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ORIGINAL ARTICLE

Impact of different surface treatment methods on bond strength between fiber post and composite core material

Abdullah Alshahrani [a,*], Muthyib Albaqami [b], Zeyad Naji [b], Yazeed Al-Khunein [c], Khalid Alsubaie [d], Abdulaziz Alqahtani [a], Ahmad M. Al-Thobity [e]

[a] *Prosthetic Dental Sciences Department, College of Dentistry, King Saud University, Riyadh, Saudi Arabia*
[b] *College of Dentistry, King Saud University, Riyadh, Saudi Arabia*
[c] *Armed Forces Hospital, Dhahran, Saudi Arabia*
[d] *King Abdulaziz Dental Center, National Guard Health Affairs, Riyadh, Saudi Arabia*
[e] *Substitutive Dental Sciences Department, College of Dentistry, Imam Abdulrahman Bin Faisal University, Dammam, Saudi Arabia*

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KEYWORDS

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Hydrogen peroxide

Abstract *Purpose:* To assess the impact of different surface treatments on the push-out bond strength between fiber post and a composite resin core material.

Material and methods: Seventy-two glass-fiber posts were randomly assigned into six groups according to the method of surface treatment: Control (no treatment), silane, sandblasting, hydrofluoric acid, hydrogen peroxide, and hydrogen peroxide with sandblasting. Two posts from each group were inspected under a scanning electron microscope to assess the surface modifications and 10 posts were employed for the push-out test. Each post was placed vertically in the middle of a cylindrical putty matrix and a dual-cure composite resin material was applied for core build-up. Two discs of each specimen were cut using a low-speed diamond saw (total 120 discs). The push-out test was executed using a universal testing machine at a crosshead speed of 0.5 mm/min. Statistical analysis was performed using one-way ANOVA and Tukey's test ($p \leq 0.05$). The mode of failure of each disc was evaluated under SEM.

Results: The sandblasting and hydrofluoric acid groups presented significantly higher bond strength than control and hydrogen peroxide groups. The hydrogen peroxide groups exhibited

* Corresponding author at: Prosthetic Dental Sciences Department, College of Dentistry, King Saud university, P.O. Box: 60169, Riyadh 11545, Saudi Arabia.
E-mail address: asalshahrani@ksu.edu.sa (A. Alshahrani).
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significantly the lowest bond strength of all groups. There was no significant difference between the control and silane groups. All groups showed predominantly adhesive failure except the hydrogen peroxide with sandblasting, where the cohesive failure of the post was predominant.

Conclusions: Sandblasting and hydrofluoric acid surface treatments demonstrated superior results to silane and hydrogen peroxide. The combined method of hydrogen peroxide and sandblasting could weaken the fiber post and lead to clinical fractures.

1. Introduction

Restoration of an endodontically-treated tooth often requires a post and core placement as a substitute for lost tooth structure, to give support and retention for the definitive restoration (Caputo and Standlee, 1976). Several materials are available for post and core systems, such as metal post and core, zirconium post and core, fiber post (FP) and composite core (CC) (Schwartz and Robbins, 2004).

FP and CC are usually used due to esthetic, easily placement and removal, minimal requirement of tooth structure removal, have a modulus of elasticity similar to dentin (Aksornmuang et al., 2004; Asmussen et al., 1999), single-visit and cost-effective procedure (Bitter and Kielbassa, 2007; Shori et al., 2013; Pyun et al., 2016). A low incidence of root fracture and more repairable situations in the instance of failure have been reported with FP and CC system, which subsequently increase the survivability of the tooth as compared to cast post and core or zirconium posts (Martinez-Insua et al., 1998; Mannocci et al., 1999; Akkayan and Gülmez, 2002; Ferrari et al., 2000b; Ferrari et al., 2000a; Sarkis-Onofre et al., 2014; Malferrari et al., 2003; Cagidiaco et al., 2008; Monticelli et al., 2008; Yavirach et al., 2009). FP gives a comparative distribution of stress to the tooth and the surrounding structures due to its elastic properties that are similar to dentin, and the tooth is protected against fracture (Goracci and Ferrari, 2011; Mosharraf and Baghaei Yazdi, 2012; Monticelli et al., 2008). The most common cause of clinical failures of FP and CC system has been found to be adhesion failure or debonding of the post at either the resin or dentin interfaces (Monticelli et al., 2003; Barfeie et al., 2015; Cagidiaco et al., 2008; Ferrari et al., 2000b). Therefore, the bond strength between FP, CC, and luting agent is essential for clinical success (Sahafi et al., 2003; Cardoso et al., 2002; Bitter and Kielbassa, 2007; Prado et al., 2017).

Several methods of FP surface treatment have been advocated to increase the bond strength (Mosharraf and Baghaei Yazdi, 2012; Kim et al., 2013). These methods are generally categorized into three categories (Monticelli et al., 2008), namely (1) chemical treatment with various concentrations of one of the following materials: phosphoric acid, hydrofluoric acid, hydrogen peroxide, methylene chloride (Yenisey and Kulunk, 2008; Elsaka, 2013; Prado et al., 2017), potassium permanganate, sodium ethoxide (Monticelli et al., 2006a) or hydrochloric acid (Monticelli et al., 2006b); (2) micromechanical roughening of the surface by sandblasting technique using aluminum oxide or silica particles (Zicari et al., 2012; Sahafi et al., 2003; Sahafi et al., 2004; Cekic-Nagas et al., 2011); and (3) combination of chemical and micro-mechanical treatments (Monticelli et al., 2008). These methods of treatments increase the FP surface roughness to enhance the mechanical attachment of the bonded surfaces, and/or expose the fibers through the removal of the matrix enabling salinization and chemical adhesion of the bonded surfaces. However, these methods can be detrimental if implemented over a long period of time (Yavirach et al., 2009). Furthermore, the application time is preferred to be clinically feasible (Daneshkazemi et al., 2016). Lately, laser irradiation has been applied for FP surface treatment. However, a recent systematic review and meta-analysis showed that laser radiation provide no significant improvement on bond strength between FP and C.C (Davoudi et al., 2019).

Silane application enhances the wettability of FP and promotes a chemical bonding between the CC and the glass component of the FP (Zicari et al., 2012; Monticelli et al., 2008). However, the role of silane would be diminished in FPs with unexposed glass fibers surrounded by a matrix of highly cross-linked epoxy resin, which is not reactive to silane (Goracci et al., 2005; Mosharraf and Ranjbarian, 2013; Perdigao et al., 2006; Monticelli et al., 2008). The sandblasting technique has been found to increase the FP roughness and expose the superficial glass fibers (Sahafi et al., 2003; Sahafi et al., 2004; Valandro et al., 2006; Zicari et al., 2012). Hydrofluoric acid application etches the FP surface and create a rough surface of the matrix and the glass fibers (Vano et al., 2006). Hydrogen peroxide has been evaluated at different concentrations and application time and has been reported to dissolve the epoxy-based matrix and uncover the superficial fibers (Vano et al., 2006; Naves et al., 2011), Conceivably, a combined successive application of hydrogen peroxide, sandblasting and silane might result in more exposed fibers with increased surface area and roughness of the FP. However, this method has not been investigated.

The current study aimed to evaluate the impact of various methods of FP surface treatment on the bond strength to a CC material. The surfaces modifications of the FP were evaluated using SEM. Therefore, the null hypothesis was that bond strength would not be affected by the different methods of FP surface treatments.

2. Materials and methods

2.1. Test groups and specimens' preparation

Seventy-two glass-fiber posts (RelyX Fiber Post, size 2; 3 M ESPE, St. Paul, MN) were used and assigned randomly into 6 groups according to the method of surface treatment. Each

group had twelve FPs, where 10 FPs were employed for push-out test and 2 FPs for assessment of surface modifications under SEM.

Control (C): had no surface treatment.

Silane (S): a silane solution (Ultradent Silane; Ultradent Products, Inc. South Jordan, UT) was applied to the FP for 60 s using a brush and air-dried for 10 s.

Sandblasting and Silane (SBS): aluminum oxide particles of 50 μm were used for sandblasting at a pressure of 60 psi for 10 s perpendicular to the FP at 10 mm distance using an intraoral sandblaster (MicroEtcher™ II, Danville Material, Zest Dental Solution, USA). The FPs were rinsed with deionized water and air-dried for 10 s. Then, the silane was applied as in group S.

Hydrofluoric acid and Silane (HFS): 9% hydrofluoric acid (Ultradent Porcelain Etch; Ultradent Products, Inc. South Jordan, UT) was applied first for 90 s to the FPs with a brush, rinsed with deionized water, and air-dried for 10 s. Then, the silane was applied as in group S.

Hydrogen Peroxide and Silane (HPS): The FPs were immersed in 24% hydrogen peroxide solution (Loba Chemie Pvt. Ltd., India) at room temperature for 60 s and air-dried for 10 s. Then, the silane was applied as in group S.

Hydrogen peroxide, sandblasting and silane (HPSBS): After FPs were immersed in hydrogen peroxide solution as in group HPS, sansblasting was performed as in group SBS, rinsed with deionized water and air-dried for 10 s. Then, the silane was applied as in group S.

Each FP was placed vertically with the tapered part in a hole in the middle of a cylindrical putty matrix (ESPE Express™ STD, 3 M ESPE, St. Paul, MN) using a surveyor. The matrix was 7 mm in width and extend vertically to contain the non-tapered part of the FP to standardize the diameter in order to simplify surface area calculation (Kurt et al., 2012). A dual-cure CC (Multicore Flow; Ivoclar Vivadent, Schaan, Liechtenstein) was dispensed incrementally to the matrix and the FP and light-cured for 20 s (Bluephase G2, 1200 mW/cm^2, Ivoclar Vivadent, Schaan, Liechtenstein). The CC was left to self-cure for 5 min. The specimen was retrieved from the matrix and light-cured with direct contact with the light curing unit for a further 20 s.

Each specimen was sectioned perpendicular to the length using a low-speed diamond saw under water-cooling (Isomet 2000; Buehler Ltd, Lake Bluff, NY) to give two discs of 2 mm in thickness, 20 discs for each group (total 120 discs). A digital caliber was used to standardize the thickness of each disc. All discs were kept in a saline solution at 37 ℃ for 24 h before testing.

2.2. Push-out test

A universal-testing machine (Instron 5965 Material Testing System; Instron Corp, High Wycombe, UK) was used to carry out the push-out test. A plunger with 1 mm cylindrical diameter was used to apply the force and was centered on each disc to avoid contact with the CC material at a crosshead speed of 0.5 mm/min. The load at failure was recorded in Newton (N) and converted to Mega Pascal (MPa) by dividing the load (N) by the surface area (A, mm2) of FP/CC interface. The equation A = 2*π*r*h was used to determine the cylindrical interface area (A) between the FP and the CC, where π is a constant 3.14, r is the FP radius and h is the disc thickness (Cekic-Nagas et al., 2011; Elsaka, 2013; Bitter et al., 2007). Fig. 1 shows the steps of specimens' preparation for push-out test.

2.3. Scanning electron microscopic assessment

Failure of each disc was assessed using a SEM (JEOL JSM-6360 LV; x 300, 50 μm). All discs were cleaned using ultrasonic for 3 min with deionized water, immersed for 2 min in 96% ethanol and air dried. All discs were treated with gold sputter-coating and inspected under SEM at a magnification of 300X. Mode of failures were categorized as adhesive between the FP and CC, cohesive of the FP, cohesive of the CC, or mixed failures. The remaining two FPs from each group were inspected under SEM to assess the surface modifications both longitudinally and cross-sectionally.

2.4. Statistical analysis

Data of the push-out test was statistically analyzed using SPSS 22.0 software (IBM, Chicago, IL, USA). The results of the bond strength of all groups were presented in means and standard deviations (SD) (Table 1). One-way ANOVA was applied followed by Tukey's test for multiple compar-

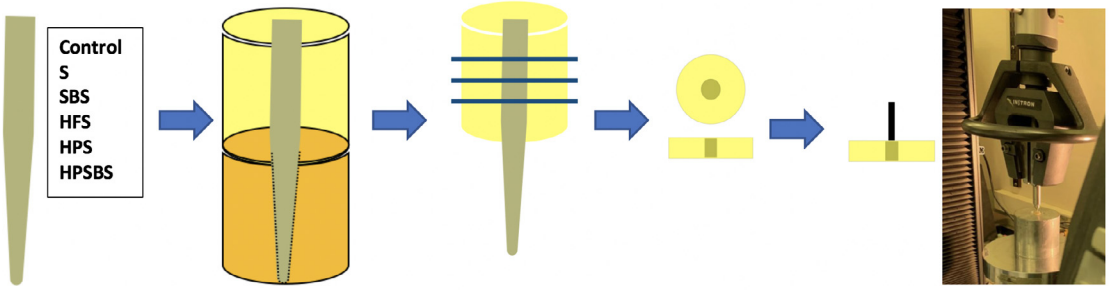

Fig. 1 Schematic representation of specimens' preparation for push-out test.

Table 1 Push-out bond strength means, standard deviations (MPa), one-way ANOVA and multiple comparison test.

Group	n	Mean	SD	P-Value	95% Confidence Interval		Multiple Comparison Test					
					Lower Bound	Upper Bound	C	S	SBS	HFS	HPS	HPSBS
C	20	16.54	2.06	0.000	15.57	17.5	1					
S	20	16.79	2.98		15.39	18.18	0.99	1				
SBS	20	19.07	3.9		17.24	20.9	0.01	0.03	1			
HFS	20	18.73	1.73		17.92	19.54	0.04	0.10	0.99	1		
HPS	20	13.07	1.09		12.56	13.57	0.00	0.00	0.00	0.00	1	
HPSBS	20	12.57	0.63		12.28	12.87	0.00	0.00	0.00	0.00	0.99	1

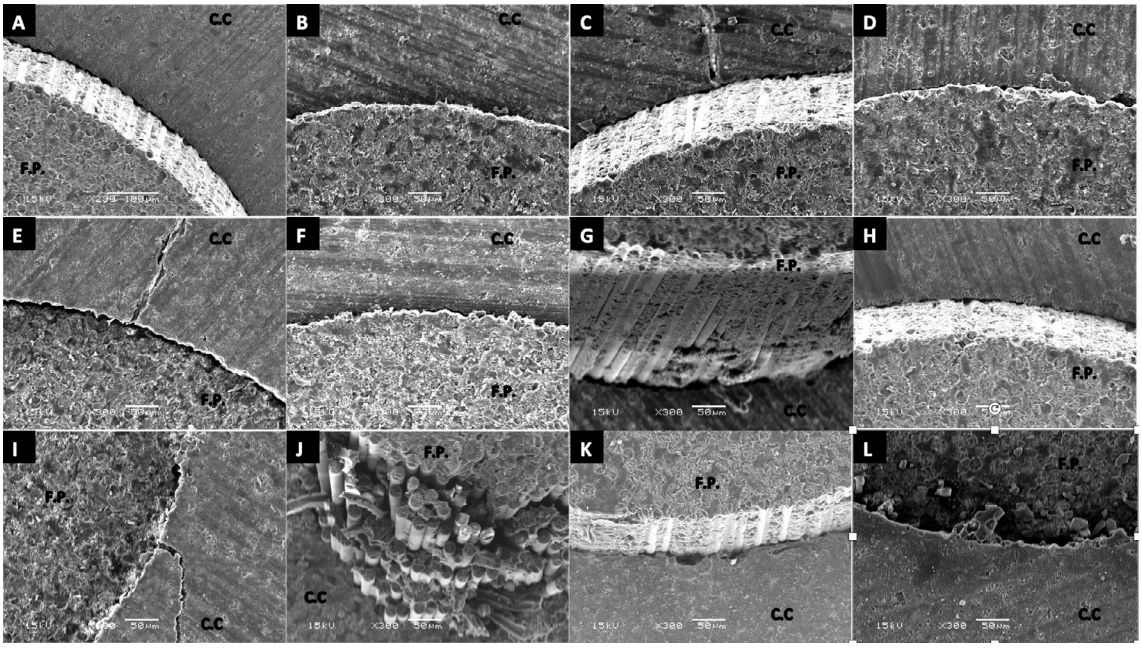

Fig. 2 Representative SEM photomicrographs of the mode of failures (**F.P.** indicates fiber post and **C.C** indicates composite core material): A, **C group**, adhesive failure; B, **S group**, adhesive failure; C, **S group**, mixed failure; D, **SBS group**, adhesive failure; E, **SBS group**, mixed failure; F and G, **HFS group**, adhesive failure; H, **HPS group**, adhesive failure; I, **HPS group**, mixed failure; J, **HPSBS group**, cohesive failure of the post; K, **HPSBS group**, adhesive failure; and J, **HPSBS group**, mixed failure.

isons. When $P \leq 0.05$, the results were considered statistically significant.

3. Results

ptThe mean values and standard deviation of all groups are given in Table 1. One-way ANOVA indicated a significant effect of the different methods of FP surface treatments ($P < 0.0001$). Further analysis by comparison test using the Tukey test revealed significant differences between the control (C) group and SBS, HFS, HPS and HPSBS groups ($P < 0.05$). However, no significant difference between C and S groups ($P = 0.99$). The SBS and HFS groups had significantly higher bond strength than the control group ($P = 0.01$ and 0.04, respectively). HPS and HPSBS groups had significantly lower bond strength than the other groups ($P < 0.0001$), while no significant between HPS and HPSBS groups ($P = 0.99$). There was no significant difference between S and HFS groups ($P = 0.1$).

SEM analysis revealed adhesive failures including displaced or debonded interfaces between the FP and the CC (Fig. 1A, B, D, F, G, H, K). Dislodged fibers where associated with the cohesive failure of the FP (Fig. 2J) Mixed adhesive-cohesive failures (Fig. 2C, E, I, L). Adhesive failure was the predominant mode of failure in groups C, S, SBS, HFS and HPS. Only 1 disc in group S, 3 discs in group SBS and 2 discs in HPS displayed mixed failures. Most of the discs in HPSBS group displayed cohesive failure of the FP, while 1 disc displayed an adhesive failure and another showed mixed failure.

SEM evaluation of the treated FPs revealed that surface modifications differed between the different groups. In the control group (Fig. 3A and B), SEM showed micropores and grooves features on the surface of untreated FP with superfi-

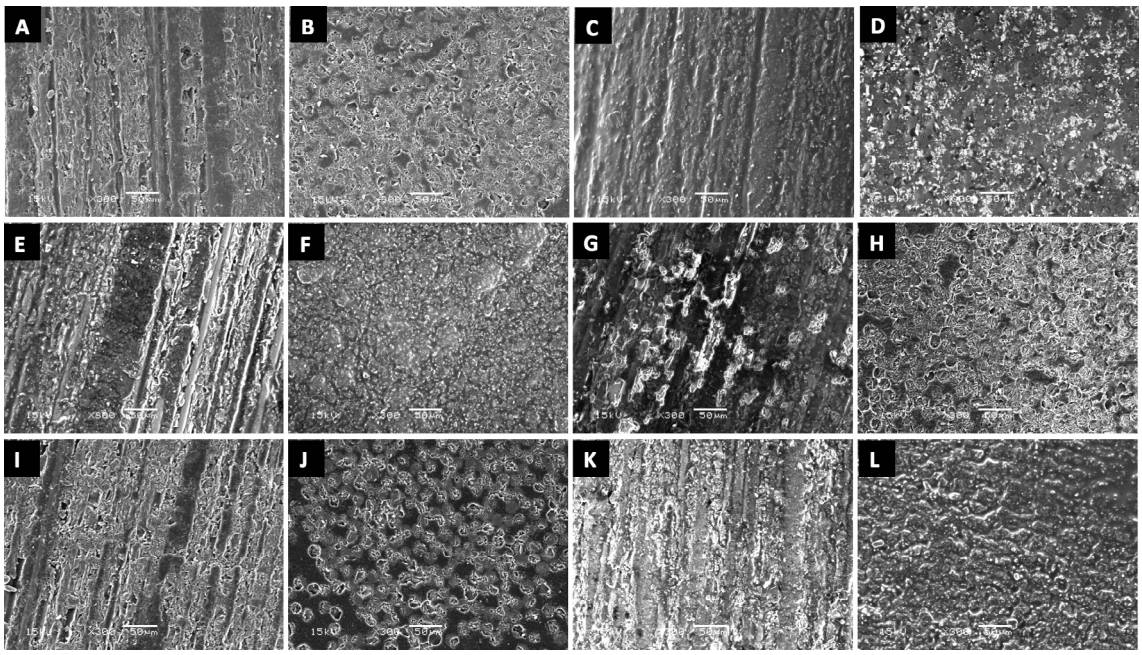

Fig. 3 Representative SEM photomicrographs of the treated surfaces both longitudinal and cross-sectional views: A and B, **C group**; C and D, **S group**; E and F, **SBS group**; G and H, **HFS group**; I and J, **HPS group**; K and L, **HPSBS group**.

cial glass fibers covered by resin matrix. The S group (Fig. 3C and D) exhibited impregnated micromechanical features of the FP by silane. The SBS group (Fig. 3E and F) exhibited a rough surface with exposed and intact superficial glass fibers. The HFS group (Fig. 3G and H) had a relatively rough etched surface of FP. The HPS group (Fig. 3I and J) had a partially dissolved matrix. The HPSBS group (Fig. 3K and L) showed a rough surface with partially dissolved matrix and exposed cracked glass fibers.

4. Discussion

The different methods of surface treatment were intended to enhance the bond between the FP and the CC material. The results of the present study demonstrated that the various methods of FPs surface treatment exhibited significantly different effects upon the bond strength to the CC ($P < 0.0001$), consequently, the null hypothesis was rejected.

The glass-fiber posts comprised of a matrix of resin and fillers of inorganic particles and glass fibers. A matrix of either epoxy or methacrylate resin matrix is often used. The FP in the current study had glass fibers in a parallel alignment to the length of the post and surrounded by a matrix of high-cross linked epoxy resin. However, the specific composition of the FP is not always disclosed by the manufacturers (Zicari et al., 2012). The CC material in this study was a dual-cured flowable composite core resin. Previous studies have reported that the flowability permits better adaptation of the CC material to the irregularities on the FP surface as compared to hybrid composites (Monticelli et al., 2003; Monticelli et al., 2005; Goracci et al., 2005). Additionally, the low viscosity of the CC material permits its use for FP luting with a comparable 4 years clinical performance to self-adhesive resin (Juloski et al., 2014).

The bond strength in the current study was investigated using a push-out testing method, which has been reported to apply a shear stress parallel to the FP and CC interface that is comparable to the clinical situation (Bitter et al., 2007; Saraiva et al., 2013; Santos et al., 2017). Furthermore, the recorded push-out bond strength values revealed limited variability which indicated high reproducibility of the measurements using the push-out testing method (Goracci et al., 2004; Goracci et al., 2007; Arslan et al., 2016). On the other hand, specimen's preparation in microtensile testing method is a technique sensitive with possible development of microfractures at the interface between bonded surfaces. Subsequently, led to early failure of specimens before testing or weakened the bond and produced low values of bond strength (Van Meerbeek et al., 2003; Goracci et al., 2004; Arslan et al., 2016).

The results showed that the bond strength was not enhanced by the application of silane alone (S). Several previous studies showed similar results (Yenisey and Kulunk, 2008; Choi et al., 2010; Elsaka, 2013; Zicari et al., 2012) and this might be due to the weak bond between silane and the FP when glass fibers were unexposed and surrounded by a highly cross-linked epoxy resin matrix that is not reactive to silane (Bitter and Kielbassa, 2007), while chemical reaction could be established by silane coupling agent between exposed glass fiber and methacrylate-based composite resin (Elsaka, 2013; Zicari et al., 2012; Bitter et al., 2007; Goracci et al., 2005). However, other studies have found improved bond strength with silane and could be attributed to the use of different FP systems with possible presence of exposed superficial glass fibers (Goracci et al., 2005; Aksornmuang et al., 2006; Aksornmuang et al., 2004; Daneshkazemi et al., 2016; Prado et al., 2017).

Surface treatment with sandblasting (SBS) and hydrofluoric acid (HFS) have significantly enhanced the bond strength as compared to untreated C group, which are consistent with the findings of previous studies (Cekic-Nagas et al., 2011; Valandro et al., 2006). This was likely due to the increased roughness, surface area of the FP and exposure of superficial glass fibers, which led to increased micromechanical and chemical adhesion of the CC and the FP (Zicari et al., 2012). This finding is supported by the SEM observations which exhibit increased surface roughness and exposure of glass fibers (Fig. 3). Sandblasting and hydrofluoric acid application attack the glass fibers and the matrix simultaneously. Unlike hydrogen peroxide which selectively dissolve the epoxy resin matrix leaving the glass fibers intact and exposed (Monticelli et al., 2006c). Sandblasting procedure could extensively modifies the post shape and volume which is considered as an aggressive procedure (Sahafi et al., 2004). Therefore, time, distance and pressure of application influence the outcomes of sandblasting (Zicari et al., 2012). However, it is still not known if these changes affect the strength and the clinical performance of the FPs (Zicari et al., 2012), nevertheless in vitro studies have reported that the strength of FP have not been altered by silane, hydrofluoric acid, sandblasting and hydrogen peroxide in comparison to untreated FPs (Soares et al., 2008; D'Arcangelo et al., 2007; Aksornmuang et al., 2017).

The application of hydrogen peroxide in HPS and HPSBS groups decreased the bond strength significantly in comparison to the other groups. This could be attributed to the matrix dissolution by hydrogen peroxide as shown in Fig. 3(I, J, K and L). Another possible explanation for this as stated by Daneshkazemi et al. (2016) might be the presence of residual oxygen by-products have inhibited the polymerization of CC. Previous studies have showed both similar (Pyun et al., 2016; Daneshkazemi et al., 2016) and contrary results (Monticelli et al., 2006b; Yenisey and Kulunk, 2008; Monticelli et al., 2006c; Vano et al., 2006). HPSBS group had the lowest bond strength, although SEM showed increased surface roughness as shown in Fig. 2(K and L). This finding was unexpected and could be attributed to a possible substantial damage to FP structure by matrix dissolution and potential damage of glass fibers by hydrogen peroxide and sandblasting, respectively. This explanation can be supported by the predominant cohesive failure of this group as shown under SEM Fig. 2(J). While, adhesive failure was the predominant mode of failure of the other groups which matched those observed in previous studies (Elsaka, 2013; Cekic-Nagas et al., 2011; Zicari et al., 2012).

The controversy and inconsistency of the findings of the previous studies are likely to be related to the differences in the composition of posts, core materials, application time and concentrations of chemical treatments, and methods of testing. In this study, the application time and the materials which have been used for surface treatments are possible to be performed during the regular dental visits. A previous study has reported that hydrogen peroxide application in 24% concentration for 60 s provides a bond strength similar to hydrogen peroxide in 50% concentration for 5 to 10 min (de Sousa Menezes et al., 2011). Lengthy application time is a waste of valuable clinical time with no enhancement in bond strength. Furthermore, other techniques such as sandblasting for 60 s or hydrofluoric acid application for 90 s are clinically feasible and were able to enhance bonding (Cekic-Nagas et al., 2011).

SEM evaluation revealed that different methods of treatment exhibited different surface modifications on the FP as explained in the results (Fig. 3). Consequently, the outcomes of the current study demonstrated that fiber posts which exhibited increased roughness with exposed fibers resulted in increased push-out bond strength as compared to surfaces with unexposed fibers or dissolved matrices. The manufacturer could supply pretreated FP, which are ready for clinical use in order to standardize the surface treatments and save the valuable clinical time (Elsaka, 2013).

This in vitro study does not replicate the exact clinical performance of the FP, which is considered as a limitation. The comparison of different types of FP and CC would be interesting. Although the surface roughening of the FP was revealed to be significant with regard to push-out bond strength, clinical studies are essential to assess the impact of the various surface treatments with regard to the incidence of debonding.

5. Conclusion

Within the limitations of the present study, it can be concluded that:

1. Surface treatment of fiber post with sandblasting or hydrofluoric acid are clinically convenient methods and showed results superior to silane and hydrogen peroxide.
2. The combined method of hydrogen peroxide and sandblasting could weaken the fiber post and lead to clinical fracture.

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Declaration of Competing Interest

No conflict of interest.

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