



Post-surface conditioning improves interfacial adhesion in post/core restorations

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Abstract Objectives: To verify the influence of different etching procedures of the post-surface on microtensile bond strength values between fiber posts and composite core materials.

Methods: 60 DT Light Posts were divided into 10 subgroups using five different chemical surface treatments and two composite materials to build-up the abutment. Chemical surface treatments including etching with potassium permanganate; treatment with 10% hydrogen peroxide; treatment with 21% sodium ethoxide; etching with potassium permanganate and 10 vol.% HCl; silanization (control group) were performed on the post's surface. The build-up was performed using (A) Core Paste XP (Dent Mat) and (B) Unifil Flow (GC). Two samples of each group were randomly selected to investigate the morphologic aspect of the post/core interface with a scanning electron microscope (SEM). The remaining specimens were cut so as to obtain microtensile sticks that were loaded in tension at a cross-head speed of 1 mm/min until failure. The statistical analysis was performed using two-way ANOVA and the Tukey's test for post-hoc comparisons ($\alpha=0.05$).

Results: SEM examination showed an interpenetrating adhesion network between the treated fiber post-surface and the composite material in all the groups tested. The results achieved with potassium permanganate had a significant influence on microtensile interfacial bond strength values with both the tested materials. Post-superficial treatments enhanced the bond strength particularly of Core Paste XP.

Significance: Etching procedures showed a similar effect on the post-surface and enhanced the adhesion of composite core build-ups as a result of micromechanical and chemical retention.

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Introduction

The use of fiber posts in combination with composite core build-up materials has increased in popularity for the restoration of endodontically treated teeth. Many laboratory and clinical studies investigated their mechanical properties and clinical behaviour.[1-4] The clinical success of a post/core restoration depends on the composite selected, but also on the quality of the post/core interface, where materials of different compositions are in intimate contact.[5]

Studies on fiber-reinforced composite materials have led to the modification and improvement of the properties of the interface between the resinous matrix and the fibers. In many cases, interfacial failure was attributed to chemical incompatibility or the plasticising phenomenon where impurities (commonly water) penetrate the interface.[6-8] In dentistry, the durability of a composite core restoration depends on the formation of a strong bond between the resin composite and the residual dentin,[9] as well as between the composite and the fiber post,[6] enabling the interface to transfer stress under functional loading.[10]

Surface treatments are common methods for improving the general adhesion properties of a material, by facilitating chemical and micromechanical retention between different constituents. Advances in adhesive dentistry have resulted in the development of surface conditioning techniques for natural substrates (i.e. enamel, dentin) [11-13] and restorative materials. The latter includes the use of acids to condition the surfaces of non-noble alloys [14] or ceramics,[15] to partially dissolve the substrate and generate a rough surface to enhance adhesion.[16]

With respect to post/core restorations, most studies were designed for improving the performances of these restorations via enhancing the mechanical properties of the composite core build-up materials.[17,18] However, from acid-etching and silanization of the glass fiber phase of these posts,[16,19] little is known on how the resin phase of these fiber posts may be improved for interfacial adhesion. The application of a silane coupling agent as adhesion promoter in fiber post/core units was recently investigated.[5,20] Nevertheless, adhesion of composites to fiber posts was still inferior when compared with the results achieved on dental substrates.[21,22] This is probably due to the absence of any chemical interaction between methacrylate-based resin composite and the epoxy resin matrix of fiber

posts.[16] The authors hypothesized that surface pre-treatment of the resin phase of fiber posts may improve their adhesion to methacrylate-based resin composites. For industrial applications such as epoxy resin-based circuit boards, many chemical treatment techniques have been introduced to improve the adhesion between the components of fiber-reinforced resin composites.[23-25] Different solutions and solvents are known to be effective on epoxy resin.[26,27] It is speculated that a similar approach may be applied in dentistry for surface pre-treatment of fiber posts to increase their responsiveness to silanization.

Thus, the aims of this study were: (1) to evaluate the effect of different chemical etching procedures on the morphological aspects of the fiber post-surface, and (2) to examine the influence of different post-surface treatments on the interfacial strength between epoxy resin-based fiber posts and methacrylate-based resin composites that are employed as core build-up materials. The null hypothesis tested was that different types of post-surface treatment do not affect the interfacial strength between fiber posts and composite core build-up materials.

Materials and methods

Sixty DT Light Posts, #3 with a maximum diameter of 2.14 mm (batch no. 120US0401A, RTD, St Egrève, France), were used for this study. The DT Light Posts are made of unidirectional pre-tensed quartz fibers (60 vol%) bound in an epoxy resin matrix (40 vol%). Five different chemical treatments were tested for their efficacy on etching the resin phase of the fiber post-surface ($n=12$ in each group):

- Group 1: etching with potassium permanganate;
- Group 2: etching with 10% hydrogen peroxide for 20 min;
- Group 3: etching with 21% sodium ethoxide for 20 min;
- Group 4: etching with potassium permanganate and 10 vol% HCl for 1 h;
- Group 5: silanisation of the post-surface for 60 s without chemical treatment of the resin phase (control group).

The etching procedure of group 1 consists of three successive steps: (1) immersion of the posts in a conditioning solution (60 vol% of methyl-pyrrolidone in deionised water) for 3 min at 50-60 °C (E-K Hole Cleaner, Elkem, Torino, Italy). This initial step enhances the removal ability of permanganate with the epoxy resin being swollen and its surface

chemical structure altered. Extensive rinsing with tap water was performed for 3 min; (2) etching in an alkaline potassium permanganate solution (20 vol% in deionised water; pH 12-13) (E-K Hole Oxidizer, Elkem) for 10 min at 70-80 °C to oxidise and remove the epoxy resin matrix previously degraded by the solvent; (3) immersion in a sulphate neutraliser solution (10 vol% in deionised water) (E-K Hole Reducer, Elkem) for 5 min at 40-50 °C to reduce and neutralise the excess permanganate and clean the surface of the post. Each surface-treated post was rinsed with deionised water for 3 min, followed by air-drying.

The fiber posts in group 2 were immersed in 10% hydrogen peroxide (Sigma, Aldrich Chemic, GmbH, Steinheim, Germany) for 20 min at room temperature and then rinsed with deionised water. The posts in group 3 were immersed in a 21 wt% sodium ethoxide solution (i.e. sodium hydroxide in ethanol; Sigma, Aldrich Chemic, GmbH, Steinheim, Germany) for 20 min at room temperature, then rinsed with pure ethanol, 50% ethanol solution in deionised water and finally deionised water (5 min for each cleaning bath) to reach a stable pH of 7.

For the posts in group 4, the same potassium permanganate treatment employed in group 1 was initially performed. This was followed by immersing the permanganate-treated post in a 10 vol% solution of hydrochloric acid (HCl; Panarec Quimica SA, Barcelona, Spain) in deionised water for 1 h.

Before performing the composite core build-ups, an additional exhaustive rinsing procedure was performed. All the posts were ultrasonically cleaned for 10 min in deionised water (P Selecta S.A. Abrera, Spain), immersed in 95% ethanol and dried with an air stream.

A single layer of silane coupling agent (Monobond-S, batch no. E53184 Ivoclar-Vivadent, Schaan, Liechtenstein) was then applied with a brush to the post-surface of each of the five experimental and control groups, and gently air-dried after 60 s, according to manufacturer's recommendations. Monobond-S is a pre-hydrolyzed single component silanising agent and contains 1 wt% of 3-methacryloxypropyl trimethoxysilane (3-MPS) in an ethanol/water-based solvent.

Core build-up procedure and microtensile test

Core build-up was performed using (A) a dual-cured composite core material (Core Paste XP batch no. 030653101, Den-Mat, Santa Maria, CA, USA), and (B) a flowable resin composite material (UniFil Flow, batch no. 04001061, GC Corp., Tokyo, Japan), using

an *in vitro* technique previously reported by Goracci et al. [20] for similar purposes. Each post was positioned perpendicularly on a glass slab and maintained with a needle holder at the apical end. A cylindrical plastic matrix was placed around the post and an incremental technique was followed to build up the core. Each 2-mm increment of the core composites was cured for 40 s with a halogen light-curing unit (Optilux, Demetron Res. Corp, Danbury CT, USA) with an output of 600 mW/cm². The material was polymerized directly from the upper side of the matrix. The matrix was subsequently removed after being filled completely with polymerised composite. This resulted in a cylinder of resin composite that was built up around the fiber post. The bottom side of the cylinder that was previously in contact with the glass slab was light-cured for an additional 20 s to ensure complete polymerization of the composite material.

After storing in distilled water for 24 h, each bonded specimen was mounted on the holding device of a slow-speed diamond saw (Isomet 4000, Buehler, Lake Bluff, USA). Two longitudinal cuts were initially made on the two opposite sides of the post/composite assembly with the diamond saw under water cooling, to expose the post-surface throughout its length. A slab of uniform thickness, with the post in the centre and the core build-up composite on either side was created. Each slab was then serially sectioned to obtain 4-5 beams of 1-mm in thickness (Fig. 1). Each beam was secured to the flat grips of a Bencor Multi-T device (Danville Engineering, San Ramon, CA, USA) with cyanoacrylate adhesive, and subjected to a tensile load at a cross-head speed of 1 mm/min until failure, using a universal testing machine (Model 4411 Instron Corp., Canton, MA, USA). Interfacial strength was calculated using the mathematical formula previously described by Bouillaguet et al.[28]

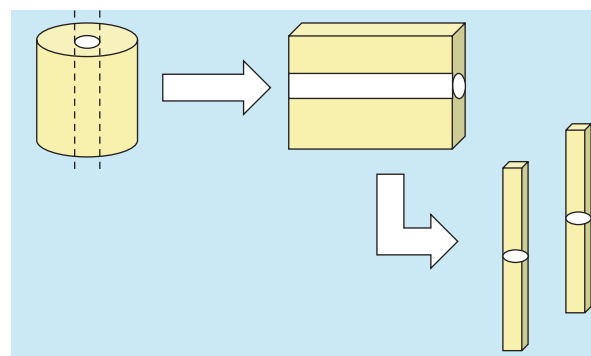


Figure 1 Schematic illustration of the specimen preparation procedure for microtensile bond strength test.

The normally distributed data (Kolmogorov-Smirnov test) were analysed using a two-way ANOVA to examine the effect of surface treatment and choice of core material on the interfacial strength to fiber posts. Tukey's test was performed for post-hoc comparisons. Statistical significance was set at $\alpha = 0.05$.

Scanning electron microscopy (SEM)

Two specimens from each group were examined with an SEM (JSM 6060 LV, JEOL, Tokyo, Japan) to study the characteristics of the post/core interface after different surface chemical treatments. The specimens were cross-sectioned by means of the water-cooled diamond saw to produce 2-mm thick slabs. The prepared specimens were mechanically polished with wet, 600-, 1200- and 4000-grit silicon carbide papers. Final polishing was achieved using a diamond polishing-paper (Lab-pol 8-12, Bantec Leicester, UK) for 10 s. The polished specimens were ultrasonicated for 5 min in deionised water (CP-104 EIA Intern. Paris, France), immersed in 95% ethanol, and gently air-dried. Each specimen was sputter-coated with gold-palladium (Polaron Range SC7620, Quorum Technology, Newhaven, England) and examined with an SEM at different magnifications.

Results

SEM analysis

SEM evaluation revealed surface modification of the epoxy resin matrix of the fiber posts in groups 1-4 that were comparable in their effect. Dissolution of the epoxy resin matrix resulted in the exposed quartz fiber component of these posts being surrounded by the core build-up resin composite. Partial removal of the epoxy resin matrix of the fiber posts created 'retention spaces' among the fibers that appeared to be completely infiltrated by the core materials (Figs. 2A and 2B). No cracking was evident from the underlining untreated epoxy resin of these four groups of fiber posts, and the exposed quartz fibers were not damaged by the chemical treatments.

Low magnification micrographs revealed the absence of any defect and/or discontinuities along the interface between the fiber post and the composite (Fig. 3).

Interfacial bond strength

The results of the microtensile bond strength test are shown in Table 1. Surface chemical pre-treatment had a significant influence on the interfacial bond strengths of these fiber posts ($P < 0.001$). The results achieved with potassium permanganate were significantly better than those obtained with other surface chemical treatment techniques for both of the composite materials tested. The type of core material did not have a significant influence on the results. However, post-surface treatments enhanced the bond strengths of the core build-up materials, especially for Core Paste XP ($P < 0.05$).

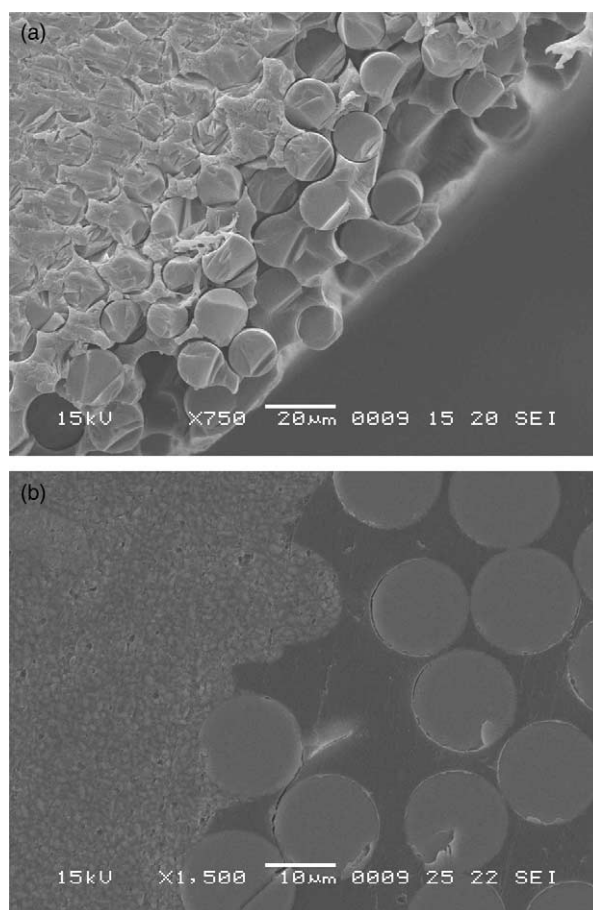


Figure 2 Representative SEM micrographs of the cross sections of fiber posts after etching with potassium permanganate. (A) At low magnification, exposure of the quartz fibers could be seen, as a result of the dissolution of the superficial epoxy resin (750X; Bar = 20 μm). (B) A high magnification of the 'free spaces' between the fibers that appeared to be completely infiltrated by the core material (1500X; Bar = 10 μm).

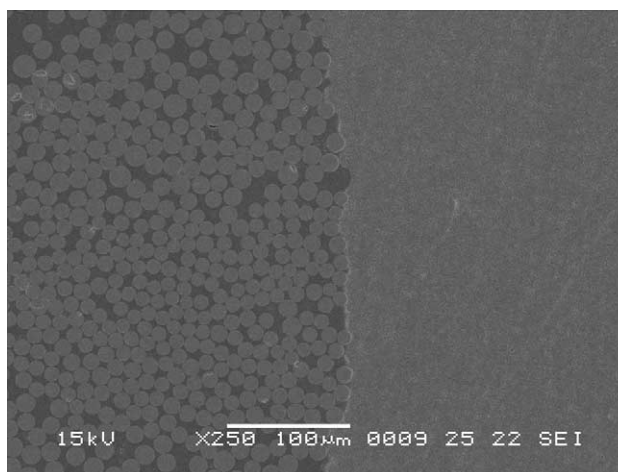


Figure 3 Low magnification micrographs revealed the absence of any defects and/or discontinuities at the interface between the fiber post and the resin composite (250X; Bar=100 µm).

Discussion

The use of chemical surface treatments influenced the interfacial bond strength between fiber posts and core build-up materials. Potassium permanganate significantly enhanced the interfacial bond strength between fiber posts and both the tested composite materials. Thus, the null hypothesis is rejected.

Epoxy resin etching techniques are commonly employed in industrial and laboratory fields. Sodium ethoxide and hydrogen peroxide are frequently used in transmission electron microscopy for immunolabelling enhancement, by partially removing the resinous superficial layer of resin-embedded tissue sections.[26,27,29] Potassium permanganate is usually applied in an industrial process designed for conditioning epoxy resin surfaces for metal plating of printed circuits

boards.[30-32] Epoxy polymers exhibit a high degree of conversion and highly cross-linked structures.[33] These agents exert their etching activity by oxidising the substrates and breaking the epoxide bonds.[26,29]

In terms of application time of potassium permanganate, a mild treatment protocol was chosen in this study, as it was initially speculated that the superficial etching developed during this conditioning process would render it suitable for the adaptation of resin composite to enhance the adhesion of fiber posts.

The concept of conditioning artificial substrates to improve bond strength has antecedents in dentistry that are exemplified by the etching of the Maryland bridge [14,34] and feldspathic porcelain restorations.[15,16,35] Based on this principle, different conditioning procedures initially proposed for ceramics have also been tested on fiber posts.

Table 1 Interfacial strength of the experimental and control groups after different post-surface treatments.

Group designation	Surface chemical treatment	Core material	N*	Mean	SD	Statistical Significance ^a
1	Potassium permanganate + silane	Core Paste XP	25	14.50	4.07	a
		UniFil Flow	26	13.02	2.49	ab
2	Sodium ethoxide + silane	Core Paste XP	27	11.04	2.98	bc
		UniFil Flow	26	8.48	2.42	cd
3	10% H ₂ O ₂ + silane	Core Paste XP	27	10.70	2.74	bcd
		UniFil Flow	24	8.18	3.20	d
4	Potassium permanganate + HCl + silane	Core Paste XP	25	10.11	3.60	cd
		UniFil Flow	25	10.20	2.81	cd
5 (control)	Silane only	Core Paste XP	26	8.49	2.91	cd
		UniFil Flow	25	10.66	2.22	bcd

N, number of microtensile sticks prepared from the bonded fiber post-composite assemblies. Means and standard deviations of the microtensile bond strengths (MPa) of the experimental and control groups after different post surface treatments.

^a Groups identified with the different letters are statistically different ($P < 0.05$).

Hydrofluoric acid has recently been proposed for etching glass fiber posts.[36] However, this technique produced substantial damage to the glass fibers, which affected the integrity of the post. This is due to the extremely corrosive effect of hydrofluoric acid on the glass phase of a ceramic matrix.[16,37]

Sahafi et al. recently tested the efficacy of blasting the surface of zirconia and fiber posts with silica oxide (Co-Jet System).[21,38] Despite the satisfactory bond strengths achieved, the treatment was considered too aggressive for fiber posts with the risk of significantly modifying their shape and consequently, their fit within the root canals.[39]

On the contrary, the etching procedures proposed in this investigation only affected the superficial part of the epoxy resin matrix of the fiber posts, leaving exposed smooth quartz fibers intact in all the groups tested. This differential etching effect reflects the difference in reactivity between the epoxy resin phase and the quartz fiber phase of the fiber posts (Fig. 2A).[24] It is further speculated that potassium permanganate, besides exposing the quartz fibers, may also activate the latter by improving their hydrophilicity. Such a condition may be due to the use of a specific neutralising solution after conditioning in order to 'clean' the fibers from residual MnO_2^- ions.[31] An increased deposition of silane may take place in the presence of a surface with more hydroxyl groups.[25]

Amino-silane coupling agents are generally used as adhesion promoters in the presence of epoxy resin polymers, as they provide both a chemical bond between inorganic substrates and the polymer, as well as increase surface wettability.[7] MPS silanes are commonly applied in dentistry.[16,40] Since MPS silane does not bond well with the epoxy matrix, the bond strength between the epoxy resin phase of the fiber post and the methacrylate-based resin composite should not be enhanced. With the removal of the superficial layer of epoxy resin via chemical treatment, more exposed quartz fibers in terms of surface area, are available for reacting with the silane molecules. The increased chemical union between the silanised quartz fibers and the methacrylate-based core material would significantly improve the interfacial bond strength.[40] When the surface of the post is etched and rinsed, a more reactive surface is thus generated for both chemical and micromechanical retention. As quartz fibers are comparable in chemical structure with ceramic materials, it is reasonable to speculate that siloxane bonds would be achieved, as they

represent the binding sites for the coupling agents to the post-surface.[16]

Two different resinous core materials were evaluated in this study: a flowable composite and a low-viscosity core material, which are commonly employed for this purpose.[9,18,41] Although flowable resin composites exhibit lower mechanical properties,[42,43] good adaptation to the post-surface was recently reported.[20,44,45] These previous results were also confirmed in this investigation, showing that the flowable composite was able to achieve a satisfactory bond with the fiber posts in the absence of any surface chemical treatment (Table 1).

Moreover, the two core build-up materials may be considered as comparable by virtue of the similar interfacial morphology achieved; their low viscosity enabled a satisfactory penetration into the porosities created between the exposed quartz fibers. This micromechanical interlocking between the infiltrating composite and the exposed glass fibers created a structure that is reminiscent of dentine hybridisation, in which adhesive resin monomers infiltrate a partially demineralised collagen matrix and polymerize in situ to create a mechanism for dentine adhesion.[12,13] Likewise, the simultaneous increase in the surface roughness of the post and the surface area of the quartz fibers also provide additional frictional resistance and sites for silanization, further enhancing the bonding of the fiber posts to the methacrylate-based composites.[24]

The main goal of fiber-reinforced composite material science is to obtain a strong bond between the different components, so as to obtain a unique material with improved performance.[46] A similar concept may be applied in dentistry to improve the quality of the post/core unit, with the objective in creating a 'monobloc' between the tooth and the restorative material.

Conclusions

Surface chemical treatments of the resin phase of fiber posts enhance the silanization efficiency of the quartz fiber phase, so that the adhesion in the post/core unit may be considered a net sum of chemical and micromechanical retention.

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