EFFICACY OF A COMBINED SILICA/METHACRYLATE COUPLING ON THE FIBER POST BONDING TO COMPOSITE CORES

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Abstract

Objectives. The aim of the study was to evaluate the effect of surface pre-treatments on different fiber posts when bonded to composite cores. **Methods and Materials.** Conventional (DT Light Post, VDW) and pre-coated quartz fiber posts (DT Light SL, VDW) were tested. Chemical surface treatments included: 1. silanization (Monobond-S; Ivoclar-Vivadent); 2. adhesive application (Prime& Bond NT; DeTreyDentsply); 3. silane + adhesive application; 4. no treatment was performed on the surface of both types of posts. A flowable composite was used for build-up (X-Flow; Dentsply). Specimens were cut into microtensile sticks that were loaded under tension (cross-head speed 0.5 mm/min) until failure. Bond strength data were statistically analyzed with two-way ANOVA and Tukey tests for post-hoc comparisons (a = 0.05). The morphology of the post/core interface was evaluated under a scanning electron microscope (SEM). **Results.** Post surface treatment influenced the bond strength ($p<0.05$). SEM evaluation revealed a modification of the superficial morphology of pre-coated fiber posts when compared to conventional posts. **Discussion.** The combined use of silane and adhesive improved bonding to conventional fiber posts. Pre-coated fiber posts may provide a satisfactory chemical bond to composite cores, while an additional adhesive layer may contribute to improved bonding, possibly "reactivating" the resinous pre-coating of these posts.

Conclusions and Clinical significance. Fiber post luting procedures may be simplified by industrial surface per-coatings, simplifying the procedure and avoiding technique-sensitive clinical steps.

Short title: Priming fiber post surface to enhance bonding

Key words: fiber posts, surface conditioning; silane coupling, silica coating, core build-ups.

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Introduction

The clinical success of fiber post-retained restorations depends on the establishment of a reliable bond at the root-post-core interfaces[1,2]. In spite of the improvements in dental material technology, post to composite adhesion is still considered relatively low[3]. Advances in silane coupling agents enhance bond strength by promoting adhesion between OH-covered inorganic surfaces and polymeric molecules[4-6]. However, the nature of the fiber post-resin joint is critical: only the exposed fibers (quartz or glass) of the post are available for silane coating and may contribute to retention[7,8]. Chemical incompatibility between methacrylate-based resins of the core and the epoxy resin matrix of certain types of fiber posts is likely to occur[9]. Alternative approaches, such as the application of an intermediate layer of fluid resin on the post, have been proposed to overcome this limitation[10].

However, the need to create a chemophysical retention has been recognized to ensure a more reliable bond at the post-core interfacial level[9,11]. Chair-side silica coating techniques (i.e. Co Jet System®), in which the tribochemical effect of air particle abrasion results in superficial deposition of SiO_2, have been recently applied for this purpose on fiber posts, with controversial results[12,13]. Although satisfactory improvements in bond strength were achieved, a weakening effect on the fiber post was reported as a consequence of a perceptible volume loss[13]. In the attempt to simplify clinical procedures, an

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industrial pre-treatment of the fiber post surface may be effective in avoiding excessively technique-sensitive steps.

The aims of the study were therefore to evaluate 1. the influence of an industrial pre-coating of a fiber post on post-core bond strength and 2. the possible effect of the application of an additional layer of silane coupling and/or adhesive system on fiber posts. The null hypothesis tested was that there are no differences between pre-coated and conventional fiber posts under the tested experimental conditions.

Materials and Methods

Conventional translucent quartz fiber posts (Group 1; DT Light Post #3, VDW GmbH, Munich, Germany) and experimental pre-coated translucent quartz fiber posts (silica-modified surface covered with a methacrylic outer layer) (Group 2; DT Light SL #3, VDW GmbH) were tested in the study. Both groups (n= 20) were divided into four subgroups (n=5) according to the different superficial treatment of the post performed: 1. application of a pre-hydrolyzed silane coupling agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein); 2. application of a bonding agent (Prime&Bond NT, Dentsply DeTrey GmbH, Konstanz, Germany); 3. subsequent application of silane and bonding agent (Monobond-S + Prime&Bond NT); 4. no additional surface treatment. The composition of the tested materials and their application modes are described in Table 1.

After post surface treatment, a core build-up was performed according to a technique described by Goracci et al.[7]. Each post was positioned perpendicularly on a glass slab and maintained with a needle holder at the apical end. A cylindrical plastic matrix (10mm in diameter) was placed around the cylindrical portion of the fiber post (with a constant diameter), incrementally filled by a low viscosity composite (X-Flow, Dentsply DeTrey) and light-cured for 20 s (output 600mW/cm^2;VIP, Bisco. The removal of the matrix resulted in a build up of a cylinder of resin composite around the fiber post (Figure 1). Samples were stored 24h at room temperature before testing.

Microtensile test specimens were prepared by sectioning each sample with a water-cooled diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Two longitudinal cuts were initially made on the two opposite sides of the post/composite assembly to expose the entire length of the post surface. A slab of uniform thickness, with the post in the center and the core build-up on each side was created (Figure 1). Each slab was then serially sectioned perpendicularly to the post into sticks: an average of 25 sticks of 1-mm in thickness was tested for each group. For the microtensile bond strength test, each stick was glued to the ends of a Geraldeli's jig[14] with cianoacrylate (Zapit, Dental Ventures of America, Corona USA) and loaded in tension at a cross-head speed of 0.5mm/min

Table 1. Mode of application and composition of tested materials.

Materials	Composition	Application
DT Light Post Batch no.120US0407B	Epoxy resin (60%); quartz fibers (40%)	
DT Light SL Batch no. W000051-4	Epoxy resin (60%); quartz fibers (40%) Silica coating and silane	
Monobond-S Batch no. F68158 pH≈4	3-MPS (1%); Ethanol/water-based solvent; acetic acid.	Apply with a dispense brush; Gently air-dry after 60s;
Prime & Bond NT Batch no. 0408000115 pH≈2.4/2.7	Acetone; Di- and Tri methacrylate resins; UDMA; PENTA; Nanofiller- amorphous silicone dioxide; Photoinitiators; Stabilizers; Cetylamine hydrofluoride	Single coat application; Gently air dry after 5 s; Light-cure for 20 s
X-Flow Batch no. 0412000740	Strontium alumino sodium fluoro phosphor silicate glass; Di- and Multifunctional acrylates and methacrylates resins; DGDMA; highly dispersed silicon dioxide; UV stabilizer; Ethyl-4-dimethylaminobenzoate; CQ; BHT; Iron pigments; Titanium oxide	Apply on the restoration. Light cure for 20 s.

3-MPS: 3-methacryloxypropyltrimethoxysilane; UDMA: urethan dimethacylate monomer; PENTA: dipentaerythritol penta acrylate monophosphate; DGDMA: diethylene glycod dimethacrylate; BHT: Butylated Hydroxytoluene; CQ: Camphorquinone.

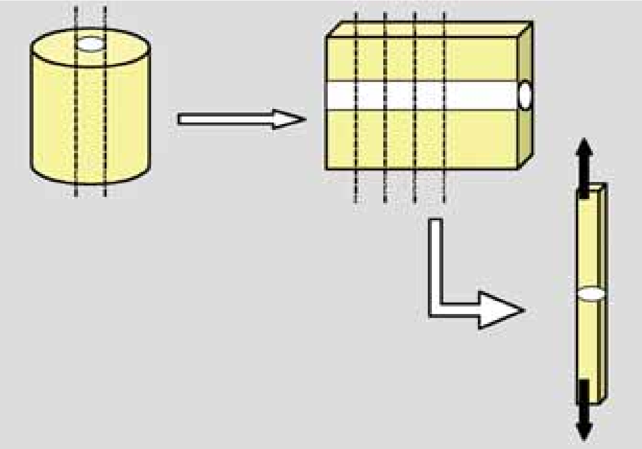

Figure 1: Schematic drawing of specimen preparation for microtensile bond strength testing. Each cylinder was sectioned with a diamond saw under water cooling: two longitudinal cuts were made on the two opposite sides of the post/composite assembly to expose the post surface throughout its length. A slab of uniform thickness, with the post in the center and the core build-up on each side was created. Slabs were serially sectioned perpendicularly to the post into sticks.

50, Control, Milan, Italy). No alteration of the fiber post inner structure was recorded during cutting and/or loading. Failure modes were evaluated with a stereomicroscope (Nikon SMZ645) 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 of failure in the same interface).

As the bonded interfaces represented a part of the circumference of a post with a circular cross section, bond strengths were expressed in MPa using a mathematical formula reported by Bouillaguet et al.[15]. After analyzing the bond strength data for the normality of data distribution (Kolmogorov-Smirnov test) and the homogeneity in variances (Levene's test), a two-way ANOVA was applied to evaluate the effect of the factors (post type and superficial treatment) on the dependent variable (bond strength). Interactions were included in the analysis. Post-hoc multiple comparisons were made with a Tukey test. The level of significance was set at ? = 0.05. The sample size was calculated to ensure a power of 0.8 in the statistical analysis.

Scanning electron microscopy analysis (SEM)

Two specimens from each group were examined with a SEM (JSM 6060 LV, JEOL, Tokyo, Japan) to evaluate the state of the post composite interfaces after the different post superficial treatments. Specimens were cut into 1.5 mm thick cross-sections (Isomet 1000) and then polished with wet silicon carbide papers, ultrasonicated in deionised water for 5 min, immersed in 96% ethanol, air dried and sputter coated with gold (Polaron Range SC7620, Quorum Technology, Newhaven, England). Micrographs were taken at different magnifications. In addition, an evaluation of the surface morphology of the tested fiber posts before building-up the core was made.

Results

Interfacial Bond Strength

The results of the microtensile bond strength test achieved for each experimental group are reported in Table 2. Although the type of post was not a single significant factor, post surface treatment and the interaction of this factor with the post type significantly influenced the measured bond strengths ($p < 0.001$). Pre-coated fiber posts showed superior results without additional treatments. Statistically similar bond strengths were achieved by applying a layer of bonding agent to the surface of

Table 2. Interfacial strength after different post surface treatments and distribution of failure modes obtained in the experimental groups.

Group treatment	Surface chemical	Post Type	Mean (SD)
1	Silane	DT Light Post + X-Flow	9.3 (2.9) bc
		DT Light SL + X-Flow	7.6(3.5) c
2	Adhesive	DT Light Post + X-Flow	12.2(3.4) a
		DT Light SL + X-Flow	12.2 (4.9) a
3	Silane + Adhesive	DT Light Post +X-Flow	14.32 (3.2) a
		DT Light SL + X-Flow	9.22 (2.1) bc
4	No treatment	DT Light Post+ X-Flow	6.9 (3.8) c
		DT Light Post SL + X-Flow	10.9 (4.1) ab

DT Light Post: Translucent conventional quartz fiber posts
DT Light SL: Translucent silica/resinous pre-coated quartz fiber posts
¶ Groups identified with the different letters are statistically different (P<0.05).

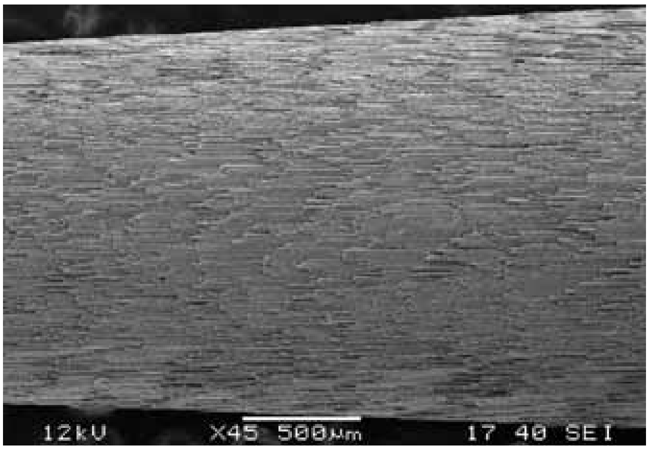

Figure 2a: Scanning electron microscopy image of the superficial aspect of a conventional quartz fiber post before performing the build-up (45x; bar= 500 µm).

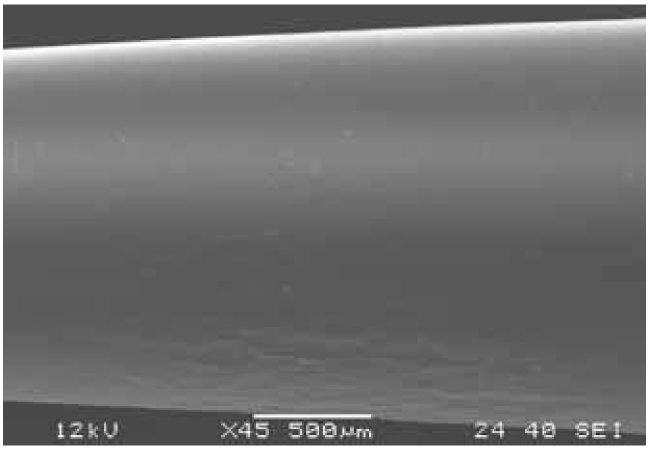

Figure 2b: SEM image of a pre-coated fiber post: a uniform thick silica-resinous coating is evident around the post. Neither epoxy resin matrix nor quartz fibers are directly exposed on the post surface (45x; bar= 500 µm).

both tested fiber posts (Table 2). Conversely, there was a significant decline in bond strength to pre-coated fiber posts (DT Post SL) after silanization. The combined application of silane anda bonding agent increased bonding to conventional fiber posts. A percentage of adhesive failures ranged between 90 and 100% was recorded in all tested groups.

SEM Analysis

SEM evaluation revealed a modification of the surface morphology of pre-coated fiber posts when compared to conventional posts (Figure 2A). This resulted in the formation of a thick smooth silica/resin coating along the length of the post (Figure 2B). The inner components of the fiber post (i.e epoxy resin matrix and quartz fibers) are not directly exposed to the post surface. Cross-sections of post-core interfaces demonstrated a good adaptation of the flowable composite to the post surface in all tested groups: no defects and/or discontinuities along the interface between the fiber post and the composite occurred (Figure 3 A and B). However, a thicker interfacial layer was more evident on pre-coated fiber posts (Figure 3 C and D) as a consequence of the silica-resinous coating. The additional silane and/or bonding agent application produced no morphological changes.

Discussion

The establishment of a reliable adhesion to fiber posts is necessary for the clinical success of composite core build-ups. The results achieved indicate that post to composite bond strength was significantly affected by the superficial treatment. The null hypothesis is therefore rejected.

Conventional and surface-modified epoxy matrix quartz fiber posts were evaluated in the study. In the case of the latter, a superficial coating made of silica and a monofunctional non-acidic monomer (MMA) as an outer protective layer had already been performed by the manufacturer.

The priming of conventional fiber posts before luting is beneficial to ensure adequate bond strengths. However, inconsistent results were reported following silanization, probably due to the limited area of exposed quartz fibers on the post surface[9]. The application of an adhesive layer or the synergistic combination of silane and bonding agent may have strengthened the joint[8]. Nevertheless, chairside post pre-treatments are considered a technique sensitive step. Poor solvent evaporation in the bonding agent or the tendency of some silane solutions to degrade over time, forming oligomers, may affect their coupling efficacy[16,17]. In particular, the hydrolytic degradation of some simplified adhesives or hydrophilic silanes has been recently reported as the cause of accelerated interfacial water sorption, offering an easy path for fiber post structural weakening[18, 19]. The use of a more hydrophobic fluid resin layer may assist in reducing the risk of water uptake.

The silica-resinous superficial coating provided by the manufacturer may contribute to the simplification of the operative procedures, achieving sufficient interfacial strength with composite cores. It is assumed that silica films give excellent surface properties, due to the stability of their bonds and the ability to form highly hydrophobic substrates[20, 21]. This may result in a protective barrier preventing diffusion of water into the post. Such a coating has to be thin enough to be flexibleto with stand the functional stress of fiber posts, without the risk of cracking and the consequent exposure of the post to moisture[22].

The epoxy resin matrix of the post is not directly involved in the adhesion mechanism, due to its incompatibility with methacrylate-based restorative materials[9]. The presence of available free radicals on the partially-polymerized outer resinous coating, could have initiated such chemical bonding with the composite[23]. However as posts are handled during clinical procedures, the resinous coupling may have a changed result: in the absence of an oxygen inhibition layer, MMA tends to form homo-polymers quickly, reducing its reactivity [(24)]. A

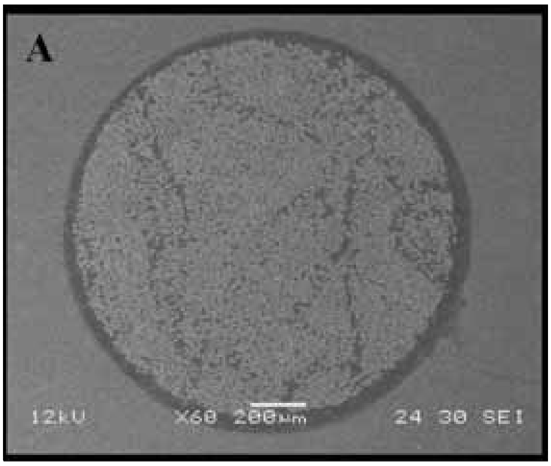

Figure 3a: Representative SEM micrographs of the post-composite interface of conventional posts after the application of the bonding agent. The adhesive layer is uniformly distributed on the post surface (60x; bar= 200 µm).

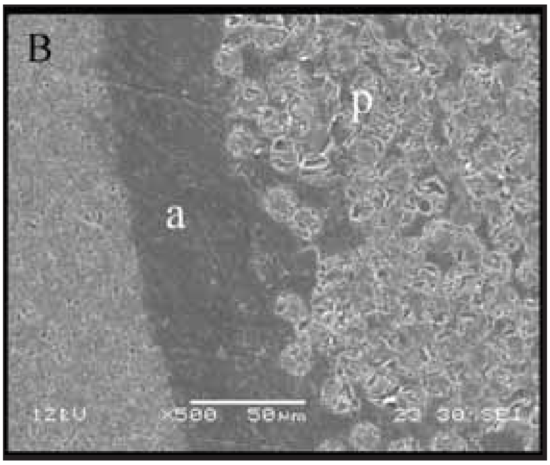

Figure 3b: No defects along the interface are detectable at higher magnification (500x; bar= 50 µm).

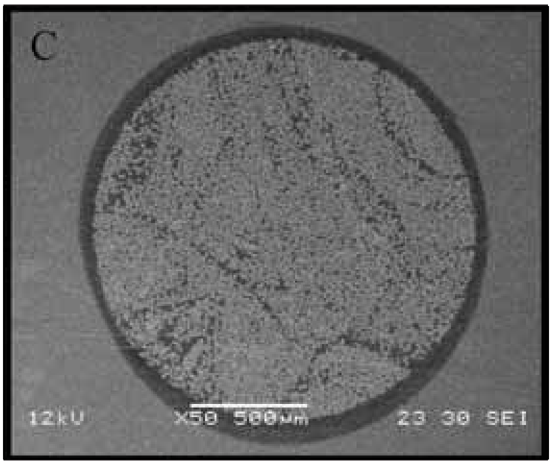

Figure 3c: Representative SEM micrographs of the post-composite interface after industrial pre-coating. The adaptation of the composite core to the post surface through the silica-resinous coating could be seen (50x; bar= 500 µm)

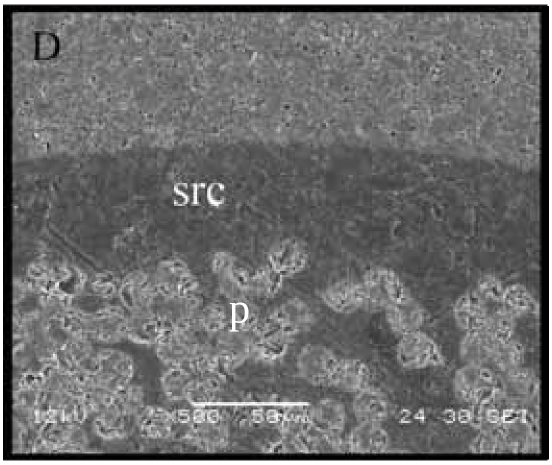

Figure 3d: A higher magnification view revealed the absence of any defects and /or discontinuities at the interface between the fiber post and the resin composite showing the extent of the industrial coating (500x; bar= 50 µm) (a = adhesive layer; p = fiber post; src = silica-resinous coating).

"freshly" applied layer of bonding agent appears to "activate" the coating, offering considerably more favourable conditions for adhesion to the composite core. The low interfacial strength obtained with silane application on the pre-coated posts suggests that such a priming regime is inadequate. The absence of a chemical state of silicon and oxygen on the post surface may prevent the formation of a polysiloxane network[25]. Furthermore, a release of resinous compounds of the coating in the ethanol/water-based silane solution is likely to occur[23].

High interfacial bond strengths between the fiber post and the core material may not be sufficient to ensure the quality of a restoration[26], since the adhesion mechanism through the smooth resinous coating of the post is only chemical in nature; the poor hydrolytic stability of methacrylate could compromise the longevity of the bond. From a manufacturing prospective, the choice of more stable monomers, such as acrylic ether phosphonic acids, might be considered[24]. Alternative strategies based on the combination of micromechanical and chemical conditioning on the post surface are also advisable to improve retention, achieving a more reliable adhesion[9,11]. Further studies are needed to examine the long term stability of these bonds through accelerated aging conditions.

Conclusions

Within the limitations of the study, the following conclusions can be drawn:

- the application of an intermediate coupling agent is necessary for promoting the adhesion between translucent fiber posts and composite cores;
- a post industrial surface pre-coating may simplify the clinical procedure, avoiding technique-sensitive steps;
- the choice of stable surface couplings is advisable for ensuring the durability of post and core restorations.

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