Bonding durability of titanium tetrafluoride treated glass fiber post with resin cement

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INTRODUCTION

Restoration of endodontically treated teeth with glass-fiber posts has been widely used. Glass-fiber posts have several advantages including similar modulus of elasticity to root dentine, improved esthetics, bond directly to tooth structure, and easy removal if endodontic retreatment is needed. However, main concern regarding the glass-fiber posts is the inadequate adhesion because of debonding at the post-resin interface. Fiber posts have a rather smooth surface, which adhere mechanically with luting agents, compared with threaded or serrated fiber posts. In addition, the epoxy resin component of glass fiber posts is highly cross-linked that reduces the copolymerization with monomers of different resin-based materials. Consequently, various surface treatments have been evaluated in order to improve the bond strength between fiber posts and luting agents including airborne particle abrasion, hydrogen peroxide, hydrofluoric acid, silanization, methylene chloride, silica coating, and laser treatments with varying outcomes. Continue searching and assessment of adequate surface treatment of fiber posts without impairing their properties are required to improve the adhesion with luting agents.

Titanium tetrafluoride (TiF₄) has been investigated to be used in dentistry in various applications including fissure sealant, desensitizing agent, etchant for dental ceramics, and varnish for prevention of enamel demineralization. Different concentrations of TiF₄ solutions have been used ranging between 0.4–5%. TiF₄ solution is highly acidic. In a previous study, an aqueous TiF₄ solution improved the bond strength of luting agent to ceramic and it was claimed that TiF₄ solution could be used as an etchant for ceramics instead of hydrofluoric acid (HF).

Based on previous studies, the possibility of using TiF₄ in the dental clinic and laboratory could make it appropriate for treating glass fiber post. Consequently, the purpose of this study was to evaluate the effect of TiF₄ solution on the adhesion of resin cement to glass fiber post. The null hypothesis tested was that the surface treatments have no influence on the bond strength between glass fiber post and resin cement.

MATERIALS AND METHODS

In the present study, two different concentrations of TiF₄ solution (2 and 4 wt/v%) with different time of applications (2 and 4 min) on fiber post were used. It was suggested that TiF₄ solution (2.5%-60 s and 2.5%-120 s) could be used as an etchant for ceramics instead of HF. However, it was shown that higher concentrations of TiF₄ solution (5%-60 s and 5%-120 s) decreased the bond strength of ceramic with luting agent. Consequently, TiF₄ solutions with different concentrations (2 and 4 wt/v%) at different dwell times (2 and 4 min) were chosen for this study.

Microtensile bond strength (µTBS)

Three hundred and twenty of glass fiber reinforced composite posts (Rebilda (RP) size # Ø 1.5, Voco, Cuxhaven, Germany) were used in this study. The RP post is composed of 70% glass fiber, 10% filler, and 20% urethane dimethacrylate. The fiber posts were divided into eight treatment groups as shown in Table 1.

Subsequently, all the posts were cleaned ultrasonically for 1 min in distilled water and air-dried except control, silanization, and sandblasted groups. Two square-shaped plates (6×6×1 mm) were attached to the post; in which one plate opposite the other.
Table 1  Grouping of specimens

| Group          | Treatment                                                                 |
|----------------|---------------------------------------------------------------------------|
| Control        | No surface treatment                                                      |
| Silanization   | The posts were treated with a silane coupling agent (Ceramic Bond, VOCO, Cuxhaven, Germany, silanization) and gently air-dried for 60 s |
| Sandblasted    | The posts were tribochemical silica coated (CoJet system; 3M ESPE, St. Paul, MN, USA) with aluminum trioxide particles (30 µm) at 2.5 bar pressure for 15 s |
| HF             | The posts were treated with HF at 9% (Ultradent Porcelain Etch, Ultradent Products, South Jordan, UT, USA) for 1 min |
| TiF₄ (2 wt/v%-2 min) | The posts were treated with TiF₄; the posts were immersed in 2 wt/v% TiF₄ solution for 2 min. |
| TiF₄ (2 wt/v%-4 min) | The posts were treated with TiF₄ (2 wt/v%-4 min) |
| TiF₄ (4 wt/v%-2 min) | The posts were treated with TiF₄ (4 wt/v%-2 min) |
| TiF₄ (4 wt/v%-4 min) | The posts were treated with TiF₄ (4 wt/v%-4 min) |

Each block was then sectioned into 5 bar-shaped sticks with 1 mm thickness of resin cement-post sections using a low speed diamond saw (Isomet 1000, Beuhler, Lake Bluff, IL, USA). Consequently, each subgroup provided a total of 100 bar-shaped sticks.

Each specimen was attached to a microtensile testing attachment using a universal testing machine (Model TT-B, Instron, Canton, MA, USA) at cross-head speed of 0.5 mm/min. The µTBS (MPa) was obtained by dividing the load at failure (Newton) by the fractured surface area (mm²). As seen in Fig. 1, to calculate the fractured cross-sectional area, the arc length (A) is needed which is given by the following formula:

\[ A = 2\pi R \left(\frac{\theta}{360}\right) \]

Where \( R \); is the radius of the post which is 0.75 mm, \( \pi \); is 3.142 and \( \theta \); is the central angle of the arc. By known the three sides of the triangle \( a=0.75 \text{ mm}, b=0.75 \text{ mm} \) and \( c=1 \text{ mm}; \) Fig. 1), the \( \theta \) angle was calculated by using the Cosine rule; which was equal 83.62°. Failure analysis of debonded specimens was assessed using a stereomicroscope (Nikon SMZ445, Melville, NY, USA) at 50× magnifications. Failure mode analysis was classified as Type 1; adhesive failure between the post and resin cement, Type 2; cohesive failure within the post, Type 3; cohesive failure within the resin cement and Type 4; mixed failure.

Scanning electron microscopy (SEM)-energy-dispersive X-ray spectroscopy (EDX) analysis

Ten additional fiber posts from each group were prepared to determine the morphological aspect using a SEM (JSM-6510LV, JEOL, Tokyo, Japan). Elemental composition analysis for each group was determined using EDX (INCA X-Sight, Model 7603, Oxford Instrument, Abingdon, England). The posts were
sputtered with carbon prior to SEM-EDX analysis.

**Flexural strength (F_s), flexural modulus (F_m) and stiffness (S)**

A three-point bending test was performed on the universal testing machine according to ISO 10477 standard. A total number of 40 posts from each group were tested using a 500 N load cell, 1.0 mm/min crosshead speed, 6 mm span distance and 2 mm loading tip cross-sectional diameter. Half of the specimens were tested immediately, and the other half of specimens were tested after storage in artificial saliva for 150 days with 10,000 thermal cycles between 5 and 55°C with a 30 s dwell time. Flexural strength (F_s), flexural modulus (F_m) and stiffness (S) were determined by using the following equations:

\[ F_s = \frac{3FL}{4d^3} \]
\[ F_m = \frac{8F_mL}{\pi d^4} \]
\[ S = \frac{FS}{d} \]

Where, \( F_s \) is the applied load (N) at the highest point of load deflection curve, \( L \) is the span length (6 mm) and \( d \) (mm) is the diameter of the post.

**Statistical analysis**

Data of \( \mu TBS \) (MPa), flexural strength \( (F_s) \), flexural modulus \( (F_m) \) and stiffness \( (S) \) were statistically analyzed (SPSS 23.0; IBM Software, Armonk, NY, USA) using a two-way analysis of variance (ANOVA) based on two factors (type of surface treatment and aging) and their interaction. Multiple comparisons were performed by the Tukey's post-hoc test \((\alpha=0.05)\).

**RESULTS**

Data of \( \mu TBS \) (MPa), flexural strength \( (F_s) \), flexural modulus \( (F_m) \) and stiffness \( (S, \text{N/m}) \) are presented in Table 2. The \( \mu TBS \) of different groups was significantly affected by the surface treatment \((p<0.001)\), aging \((p<0.001)\) and interaction between these factors \((p<0.001)\). All surface treatments improved significantly the bond strength compared with the control group \((p<0.05)\). The highest \( \mu TBS \) achieved with the TiF_4 (4 wt/v%-4 min) group compared with the other groups \((p<0.05)\). Failure modes analysis of different groups is presented in Fig. 2. Failure mode analysis revealed that the predominant mode of failure was adhesive failure between the post and resin cement (Type 1) in all groups except for the groups treated with TiF_4 (4 wt/v%-4 min) whereas the cohesive failure within the resin cement.

### Table 2 Mean (standard deviations) of \( \mu TBS \) (MPa), flexural strength \( (S_f, \text{MPa}) \), flexural modulus \( (E_f, \text{GPa}) \), stiffness \( (S, \text{N/m}) \) of different groups and statistical analysis

| Groups            | \( \mu TBS^* \) (MPa) | Flexural strength** \( (S_f, \text{MPa}) \) | Flexural modulus** \( (E_f, \text{GPa}) \) | Stiffness** \( (S, \text{N/m}) \) |
|-------------------|------------------------|---------------------------------------------|-------------------------------------------|---------------------------------|
|                   | No aging*** | Aging                                     | No aging*** | Aging                                     | No aging*** | Aging                                     |
| Control           | 10.49 (0.64)\(^a\) | 8.53 (0.83)\(^a\) | 818.5 (42.15) | 616.3 (33.32) | 19.69 (1.25) | 16.08 (1.15) | 273.95 (12.92) | 251.65 (11.82) |
| Silanization      | 13.76 (0.93)\(^bc\) | 10.69 (0.53)\(^bd\) | 801.8 (48.32) | 615.35 (27.24) | 19.62 (1.10) | 15.96 (0.86) | 271.75 (12.41) | 249.5 (14.23) |
| Sandblasted       | 14.22 (0.9)\(^b\) | 12.29 (0.55)\(^c\) | 790.1 (38.18) | 613.45 (24.67) | 19.46 (0.79) | 15.93 (1.05) | 269.15 (9.59) | 247.85 (15.32) |
| HF                | 15.74 (0.89)\(^d\) | 13.85 (0.71)\(^e\) | 783.35 (31.69) | 612.15 (22.92) | 19.44 (0.87) | 15.82 (0.85) | 265.75 (14.36) | 245.45 (14.72) |
| TiF_4 (2 wt/v%-2 min) | 12.25 (0.79)\(^e\) | 10.7 (0.85)\(^d\) | 813.05 (33.69) | 613.2 (30.40) | 19.56 (1.09) | 15.92 (0.68) | 272.95 (9.98) | 250.7 (11.10) |
| TiF_4 (2 wt/v%-4 min) | 13.15 (1.16)\(^e\) | 11.36 (0.84)\(^d\) | 804.45 (20.93) | 612.3 (25.34) | 19.61 (0.90) | 15.89 (0.54) | 270.5 (11.56) | 248.15 (13.75) |
| TiF_4 (4 wt/v%-2 min) | 16.42 (0.99)\(^d\) | 14.55 (0.93)\(^e\) | 795.05 (25.83) | 613.1 (28.11) | 19.53 (0.92) | 15.86 (0.75) | 268.1 (11.19) | 247.3 (8.85) |
| TiF_4 (4 wt/v%-4 min) | 22.88 (1.08)\(^e\) | 18.4 (0.82)\(^d\) | 792.3 (20.68) | 611.4 (19.36) | 19.51 (0.80) | 15.84 (0.62) | 267.25 (13.97) | 246.55 (8.37) |

* Different superscript uppercase letters in the column are significantly different \((p<0.05)\).
** No significant difference between different treatments (column) for each property \((p>0.05)\).
*** Significant difference in the aging factor (row) for each property \((p<0.05)\).
Fig. 2  Percentages of failure modes of different groups.
Type 1; adhesive failure between the post and resin cement, Type 2; cohesive failure within the post, Type 3; cohesive failure within the resin cement, Type 4; mixed failure.

Fig. 3  SEM micrographs (×500) of RP post surfaces with different treatment.
A; Control, B; Silanization, C; Sandblasted, D; HF, E; TiF₄ (2 wt/v%-2 min), F; TiF₄ (2 wt/v%-4 min), G; TiF₄ (4 wt/v%-2 min), H; TiF₄ (4 wt/v%-4 min). A layer was deposited on the surface of the post (asterisks) (E–H) after treatment with TiF₄.

Increased (Type 3). Regarding flexural properties, the surface treatments did not affect the $F_s$, $F_m$ and $S$ properties ($p=0.084$, $p=0.953$, $p=0.131$, respectively); however, the aging factor had significantly affected the flexural properties of different treated groups ($p<0.001$).

There was no significant interaction between the surface treatments and the aging factors for the $F_s$, $F_m$ and $S$ properties ($p=0.257$, $p=1.000$, $p=1.000$, respectively).

Surface topography analysis of different treated groups revealed that the surfaces of the untreated posts
were mostly covered by the resin matrix (Fig. 3A). The other treated groups revealed variations on the surface topography with more glass fibers were exposed compared with the control group. The surface of the sandblasted group showed broken glass fibers (Fig. 3C). For the HF group, some of the glass fibers were dislodged from the surface and broken (Fig. 3D). The glass fibers were also exposed for the posts treated with TiF$_4$ (2 and 4 wt/v%-2 and 4 min, Figs. 3E–H). It was observed that a layer was deposited on the surface of the posts treated with TiF$_4$ (2 and 4 wt/v%-2 and 4 min, Figs. 3E–H) that revealed the presence of Ti element on the surface of the post as obtained from EDX analysis (Table 3).

**DISCUSSION**

In the present study, an experimental solution of TiF$_4$ was used as a surface treatment to a glass fiber post in order to enhance the bond strength of the post to resin cement. Based on the results, the null hypothesis was rejected as the surface treatments performed enhanced the bond strength between glass fiber post and resin cement compared with the untreated group.

TiF$_4$ was chosen as it was tested for different dental applications in previous studies$^{13-19}$. One of these tested applications of TiF$_4$ solution was for etching the ceramic to enhance its bond strength with luting agent and it was suggested that TiF$_4$ solution could be used as an etchant for ceramics instead of HF$^{16}$. All the tested surface treatments enhanced the bond strength between the glass fiber post and resin cement compared with the untreated group. TiF$_4$ (4 wt/v%-4 min) treatment revealed the highest bond strength compared with the other treatments (Table 2). This finding could be attributed to the effectiveness of the TiF$_4$ (4 wt/v%-4 min) treatment by removing the surface layer of the resin matrix of fiber posts that provides more uncovered surface areas of glass fiber posts which improved the micromechanical retention of the resin cement$^{11}$. Additionally, it was observed that TiF$_4$ solution forms a precipitate layer of titanium-coating on the surface of the post. The hydrolysis of TiF$_4$ resulted in the formation of surface titanium dioxide (TiO$_2$) layer which is formed by the reaction of titanium (Ti) with the oxygen (O$_2$)$^{26}$. However, fluorine was not observed due to its low atomic weight (Table 3)$^{27,28}$. It was observed that Ti-layer was not consistently covers all the surfaces of the treated posts. This could be attributed to the lack of homogeneity of the surface of the post which impaired the formation of continuous homogenous Ti-layer. Failure mode analysis supported the enhanced quality of the bonding interface formed by the experimental solution TiF$_4$ (4 wt/v%-4 min) as the predominant mode of failure was cohesive failure within the resin cement (Type 3).

Silanization surface treatment enhanced the bond strength between the glass fiber post and resin cement compared with the control group and TiF$_4$ (2 wt/v%-2 min, Table 2). This finding could be attributed to the improved surface wettability of the post following silane coating$^{29,30}$. The low viscosity of silane enhanced the wetting of post and consequently improved the intimate contact with the resin cement. Also, the van der Waals’ forces would become effective that enhanced the physical adhesion$^{30,31}$.

Regarding sandblasting surface treatment (tribochemical silica coating), it was observed that the bond strength between the post and resin cement was improved compared with the control and TiF$_4$ (2 wt/v%-2 and 4 min) groups. This could be attributed to the silica particles that react chemically with the adhesive monomer of MS resin cement$^{32}$, which enhanced the adhesion with the post. Additionally, it could be postulated that the increased surface roughness and surface area of the post surface enhanced the mechanical interlocking with the resin cement$^{33}$. Roughening with the sandblasting by using CoJet system and HF treatments increased the number of exposed glass fibers and consequently the surface area available for reacting with the resin cement, allowing for higher bond strength$^{33}$. Etching
the post with HF enhanced the bond strength with resin cement by modifying the outer surface layer of the post surface that provides adequate mechanical retention without affecting the internal strength of the post\(^6\). The topographic features of the treated surfaces showed some of the glass fibers were dislodged from the surface and broken. However, in previous studies, it was shown the aggressive effect of HF treatment on the surface integrity of fiber post\(^5,23\), which reduced the adhesion with the resin cement\(^35\). This contradictory finding could be attributed to the different types of fiber post, luting agents and testing methodology used in these previous studies\(^34,35\). For health safety, the intraoral use of HF acid is limited due to its hazardous effect as it penetrates rapidly into skin and other soft tissues\(^36,37\).

All tested groups revealed lower bond strength values after aging (long water storage and thermal cycling). This finding could be attributed to the hydrolytic degradation that negatively affected the adhesion between glass fiber post and resin cement\(^5,23\). The failure analysis revealed more adhesive failure (Type 1) after aging which was consistent with the lower bond strength values compared with no aging in all groups. In the present study, aging conditions performed for the bonded post-resin cement might provide potential estimates of adhesion durability. However, other factors including oral environment, masticatory forces and type of luting agent could also affect adhesion and durability in vivo.

A dual-cure self-adhesive luting agent was tested with the glass fiber post as the self-adhesive luting agent is less-sensitive technique and ease to use clinically in narrow and deep root canals\(^21,29\). MS resin cement is based on adhesive phosphate methacrylate monomer that provides the cement with self-adhesive features. It has been reported that resin-cements that contained phosphate monomer enhanced the bond strength compared with cement lacking methacrylated phosphoric acid ester\(^32,38\).

Based on the results of flexural properties, it was observed that the alteration formed at the outer surface of the posts by different treatments did not affect the flexural properties of the posts. However, after aging the flexural properties were significantly reduced. This could be attributed to the hydrolytic stresses that formed which resulted in strength degradation of fiber post\(^39\). It has been reported that fiber posts that were stored in water revealed lower flexural strength than dry-stored fiber posts\(^39\). Water sorption of the resin matrix of fiber post might result in plasticization of the resin which was revealed to adversely affect the flexural properties of the post\(^40\). In addition, water molecules might result in weakening the bond between the resin matrix and the fibers which deteriorate the flexural strength of the post\(^40\). However, in clinical situation, the posts are placed inside the root canal and luted to the tooth with a luting agent\(^41\). So, the post is protected from contamination by water. If the adhesion between the post-cement, post-root or post-core systems fails somehow, there will be a risk of water contamination of the fiber post which may adversely affect the flexural properties\(^5,41\).

The present study suggested the potential use of TiF\(_4\) (4 wt/v%-4 min) solution for treating glass fiber post in order to enhance the adhesion with resin cement. One of the limitations of this study was that one type of post and resin cement was only tested and consequently, the findings of the present study could not be generalized to similar materials. Further investigations are required in order to investigate the thickness and topography of the formed TiF\(_4\) layer.

CONCLUSIONS

Surface treatment of fiber post with TiF\(_4\) (4 wt/v%-4 min) solution exhibited higher bond strength to resin cement compared with other surface treatments. There were no adverse effects on the flexural properties of fiber post from different applied surface treatments. However, aging had significantly affected the bond strength and flexural properties of different treated groups.

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