Adhesion of two dual-cure core resins to silica fiber posts treated with different bonding agents

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Abstract

Objectives: To evaluate the adhesion of core resin to silica fiber posts treated with different bonding agents.

Materials and Methods: Twenty-eight silica fiber posts (Tech21 Xop®) were divided into four groups according to the different bonding agents to be applied to the post surface: Group 1: Clearfil SE Bond; Group 2: Clearfil SE Bond plus Clearfil Porcelain Bond Activator; Group 3: PR.-Bond Ethanol; Group 4: PR.-Bond Ethanol plus PR.-Bond Activator. Two dual-cure core resins were used for core build-up: Clearfil DC Core Automix (Groups 1 and Group 2) and DC Flow Core (Groups 3 and Group 4). A non-trimming microtensile test was performed and the data were analyzed using one-way ANOVA and Tamhane post-hoc tests. The bonding interface in each group was also evaluated under SEM.

Results: The µTBS of Group 2 (22.30 ± 8.26 MPa) was significantly higher than that of Group 3 (15.34 ± 5.91 MPa, p<0.05). No significant difference in µTBS was found between the other two groups (p>0.05). The percentage of adhesive failures recorded in Group 2 was significantly lower than that of other groups (p<0.001). SEM Observation revealed a continuous and tight bonding interface in Groups 1, 2, and 3 and dispersed interfacial gaps and voids in Group 4.

Conclusions: The bond strength between core resin and fiber posts was affected by different combinations of bonding agents and core resins. The type of bonding agent had no effect on post-core bond strength for either of the two core resins used in this study.

Clinical significance: Within the limitations of this in vitro study, a dual-cure core resin coupled to a post treated with a combined silane/adhesive agent may be a good choice for restoring a tooth using a fiber post.

Short title: Adhesion of core resin to fiber posts.

Key words: fiber posts, microtensile bond strength, bonding agent, silane coupling agent

Introduction

A post-core restoration is usually recommended for endodontically treated teeth in cases where little coronal tooth tissue remains or where the teeth need to be used as abutments [1-4]. Recently, the use of fiber-reinforced composite posts has increased due to their good biomechanical compatibility with dentin, which produces more favorable stress distribution, decreases the occurrence of root fractures in general and decreases the risk of catastrophic root fracture [5-10].

Unlike metallic posts, the most frequent failure of fiber post restoration was not due to fracture, but to debonding, which may occur between fiber post and resin or between resin and intraradicular dentin [8-10]. The adhesion between resin and intraradicular dentin is considered to be a weak point in luting a fiber post [11,12]. However, it should be noted that a reliable bond between fiber post and core resin also plays an important role in the post-core restoration of endodontically treated teeth.

Certain mechanical and chemical treatments of post surface such as sandblasting, airborne-particle abrasion and silane coupling have shown favourable results in terms of improving the bond strength between fiber posts and core resins [13-15]. Compared with bonding to dental substrates, the adhesion of fiber posts to resin cement was still inferior due to an absence

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Table 1. Bonding agent and procedure of post surface treatment for each group

Group	Bonding agent	Type	Manufacturer	Procedure
1	Clearfil SE Bond	Two-step self-etch bonding agent	Kuraray Medical Inc., Japan	Apply SE primer for 20s, gently air dry, apply SE Adhesive, gently air dry, light cure for 10s.
2	Clearfil SE Bond plus Clearfil Porcelain Bond Activator	Two-component silane coupling bonding agent	Kuraray Medical Inc., Japan	Apply a mixture of SE primer and Clearfil Porcelain Bond Activator (1:1) for 20s, gently air dry, apply SE adhesive for 10s, light cure for 10s.
3	PR.-Bond Ethanol	Light-cure one bottle bonding agent	S&C Polymer GmbH, Elmshorn, Germany	Apply PR.-Bond Ethanol for 20s, gently air dry, light cure for 20s.
4	PR.-Bond Ethanol plus PR.-Bond Activator.	Self-cure one bottle bonding agent	S&C Polymer GmbH, Elmshorn, Germany	Apply a mixture of PR.-Bond Ethanol and PR.-Bond activator (1:1) for 20s, gently air dry.

of chemical bonding[16-18]. Goracci et al[15] found that the application of a silane coupling agent could improve the bond strength of fiber posts to flowable composites by creating a chemical bonding at the bonding interface. Aksornmuang et al [19, 20] reported that the application of the bonding agent to the post surface could improve the bond strength between fiber post and core resin, which is dependent on the curing mode of bonding agent and type of fiber post. Furthermore, the application of a bonding agent in combination with a silane coupling agent was also found to be efficient in improving the bond strength between fiber posts and core resin [19, 20, 26]. However, few studies on the effect of different bonding agents applied to the post surface on the adhesion to fiber posts have been reported.

The purpose of this in vitro study was to evaluate the adhesion of two core resins to silica-fiber posts treated with different bonding agents. The null hypotheses to be tested were: 1. there was no significant difference of bond strength in groups with different combinations of core resin and bonding agent; 2. for each core resin, the different bonding agent has no effect on its bond strength to fiber posts.

Materials and methods

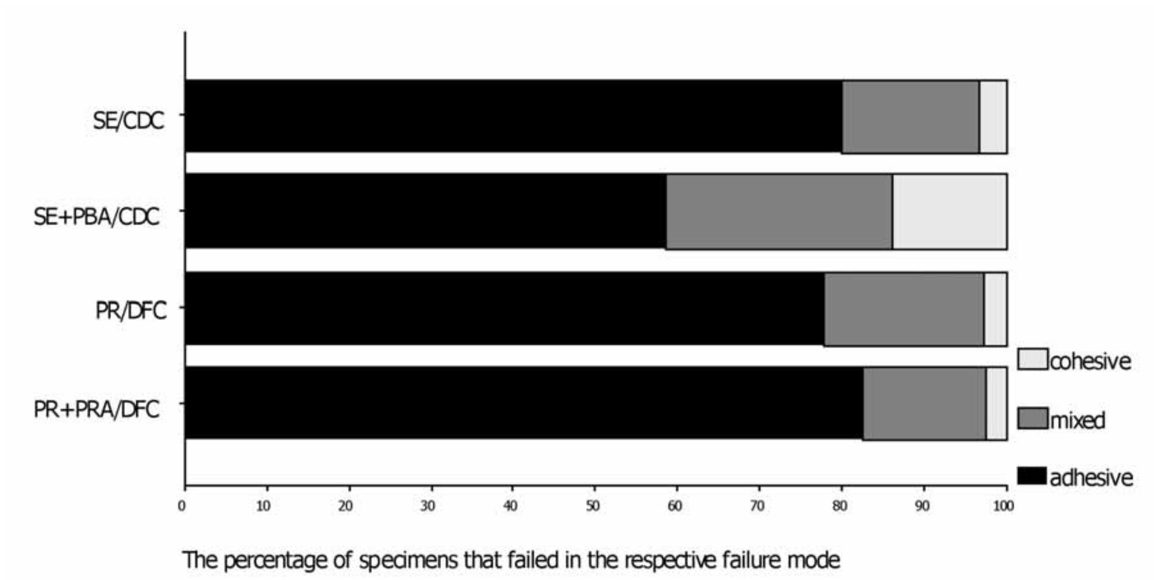
Specimen preparation

Twenty-eight silica fiber posts (Tech21 Xop®, Carbotech Ganges, France) with a maximum diameter of 1.6 mm were used in this study. The posts were divided into four groups according to the different bonding agents to be applied to the post surface. The bonding agent and procedure of post surface treatment for each group is reported in Table 1.

After post surface treatments, a core build-up was performed following the procedure described by Goracci et al [15]. Two dual-cure resin core materials were used: Clearfil DC Core Automix® (Kuraray Medical Inc., Tokyo, Japan) for Group 1 and Group 2 and DC Flow Core® (S&C Polymer GmbH, Elmshorn, Germany) for Group 3 and Group 4. The core resins were mixed (catalyst and universal 1:1) and dispensed into a transparent cylindrical plastic matrix with an automix syringe provided by the core resin manufacturer. The matrix was placed on a glass plate to ensure the bottom of the core was level. The fiber post was inserted carefully into the middle of the matrix filled with the core resin and held by forceps at the apical end. The direction and position of the post were adjusted to make it perpendicular to the bottom of the core. The core material was light cured for 20 s with a halogen curing light (VIP, Bisco, Schaumburg, IL, USA) directly from the open upper side of the matrix and through the post. In order to promote complete polymerization of the core material, additional irradiations for 20 s were performed from each side of the cylinder prior to the removal of the matrix and from the bottom of the cylinder immediately after the removal. The specimens were then stored in water for 24 h at room temperature.

Microtensile bond strength (μTBS) tests

After storage in water, five specimens from each group were randomly selected for μTBS tests. The specimens were stabilized in the grip of a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) and sectioned under water cooling. Two longitudinal cuts along the post axis were made



Graph 1: Failure distribution
CDC: Clearfil DC Core Automix; DFC: DC FlowCore; SE: Clearfil SE Bond; PR: PR.-Bond Ethanol; PBA: Clearfil Porcelain Bond Activator; PRA: PR.-Bond Activator.

to form a slab with the post in the center of the resin while two parallel surfaces of the post were exposed throughout its length. The slab was then sectioned perpendicularly to the post axis into sticks with a thickness of 1mm. Seven to nine sticks were obtained from each specimen - thirty-one to forty sticks for each group. The dimension of the bonding area of each stick was measured using a digital caliper. The bonding area was calculated using a mathematical formula previously used by Bouillaguet et al [21]. Two free ends of each stick were fixed by a cyanoacrylate glue (Super Attak Gel®, Henkel Loctite Adesivi S.r.l., Milano, Italy) to the two sliding components of a jig, which was mounted on a universal testing machine (Triax, Controls S.P.A., Milano. Italy). A microtensile bond strength test was performed with a cross-head speed of 0.5mm/min. The force at the point of a sharp drop along the load/time curve was observed and recorded as the fracture load. The bond strength was finally expressed in MegaPascals (MPa) by dividing the load with the bonding surface area. The microtensile bond strengths of prematurely failed specimens (debonded during the cutting procedure) were recorded as zeros. The failure mode of each specimen was assessed under a stereomicroscope (Nikon SMZ645, Tokyo, Japan) at 40x magnification and classified as adhesive, cohesive and mixed. The percentage of each kind of failure mode within each group was then calculated. The following criteria were used to assess the failure mode:

Adhesive: failure at the post-resin interface.

Cohesive: failure within the post or failure within the core resin.

Mixed: a combination of an adhesive and cohesive failure at the same interface

Scanning electronic microscope (SEM) observation

The remaining two specimens from each group were used for SEM observation (JSM 6060 LV, JEOL, Tokyo, Japan). Using a slow speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling, the specimens were sectioned perpendicularly to the post axis into slabs of 1.5mm thickness. One surface of the slab was selected to make the observation and polished with wet silicon carbide abrasive papers with 560, 1100, 4000-grit respectively. The polished surface was then etched with 32% phosphoric acid gel (Unietch®, Bisco.Inc, Shaumburg, IL, USA) for 30s, thoroughly rinsed with water, air dried, gold-sputtered (Polaron Range SC7620, Quorum Technology, Newhaven, UK) and observed at 55x and 300x magnification.

Statistical analysis

The microtensile bond strength data were first verified by the Kolmogorov-Smirnov test for their normal distribution and by Levene's test for the homogeneity of variances. Then one-way ANOVA was performed and Tamhane Post Hoc Test was used for multiple comparisons. The failure mode data were analyzed using the Chi-Square test. In all the tests, the level of significance was set at $p<0.05$ and calculations were handled by the SPSS 11.0 software (SPSS Inc.; Chicago, IL, USA).

Results

Microtensile bond strength tests

The results of microtensile bond strength tests are reported in Table 2. The one-way ANOVA revealed that there was a significant difference in µTBS between the groups ($p<0.05$). The µTBS in Group 2 was significantly higher than that of Group 3. No significant difference in µTBS was found between the other two groups ($p>0.05$). The Chi-Square test revealed that there were significant differences in failure distribution between the groups ($p<0.05$). The percentage of adhesive failures in Group 2 was significantly lower than in Groups 1, 3

Table 2. Microtensile bond strength values (MPa) in the experimental groups

Group	Core resin	Post surface treatment	Mean (SD)	Tamhane post-hoc test
1	Clearfil DC	Clearfil SE Bond	20.25 (11.82)	AB
2	Core Automix	Clearfil SE Bond plus Clearfil Porcelain Bond Activator	22.30 (8.26)	A
3	DC	PR.-Bond Ethanol	15.34 (5.91)	B
4	FlowCore	PR.-Bond Ethanol plus PR.-Bond Activator.	17.22 (7.69)	AB

*Different letters indicate statistically significant differences (p<0.05)

and 4 (Graph 1).

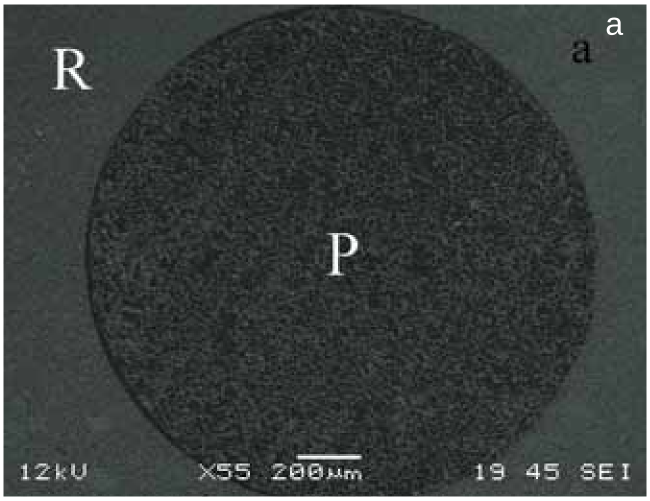
SEM Observation

The SEM images are presented in Figures 1, 2 and 3. The images of specimens from Group 1 and Group 2 were similar: a continuous bonding interface was observed with no interfacial gaps and voids, and a visible adhesive layer along the interface was detected as well (Figure 1). No visible adhesive layer along the interface was observed in both Group 3 and Group 4. A continuous bonding interface with no interfacial gaps and voids was observed in specimens from Group 3 (Figure 2), while dispersed interfacial gaps and voids were detected in specimens from Group 4 (Figure 3).

Discussion

There was a significant difference in µTBS between Group 2 and Group 3. Thus, the first null hypothesis was rejected. For any of the two groups using the same core resin, the µTBS was not significantly affected by the bonding agent applied to the post surface. The second null hypothesis could not therefore be rejected.

The effect of applying a bonding agent to the post surface on the adhesion of composite resins to fiber posts, which is related to the chemical composition and viscosity of a bonding agent, compatibility between resin cement and post matrix, etc is inconsistent with literature [19, 20, 22]. Clearfil SE Bond used in Group 1 is a two-step self-etch bonding agent. In Group 2 it was used in combination with Porcelain Bond Activator and therefore functioned as a silane coupling agent. Although insignificantly, the µTBS of Group 2 was higher than that of Group 1, and the percentage of adhesive failure recorded in Group 2 was significantly lower than in Group 1. The authors speculate that using the silane-containing bonding agent Clearfil SE Bond plus Clearfil Porcelain Bond Activator could improve the adhesion of core resin to fiber post. This is in accordance with the results of Aksornmuang et al and



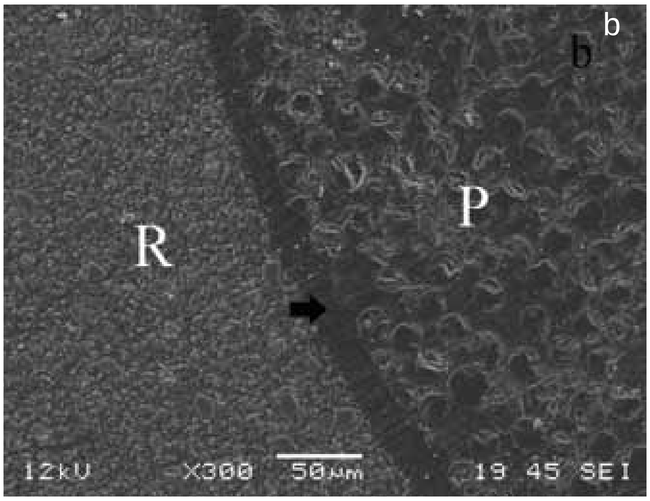


Figure 1a and 1b: SEM micrographs of post-core interfaces in groups using Clearfil DC Core Automix as the core resin (P: fiber post; R: core resin): a: Low magnification showed a continuous bonding interface with no interfacial gaps and voids; b: High magnification showed a visible adhesive layer (arrow).

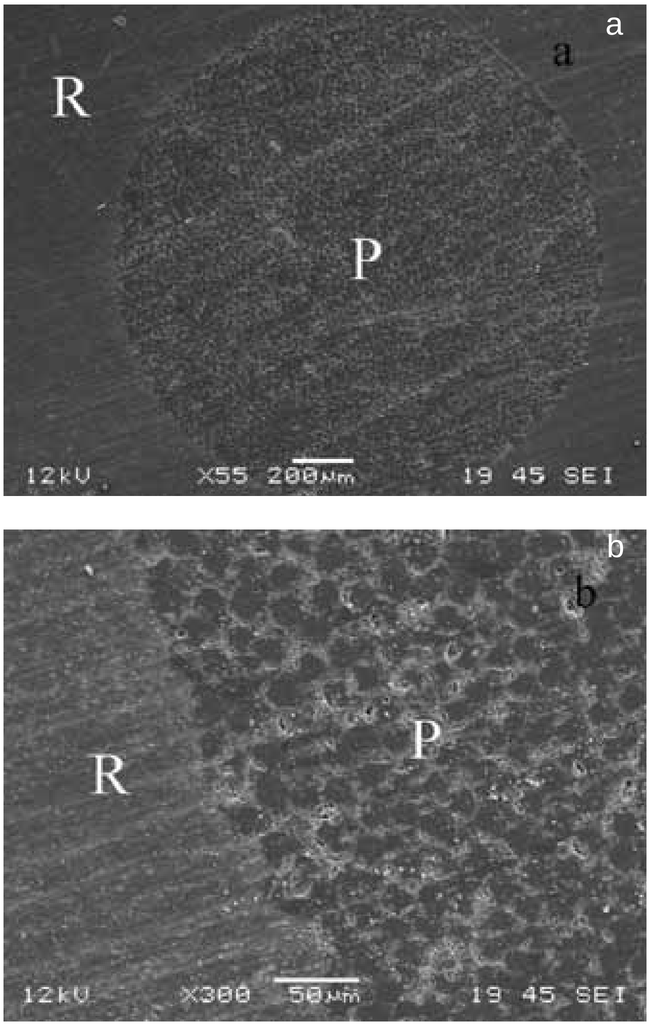


Figure 2a and 2b: SEM micrographs of post-core interfaces in Group 3 (P: fiber post; R: core resin): a: Low magnification showed a continuous bonding interface with no interfacial gaps and voids. b: High magnification: no visible adhesive layer was observed.

Monticelli et al [19, 20, 23]. Silanization is a conventional surface conditioning method used in dentistry, which can promote adhesion through the formation of covalent bonds between silanol group of silanes and hydroxyl –OH groups of inorganic substrates [24]. Porcelain Bond Activator used in Group 2 contains γ-methacryloxy propyltrimethoxy silane (γ-MPS), which can rapidly hydrolyze after mixing with acidic SE primer and may form a siloxane bond with the silica contained in Tech21 Xop® posts. However, the effect of a silane coupling agent on the adhesion to fiber posts is still uncertain. Some researchers reported that silane did not significantly increase the bond strength of fiber posts to resin cement [13, 25]. They considered the reason to be a weak or absent bond of the silane functional group to the nonsilicate-based epoxy resin which is highly-crossed linked around the glass or quartz fibers. Besides the chemical bridge function of a silane coupling agent, the wetting ability of the agent itself may also help to improve the bond strength. That may be the reason why the effect of Clearfil SE Bond plus Clearfil Porcelain Bond Activator on the bond strength of core resin to fiber posts was not significant compared with that of Clearfil SE Bond. The continuous bonding interface with no interfacial gaps and voids in Group 1 and Group 2 also revealed a good adaptation of Clearfil SE Bond and Clearfil SE Bond plus Clearfil Porcelain Bond Activator to the post surface, which may be related to the wetting ability of the bonding agent.

PR.-Bond Ethanol, used in Group 3, is a one-bottle light-cured bonding agent that can be self-cured after being mixed with PR.-Bond Activator. There was no significant difference in the bond strength between Group 3 and Group 4. It appeared that the bond strength of DC FlowCore to fiber posts was not affected by the curing mode of the bonding agent that was applied to the post surface, which does not conform to the results of Aksornmuang et al [19, 20]. A possible explanation is the different type of bonding agent, fiber post and core resin used in our study. Both PR.-Bond Ethanol and PR.-Bond Activator are experimental materials and information on their composition was unavailable. Therefore, it is still unknown whether the chemical and physical characteristics of PR.-Bond Ethanol may alter after being mixed with PR.-Bond Activator, thus having effect on the bond strength . Moreover, dispersed gaps and voids were observed at the bonding interface in Group 4 with PR.-Bond Ethanol plus PR.-Bond Activator, while a continuous and tight bonding interface was observed in Group 3 with PR.-Bond Ethanol. The intermixing of core resin with the unset PR.-Bond Ethanol plus PR.-Bond Activator on the post surface may have produced voids and gaps during post placement and may have impaired the adaptation of core resin to post surface.

With regard to the combination of a bonding agent applied to the post surface and core resin, Group 2 exhibited a higher bond strength than the other groups. Besides the effect of the silane coupling agent mentioned above, the superior chemical compatibility between Clearfil SE Bond and Clearfil DC Core Automix may have played a role. The solvent of the bonding agent which can affect its wetting ability should also be taken into account [26]. Clearfil SE Bond and Clearfil SE Bond plus Clearfil Porcelain Bond Activator, used in Group 1 and Group 2, are water-based bonding agents, while PR.-Bond Ethanol and PR.-Bond Activator, used in Group 3 and Group 4, are ethanol-based bonding agents. Clinicians are usually faced with the predicament of choosing the optimal resin materials for fiber post restoration. Flowable and light cure materials were found to have better interfacial adaptation to fiber posts and while easier to handle, also showed less reliable mechanical properties [27, 28]. Dual-cure resin core materials were recently introduced to post-core restorations. A major advantage of these materials is their similar elastic modulus to dentine and

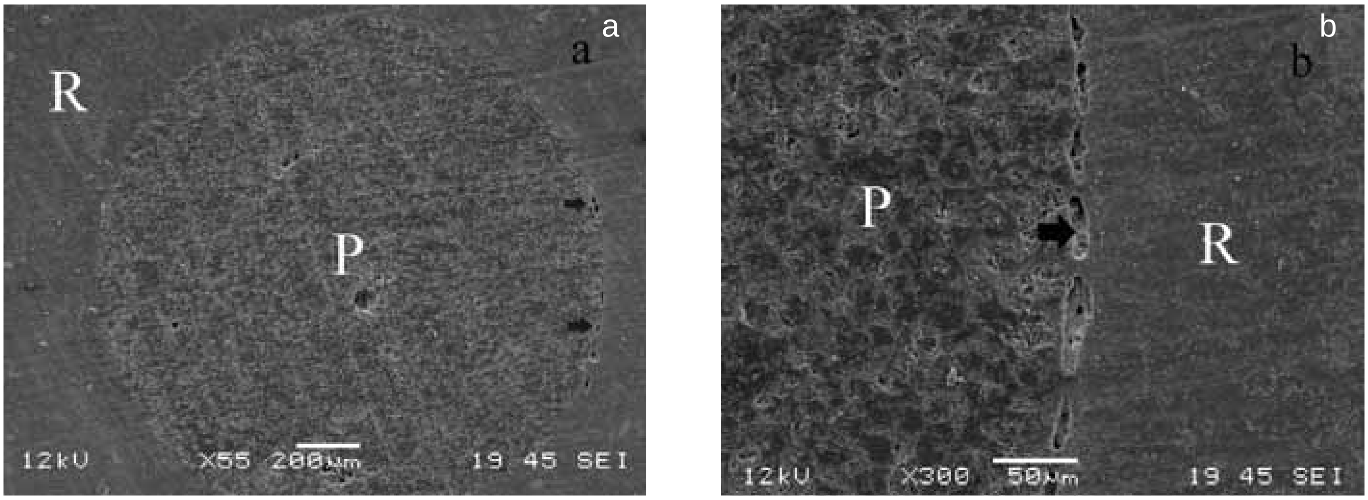


Figure 3a and 3b: SEM micrographs of post-core interfaces in Group 4 (P: fiber post; R: core resin):
a: Low magnification: dispersed interfacial gaps and voids (arrow) were detected; b: High magnification: gaps and voids (arrow) along the interface between the adhesive layer and core resin were observed.

fiber posts, which helps to establish a monobloc unit of post-core-dentine that is expected to result in favorable stress distribution under the masticatory load. They can be used simultaneously as both cement and core materials, simplifying the clinical procedure [11, 20]. Clearfil DC Core Automix and DC FlowCore, used in this study, are both such dual-cured resin core materials. Therefore, Clearfil DC Core Automix in combination with a silane coupling agent applied to the post surface would appear to be appropriate for a tooth restoration using a fiber post.

The Tech21 Xop® posts used in this study are manufactured from silica fibers with a special resin obtained through the polymerization of diphenilpropane and metiloxirane (dpp MOR). Boschian et al [11] reported that when used simultaneously with 10-methacryloyloxydodecyl dihydrogen phosphate (MDP) containing resin, a high bond strength value (29.038±0.798 MPa) could be obtained with the posts due to chemical affinity between post and luting material. It was anticipated that surface treatment with an MDP-contained adhesive and bond activator would promote chemical bonding. However, neither morphological observation nor mechanical tests confirmed this assumption.

Conclusions

Within the limitations of this study, the microtensile bond strength of core resin to fiber posts was not affected by the curing mode of a bonding agent applied to the post surface. The application of a silane coupling agent to the post surface could result in a slight improvement of the bonding of core resin to fiber posts. The bond strength between core resin and fiber posts was affected by different combinations of bonding agent applied to post surface and core resin.

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