Retention force of fiber-reinforced composite resin post on resin composite for core buildup — Effects of fiber orientation, silane treatment and thermal cycling

Tomoko SOMEYA¹, Masaaki KASAHARA¹, Shinji TAKEMOTO² and Masayuki HATTORI¹

¹ Department of Dental Materials Science, Tokyo Dental College, 2-9-18 Kandamisaki-cho, Chiyoda-ku, Tokyo 101-0061 Japan
² Department of Biomedical Engineering, Iwate Medical University, 1-1-1 Iida-dori, Yahaba-cho, Shiwa-gun, Iwate 028-3694, Japan

Corresponding author, Tomoko SOMEYA; E-mail: someyatomoko@tdc.ac.jp

The purpose of this study was to investigate the effects of fiber orientation, silane treatment, and thermal cycling on the retention force of fiber-reinforced composite resin (FRC) posts on resin composite. Two types of FRC posts (linear and woven) were prepared with and without silane treatment. Pull-out test specimens were made of FRC posts and resin composite for core buildup. Specimens were either incubated in distilled water for 24 h or subjected to 10,000 thermal cycles. The maximum fracture load obtained by a pull-out test was defined as the retention force. Fracture modes were observed after the test. Fiber orientation and thermal cycling did not affect the retention force on resin composite, and the retention force was improved by silane treatment. Whereas, fiber orientation affected the fracture mode. The result showed woven fiber orientation could contribute to the mechanically interlocking with the resin composite.

Keywords: Fiber-reinforced composite resin post, Resin composite for core buildup, Retention force, Silane treatment, Thermal cycling

INTRODUCTION

Post materials, in conjunction with factors such as adequate root canal treatment, prosthetic restorative materials, and ferrule effects, play an important role in determining the long-term success of endodontically treated teeth¹⁻⁰. Metals have long been used as post materials, however in recent years, fiber-reinforced composite resin (FRC) posts have become increasingly more commonplace. This is primarily due to the decreased risk of severe root fractures associated with FRC posts compared to metal posts due to a lower Young’s modulus⁶⁻⁸. There are two methods for post and core restoration with FRC posts on root canal treated teeth: direct and indirect⁹. In the direct method, the post and core restoration is fabricated intraorally. In the indirect method, the post and core restoration is fabricated on model die and cemented in root canal cavity. There are two bonding interfaces in the post and core restoration with FRC post: one between the dentin and resin composite and the other between the resin composite and the FRC post. Previous studies have reported that adhesive failures occurred between the FRC post and resin composite when pull-out tests were performed on root canal treated teeth¹⁰,¹¹. This shows that the retention force of FRC post on resin composite is an important factor when considering the longevity of FRC posts.

Pretreatment methods have been studied in order to improve the retention force of FRC post to resin composite. Pretreatment methods are classified into two categories: one aimed at improving chemical bonding and the other aimed at improving mechanical retentive force. The former pretreatment method includes silane treatment with silane coupling agents, while the latter includes etching with hydrofluoric acid and alumina-blasting, which improves the mechanical retentive force at a microscopic level between the FRC post and resin composite¹²⁻¹⁷. An important observation was that etching and alumina-blasting dissolved and destroyed the glass fibers in the FRC post, which reduces the mechanical strength of the FRC post itself¹₈⁻²₀. Therefore, increasing microscopic mechanical bonds through etching and alumina-blasting in order to improve FRC post retention is a questionable process.

The fiber orientation of conventional FRC posts is linear. Whereas, FRC posts that contain woven fiber orientation that improves the macroscopic interlocking force are introduced recently. However, the retention force of FRC posts with woven fiber orientation on resin composite, as well as the effects of silane treatment, a method used to chemically improve the adhesion of FRC post and resin composite, still remained unclarified. The objective of this study was to compare and contrast the effects of fiber orientation (woven vs. liner) and silane treatment on the retention force of FRC posts on resin composite used for core buildup. In addition, the effect of thermal cycling for the long-term use of FRC posts was also investigated. The null hypothesis was as follows: fiber orientation, silane treatment, and thermal cycling have no effect on the retention force of FRC post on resin composite for core buildup.

Keywords: Fiber-reinforced composite resin post, Resin composite for core buildup, Retention force, Silane treatment, Thermal cycling
MATERIALS AND METHODS

Specimen preparation

The retention force of the FRC post on resin composite for core buildup (resin composite) was evaluated by using a pull-out test. Table 1 shows the FRC posts, silane coupling agents, and resin composite used in this study. FRC posts with both a woven structure (code: TF, i-TFC Luminous Fiber, Sun Medical, Shiga, Japan) and a linear structure (code: FP, GC Fiber Post, GC, Tokyo, Japan) were used. The FRC post was studied under a scanning electron microscope (SEM; SU-6600, Hitachi, Tokyo, Japan) after carbon deposition for surface observation. After cleaning the FRC post with ethanol, two types of silane coupling agents (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental, Tokyo, Japan, and RelyX Ceramic Primer, 3M ESPE, Tokyo, Japan) were applied and dried according to the manufacturer’s instructions (denoted as w/CF and w/RX). A mold was prepared to place the FRC post at the center of the specimen with 2 mm of the post exposed above the surface (Fig. 1(a)). An acrylic ring (inner diameter: 8 mm, height: 2 mm) was attached to the mold (Fig. 1(b)) and filled with a resin composite (Clearfil DCcore Automix, Kuraray Noritake Dental). A glass plate was pressed against the acrylic ring and irradiated for 10 s each from 4 points at 90 degrees angle to each other with a light irradiator (Pencure, Morita, Kyoto, Japan). After irradiation, the specimen was taken out of the mold and irradiated again from the bottom for an additional 10 s each from 4 points at 90 degrees angle to each other. This specimen was used for the pull-out test (Fig. 1(c)). The thickness of the resin composite was 2.00±0.20 mm as measured with a digital caliper (Digimatic caliper, Mitutoyo, Kanagawa, Japan). Half of the specimens in each group were placed in distilled water at 37°C for a day (w/o TC), and the other half were subjected to 10,000 thermal cycles of alternate immersion in 5°C and 55°C water for 30 s each.

![Specimen preparation](image)

**Fig. 1** Specimens fabrication for pull-out test. (a): FRC post placed in mold (b): Acrylic ring placed in mold (c): FRC post and resin composite specimen for pull-out test (d): Specimen during pull-out test

| Table 1 Materials used in this study |
|--------------------------------------|
| **Bland** | **Code** | **Lot No.** | **Manufacturer** | **Component (Manufacture’s instruction)** |
| FRC post | | | | |
| Woven type | TF | RW12 | Sun Medical | Silicate glass, Barium oxide, Dimethacrylate |
| i-TFC Luminous Fiber | | | | |
| (1.6 mm) | | | | |
| Liner type | FP | 1512121 | GC | Silicate glass, Bis-GMA, Methacrylic ester |
| GC Fiber Post | | | | |
| (1.6 mm) | | | | |
| Silane coupling agent | | | | |
| Clearfil Ceramic Primer Plus | CF | 1k0032 | Kuraray Noritake Dental | γ-MPTS, MDP, Ethanol |
| RelyX Ceramic Primer | RX | N988623 | 3M ESPE | γ-MPTS, Ethanol, Water |
| Resin composite for core buildup | | | | |
| Clearfil DCcore Automix | C50271 | Kuraray Noritake Dental | Bis-GMA, TEGDMA, Filler, Chemical catalyst, Photopolymerization catalyst, Chemical catalyst |

Bis-GMA: 2,2-bis-[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane
γ-MPTS: 3-(methacryloyloxy)propyltrimethoxysilane, MDP: 10-methacryloyloxydecyl dihydrogen phosphate
TEGDMA: Triethyleneglycol dimethacrylate
Fig. 2 Fracture mode after pull-out test. (a): Adhesive failure between FRC post and resin composite (b): Mixture of adhesive and cohesive failure of resin composite

Table 2 Specimen preparation (n=7)

| FRC post | Silane treatment | Thermal cycling |
|----------|-----------------|-----------------|
| TF       | w/o TC          | w/TC            |
| FP       | w/o TC          | w/TC            |

Table 2 continues...
Fig. 4 Retention force of FRC post and resin composite
Means with same letter are not significantly different in each group (TF (w/o TC), TF (w/TC), FP (w/o TC), FP (w/TC)).
FRC post: TF (woven type), FP (liner type), Silane treatment: w/o (without silane treatment), w/CF (with CF), w/RX (with RX), Thermal cycling: w/o TC (without thermal cycling), w/TC (with thermal cycling). TF w/TC showed a significant difference between w/o and w/RX ($p<0.05$).

Table 3 Statistical analysis using three-way ANOVA (Retention force of FRC post and resin composite)

| Source                          | Sum of squares | DF  | Mean square | F value | p value | Judge \(^*\): \(p<0.05\) |
|---------------------------------|----------------|-----|-------------|---------|---------|--------------------------|
| Fiber orientation (a)          | 2,708.679      | 1   | 2,708.679   | 1.062   | 0.306   |                          |
| Silane treatment (b)           | 30,953.786     | 2   | 15,476.893  | 6.067   | 0.004   | *                        |
| Thermal cycling (c)            | 480.964        | 1   | 480.964     | 0.189   | 0.665   |                          |
| a*b                            | 3,063.071      | 2   | 1,531.536   | 0.600   | 0.551   |                          |
| a*c                            | 2,170.583      | 1   | 2,170.583   | 0.851   | 0.359   |                          |
| b*c                            | 325.929        | 2   | 162.964     | 0.064   | 0.938   |                          |
| a*b*c                          | 7,704.167      | 2   | 3,852.083   | 1.510   | 0.228   |                          |
| Error                           | 183,683.143    | 72  | 2,551.155   | —       | —       | —                        |
| Total                           | 231,090.321    | 83  | —           | —       | —       | —                        |

Fig. 5 Fracture mode ratio after pull-out test.
FRC post: TF (woven type), FP (liner type), Silane treatment: w/o (without silane treatment), w/CF (with CF), w/RX (with RX), Thermal cycling: w/o TC (without thermal cycling), w/TC (with thermal cycling). Fracture mode 1 was observed in all FP specimens. Fracture mode 2 in addition to 1 was observed in TF specimens.

Observation after pull-out test
Figure 5 shows the ratio of fracture modes after the pull-out test. Fracture mode 1 (Fig. 2(a)) was observed in all FP specimens. Fracture mode 2 (Fig. 2(b)) in addition to 1 was observed in TF specimens. According to the composition of FRC posts and resin composite for core buildup by EPMA, Si of FRC post and Ba of resin composite for core buildup were characterized for mapping on FRC post after pull out test with SEM observation. Figure 6 shows a typical SEM image of the FRC post surface after the pull-out test and EPMA elemental mapping images (Si, Ba). In the SEM images, the direction glass fibers were identified, and partial aggregation was observed where the fibers intersected (yellow arrow). Large amounts of Ba (a component of resin composite for core buildup) were detected on TF specimens in regions where the fibers intersected (yellow arrow).
DISCUSSION

The purpose of this study was to compare and contrast the effects of fiber orientation (woven vs. linear), silane treatment, and thermal cycling on the retention force of FRC post on resin composite used for core buildup. Statistical analysis showed that silane-treated FRC posts had significantly greater retention on resin composite. Thus, the null hypothesis that fiber orientation and thermal cycling had no effect on the retention force of FRC post on resin composite was accepted, while the null hypothesis that silane treatment did not affect retention force of FRC post on resin composite was rejected. The retention force of FRC post to resin composite was evaluated with a pull-out test. Bond strength is usually evaluated through shear, push-out, or pull-out tests21. However, there are a number of limitations to evaluate the bond strength of FRC posts and the resin composite. Because it is difficult to measure area of the post surface with containing the cylindrical glass fibers. Previous studies used pull-out tests to evaluate the retention of post and core on bovine teeth roots that underwent various surface treatments21-25). These studies reported that fractures occurred between the FRC post and resin composites21-25). Therefore, in this study, the retention force between the FRC post and resin composite was established as the maximum load during pull-out test.

Fiber orientation was associated with mechanical interlocking, whereas silane treatment is related to chemical adhesion12-14). In this study, three-way ANOVA revealed that silane treatment of the FRC post had an effect on the retention force of FRC post on resin composite (Table 3), but only under certain conditions. The result is in accordance with those of previous studies showing a significant increase in the retention between FRC post and resin composite after silane treatment12,13,19). However, silane treatment with CF did not significantly improve the retention force of FRC post. Silane coupling agents used in this study contain 3-(methacryloyloxy) propyltrimethoxysilane which bridges resin and inorganic substances covered by OH at FRC post. In addition to that, CF contains MDP monomer. The reason for the difference in results between CF and RX may be due to the difference in ingredients. The concentration of 3-(methacryloyloxy)propyltrimethoxysilane needs to be investigated further.

Thermal cycling has been used to evaluate the degradation of resin materials due to thermal stress and absorption of water14,23,26-28). Lucas et al. reported that thermal cycling reduced the bonding strength of adhesive resin cements26). Sasaki et al. also reported that the impact strength of acrylic resin was reduced by thermal cycling28). Bitter suggests that the retention between FRC post and resin composite after thermal cycling might have been affected due to a variation in coefficients of thermal expansion between fibers and matrix resin in FRC post10). In this study, 10,000 times of thermal cycling did not affect the retention force of FRC post on resin composite. Gale suggests that 10,000 cycles represents a year26). Sasaki reported that the mechanical properties of resin materials decreased after 50,000 cycles26). Further study involving larger numbers of thermal cycling is needed to clarify the long- time stability of retention force between FRC post and resin composite.

Results indicated that fiber orientation and thermal cycling did not affect the retention of FRC posts on resin composite. Silane treatment was partly effective for improving FRC post retention. These results indicated that chemical bonding force by the silane treatment is effective for improving retention force regardless of fiber orientation.

The fracture mode after the pull-out test varied greatly depending on the fiber orientation of the FRC post (Fig. 5). For FP with linear fibers, adhesive failure between the FRC post and resin composite was observed in all specimens (Fig. 2(a)), whereas for TF with woven fibers, mixed fracture was observed with adhesive and cohesive failure in the resin composite (Fig. 2(b)).

The aggregates were found to contain large amounts of Ba from the resin composite for core buildup (Fig. 6(c)). This finding indicates that the resin composite underwent cohesive failure within the woven structure of TF. The resin composite at the intersection of fibers in TF had been loaded by shear force occurring during the pull-out test. The local stress in the resin composite by the direction of shear force could be higher than mechanical strength of the resin composite. In other words, a force exceeding the mechanical strength of resin composite was happened to the pull-out direction of the FRC post resulting in cohesive failure of resin composite. Generally, cohesive failure occurs when the strength of the material is weak, however the compressive strength of the resin composite used in this study was approximately 230 MPa, which is considered to be adequate for core materials.

Adequate retention of post and core material on tooth structure is required in a clinical setting, however retention of FRC post on resin composite is also important. Singh et al. reported that the clinically required retention force of post in a root canal was 200 N30). Past studies have indicated that the retention force of post was 150 N or more in root canals with a diameter of 2 to 3 mm and a depth of 4 to 5 mm21,23,25). In addition, although the width of the bonding region between the FRC post and resin composite was only 2 mm, retention force of FRC post on resin composite was more than 250 N in all conditions in this study. Therefore, the retention of FRC post on resin composite can be considered sufficient for clinical use.

CONCLUSION

Fiber orientation and thermal cycling did not affect the retention force on resin composite, and the retention force was improved by silane treatment. Whereas, fiber orientation affected the fracture mode after the pull-out test. That result showed woven fiber orientation could contribute to the mechanically interlocking with the resin composite.
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