resin and Bis-GMA posts had comparable bond strengths in the coronal and apical sections (P > 0.05). Additionally, their bond strengths were comparable in the middle section when the post surfaces were treated with air abrasion and silanization, or $35\% H_3PO_4$ (P > 0.05).

Conclusions: Air abrasion and silanization could be suggested as the optimal surface treatment strategy to improve the bond strength of FRC posts to resin cement. Proper mechanical and chemical surface treatments might be more important than the resin matrix composition in determining the bond strength of FRC posts.

Keywords: Air abrasion, Bisphenol A-Glycidyl Methacrylate, Bond strength, Epoxy resins, Fiber post, Silane

Introduction

Endodontically treated teeth can be restored with fiber-reinforced composite (FRC) posts instead of traditional cast post-and-core systems, particularly when stress distribution, aesthetics, and conservation of the remaining tooth structure are prioritized (1). FRC posts are composed of resin matrix composites embedded with silica fibers. They exhibit favorable flexibility and stress distribution along the root. Their elastic modulus closely aligns with that of dentin, which allows for more natural force absorption and reduces

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stress concentration (2). A recent meta-analysis showed a success rate of 92.8% for FRC posts, which is satisfactory (3).

Despite the advantages of FRC, one of the major challenges in clinical practice is ensuring a reliable longterm bond between the FRC post and resin cement (4). FRC post-debonding from the root canal remains the most frequent cause of its clinical failure (5). The debonding primarily stems from the highly polymerized nature of the polymer matrix in prefabricated posts, especially those made with Bis-GMA or epoxy-based matrices. The high degree of cross-linking in these matrices prevents the monomers in luting cement from effectively penetrating the surface, leading to a weaker bond at the interface (6). Approximately 60% of fiber post failures occur at the interface between the fiber post and the resin cement (7). Bonding between crosslinked posts and dimethacrylate-based cement relies mainly on mechanical interlocking (8). Therefore, improving the bond strength between the FRC posts and



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the resin cement is crucial for clinical success, highlighting the importance of surface treatments to enhance adhesion.

Several surface treatment techniques are recommended to improve the bond strength of FRC posts to the resin cement. These include chemical agents such as hydrogen peroxide (H₂O₂), hydrofluoric acid, and phosphoric acid (H₃PO₄), as well as mechanical methods like sandblasting (4, 9, 10). These treatments aim to roughen the post surface, alter or remove the cross-linked superficial layer, expose the glass fibers, and enhance the mechanical and chemical interlocking between the post and cement (4). The use of silane coupling agents is also a chemical treatment method that can increase bond strength by 20%. This enhancement occurs because silane agents mediate adhesion between the inorganic fiber matrix and the organic resin matrix through the formation of siloxane bonds. As a result, silanization improves the compatibility of the post surface with resin bonding agents (11).

The matrix composition of FRC posts plays a critical role in the adhesion to the bonding agents and their mechanical performance. Asakawa et al. (12) investigated the effects of various matrix resins and surface treatments on the bond strength of experimental FRC posts. They found that both the matrix resin and surface treatment influenced adhesion to the core build-up resin. Ibtisam et al. (8) evaluated the pushout bond strength of commercial FRC posts with epoxy resin, Bis-GMA, and semi-IPN matrices, and found that epoxy-based posts had significantly higher bond strength. Limited studies have examined the effect of various surface treatments on FRC posts with different matrix compositions. Therefore, the current study investigated the effect of various surface treatment methods on the push-out bond strength of FRC posts with epoxy resin-based or Bis-GMA-based matrices.

Materials and methods

The protocol of this *in vitro* study was approved by the ethics committee of Mashhad University of Medical Sciences (IR.MUMS.DENTISTRY.REC.1398.033).

Sample collection and preparation

Based on the study by Asakawa et al. (12), the sample size was calculated to be seven teeth per group, considering α = 0.05 and β = 0.2. Fifty-six human single-rooted premolar teeth were obtained and stored in normal saline. The inclusion criteria were intact teeth with straight root canals of an average 18 mm length

extracted due to orthodontic reasons. Each tooth was decoronated 1 mm above the cementoenamel junction (CEJ) with a milling machine under water coolant. The roots were embedded in cylinders using a self-curing acrylic resin.

Endodontic treatment

The root canals were prepared using hand-held stainless-steel K-files (Mani Inc, Tokyo, Japan). They were irrigated with normal saline and 2.5% sodium hypochlorite. Additionally, 17% EDTA (Cerkamed Medical Company, Stalowa, Poland) was applied to the root canals to remove the smear layer. After thorough irrigation, the root canals were dried. They were then obturated with gutta-percha cones (Meta Biomed Co., Seoul, Korea) and AH-26 root canal sealer (Dentsply, Tulsa, OK, USA) using the lateral condensation technique. Finally, the specimens were stored for 72 hours at 37°C.

Post space preparation

Root canal fillings were removed partially with a hot instrument. The post space was prepared using Peeso remears #1 to 3 (Mani Inc, Tachigiken, Japan) up to 12 mm. The root canals were rinsed thoroughly, cleaned with alcohol, and dried using size 40 paper cones.

Grouping and surface treatment

Teeth were randomly assigned to two groups based on the matrix composition of the FRC posts (n = 28):

1) Epoxy resin-based posts (White Post DC; FGM Dental, Joinville, Brazil)

2) Bis-GMA resin-based posts (FRC Postec Plus; Ivoclar Vivadent, Zurich, Switzerland)

Table 1 provides a description of the FRCs and resin cement used in this study, including their manufacturers and compositions.

Within each post group, the samples were further divided into four subgroups based on the surface treatments applied (n = 7 each):

Subgroup A (70% Ethanol): FRC posts were cleaned with a gauze that was soaked in 70% ethanol. Then, they were gently air-dried for 30 seconds. This group was considered as the control group.

Subgroup B (Air abrasion and silanization): Posts were subjected to air abrasion with 50 μ m aluminum oxide particles for 10 s. The posts were held perpendicular to the incoming particle stream, 20 mm from the device tip, using a pressure of 2 bar. Then, the post surface was silanized using a silane coupling agent (Ultradent, Utah, USA).

Table 1. Fiber-reinforced composite (FRC) posts and cement used in the present study

Material	Commercial name	Manufacturer	Composition
Resin	G-CEM	GC Corporation, Tokyo,	Paste A: fluoroaluminosilicate glass, initiator, urethane dimethacylate (UDMS), dimethacrylate, pigments, silicon dioxide, and inhibitor
cement	Linkace	Japan	Paste B: silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor
Fiber glass post	Whitepost DC	FGM Dental, Joinville, Brazil	Glass fiber, epoxy resin, inorganic filler, silane, and polymerization promoters
Fiber glass	FRC Postec	lvoclar Vivadent, Zurich,	E-glass fibers, Dimethacrylates (ethoxylated bisphenol A dimethacrylate, Bis-GMA, 1,4-butanediol dimethacrylate), and ytterbium fluoride fillers
post	Plus	Switzerland	

Subgroup C (35% H₃PO₄): Posts were etched with 35% H₃PO₄ (Ultraetch, Ultradent, USA) for 15 s, followed by rinsing with water for 30 seconds and gentle air-drying.

Subgroup D (24% H_2O_2): H_2O_2 was applied to the FRC surface using a micro brush for 5 minutes, followed by rinsing with water for 30 seconds and gentle air-drying.

FRC cementation

To cement the FRC posts, the root canals were filled with self-adhesive cement (G-Cem LinkAce; GC Corp, Tokyo, Japan), and posts were slowly inserted by finger pressure. Excess cement was removed with an explorer. The cement was light-cured for 40 seconds with the light-emitting diode device (JR-CL37, JERRY Co., China) with a light intensity of 1200 mW/cm2 according to the manufacturer's protocol. The samples were incubated in a 37° C water bath for 24 hours. Then, they were thermocycled for 1000 cycles between 5 to 55°C with a dwell time of 30 seconds.

Push-out test

Each specimen was sectioned perpendicular to the root axis using a cutting machine, creating three 2 mm sections from apical, middle, and cervical thirds. The push-out test was performed with a universal testing machine (Santam, STM 20, Tehran, Iran) at a crosshead speed of 0.5 mm/min. The push-out bond strength values, expressed in MPa, were calculated by dividing the applied load at specimen failure (N) by the bonded area (mm²). The push-out bond strength was then compared within each root canal section between the different matrix types and surface treatment groups.

Statistical analysis

Two-way ANOVA was run in each root canal section to compare the push-out bond strength of the two post types pre-treated with different methods. Tukey post hoc test was used for pairwise comparisons. Statistical tests were performed using IBM SPSS 26.0 software (IBM, Armonk, NY, USA) and a significance level of 0.05 was set for all analyses.

Results

Table 2 presents the push-out bond strength (MPa) of different posts treated with various methods in the cervical and apical thirds. In both sections, the two-way ANOVA revealed a significant difference in bond strength between the different treatment methods (P < 0.05; Table 2), but the post type had no significant effect on bond strength (P > 0.05). The interaction between the two factors also was not significant (P > 0.05).

 Table 2. Mean± standard deviation (SD) of push-out bond strength (MPa) of BIS-GMA- and epoxy resin-based FRCs with different treatment techniques in the cervical and apical thirds

Surface treatment	Cervical			Apical		
	Epoxy resin	Bis-GMA	Total	Epoxy resin	Bis-GMA	Total
70% ethanol	25.03±1.76	24.63±2.52	24.83±2.26 ^a	5.67±1.23	5.92 ± 1.32	5.79±1.33ª
Air abrasion+ salinization	27.71±1.28	27.55±1.44	27.63±1.41 ^b	7.44±0.63	6.93 ± 1.18	7.18±1.01 ^b
35% H3PO4	25.26±2.83	25.71±1.77	25.49±2.45 ^a	5.54±1.03	5.90 ± 1.05	5.72±1.09ª
24% H2O2	25.89±2.01	25.92±1.66	25.91±1.91 ^{ab}	5.82±0.75	6.18 ± 0.84	6.00±0.84ª
The effect of post type	P=0.972			P=0.700		
The effect of surface treatment	P=0.008*			P=0.003*		
Interaction	P=0.960			P=0.685		

Data were analyzed by two-way analysis of variance and Tukey test.

* Statistically significant differences were noted at P<0.05.

In each column, different lowercase superscript letters denote statistically significant differences between surface treatments at P<0.05.

Surface treatment	Middle	Middle				
	Epoxy resin	Bis-GMA posts	P value			
70% ethanol	15.8 ± 1.26 ^{ab}	13.39±1.41 ª	0.009*			
Air abrasion+ salinization	16.82 ± 0.87 ª	16.47±0.73 ^b	0.465			
35% H3PO4	15.99 ± 1.17 ^{ab}	15.58 ± 1.70 ^b	0.634			
24% H2O2	14.50±1.22 b	16.39 ± 1.31 ^b	0.024*			
P-value	0.015*	0.002*				

 Table 3. Mean± standard deviation of push-out bond strength (MPa) of BIS-GMA- and epoxy resin-based FRCs with different treatment techniques in the middle third

Data were analyzed by two-way analysis of variance and Tukey test.

* Statistically significant differences were noted at P<0.05.

In each column, different lowercase superscript letters denote statistically significant differences between surface treatments at P<0.05.

In the middle section, the two-way ANOVA indicated a significant interaction between the two factors (P = 0.001). Therefore, subsequent analysis was done by one-way ANOVA and an independent-samples t-test, as presented in Table 3.

In the cervical section, post-hoc analysis using the Tukey test showed that the bond strength of posts pretreated with air abrasion and silanization (27.63 ± 1.41 MPa) was significantly higher than that of samples pretreated with 70% ethanol (24.83 ± 2.26 MPa) and 35% H_3PO_4 (25.49 ± 2.45 MPa) (P < 0.05; Table 2). The bond strength of the 24% H_2O_2 -treated group was similar to that of the other groups (P > 0.05).

In the apical section, pairwise comparisons revealed that posts pre-treated with air abrasion and silanization (7.18 \pm 1.01 MPa) exhibited significantly higher bond strength compared to samples treated with 70% ethanol, 35% H₃PO₄, and 24% H₂O₂ (P < 0.05; Table 2).

Table 3 shows the push-out bond strength of the samples in the middle section. ANOVA revealed a significant difference between surface pre-treatments in both types of FRC posts (P<0.05; Table 3). In the epoxy resin post, samples treated with air abrasion and silanization (16.82 \pm 0.87 MPa) had significantly higher bond strength compared to those treated with 24% H₂O₂ (14.50 \pm 1.22 MPa) (P<0.05). The other groups had comparable bond strengths (P > 0.05; Table 3). In the Bis-GMA-based posts, the bond strengths of the samples treated with air abrasion and silanization, 35% H₃PO₄, and 24% H₂O₂ were significantly higher than that of the control group (P < 0.05; Table 3).

In terms of differences between post types, the bond strength of epoxy resin posts was significantly higher than that of Bis-GMA posts in the middle section when treated with 70% ethanol (P = 0.009). However, epoxy resin posts showed significantly lower bond strength than Bis-GMA posts when treated with 24% H_2O_2 (P = 0.024). When the posts were treated with air abrasion or 35% H_3PO_4 , there was no significant difference in bond strength between the two post types.

Discussion

The current study assessed the push-out bond strength of Bis-GMA-FRCs and epoxy resin-FRCs in the cervical, middle, and apical root canal thirds, following various surface treatments. The treatments included cleaning with 70% ethanol, air abrasion and silanization, and applying 35% H_3PO_4 or 24% H_2O_2 . The specimens were subjected to thermal aging to improve the reliability of the findings. It is believed that the thermal aging leads to a hydrolytic breakdown in the resin matrix, increasing adhesive failures between the FRC posts and dentin (13-15).

In the present study, the push-out bond strength of the two post types was comparable in the coronal and apical sections. The bond strength of the two post types was also similar in the middle section when surface treatment was performed with air abrasion and silanization, or 35% H₃PO₄. This suggests that proper mechanical and chemical surface treatments might be more important than the matrix resin in determining the bond strength.

In the middle section, the bond strength of epoxybased posts was significantly higher than that of Bis-GMA posts when treated with 70% ethanol. On the other hand, epoxy resin posts showed significantly lower bond strength than Bis-GMA posts when treated with 24% H_2O_2 . Other studies have also indicated that the post matrix can affect the push-out bond strength of the FRC posts (8, 12). Alnaqbi et al. (8) reported that epoxybased prefabricated posts (Rely X) had a significantly higher push-out bond strength than Bis-GMA-based posts (FRC Postec Plus) in the middle section when they were cleaned with alcohol.

In the cervical and apical sections, air abrasion and silanization demonstrated significantly higher bond strength than other groups. The only exception was 24% H_2O_2 treatment in the cervical section which showed comparable bond strength to air abrasion and silanization. In the middle section, air abrasion followed

by silanization exhibited the highest push-out bond strength, although the difference was only significant to the 24% H₂O₂ treatment in the epoxy resin posts, and the control treatment in the Bis-GMA-based posts. Therefore, air abrasion and silanization appear to be the most effective surface treatment techniques for FRC posts. Silanization increases the chemical reactivity of the surface by forming siloxane bonds between the fiberglass silica and the composite resin or the resin cement (16). Several studies have reported that silanization enhances the bond strength between FRC posts and resin cement or composite resin cores (17-20).

In the present study, silanization was performed after sandblasting. Mechanical or chemical treatments are essential to alter or remove the superficial layer and expose the internal glass fibers before silane application. This is because the FRC post matrix is highly cross-linked and lacks free functional groups needed for interacting with silane (21). In a systematic review, Moraes et al. (22) concluded that pretreating the post surface to expose glass fibers before applying silane significantly improves the post retention. These treatments include sandblasting or chemical etching (20). Aluminum particles create irregularities on the FRC surface, increase surface roughness and micromechanical interlocking, and expose the glass fibers (23, 24). Several studies have identified air abrasion as the preferred method for enhancing silanization efficiency (15, 24, 25). Karunakaran et al. (10) observed that sandblasting combined with silanization significantly increased the bond strength of both glass and guartz fiber posts to all root sections compared to etching with 4% titanium tetrafluoride plus silanization, silanization alone, and the control group.

Surface treatment with 35% H₃PO₄ resulted in bond strengths similar to the control group in the cervical and apical sections and the middle section when applied to epoxy resin posts. In contrast, some studies have shown that 35% H₃PO₄ enhances the bond strength of epoxy glass fiber posts to root dentin when applied for 15 seconds compared to no treatment (26, 27). Güler et al. (28) reported that etching FRC Postec Plus with 35% H₃PO₄ for 3 minutes improved push-out bond strength compared to the control group, but no significant difference was found with shorter durations (30, 60, and 120 s). This suggests that the short etching time used in the present study (15 seconds) may account for the nonsignificant difference in bond strength with the control group. The outcomes of this study are consistent with those of Albashaireh et al. (29), who found that treating the post surface with $36\% H_3PO_4$ for 15 seconds before cementation did not improve post retention, likely because the removal of the top layer of epoxy resin was minimal, resulting in weak micro-mechanical retention.

The push-out bond strength of the samples treated with 24% H₂O₂ was comparable with the control group at all root sections. The only exception was observed in the middle section of Bis-GMA posts, where H₂O₂ treatment caused significantly higher bond strength than the control group. At all root sections, bond strength in the 24% H₂O₂ and 35% H₃PO₄ subgroups were comparable. H₂O₂ partially dissolves the FRC matrix through a substrate oxidation mechanism (30). Daneshkazemi et al. (19) observed that treating epoxybased White Posts with H₂O₂ resulted in bond strengths comparable to H₃PO₄. Their SEM results showed that H₂O₂-treated posts had a roughened porous surface, which may retain residual H₂O₂ and oxygen byproducts, potentially weakening the bond.

The present study had limitations due to its in vitro nature, as it could not account for different biting forces or thermal variations in the oral environment. Further studies are needed to investigate the effect of different types of cement on the push-out bond strength of FRC posts to root dentin using various surface treatment techniques.

Conclusions

Based on the findings of this study, the following conclusions are drawn:

- 1- The highest push-out bond strength in the cervical, middle, and apical sections was observed in subgroups treated with air abrasion and silanization. Therefore, air abrasion and silanization could be suggested as the optimal surface treatment strategy to improve the bond strength of FRC posts to resin cement.
- 2- Epoxy resin-based and Bis-GMA-based FRC posts had a similar push-out bond strength in the coronal and apical sections of the root canal. Additionally, their bond strengths in the middle section were comparable when the post surfaces were treated with air abrasion and silanization, or 35% H₃PO₄. This suggests that proper mechanical and chemical surface treatments might be more important than matrix resin in determining the bond strength of FRC posts.

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Conflict of interest

The authors declare no competing interests.

Authors contributions

M.H., E.B.N., M.J.M., and N.K. conceptualized the main idea, designed, supervised the study, and reviewed the manuscript. M.H and S.M. acquired data, conducted data analysis, and wrote the original draft. All authors read and approved the final manuscript.

Ethics approval

The study protocol was approved by the ethics committee of Mashhad University of Medical Sciences (IR.MUMS.DENTISTRY.REC.1398.033).

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