



Original Article

Effect of various chemical post surface treatments on the microtensile bond strength of fiber posts

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Abstract

Background/Objectives To evaluate the microtensile bond strength (μ TBS) between epoxy-based or dimethacrylate-based fiber posts and resin cores after various post surface treatments.

Materials and methods Eighty DT light (DT) and forty FRC Postec Plus (FRC) posts were divided into 8 groups; group 1: silanization (S), group 2: silanization and application of bonding agent (SB), groups 3, 5, 7: etching with 37% phosphoric acid for 1 minute (P), 30% hydrogen peroxide for 10 minutes (H30), 35% hydrogen peroxide for 1 minute (H35), respectively, followed by S, groups 4, 6, 8: etched as in groups 3, 5, and 7, but followed by SB. The cores were built up with Multicore flow. Twenty stick-shaped specimens per group were randomly selected for the μ TBS test with a universal testing machine. The failure modes were classified by stereomicroscope. The post surfaces after chemical treatment and cross-sectioned specimens of the fiber posts were examined by scanning electron microscope. The data were analyzed with two-way analysis of variances and Tukey's test.

Results Types of resin matrix and surface treatment and the interaction between them significantly affected μ TBS ($p < 0.05$). The DT groups showed significantly higher bond strength than those of the FRC groups. Post surface treatment with SB, phosphoric acid or hydrogen peroxide followed by S or SB significantly increased the μ TBS compared to S without other surface treatments.

Conclusion Post surface treatments and types of resin matrix of the fiber posts affected on the μ TBS between fiber posts and resin composite cores.

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Keywords: dimethacrylate; epoxy; fiber post; microtensile bond strength; surface treatment

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Introduction

Fiber-reinforced composite posts are mainly composed of fiber and matrix. The fiber is composed of carbon, glass, or quartz, whereas the matrix is mostly composed of cross-linked polymers such as epoxy resin, dimethacrylate resin, or methacrylate resin. However, due to their highly cross-linked and smooth surfaces, the main problem when using these fiber posts is debonding between fiber posts and resin composite cores (Ferrari et al., 2000; Cagidiaco et al., 2008). Studies have demonstrated that post surface treatments such as silanization, sandblasting, or chemical treatment can improve the bond strength between fiber posts and resin composite cores (Monticelli et al., 2008; Mosharraf and Baghaei Yazdi, 2012; Elsaka, 2013; Pyun et al., 2016).

Silanization can increase post surface wettability and promote chemical bonding between the fibers of the post and resin composite core. Silanes are linear molecules that have a functional group at each end. The organic functional group, e.g. vinyl, allyl, amino, and isocyanato, can polymerize with an organic matrix such as the methacrylate in resin composite restorative materials; and the inorganic functional alkoxy group can react with the glass or quartz fiber hydroxyl groups (Matinlinna et al., 2004). As a result, the bond strength between a fiber post and resin composite core is increased (Aksornmuang et al., 2004). However, the effect of silanization is still controversial. Normally the fibers of the post are covered with matrix, thus they cannot directly react with silane. Accordingly, the bond strength between silanized fiber posts and resin composite cores did not increase (Perdigao et al., 2006; Bitter et al., 2007; Machado et al., 2015).

Bonding agents are mainly composed of methacrylate monomer that are similar to that of resin composite cores. Bonding agents improve the bond strength between a fiber post and resin composite core by promoting micromechanical retention and/or

chemical interaction between the fiber post and resin composite core (Ounsi et al., 2009). Moreover, the application of silane and bonding agents increased the bond strength between the fiber post and resin composite core (Ferrari et al., 2006). However, Ferrari et al. found no difference in bond strength between with and without using a bonding agent (Ferrari et al., 2006).

Hydrofluoric acid, phosphoric acid, and hydrogen peroxide have been recommended for treating fiber posts to create post surface roughness and remove the resin matrix to expose the fibers before silanization (Vano et al., 2006; Sumitha et al., 2011; Mosharraf and Ranjbarian, 2013; Majeti et al., 2014; Sharma et al., 2014). Hydrogen peroxide and phosphoric acid were recommended as post surface treatments because they did not damage the exposed fiber surfaces (Menezes et al., 2011; Sumitha, et al., 2011; Guler et al., 2012; Menezes et al., 2014). However, hydrofluoric acid treatment resulted in the generation of crack lines on the fiber post.

The effect of post surface treatment on post-core bond strength has been widely studied in epoxy-based fiber posts; however, posts with a different type of resin matrix, dimethacrylate-based fiber posts, are less well investigated. Although both dimethacrylate-based and epoxy-based fiber posts are cross-linked fiber posts, dimethacrylate-based fiber posts are composed of methacrylate functional groups that can also be found in resin composite cores. Thus, the effect of surface treatment of dimethacrylate-based fiber posts may be different from those of epoxy-based fiber posts. The null hypothesis was that the types of post matrix and post surface treatment would not affect the bond strength between the fiber post and the resin composite core. The aim of this study was to evaluate the effect of hydrogen peroxide or phosphoric acid treatment followed by silanization or silanization and application of bonding agent on the microtensile bond

Table 1 Experimental groups for μ TBS test in this study.

Group	Chemical surface treatment	DT light post	FRC Postec Plus
1 (S)	Silanization	DT-S	FRC-S
2 (SB)	Silanization + bonding agent	DT-SB	FRC-SB
3 (PS)	37% H_3PO_4 for 1 minute + silanization	DT-PS	FRC-PS
4 (PSB)	37% H_3PO_4 for 1 minute + silanization + bonding agent	DT-PSB	FRC-PSB
5 (H30S)	30% H_2O_2 for 10 minutes + silanization	DT-H30S	FRC-H30S
6 (H30SB)	30% H_2O_2 for 10 minutes + silanization + bonding agent	DT-H30SB	FRC-H30SB
7 (H35S)	35% H_2O_2 for 1 minutes + silanization	DT-H35S	FRC-H35S
8 (H35SB)	35% H_2O_2 for 1 minutes + silanization + bonding agent	DT-H35SB	FRC-H35SB

strength (μ TBS) between two types of fiber posts, epoxy-based and dimethacrylate-based fiber posts, and resin composite cores to determine the appropriate post surface treatment for each type of fiber post.

Materials and Methods

Eighty DT Light Post Illusion X-RO (DT) size 3 and 40 FRC Postec Plus (FRC) size 3 with a coronal diameter of 2.2 and 2 mm, respectively, were used for the μ TBS test. Only the parallel portion of each fiber post (DT = 5 mm, FRC = 10 mm) were used for specimen preparation. The posts were cleaned with deionized water in an ultrasonic cleaner for 2 minutes and air-dried. The posts were divided into 8 groups (DT n = 10, FRC n = 5) according to the following surface treatments in table 1. The composition and application of the materials used in this study are shown in table 2.

The core build-up procedure was performed according to Goracci et al. (Goracci et al., 2005). Each post was placed upright on a glass slide and secured with cyanoacrylate. A cylindrical plastic matrix was placed around the parallel portion of the post and adjusted so that the post would be exactly in the center of the matrix. The core material (Multicore Flow) was injected inside the matrix until it was full. A light emitting diode (LED) curing light with an output of 1,200 mW/cm² (Elipar S10, 3M ESPE, Minnesota, USA) was used to cure along the post, each side of the matrix, and the glass-slide contacted surface for 40 seconds each. The post-core units were kept in a dry environment for 24 hours at 37°C. Each post-core unit was then mounted in a cutting machine (Isomet 1000, Buehler Ltd., Illinois, USA) and sectioned by a water-cooled diamond blade to generate approximately thirty 1 x 1 mm² cross-sectional stick-shaped specimens per group (Fig 1). The specimens

were measured using a digital vernier caliper (Mitutoyo, Tokyo, Japan). The prepared specimens were stored in a dry environment for 24 hours at 37°C before the microtensile bond strength test.

Microtensile bond strength test

The specimens were observed with a stereomicroscope (SZ61TR, Olympus, Tokyo, Japan) at 40X magnification to find specimens without voids or bubbles. Twenty specimens per group were randomly

selected for testing. The specimens were attached to the two free sliding components of a jig with cyanoacrylate (Model repair II blue, Sankin Industry, Tokyo, Japan) (Fig 2). The jig was mounted on a universal testing machine (EZ-S, Shimadzu, Kyoto, Japan) and loaded in tension at a crosshead speed of 0.5 mm/min until failure occurred. The microtensile bond strength (MPa) was computed by dividing the failure load (N) by the bond surface area (A). Due to the curved surface of the post, the area was

Table 2 Material composition and applications used in this study

Material	Batch number	Composition	Application
DT Light Post Illusion X-RO (RTD st. Egrevé Grenoble, France)	238361401	Epoxy resin matrix (40 vol%) Quartz fibers (60 vol%)	
FRC Postec Plus (Ivoclar Vivadent, Schaan, Liechtenstein)	R83913	Dimethacrylates (21%) Ytterbium fluoride (9%) Glass fibres (70%) Catalysts and stabilizers (< 0.5%)	
MultiCore Flow (Ivoclar Vivadent, Schaan, Liechtenstein)	T02659	Matrix: bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate Fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, highly dispersed silicon dioxide. Particle size 0.04-25 µm. Total volume of fillers (47%)	Dispensed from the automix syringe and light-cured
Monobond-S (Ivoclar Vivadent, Schaan, Liechtenstein)	R53818	1% 3-methacryloxypropyltrimethoxysilane (3-MPS), ethanol/water-based solvent	Applied on post surface for 60 seconds and air-dried
Excite F DSC (Ivoclar Vivadent, Schaan, Liechtenstein)	S29202	Adhesive: phosphonic acid acrylate, dimethacrylates, hydroxyethyl methacrylate (72%) highly dispersed silicon dioxide (0.5%) ethanol (24.5%) catalysts, stabilizers, fluoride (3.0%) Applicator: coated with initiators	Applied on post surface for 10 seconds, air-dried for 5 seconds and light-cured for 20 seconds
Eco-Etch (Ivoclar Vivadent, Schaan, Liechtenstein)	R63105	37% Phosphoric acid	Applied on post surface for 60 seconds, rinsed with deionized water for 2 minutes and air-dried
Hydrogen peroxide 30% (Carlo Erba, Milano, Italy)	1M736291N	Hydrogen peroxide 30% w/w	Immersed post in solution for 10 minutes, rinsed with deionized water for 2 minutes and air-dried
Hydrogen peroxide 35% (Vidhyasom, Bangkok, Thailand)	000255	Hydrogen peroxide 35% w/w	Immersed post in solution for 60 seconds, rinsed with deionized water for 2 minutes and air-dried

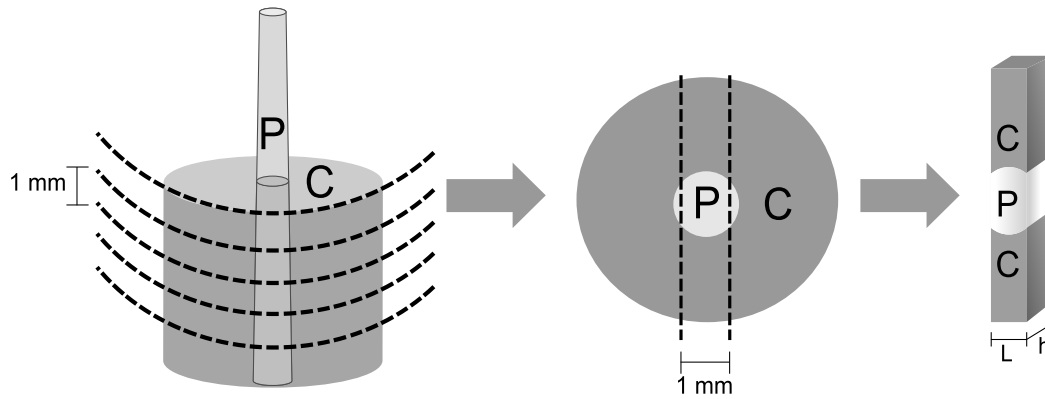


Fig. 1 Schematic of the cutting lines used in the preparing stick-shaped specimens for the microtensile bond strength test (P: post, C: core, L: width, h: thickness)

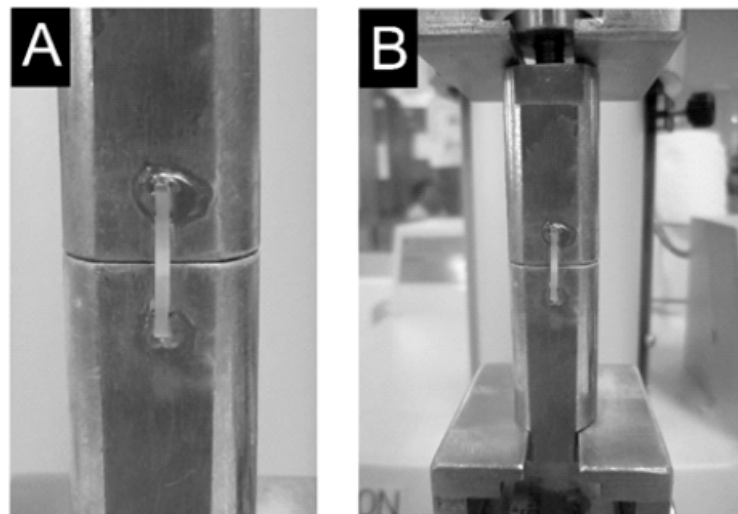


Fig. 2 A specimen was attached to two free sliding components of the jig (A). The jig was mounted on the universal testing machine (B).

calculated using the formula (Valandro et al., 2006) : $A = 2r \arcsin (L/2r) \times h$, where r, L, and h are the post diameter, width and thickness, respectively. The fractured specimens were evaluated using a stereomicroscope at 40X magnification and classified into three patterns; adhesive failure, cohesive failure, or mixed failure.

Scanning electron microscope (SEM) observation

An additional 4 fiber posts of each post type were cleaned with deionized water in an ultrasonic cleaner for 2 minutes, air-dried, and one each received

surface treatments of: non-etched, etched with 37% phosphoric acid for 1 minute, etched with 30% hydrogen peroxide for 10 minutes and etched with 35% hydrogen peroxide for 1 minute. The specimens were rinsed with deionized water using an ultrasonic cleanser for 5 minutes and air-dried for 30 seconds. Half of Each post was mounted on a metallic stub, sputter coated with gold. The cross-sectional and the surface morphology of one of each post was investigated under an SEM (JSM 5410LV, JEOL, Tokyo, Japan) set at 15 kV and 500x magnification.

Table 3 Results of two-way analysis of variance

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	37938.868a	15	2529.258	130.941	.000
Intercept	614914.750	1	614914.750	31834.526	.000
Matrix	33832.510	1	33832.510	1751.530	.000
Treatment	3813.492	7	544.785	28.204	.000
Matrix*	292.866	7	41.838	2.166	.037
Treatment					
Error	5872.055	304	19.316		
Total	658725.673	320			
Corrected Total	43810.923	319			

a. R Squared = .866 (Adjusted R Squared = .859)

Statistical analysis

The μ TBS data were analyzed by SPSS version 17.0 (SPSS Inc, Chicago, Illinois, USA). The normality and variance of the data were determined using the Kolmogorov–Smirnov and Levene’s tests, respectively. The data were normally distributed, thus, two-way analysis of variance was used to evaluate the differences in the types of resin matrix and types of post surface treatment. Because the variance of data was not significantly different, the Tukey’s test was used as a post hoc test at 95% confidence level.

Results

The μ TBS of each group were shown in figure 3. Two-way analysis of variance indicated that the types of resin matrix, the types of post surface treatment, and the interaction between them significantly affected on μ TBS ($p < 0.05$) (Table 3). The DT groups all had significantly higher mean μ TBS (47.58–57.11 MPa) than those of the FRC groups (24.19–37.43 MPa) ($p < 0.05$).

Within the same type of resin matrix groups, the μ TBS of the S group was the lowest, significantly different from the μ TBS of other groups ($p < 0.05$), except for the DT–H30S group. Analysis of the μ TBS of the silanization groups (S, PS, H30S, and H35S groups) indicated that the etching fiber posts with phosphoric acid (PS) and H_2O_2 (H30S, H35S) showed significantly higher μ TBS than that of the non-etched fiber posts (S) within the same type of resin matrix group, except for the DT–H30S group. Conversely, the application of silane and bonding agent (SB, PSB, H30SB, H35SB groups) resulted in μ TBS between non-etched and etched DT posts that were not significantly different, whereas the μ TBS of FRC–H30SB and FRC–H35SB groups were significantly higher than that of the FRC–SB group ($p < 0.05$). Moreover, there was no significant difference in μ TBS when the different types of etching agents; phosphoric acid or hydrogen peroxide, were applied and followed by either silane or silane and bonding agent ($p > 0.05$). In both resin matrix types,

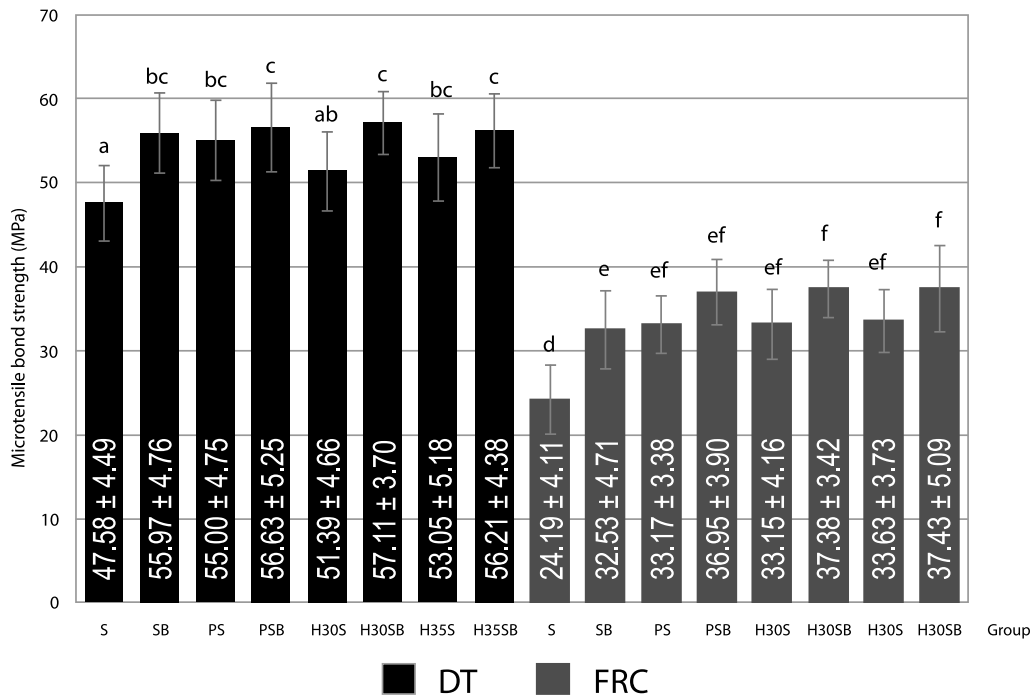


Fig. 3 Microtensile bond strength of the surface treatment groups (Mean ± SD, MPa). The same letter indicates no significant difference between the groups ($p > 0.05$).

the application of silane and bonding agent resulted in significantly higher μ TBS compared with the application of silane alone ($p < 0.05$). Etching post surface with subsequent application of silane or silane followed by bonding agent resulted in similar μ TBS, except for the DT-H30SB group that showed significantly higher μ TBS than the DT-H30S group ($p < 0.05$) (Fig 3).

The distribution of the failure modes were shown in figure 4. In the DT groups, all the specimens in the S, PS, H30S, and H35S groups fractured at the post and core interface, i.e. adhesive failure. Cohesive failure in the fiber post was not found in any DT group. In contrast, cohesive failure was the most common failure mode found in the FRC groups except for the FRC-S group, where only adhesive failure was found. Mixed failure, failure at both the post-core interface and in the resin composite core, was seen in the SB, PSB, H30SB, and H35SB DT groups and the etched FRC groups.

The effects of etching agent on the surface morphology of each group were illustrated in the SEM images in figures 5. The non-etched DT post surface showed rough and irregularity surface, whereas the non-etched FRC post surface was smooth. There were non-fibers exposed in either group. After etching surface with phosphoric acid and H_2O_2 , the dissolved DT and FRC resin matrices exhibited a rough surface with some exposed fibers (Fig 5). Moreover, application of 30% hydrogen peroxide for 10 minutes resulted in greater dissolution than the application of either 35% hydrogen peroxide or 37% phosphoric acid for 1 minute. Thus, there were more fibers exposed and rougher surfaces after treatment with 30% hydrogen peroxide for 10 minutes in both post types. The SEM image of a cross-sectioned FRC post showed the presence of voids defect between the fibers, whereas a tight junction was observed between the fibers and the resin matrix of the DT posts (Fig 6).

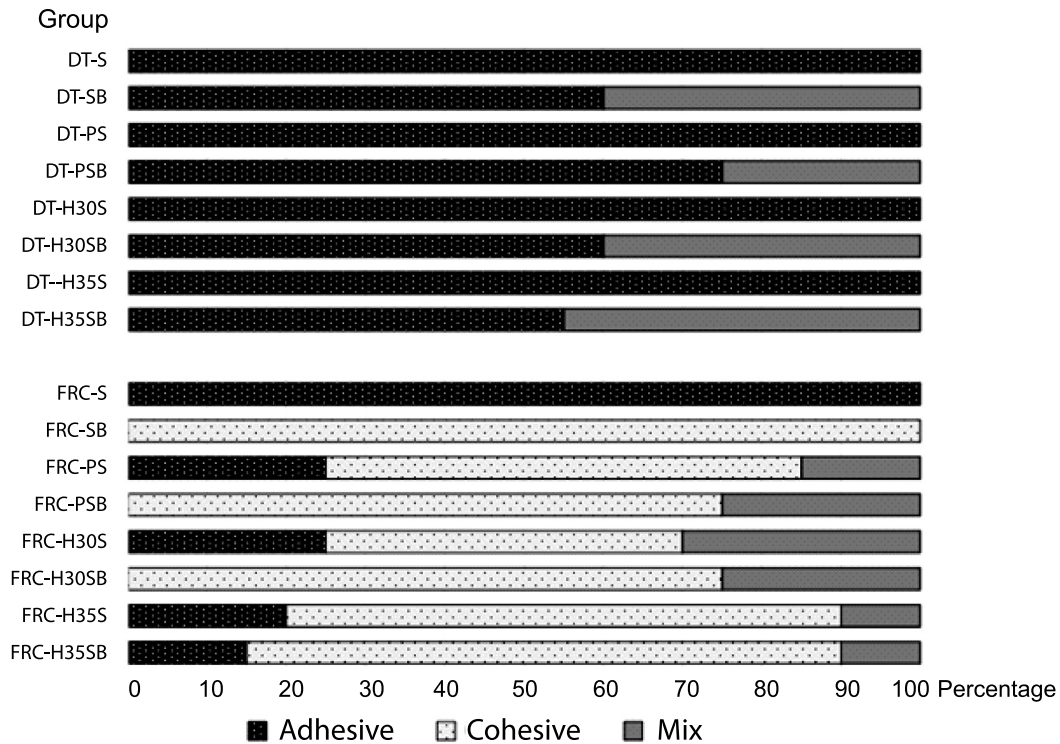


Fig. 4 Distribution of the failure modes in each group (%)

Discussion

The DT and FRC posts were used in our study because of their high fatigue resistance and good structural integrity (Grandini et al., 2005). Resin matrixes of these two posts were different which may result in the microtensile bond strength (μ TBS) between fiber posts and resin cores after various post surface treatments. From this study, the types of resin matrix and types of post surface treatment significantly affected μ TBS. Thus, the null hypothesis was rejected. In case of only silane application, the epoxy-based fiber post, DT posts (DT-S) showed significantly higher μ TBS than that of the dimethacrylate-based fiber post, FRC posts (FRC-S). This may be the result of the different surface irregularity of the post from the manufacturers as shown in figure 5. The roughness of the post surface plays a positive role on μ TBS because the flowable resin composite core can more interpenetrate into the post surface resulting in the mechanical interlocking between them (Le Bell et al., 2004; Mannocci et al., 2005; Vidhya et al., 2010).

Etching post surface is an important process to enhance the bond strength between both types of fiber posts and a resin composite core. The DT and FRC posts are composed of a highly cross-linked polymer matrix that makes them difficult to bond with a resin composite core. Etching post surface with either phosphoric acid or hydrogen peroxide increased the μ TBS between the fiber posts and resin composite core because these treatments dissolved the resin matrix to roughen the post surface and exposed the post fibers for more effective silanization. Since phosphoric acid releases hydrogen atoms and hydrolyzes the ester groups in the resin matrix (Prakki et al., 2005), whereas hydrogen peroxide oxidizes the carbon-carbon double bonds at the post surface (Papacchini et al., 2007). Vano et al (Vano et al., 2006) reported that applying 24% by volume hydrogen peroxide for 10 minutes on the post surface significantly increased the bond strength between the fiber post and resin composite core. The amount of hydrogen peroxide in 24% hydrogen peroxide is

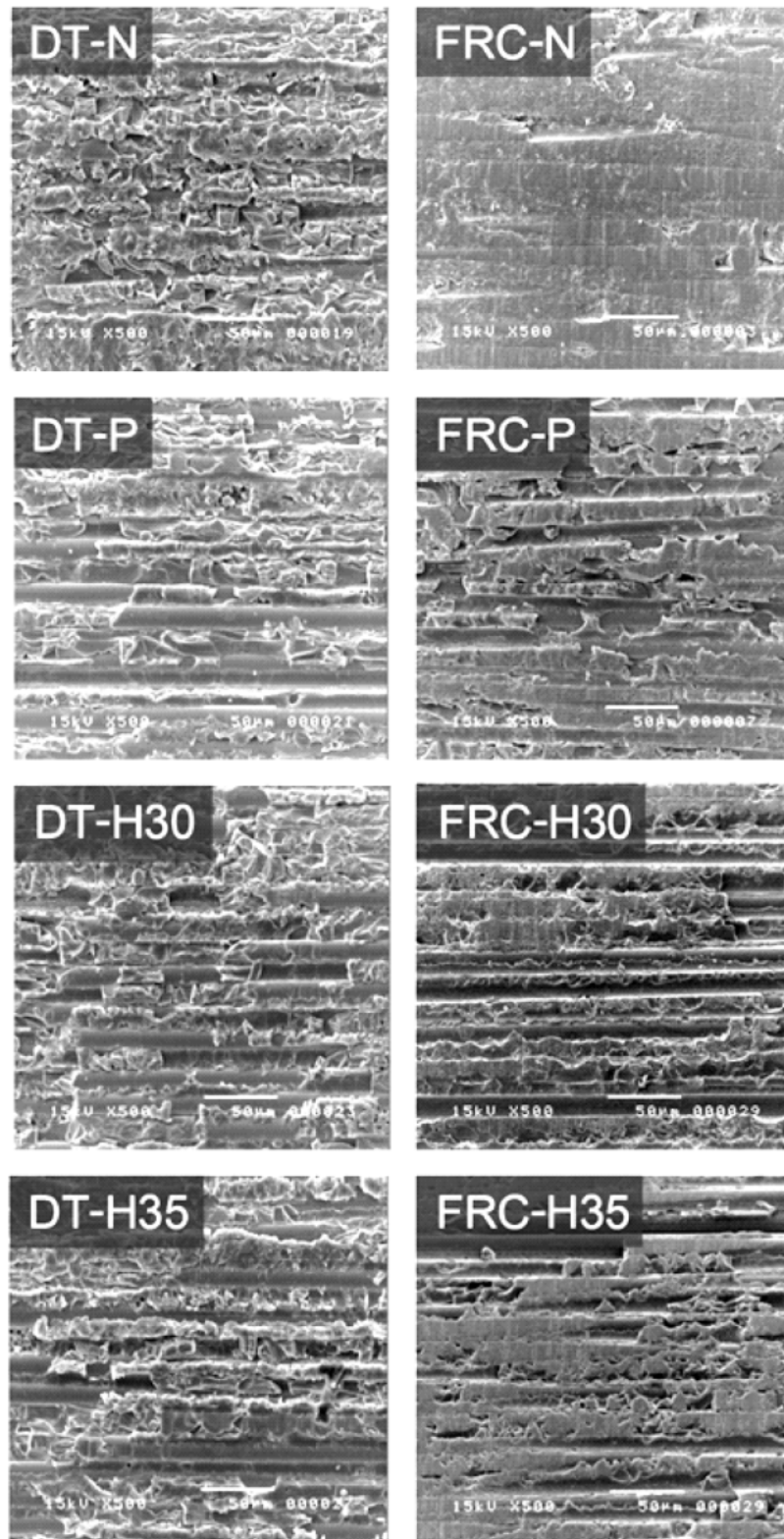


Fig. 5 SEM images of the DT light post (DT) and FRC Postec Plus (FRC)'s surfaces treated with none (N), 37% phosphoric acid for 1 minute (P), 30% hydrogen peroxide for 10 minutes (H30) and 35% hydrogen peroxide for 1 minute (H35). Stars indicated the exposed fibers.

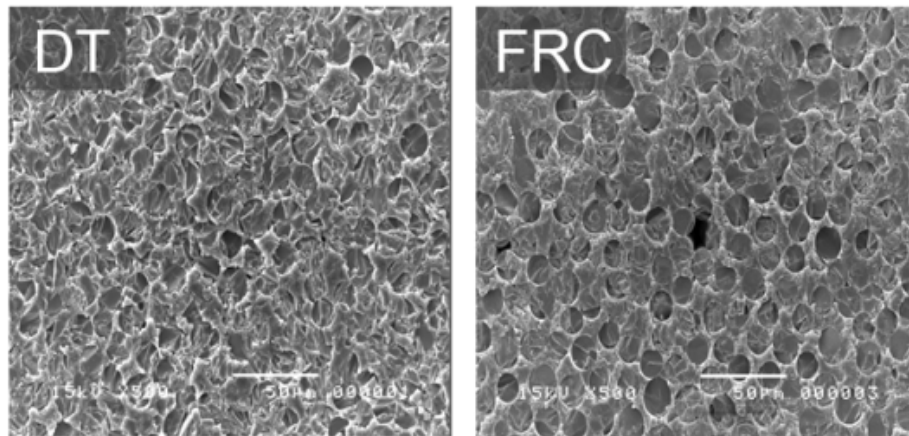


Fig. 6 SEM images of the DT light post (DT) and FRC Postec Plus (FRC) in cross section. The void between fibers of FRC is marked by the arrow.

approximately as the same concentration as that in 30% hydrogen peroxide by weight. Thus, we used 30% hydrogen peroxide by weight as the positive control group in our study. The SEM results demonstrated that pre-treatment with the etching agents resulted in a rougher surface in the both DT and FRC groups. This roughness likely increased the surface area for mechanical interlock.

Silane application enhances the bond between a fiber post and resin composite core by promoting chemical bonding between the fiber and resin composite core and the wettability of the post surface (Matinlinna et al., 2004). The Monobond-S silane used in our study is composed of 3-methacryloxypropyl-trimethoxysilane, which has two functional groups: methacrylate-side functional groups that react with resin composite, and alkoxy-side functional groups that react with post fibers. From the result, it was found that the group which only treated with silane coupling agent (DT-S and FRC-S) show significant lowest μ TBS in each resin matrix group with total adhesive failure mode. That may be result from silane could not form the siloxane bond to the unexposed quartz or glass fiber. Silanes could increase post surface wettability by enhance the flow of resin composite core materials, resulting in more intimate contact between the post

surface and core by van der Waals' forces (Marshall et al., 2010). But only the low viscosity of silane could not substitute the great space area which the resin matrix have been removed. The SEM investigation revealed more exposed the fiber after etching fiber posts with 30% hydrogen peroxide by weight for 10 minutes which might create the unfilled area with resin composite. Consequently, the μ TBS did not significant differ from etching with 35% hydrogen peroxide in water by weight for 1 minute. As shown in previous studies (Menezes et al., 2011; Menezes et al., 2014), the results of our study revealed that etching post surface improved μ TBS bond strength but different hydrogen peroxide concentrations and application times did not affect μ TBS. So the etching fiber post with 35% hydrogen peroxide for 1 minute is more practical and less clinical chair time than that with 30% hydrogen peroxide for 10 minutes.

Etching post surface with either phosphoric acid or hydrogen peroxide followed by silane or silane and bonding agent significantly increased the μ TBS of both types of resin matrix. The application of bonding agent also increased the μ TBS between fiber posts and resin composite cores. The effect of the bonding agent can be explained by the micromechanical retention from bonding agent penetration into the rough fiber

post surface, and the chemical bonding between the free radicals of the matrix of the fiber post and monomer of the bonding agent (Vidhya, et al., 2010). However, a previous study suggested that the greatest benefit of free radical polymerization bonding was found on the composite substrate during the first 24 hours after polymerization (Saunders, 1990). Therefore, the posts used in our study were unlikely to have free radicals available on their surfaces for polymerization with bonding agent. Moreover, the good wettability properties of a low-viscosity bonding agent also enhanced the bond strength (Mount, 1989).

Without etching post surface treatment, the application of silane and bonding agent showed higher bond strength than the application of silane alone. This may be due to the combined effects of both silane and bonding agent. This result was consistent with the results of previous studies (Ferrari et al., 2006; Ounsi, et al., 2009). However, after chemical post surface treatment, the application of silane and silane followed by bonding agent resulted in similar μ TBS. Therefore, it is not essential to apply bonding agent on a post surface that was previously treated with a chemical agent followed by silanization. Etching with chemical agents did not affect the μ TBS in the DT groups when post-treated with silane and bonding agent because the original roughness of the outer matrix promoted micromechanical interlocking with the bonding agents. In contrast, the FRC post surface was smooth; therefore etching with chemical agents was necessary to roughen the post surface.

Cohesive failure in the fiber posts were not detected in the DT groups, whereas it was the most common failure found in the FRC groups, indicating that the DT posts were stronger than the FRC posts. This may be because epoxy-based resin matrix has less stiffness compared with dimethacrylate-based

resin matrix (Ferrari et al., 2006). Thus, DT posts can endure tensile stress better than FRC posts. This may also be explained by the manufacturing process of endodontic posts. Our cross-section SEM images of an FRC post showed voids between the glass fibers of FRC post, whereas a tight junction was observed between the fibers and resin matrix of the DT post. These voids might initiate crack propagation in the FRC posts as reported in a previous study (Zicari et al., 2013).

The μ TBS test has been widely used to assess the bond strength between fiber posts and resin composite core because of its uniform stress distribution and because it allows the preparation of several specimens per post-core unit. Nevertheless, specimen preparation is difficult and the chance of premature failure is high (Valandro et al., 2006; Goracci et al., 2007). However, in our study we did not find premature failure after silane or silane and bonding agent application or etching and followed by silane or silane and bonding agent application. In addition, our pilot study, all the specimens in the non-etched FRC and without silane or bonding agent groups failed during specimen preparation. This implies that the dimethacrylate resin matrix of the FRC posts could not directly chemically bond with the resin composite cores. However, all specimens in the non-treated DT and without silane or bonding agent groups endured the specimen preparation process because a micromechanical bond existed between the resin composite core and rough post surface. Thus, the non-etched DT posts showed a higher μ TBS than the non-etched FRC posts.

A limitation of the present *in vitro* study was that silane, bonding agent, and resin composite core from only one manufacturer were evaluated. Further study should investigate the effects of thermocycling and water ageing on bond strength between fiber posts and resin composite cores.

Conclusion

The types of resin matrix and surface treatment affected the μ TBS between fiber posts and resin composite cores. The DT light posts showed significantly higher bond strength than that of the FRC Postec Plus posts. Etching with phosphoric acid or 35% hydrogen peroxide for 1 minute followed by application of either silane or silane and bonding agent improved the bond strength between both the epoxy-based and dimethacrylate-based fiber posts and resin composite core. The application of both silane and bonding agent significantly increase the bond strength between post and resin cement even though without etching treatment.

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