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**Original**

# Effect of different bonding procedures on micro-tensile bond strength between a fiber post and resin-based luting agents

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**Abstract: The purpose of this study was to evaluate the effect of silanization and light-irradiation on bonding between a fiber post and different resin-based luting agents. Sixty silicium fiber posts (Easy Post) were divided into 10 groups according to the type of resin-based luting agent employed, whether the post surface was silanized, whether the adhesive was light-irradiated, and whether Calibra luting agent was used. The micro-tensile bond strength and bonded interface of specimens in each group were evaluated. Specimens luted with Calibra or FluoroCore 2 resin-based luting agent systems were superior to those treated with Multilink or Variolink II, in terms of both bond strength and interfacial integrity. Application of silane, light-irradiation of the adhesive, or light-irradiation of the Calibra resin-based luting agent did not significantly increase the bond strength further. It can be concluded that Calibra or FluoroCore 2 resin-based luting agent systems are more suitable for luting prefabricated Easy Post in a clinical situation, while pre-silanization of the post surface and light-irradiation of XP Bond/SCA adhesive or resin-based luting agent may not be as important as hitherto considered. (J. Oral Sci. 49, 155-160, 2007)**

Keywords: fiber post; silanization; polymerization mode; resin-based luting agent; bond strength; interfacial integrity.

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## Introduction

Endodontically treated teeth are known to present a higher risk of biomechanical failure than vital teeth, and some of them are also severely damaged by decay, trauma, excessive wear or previous restorations. In such conditions, posts are needed to allow the clinician to rebuild enough tooth structure to retain restorations (1). The use of fiber posts has recently increased in popularity since they were introduced in the 1990s and have considerably improved the rehabilitation of endodontically treated teeth (2,3). However, some clinical studies have pointed out that insufficient bonding performance of fiber posts may still result in clinical failure (4,5). Many *in vitro* studies have investigated various factors that may affect the retention of posts. These factors include length, design, diameter, and surface treatment of the post, and the type of luting materials employed (6-8).

Surface treatment is a common method for improving the general adhesion properties of a material, by facilitating chemical and micro-mechanical retention between different constituents. Some studies have confirmed that mechanical conditioning methods (e.g., sandblasting or etching) can improve bonding between a fiber post and a resin-based luting agent (9). However, these techniques produce substantial damage to the glass fibers in the post, and thus compromise its integrity. Silane coupling agent is a hybrid organic-inorganic compound that can mediate adhesion between inorganic and organic matrices through intrinsic dual reactivity (10). Application of silane is a well known and recommended method for improvement of bonding between metal and resin (11), or between porcelain and resin (12). The application of a silane coupling agent as an adhesion promoter between a fiber post and a resin composite was recently investigated (13,14).

Although it is possible to transmit light through a translucent post, use of an exclusive dual-curable or self-curable resin-based luting agent is recommended for luting a fiber post into a root canal (15). The effect of different resin-based luting agents on post retention has been investigated extensively, and various conclusions have been drawn (16,17). Different manufacturers have different recommendations as to whether or not an adhesive agent should be used.

The present study was conducted with the aim of measuring, through the micro-tensile non-trimming technique and scanning electron microscopy (SEM), the bonding between Easy Post, a prefabricated translucent post, and different resin-based luting agents, with or without silanization of the post surface, or light-irradiation of the adhesive and luting agent. The null hypotheses tested in this study were: 1. The bond strengths achieved at the post/resin interface with various resin-based luting agent systems are not significantly different; 2. Surface silanization has no influence on the bonding between a fiber post and a resin-based luting agent; 3. The polymerization mode of an adhesive and a resin-based luting agent does not affect the bonding performance of a fiber post.

## Materials and Methods

Information about the fiber post, silane coupling agent, adhesive and resin-based luting agents used in this study is summarized in Table 1.

An Easy Post with a diameter of 2.0 mm in the cylindrical coronal portion was insulated with Teflon foil (0.2 mm thick) and positioned upright on a glass slab with a drop of sticky wax. Then a cylindrical Teflon matrix with a diameter of 10 mm was placed around the post and adjusted to ensure that the post was exactly in the middle. In height, the matrix was extended only to the cylindrical portion of the post. The two components of the resin-based luting agent (same as the material used for luting) were mixed and applied to the matrix, and light-irradiated for 20 sec with a halogen curing light (600 mW/cm$^2$ output; VIP; Bisco Inc., Schaumburg, IL, USA) from each side of the cylinder to ensure optimal polymerization. After polymerization, the insulated post was removed, and a mold with a cavity in the center for the post space was obtained (Fig. 1). Fabrication of this mold and its usage for luting was done within one day. Before luting, these molds were warmed to 37°C for 24 h.

Sixty Easy Posts were cleaned in an ultrasonic bath with 95% ethanol for 3 min and air dried. The posts were then divided into 10 groups (n = 6), according to the different resin-based luting agent systems and bonding

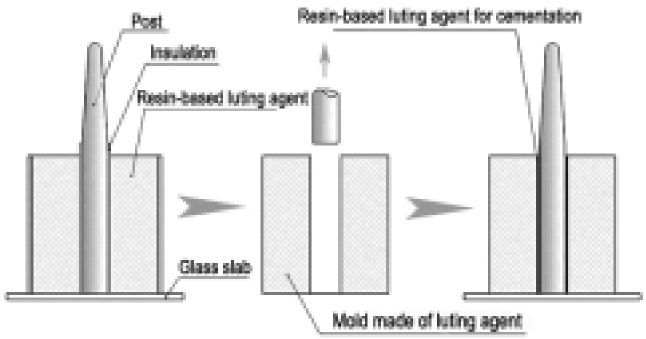


Fig. 1 Procedure used in this study for making the mold and luting the post.

Table 1 Information on the materials tested*

| Material | | Chemical composition | Manufacturer |
|---|---|---|---|
| Fiber post | Easy Post | silicium fibers enriched with zircon (60%)<br>epoxy resin matrix (40%) | Dentsply Maillefer, Ballaigues, Switzerland |
| Adhesive | XP Bond | PENTA, TCB resin, UDMA, TEGDMA, HEMA, nanofiller, camphorquinone, DMABE, tert-butanol | Dentsply DeTrey, Konstanz, Germany |
| | Self-Cure Activator (SCA) | UDMA, HEMA, catalyst, photoinitiators, acetone, water | |
| Silane coupling agent | Calibra | organo silane, acetone, ethyl alcohol | Dentsply DeTrey, Konstanz, Germany |
| | Monobond-S | 3-MPS, ethanol/water-based solvent, acetic acid | Ivoclar-Vivadent, Schaan, Liechtenstein |
| Resin-based luting agent | Calibra | BisGMA, EBPADM, TEGDMA, butylhydroxytoluol, benzoyl peroxide, barium glass, silica | Dentsply DeTrey, Konstanz, Germany |
| | FluoroCore 2 | UDMA, di-&tri-functional methacrylates, barium boron, fluoroaluminosilicate glass, silicon dioxide, benzoyl peroxide | |
| | Multilink (Self-curable) | dimethacrylate, HEMA, barium glass, $YbF_3$, spheroid mixed oxide. | Ivoclar-Vivadent, Schaan, Liechtenstein |
| | Variolink II | BisGMA, UDMA, TEGDMA, barium glass, silica fillers, $YbF_3$ | |

* Information provided by manufacturers.
PENTA: Dipentaerytritolpentacrylate-phosphoricacid-monomer; TCB resin: Carboxylic acid modified dimethacrylate resin
DMABE: Ethyl-4-dimethylaminobenzoate; EBPADM: Ethoxylated bisphenol A dimethacrylate

procedures employed (Table 2). For groups where silanization was employed, the silane coupling agent was applied to the post surface and air dried 60 sec later. For groups where adhesive was used, the XP Bond and SCA were mixed in a 1:1 ratio and applied to the post surface for 20 sec. After surface treatment, the resin-based luting agent was mixed and injected into the post space of the mold. Remaining luting agent was placed on the post surface and then the post was carefully placed in the center of the cavity. Superfluous luting agent was removed by a brush before polymerization. For groups where the luting agent was self-polymerized, the mold with the luted post was stored at 37°C for 10 min. For groups where the luting agent was dual-polymerized, the resin-based luting agent was light-irradiated for 40 sec with a halogen curing unit directly from the open upper side of the mold and through the post. The specimen cutting and loading procedures were started immediately.

## Micro-tensile bond strength test

Five specimens in each group were mounted in a cutting machine (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) with sticky wax and sectioned under water cooling to obtain a slab of uniform thickness, with the post in the center and resin-based luting agent on each side. A medium of 6-8 sticks 1 mm thick were obtained from each slab, resulting in at least 30 specimens per group that were available for micro-tensile testing. Sticks were glued with cyanoacrylate (Super Attak Gel, Henkel Loctite Adesivi S.r.l., Milan, Italy) to the two free-sliding components of a jig, which was mounted on a universal testing machine (Triax, Controls S.P.A., Milan, Italy) and loaded in tension at a speed of 0.5 mm/min until failure occurred at either side of the post-cement interface. The calculated bond strength was determined by dividing the maximal force applied during the test by the bonded area. As the bonded interface was curved, the area was calculated using a mathematical formula previously applied by Bouillaguet et al. (18). Specimens that failed prematurely during the cutting or gluing phases were considered to have a bond strength of 0 MPa.

Statistical analysis was performed using SPSS11.0 software (SPSS Inc., Chicago, IL, USA). The means of each group were analyzed by one-way analysis of variance (ANOVA) and a Tamhane test was used for all *post hoc* pairwise comparisons.

## SEM observation

The remaining one bonded specimen in each group was used for SEM observation. Each cylindrical specimen was sectioned into slabs 1.5 mm thick to observe the cross-section of the bonded interface. After being polished with wet silicon carbide paper of increasing grits (No. 360, 600, 1,000 and 1,200), all slabs were etched with 32% phosphoric acid etchant (UNI-ETCH, Bisco Inc., Schaumburg, IL, USA) for 30 sec, rinsed with water and

Table 2a Micro-tensile bond strengths (MPa) between fiber post and different resin-based luting agents with recommend bonding procedures

| Group | Silane | Adhesive | Polymerization mode of adhesive | Resin-based luting agent | Polymerization mode of cement | Bond strength (MPa) | Tamhane post-hoc* |
|---|---|---|---|---|---|---|---|
| 1 | Calibra | XP + SCA | NL | Calibra | DP | 19.82 ± 8.78 | A |
| 2 | Calibra | XP + SCA | NL | FluoroCore2 | DP | 16.15 ± 8.39 | A |
| 3 | Monobond S | | | Multilink | SP | 3.02 ± 4.09 | B |
| 4 | Monobond S | | | Variolink II | DP | 5.60 ± 5.40 | B |

Table 2b Micro-tensile bond strengths (MPa) between fiber post and Calibra resin-based luting agent with different bonding procedures

| Group | Silane | Adhesive | Polymerization mode of adhesive | Resin-based luting agent | Polymerization mode of cement | Bond strength (MPa) | Tamhane post-hoc* |
|---|---|---|---|---|---|---|---|
| 5 | | | | Calibra | DP | 3.06 ± 3.55 | B |
| 6 | Calibra | | | Calibra | DP | 7.07 ± 5.87 | B |
| 7 | | XP + SCA | NL | Calibra | DP | 16.64 ± 5.91 | A |
| 8 | | XP + SCA | NL | Calibra | SP | 14.80 ± 8.68 | A |
| 9 | Calibra | XP + SCA | NL | Calibra | SP | 14.52 ± 6.54 | A |
| 10 | Calibra | XP + SCA | LR | Calibra | DP | 19.35 ± 7.12 | A |
| 1 | Calibra | XP + SCA | NL | Calibra | DP | 19.82 ± 8.78 | A |

*: Groups identified with the same letter were not significantly different ($P > 0.05$).
NL: No light- irradiating; LR: Light-irradiating for 10 sec; SP: Self-polymerization; DP: Dual-polymerization.

air dried, which was merely for cleaning purposes and did not affect the surface structure. Then each slab was mounted on a metal stub, gold-sputtered (Polaron Range SC7620; Quorum Technology, Newhaven, UK), and observed using a scanning electron microscopy (JSM 6060 LV, JEOL Ltd., Tokyo, Japan) at different magnifications.

## Results

### Micro-tensile bond strength test

The values of micro-tensile bond strength for each group are presented in Table 2. As Table 2a shows, the mean bond strengths achieved by Calibra and FluoroCore 2 resin-based luting agent systems were significant higher than those of Multilink and Variolink® systems ($P < 0.05$). There were no significant differences among the mean values for groups 1 and 2, or between groups 3 and 4 ($P > 0.05$).

As shown in Table 2b, for the Calibra resin-based luting agent system, silanization on the post surface had no significant influence on the bond strength, irrespective of whether it was combined with adhesive or not ($P > 0.05$). Use of XP/SCA adhesive significantly improved the bond strength ($P < 0.05$). When XP Bond/SCA adhesive was used, the polymerization mode of the luting agent had no influence on the bond strength ($P > 0.05$). Light-irradiation of the XP/SCA adhesive did not improve the bond strength ($P > 0.05$).

### SEM observation

The cross-sections of groups 3-6 were similar: there were several gaps or bubbles at the interface between the resin-based luting agent and the post, but there were no defects between the luting agent and the mold (Fig. 2). For the other groups with higher bond strengths, there were no visible defects between the fiber post and the resin-based luting agent, but some gaps or bubbles were present at the interface between the luting agent and the mold (Fig. 3).

## Discussion

On the basis of the results, the first null hypothesis was rejected, while the second and third null hypotheses were accepted.

To find the best combination of resin-based luting agent with Easy Post, four different commercially available resin-based luting agent systems were used in this study. In accordance with the manufacturer's instructions, after silanization of the fiber post, Multilink and Variolink II resin-based luting agents were applied directly to the post surface, without using an adhesive. However, both of them showed low bond strengths with Easy Post. SEM observations also confirmed poor integrity at the interfaces between these two luting agents and the post surface. It may be speculated that the higher viscosity of these resin-based luting agents restricted their flow along the post space in the mold or wetting on the surface of the fiber post (19). On the other hand, Calibra and FluoroCore2 resin-based luting agents combined with relevant adhesive showed excellent bonding performance with Easy Post. Therefore, the Calibra resin-based luting agent system was employed for further investigation of the effect of different bonding procedures on micro-tensile bond strength between the fiber post and resin-based luting agent.

Some studies have found that silanization significantly improves the bond strength of fiber posts (14,20). However, in the present study, silanization had no significant effect on the bond strength of fiber posts ($P > 0.05$), consistent with the results reported by Sahafi et al. and Perdigao et al. (21,22). The main reason for these different results may be the micro-structure and composition of the different fiber posts. Most commercially available fiber posts contain epoxy resin as the matrix connecting the individual fibers, which has no functional groups to react with a silane

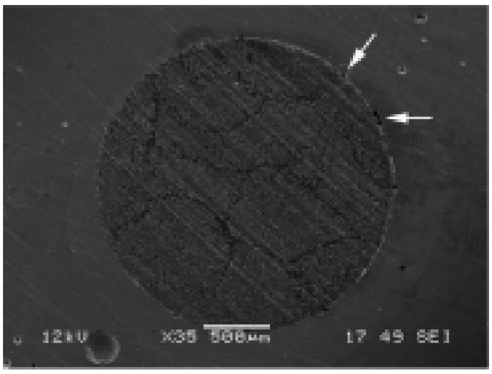


Fig. 2 Cross-section of specimen in group 3 at ×35 magnification. Bubbles are evident between the fiber post and the resin-based luting agent (arrow).

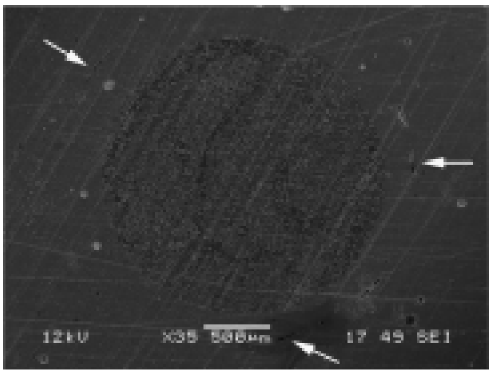


Fig. 3 Cross-section of specimen in group 1 at ×35 magnification. Gaps are evident between the resin-based luting agent and the mold (arrow).

coupling agent. However, the silicates on the surface of silicate-based fiber contain hydroxyl (-OH) groups, which can form covalent bonds with the silanol group of the silane coupling agent (23). It may be speculated that most of the silicium fibers on the surface of Easy Post were coated by epoxy resin, thus limiting the quantity of exposed fibers. It is also noteworthy that a new kind of silicium fiber enriched with zircon is employed in Easy Post, although the detailed composition is unknown. Some studies have suggested that silanization cannot improve the bonding performance of zirconia-based ceramics (24,25). Therefore, it may also be speculated that the silicium fiber employed in Easy Post cannot react with silane coupling agent.

It has been proved that sufficient bond strength or conversion of polymer matrix cannot be achieved if a dual-curable resin-based luting agent is not exposed to a light-curing unit, or if the light is attenuated (26-28). In the present study, the polymerization mode of Calibra resin-based luting agent showed no significant influence on the bond strength between the fiber post and the resin-based luting agent ($P > 0.05$), which was inconsistent with previous studies (27,28). This may be attributable to the effect of XP Bond/SCA adhesive used in the present study.

XP Bond combined with a Self-cure activator (SCA) is a dual-curable adhesive, indicated for bonding indirect restorations in conjunction with a dual-curable or self-curable resin-based luting agent. The present results confirmed that application of XP Bond/SCA adhesive was able to markedly improve the bond strength between Calibra resin-based luting agent and a fiber post ($P < 0.05$). Neither application of silane, nor light-irradiation of the adhesive mixture, nor light-irradiation of Calibra resin-based luting agent significantly increased the bond strength further ($P > 0.05$). This excellent performance may have been due to some important components in this new adhesive. The use of PENTA and TCB resin as adhesion promoters and wetting agents promotes chemical interaction between the monomers and the bonded substance. HEMA can also increase penetration into the bonded substance and ensure high bond strength. The use of tert-butanol as a solvent increases the contents of resin and nanofiller, both of which can improve a number of properties of the adhesive layer. The catalyst in SCA can promote adhesion of compatible self-curable or dual-curable resin-based luting agents to the adhesive layer, and accelerate their polymerization. Therefore, application of XP Bond/SCA adhesive to the fiber post surface not only improves the bond strength, but also simplifies the bonding procedure.

In order to simulate the clinical luting procedure, in which just a small amount of resin-based luting agent surrounds the post, molds with a post space were used in this study. Some previous studies directly made cylindrical specimens with the post in the center by applying a resin-based luting agent into a cylindrical Teflon matrix (14,29). According to the C-factor theory (30), there would be a greater chance for releasing the shrinkage stress caused by polymerization of resin-based luting agent. However, SEM observation of groups 1-2 and groups 7-10 in the present study showed that there were no defects between the fiber post and the resin-based luting agent, although some gaps or bubbles were found at the interface between the resin-based luting agent and the mold. It can be speculated that, in these groups, the shrinkage stress of the resin-based luting agent during polymerization was higher than the bond strength between the resin-based luting agent and the mold, but lower than that between the post and the resin-based luting agent. Comparison with previous studies suggests that use of a mold may more accurately reflect the bonding characteristics at the interface between a fiber post and a resin-based luting agent.

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