THE ADHESION BETWEEN FRC POSTS AND RESIN CORE MATERIALS FOLLOWING DIFFERENT TREATMENTS OF THE POST SURFACE

CLAUDIA MAZZITELLI[1], ELISA MAGNI[1], IVANA RADOVIC[2], FEDERICA PAPACCHINI[1], CECILIA GORACCI[1], MARCO FERRARI[1]

Abstract

Objective: To evaluate the effect of surface treatments on the microtensile bond strength of fiber posts with different resin-based material used as core.

Methodology: 74 fiber posts (FRC Postec, Ivoclar Vivadent) were divided into four groups according to the surface treatment performed. Group 1: sandblasting only (Rocatec-Pre, 3M ESPE). Group 2: sandblasting and a silane application (Monobond S, Ivoclar Vivadent). Group 3: silane application only. Group 4: no treatment (control). Four core materials (Multicore Flow, Multicore HB, Tetric Flow, Tetric EvoCeram; Ivoclar Vivadent) were applied on the posts to produce cylindrical specimens. Specimens were cut to obtain microtensile sticks that were loaded in tension at a cross-head speed of 0.5 mm/min until failure. The fiber post surface morphology and post-core interface were evaluated by SEM. Statistical analysis was performed with two-way ANOVA and Tukey test for post-hoc comparisons ($p<0.05$).

Results: Both core material and surface treatment had a significant influence on bond strength ($p<0.05$) with the flowable materials performing significantly more effectively. Silanization with and without sandblasting resulted in a significantly higher bond strength. The interaction of core material and treatment was also significant ($p<0.05$). The highest bond strength was obtained with Tetric Flow on silanated posts.

Conclusions: Fiber post-core bond strength was influenced by the core material and post surface treatment. The highest bond strength was recorded with flowable materials on silanated posts with and without preliminary sandblasting.

Clinical Significance: Based on the present study, when silane is used as an adhesion promoter between FRC posts and core materials, higher bond strengths may be recorded with or without preliminary sandblasting of the post.

Short title:
Adhesion between FRC post and core materials after treating post surface.

Key Words:
fiber post, surface treatment, sandblasting, core materials, microtensile bond strength.

Introduction

Caries and subsequent endodontic treatment may lead to a significant reduction in the capability of a tooth to resist different conditions to which it is exposed in the oral environment. [1] Several methods have been proposed to overcome the problems of corono-radicular stabilization, with post-and-core system currently the most common treatment.[2-3] The reconstruction of endodontically-treated teeth with post and core is of great importance, particularly in terms of the retention of the subsequent prosthetic restoration. [4] Since their introduction in 1990, fibre-reinforced post systems have become part of daily practice to reintegrate the function of endodontically treated teeth.[5]

Fiber posts were primarily introduced as an alternative to cast metal posts [6-7] that had been used predominantly for many years. The cast post has superior physical properties [8], but is at an esthetic disadvantage due to the metallic grey colour that uniformly appears under the restoration. [9] In response to esthetic demands, glass and carbon fiber posts were introduced [10], offering a more natural and pleasing esthetic appearance to the patient [11-12-13]. Furthermore, they have an elastic modulus similar to dentin, which consequently allows a better stress distribution when compared with metal posts. [14-15] This was also assessed by

[1]Department of Dental Materials and Restorative Dentistry, University of Siena, Siena, Italy.

[2]Clinic for Pediatric and Preventive Dentistry, Faculty of Dentistry, University of Belgrade, Beograd, Serbia and Montenegro

Corresponding author:
Marco Ferrari, Chairman and Professor, Department of Restorative Dentistry and Dental Materials, University of Siena. Policlinico "Le Scotte", Viale Bracci, 53100, Siena, ITALY.
Tel: +39 0577 233131, Fax: +39 0577 233117,
E-mail: md3972@mclink.it

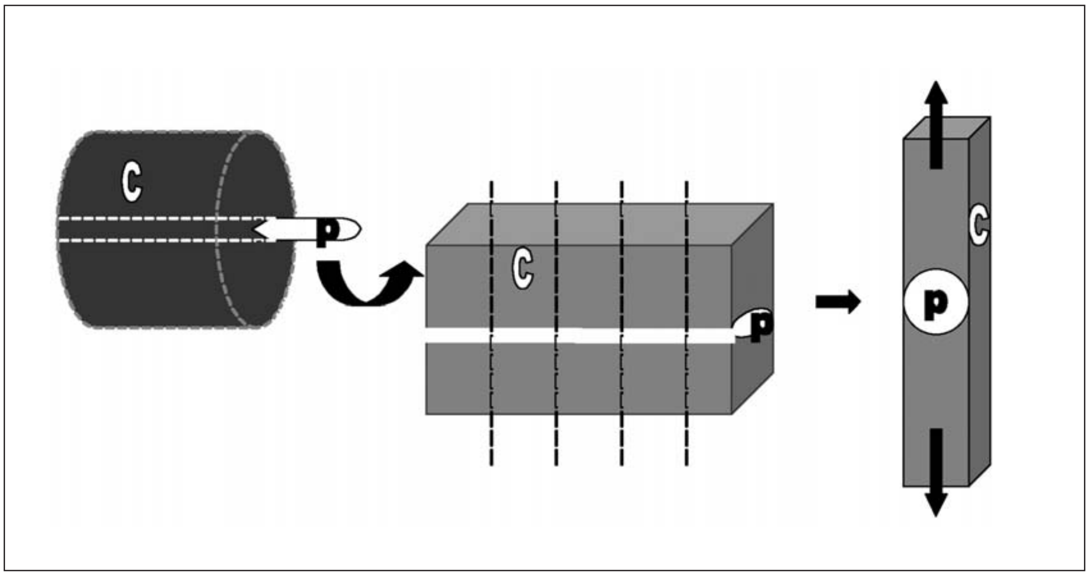


Figure 1: Schematic drawing representing the cutting procedures. The post was cut at its outermost periphery, in order to obtain sticks of 1 mm in thickness. C: composite; P: post.

recent finite element analysis studies.[16-17] In addition, fiber posts have improved clinical performance by saving chair-time and thus reducing costs[18]; increasing the transmission of light within the root[19]; making the post removal easier if re-treatment of the root canal is necessary[20-21] and eliminating the potential corrosion and allergic hypersensitivity[22].

When metal-free posts are used to restore endodontically treated teeth, the resin composite core material is usually chosen. A practical reason is that the immediate hardening of the composite allows the tooth to receive a crown restoration at the same appointment.[23] A variety of composite resin materials, including packable, microhybrid and flowable[24], is available for the core build-up procedure. According to the polymerisation mode, these can be further divided into light-activated, self-activated or dual-activated materials.

In order to improve the bond between post and core materials, many post surface treatments have been investigated, using mechanical procedures[25] or chemical agents[26]. In previous studies an enhanced adhesion between post and luting agents was observed after the surface area was increased by the roughening of the post surface[27-28]. It was observed that sandblasting and silanization of the surface increased the bond strength to zirconia posts.[29] Silane application demonstrated an enhanced adhesion between fiber posts and core materials.[30]

The prognosis of an endodontically-treated tooth also depends on the type of core reconstruction and the material used.[31] Core materials differ in terms of strength, stiffness, elasticity, and other properties[32] that may influence the structural integrity as well as the durability of the final restoration.

The aim of this in vitro study was to evaluate the effects of surface treatments of fiber reinforced composite posts on the microtensile bond strength with different core materials. The investigation tested the null hypothesis that different post surface treatments and types of build-up materials do not significantly influence the interfacial bond strength.

Materials And Methods

Seventy-four glass fiber posts with a diameter of 2 mm (FRC Postec, Ivoclar-Vivadent, Schaan, Liechtenstein) and four core materials (Multicore Flow, Multicore HB, Tetric Flow, Tetric EvoCeram, Ivoclar-Vivadent, Schaan, Liechtenstein) were used. Posts were divided into four groups, according to the surface treatments performed. GROUP 1: the surface was sandblasted (Rocatec-Pre, 3M ESPE, Seefeld, Germany) for 5 seconds at 2.8 bar. The tip of the sandblasting device was held perpendicularly to the post at a distance of 1 cm. During the procedure the post was rotated in order to blast the aluminium oxide particles on the entire post surface. GROUP 2: the post surface was sandblasted and silanized with Monobond S (Ivoclar-Vivadent, Schaan, Liechtenstein). The silane was applied with a disposable brush for 60 seconds at room temperature and gently air-dried for 5 seconds. GROUP 3: only silane was applied on the post surface. Group 4: no treatment was performed and it served as a control. Prior to the silane application in groups 2 and 3, and prior to the built-up procedures in groups 1 and 4, the posts were cleaned with phosphoric acid gel (Total Etch, Ivoclar-Vivadent AG, Schaan, Liechtenstein) as recommended by the manufacturer.

Each group was further divided into four subgroups, according to the material used for core build-up procedure. Four core materials (Multicore Flow, Multicore HB, Tetric Flow, Tetric EvoCeram, Ivoclar-Vivadent, Schaan, Liechtenstein) were applied on the posts to produce cylindrical specimens,

Table I. Investigated materials, batch numbers, chemical compositions and manufacturers

Material	Composition[1]	Manufacturer
Monobond S Batch n°: H34023	1% 3-methacryloxypropyltrymethoxysilane (3-MPS), ethanol/water-based solvent	Ivoclar-Vivadent, Schaan, Liechtenstein
Multicore Flow Batch n°: H17113	Bis-GMA,urethane dymethacrylate,triethylene glycol dimethacrylate(28,5%), barium glass, ytterbiumtrifluoride,highly dispersed silicon dioxide(71,0%),catalyst stabilizers,pigments(0,5%)	Ivoclar-Vivadent, Schaan, Liechtenstein
Multiocore HeavyBody(HB) Batch n°: J01085	Bis-GMA,urethane dymethacrylate,triethylene glycol dimethacrylate(13,5%), barium glass, ytterbiumtrifluoride, highly dispersed silicon dioxide(86,0%), catalyst stabilizers,pigments(0,5%)	Ivoclar-Vivadent, Schaan, Liechtenstein
Tetric Flow Batch n°: H35185	Bis-GMA,urethane dymethacrylate,triethylene glycol dimethacrylate(35,0%), barium glass, ytterbiumtrifluoride, highly dispersed silicon dioxide(64,6%), catalyst stabilizers, pigments(0,4%)	Ivoclar-Vivadent, Schaan, Liechtenstein
Tetric EvoCeram Batch n°: H34327	Dimethacrylates(17-18%), barium glass, ytterbiumtrifluoride,mixed oxide,prepolymer(82-83%), additives,catalysts,stabilizers,pigments(<1%)	Ivoclar-Vivadent, Schaan, Liechtenstein
FRC Postec Batch n°: H31059	Glass fibers(61,5%),urethane dimethacrylate(18,3%), triethylenglycoldimethacrylate(7,6%), Ytterbium trifluoride(11,4%),highly dispersed silicone dioxide(0,9%),catalysts,stabilizers(<0,3%)	Ivoclar-Vivadent, Schaan, Liechtenstein
Rocatec Pre	High-purity aluminium oxide 110 μm	3M ESPE, Seefeld, Germany

[1] Information from the manufacturers.

immediately after the post surface treatment. The compositions, manufacturers and batch numbers of the investigated materials are listed in Table 1.

Core build-up preparation and microtensile bond strength test

The materials used for core build up were: two flowable resin dual composites (Multicore Flow, Tetric Flow), a nano-hybrid light-activated composite (Tetric EvoCeram) and the dual heavy core material (Multicore HB). All the materials were applied in strictly accordance with the manufacturer's recommendations. The build up preparation was performed following the protocol previously described by Goracci et al[33]. Each post was positioned upright on a glass slab, and secured with a drop of sticky wax. A cylindrical plastic matrix was then positioned around the post and arranged so that the post would be exactly in the middle. The matrix was 10 mm in diameter and the length was equal to the non-tapered portion of the post. This procedure was performed to ensure that the post diameter was constant throughout the post length during the cutting procedure. The composites were applied onto the post in 1-2 mm increments, which were carefully adapted with a spatula or a syringe and tip according to the viscosity of the material employed. Each layer was cured for 40 seconds with a halogen lamp with an output of 600 mW/cm^2 (VIP, Bisco, Schaumburg, IL, USA). The curing light was directed from the open upper side of the matrix and through the post. Additional irradiation for 40 seconds was performed from each side of the cylinder prior to the removal of the matrix. When the matrix was completely filled, the composite matrix was detached from the glass plate and another 40 seconds irradiation performed from the bottom of the cylinder to ensure an optimal polymerisation of the material. At this point, it was possible to cut the transparent matrix from the specimen.

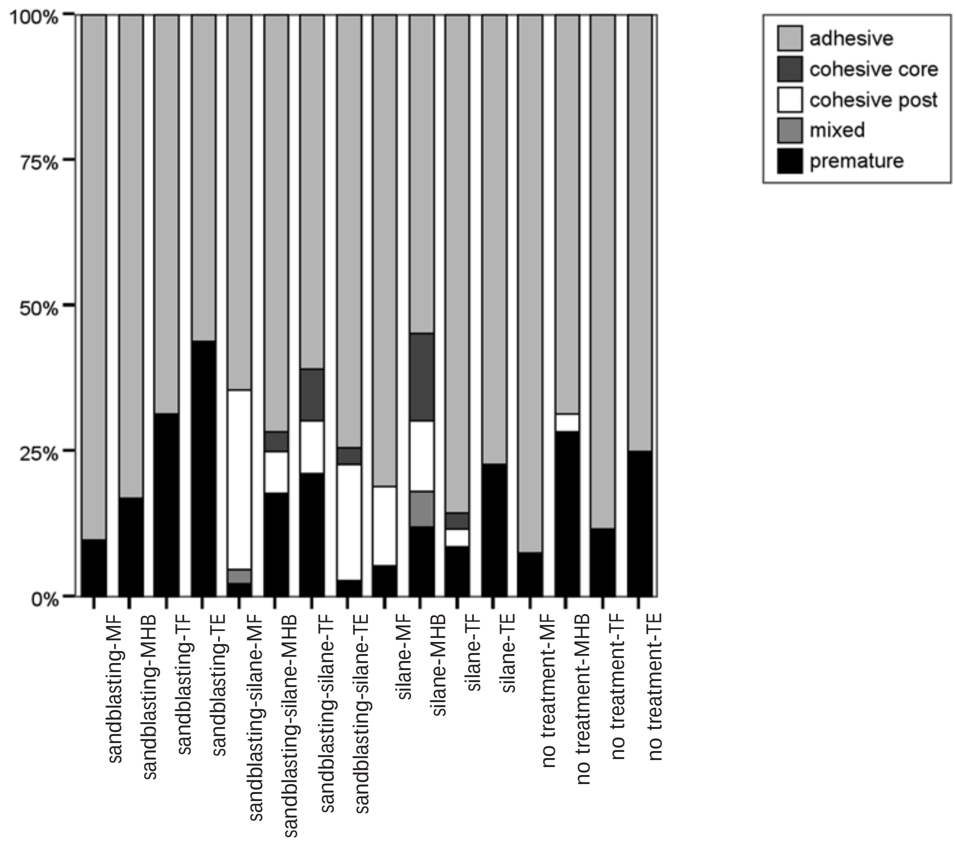


Figure 2: Percentage and type of pre-test failure for each investigated group. Premature failures were excluded from the statistical analysis in order to obtain a normal distribution of data. MF: Multicore Flow; HB: Multicore HB; TF: Tetric Flow; TE: Tetric EvoCeram,

The sectioning and loading procedures began immediately after the core build-up preparation, in order to simulate the clinical situation of immediate loading following core build-ups. Each cylinder was secured with wax on the holding device of a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Two longitudinal cuts were performed initially with a diamond blade under water cooling on two opposite sides of the post at its outermost periphery, in order to expose the post surface throughout its length. As a result, a slab of uniform thickness was obtained, with the post in the center and the core material on each side (Figure 1). A mean of 7-8 sticks of 1 mm in thickness were obtained from each slab, resulting in 30-33 specimens per subgroup. Each stick was measured with a digital calliper (Orteam, s.r.l, Milan, Italy), glued (Super Attak Gel, Henkel Loctite Adesivi, s.r.l. Milan, Italy) to the two free sliding components of a jig mounted on a universal testing machine (Triax, Controls s.p.a, Milan, Italy), and then loaded in tension at a crosshead speed of 0.5 mm/min until failure occurred at either side of the post-composite interface. Failures were evaluated with a stereomicroscope (Nikon SMZ645, Tokyo, Japan) at 40x magnification and recorded as adhesive (at the post-composite interface), cohesive (within the post or the composite) or mixed (a combination of the two modes in the same interface). Bond strength was expressed in MegaPascals (MPa), dividing the load at failure in Newton (N) by the bonding surface area (mm2). As the bonded interface was curved, its area was calculated using a mathematical formula previously applied by Bouillaguet et al. [34]

SEM evaluation

Two posts from group 1 and 4 were examined with a scanning electron microscope (SEM) in order to detect differences in the surface texture between sandblasted and non-sandblasted posts. One post from each group was observed longitudinally, while the other was cross-sectioned with a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The specimens were rinsed in deionised water, immersed in 96% ethanol and air-dried. Each specimen was mounted on a metallic stub, gold-sputtered (Polaron Range SC7620; Quorum Technology, Newhaven, UK), and observed under a JSM 6060 LV microscope (JEOL, Tokyo, Japan) at different magnifications.

Following build-up procedures, one post-core cylinder from each subgroup (surface treatment/core material combination) was randomly chosen for the SEM evaluation of the adhesive interface. Two 1.5 mm thick cross-sections and one longitudinal section through the center of the post were cut in each cylinder (Isomet 1000). Sections were polished with wet silicon carbide paper of increasing grit (600, 1000, 1200), etched with 32% phosphoric acid for 15 seconds (Uni-etch,

Table II. Post-core microtensile bond strength [MPa][2]

Core material	No Treatment		Monobond S		Sandblasting		Sandblasting + Monobond S	
	Bond strength	Pretesting failures[3]	Bond strength	Pretesting failures	Bond strength	Pretesting failures	Bond strength	Pretesting failures
MultiCore Flow	14,5(4,7) bcde	3 of 39 (7,7%)	18,0(9,0) abc	2 of 37 (5,4%)	17,5(6,7) abcd	4 of 41 (9,8%)	20,5(6,4) ab	1 of 42 (2,4%)
MultiCore HB	10,2(3,5) e	10 of 35 (28,6%)	15,8(8,1) bcde	4 of 33 (12,1%)	14,9(9,2) bcde	6 of 35 (17,1%)	12,9(7,1) cde	5 of 28 (17,9%)
Tetric Flow	15,5(6,0) bcde	4 of 34 (11,8%)	23,7(7,9) a	3 of 34 (8,8%)	9,5(3,4) e	11 of 45 (31,4%)	20,6(8,7) ab	7 of 33 (21,2%)
Tetric Evoceram	11,3(4,5) de	9 of 36 (25%)	13,4(7,2) cde	9 of 35 (25,7%)	11,8(5,6) cde	16 of 34 (47,1%)	17,4(8,9) abcd	2 of 34 (5,7%)

[2]Numbers are means. Values in brackets are standard deviations.
Different letters represent statistically significant differences.
[3]The number and the percentage of pretesting failures in each group are reported.

Bisco, Lot. 0500003648, Schaumburg, IL, USA), rinsed with deionised water, immersed in 96% ethanol and then air-dried. Each section was then mounted on a metallic stub, gold-sputtered (Polaron Range SC7620; Quorum Technology), and observed under a JSM 6060 LV microscope (JEOL) at different magnifications.

Statistical analysis of the microtensile bond strength data

To ensure a normal distribution, pre-testing failures (sticks that fractured at any stage prior to loading and considered as "zero bond") were excluded from the statistical calculation (pre-testing failures per each group are reported in Figure 2). After having checked that the data distribution was normal (Kolmogorov-Smirnov test) and group variances were homogeneous (Levene's test), the Two Way Analysis of Variance was applied with bond strength as the dependent variable, core material and post treatment as factors. The Tukey test was applied for post hoc comparisons where needed. In all the tests the level of significance was set at $p<0.05$, and calculations were handled by SPSS 13.0 software for Windows (SPSS Inc, Chicago, IL, USA).

Results

Microtensile bond strength

The means and the standard deviations of the post-core bond strength are reported in Table 2. The Two Way ANOVA revealed that the core material had a significant influence on bond strength ($p<0.05$), with the two flowable composites Multicore Flow and Tetric Flow performing comparably and significantly better than Multicore HB and Tetric EvoCeram, which were similar. Additionally, the type of post surface treatment was a significant factor (p<0-05), with silanization, and sandblasting plus silanization yielding comparable bond strengths that were significantly higher than those achieved with sandblasting or without any treatment. Under these two latter conditions similar bond strengths were measured. The interaction between the type of post surface treatment and core material was also significant ($p<0.05$). Significant differences are reported and labelled with different letters in Table 2.

None of the surface treatments significantly improved the bond strength of Multicore HB, Tetric EvoCeram and Multicore Flow to post compared to control group. The combination of Tetric EvoCeram with sandblasting+silane gave better results than only sandblasting and resulted in higher bond strengths compared to the use of the silane although the differences were not statistically significant.

Among all the experimental groups, the highest bond strength was recorded with Tetric Flow on silanated posts. The values in this group were significantly higher compared to the control. Conversely, the lowest bond strength was recorded with the same material on sandblasted posts without additional silane application. Moreover, when the posts were silanized, the bond strength of Tetric Flow was significantly higher than the bond strengths of Multicore HB and Tetric EvoCeram. On sandblasted and silanated posts, the bond strengths of flowable core materials Tetric Flow and Multicore Flow were comparable, and significantly higher than bond strength of Multicore HB.

SEM examination

Through SEM observation the beneficial effect of silane in terms of adhesion in the case of Multicore HB could be speculated (Figure 3). Multicore HB is a viscous material that

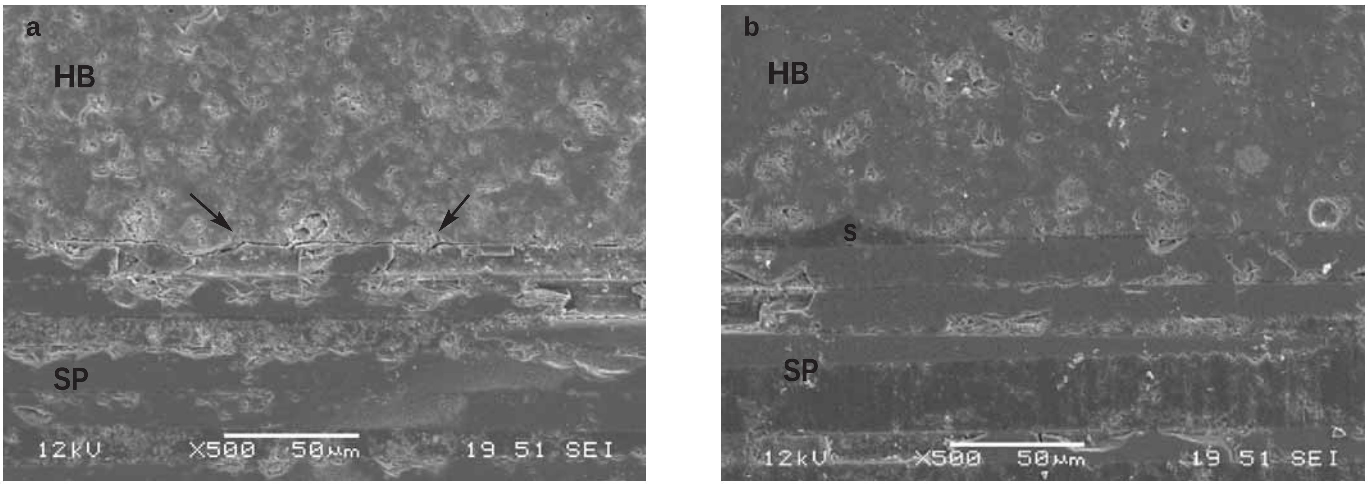


Figure 3: SEM micrographs of the fiber post-core interfaces on sandblasted posts. The more viscous core material Multicore HB appeared not capable of adapting closely to the post surface (arrows) in the absence of silane (A). When the silane was applied, the interface was tight and continious (B). HB: Multicore HB. SP: sandblasted fiber post. S: Silane (500x).

appeared to be unable to adapt closely to the post surface in the absence of silane. SEM images show the continuous interface when silane is applied. In some cases the presence of voids have caused cracks at the post and core interface, despite the application of silane was (Figure 4). That may justify the high number of premature failures recorded for Multicore HB and Tetric EvoCeram. SEM showed that the combination of Tetric Flow and sandblasting+silane also produced similar results when compared to the other materials employed (Figure 5). No cracks were observed at the post and core interface in cross-section magnifications. SEM images show the roughened surface that allows the resin of the composite to penetrate and adapt strictly.

Discussion

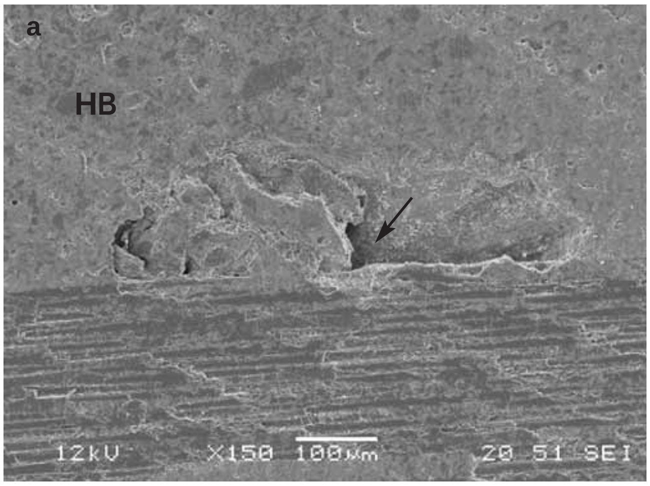
Good bonding is achieved when the material used is able to create an intimate adhesion with the post, by creating a "monobloc". [35] The interface between post and core materials has been investigated in several studies, involving both bond strength tests [36], and microscopic investigations. [37] In the present in vitro study the effect of different surface treatments, core materials and the interaction between the surface treatments and core materials were significant. The null hypothesis has to be rejected.

Since composite resins differ in terms of strength, stiffness, elasticity, and other properties, [18] different core materials were selected for the study: flowable composite, nano-hybrid and a heavy-body core material. SEM observations showed a more homogeneous interface between flowable materials and the treated post when compared with the others materials.

Sandblasting+silanization of FRC post surface increased the bond strength of core materials to post when compared to that achieved when either sandblasting alone, or no treatment of the post surface were performed. Sandblasting generated a micro-retention for anchoring the composite resin. [25] SEM observations revealed an increase in surface roughness on sandblasted posts, with an exposure of the fibers on the post surface. Despite the increase in surface roughness, the sandblasting treatment alone did not significantly improve the interfacial strength between post and core materials when it was not followed by silane application.

The dual molecule of silane is characterised by two ends with different polarity: the alkoxy group of the silanol unit chemically bonds with the silicatised surface, while the methacrylate group polymerizes with the composite resin monomers. As the silane agent is only able to chemically bridge resins and OH-covered inorganic substrates, [38] bonding is possible only between the resin of the core material and the exposed fibres of the post at the post-core interface. By removing the superficial layer of the post with sandblasting, more fibres were available for reacting with the silane molecules and this may have influenced the increase in bond strength. Another possible explanation for the beneficial effect of silane application is the improved surface wettability. [39] Once an firm contact between the interfacing materials is established, the van der Waals' forces become effective. This provides a physical adhesion, which produces chemical reactions. [40] The enhanced bond strength of composite materials to the post surface after silanization supports the results of previously published investigations. [33]

Four composite materials were used for build-up. Although flowable resin composites exhibit lower mechanical properties[41], good adaptation to the post surface was recently assessed by Monticelli et al. [24]. The results were also confirmed in this study, by revealing that the low viscosity of the flowable materials allowed them to take advantage of the improved wettability and penetrate into the roughness of the post created by sandblasting. The surface roughness of the sandblasted posts



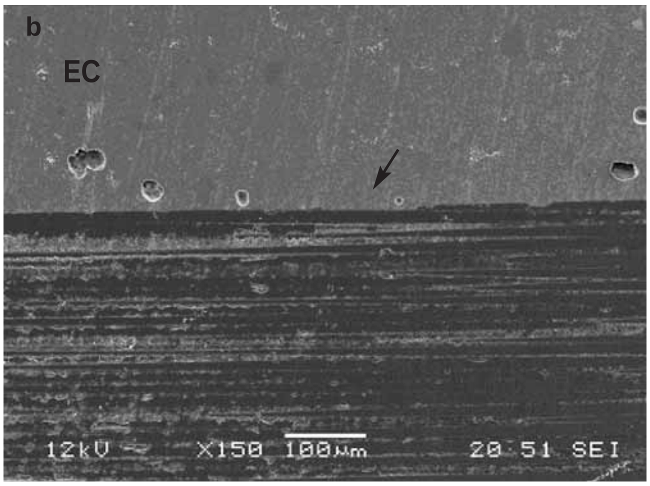


Figure 4: A) Multicore HB; B) Tetric EvoCeram. The intrinsic difficulties of the materials during the specimen preparation may have caused the presence of voids at interface and consequently the high number of failures. HB:Multicore HB. EC: Tetric EvoCeram.

provided additional sites for silanization, enhancing the bonding of the fibre post to the methacrylate-based composite.

Dual-cure composites, Multicore Flow and Multicore HB, were used in this study. Multicore Flow showed a better behaviour compared to Multicore HB when the posts were sandblasted and silanized. Multicore Flow is recommended by the manufacturer both as a core material and a luting agent. Its use may simplify restorative procedures, since it offers better handling properties in comparison to Multicore HB. The fast polymerization and difficult application of Multicore HB may have caused the formation of defects that made this material unable to closely adapt to the post surface in the absence of silane.

Tetric EvoCeram is a nano-hybrid composite that was used for the reconstruction of the core in the present study. Although a high number of pre-testing failures were recorded, Tetric EvoCeram revealed enhanced bond strength in combination with sandblasted and silanated posts.

The microtensile test was used in this investigation as it is accepted as a reliable method for bond strength testing.[42] The test was performed immediately after specimen preparation, in order to simulate the clinical situation of immediate prosthetic loading following core build-ups. The small size of the specimens is a condition which allows a uniform stress distribution during loading. A certain number of premature failures were recorded, which considerably elevated the standard deviations of the data set. The decision to exclude them from the statistical analysis was made in order to ascertain a normal data distribution. However, this laboratory study has some limitations: Results obtained in vitro are not sufficient to predict the in vivo performance of the same materials with certainty. Only one type of fibre post was used in the present study; therefore, it would be of interest to investigate different types of posts and to compare their performances. Future studies may explain the exact mechanism of adhesion between post and core and identify methods of improvement.

Conclusions

Within the limitation of this in vitro study, post surface treatments significantly influenced the adhesion with composite resin materials. The best results were obtained with silanization with or without preliminary sandblasting. Flowable composites achieved a higher bond strength than a hybrid composite and a heavy body core material. The study supports the use of a silane agent for inducing adhesion at the interface between FRC posts and flowable core materials.

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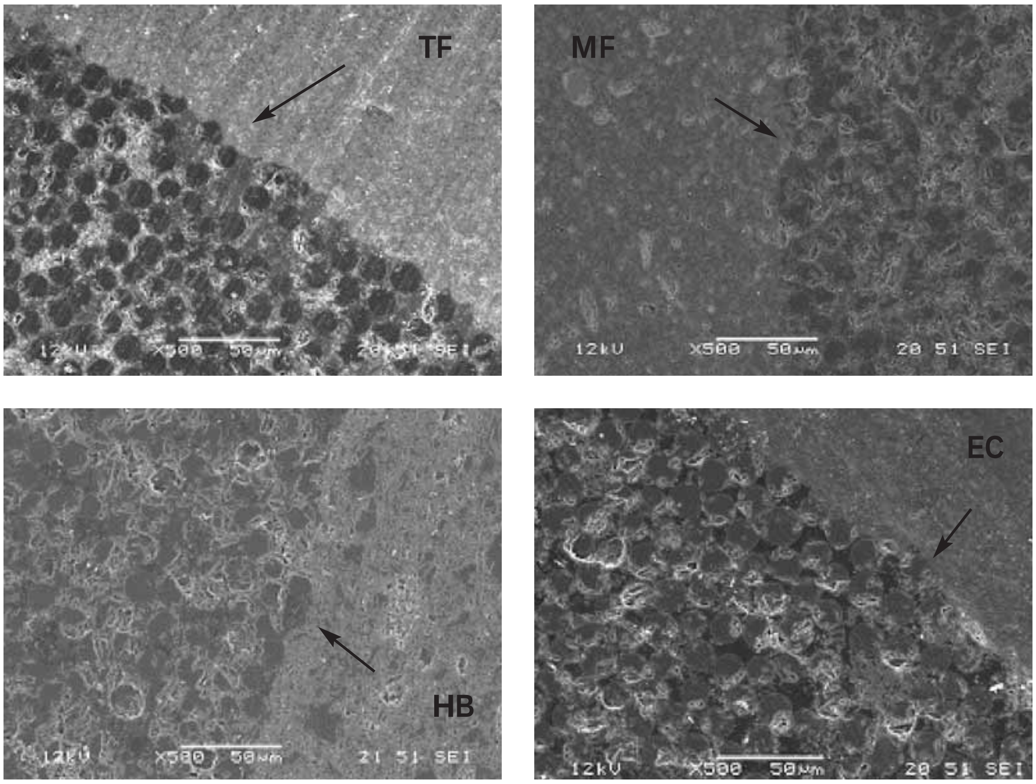

Figure 5: SEM micrographs of fiber post-core interfaces on sandblasted and silanated posts. No defects and discontinuities could be observed with all the investigated core materials. TF: Tetric Flow. MF: Multicore Flow. HB: Multicore HB. EC: Tetric EvoCeram.

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