A Simple Etching Technique for Improving the Retention of Fiber Posts to Resin Composites

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Abstract

Coupling of fiber posts to composites is hampered by absence of chemical union between epoxy resins and methacrylate-based resins. This study examined a clinically feasible protocol for creating micromechanical retention on the surface of fiber posts, using hydrogen peroxide etching to remove the surface layer of epoxy resin. This was followed by silanization of the exposed quartz fibers to enhance their chemical bonding to composites. Etching with 24% H_2O_2 for 10 min or 10% H_2O_2 for 20 min produced a 50 μm thick surface zone that is depleted of epoxy resin, leaving intact, undamaged quartz fibers for silanization. Low viscosity flowable composites were employed to infiltrate this zone, to simulate the creation of hybrid layers in acid-etched dentin by dentin adhesives. Interfacial strengths were enhanced with the adjunctive use of H_2O_2 etching and silanization, and were probably dependent on the ability of the flowable composites to completely infiltrate this interdiffusion zone. *(J Endod 2006;32:44–47)*

Key Words

Fiber post, flowable composite, hydrogen peroxide etching, interfacial strength, silane

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0099-2399/$0 - see front matter

doi:10.1016/j.joen.2005.10.005

Etching procedures for natural and artificial substrates have been developed to enhance adhesion (1–4). They include acid treatments of enamel (1), dentin (2, 3) or nonnoble alloys (4) that partially dissolve the substrates and generate micro-porosities where resin can penetrate, creating micromechanical interlocking (3).

Fiber posts are extensively used in clinical practice to restore endodontically treated teeth (5–12). The clinical longevity of endodontically treated teeth restored with fiber posts and resin composite cores was recently evaluated (6, 13). Satisfactory adaptation at the post/core interface could be achieved using flowable composites as core materials (13, 14).

The effect of post silanization on the interfacial strength between fiber posts and core build-up composites was recently evaluated with the microtensile test (15). Silane coupling agents can achieve chemical bonds with OH-covered inorganic substrates such as glass. A chemical bond may be achieved between the core resin matrix and the exposed glass fibers of the post at the interface level (15, 16). However, the interfacial strength is still relatively low when compared to the values normally achieved with coronal dentin or enamel (17, 18), because of the absence of chemical union between the methacrylate-based resin composites and the epoxy resin matrix of fiber posts.

Hydrogen peroxide (H_2O_2) is commonly employed in immunological electron microscopy to partially dissolve the resin surface of epoxy resin-embedded tissue sections, and expose tissue epitopes for immunolabeling enhancement. The etching effect of H_2O_2 depends on its capacity to partially dissolve the resin matrix, breaking epoxy resin bonds through a mechanism of substrate oxidation (19–22). A similar H_2O_2 etching procedure may be employed to improve the micromechanical retention between the epoxy resin matrix of fiber posts and methacrylate-based resin composites.

The aims of this study were to evaluate the effect of H_2O_2 on the morphological aspects of the post surface, and the influence of different surface treatment on the interfacial strengths between fiber posts and composites. The null hypothesis tested was that different types of post surface treatment and the type of flowable core build-up composites do not affect the interfacial strength between fiber posts and resin composites.

Materials and Methods

Experimental Design

Sixty DT Light-Posts (RTD, St. Egève, France), each with a 2.1 mm maximum diameter, were used in this study. These posts are made of quartz fibers (60 vol%) previously processed to a unidirectional axial tensile stress and embedded in an epoxy resin matrix (40 vol%). They were divided into five groups ($n = 12$) according to the experimental surface pretreatments performed.

In group 1 the posts were immersed in 24% H_2O_2 for 10 min at room temperature (RT). In group 2 they were immersed in 10% H_2O_2 for 20 min at RT. In group 3, after immersion in 24% H_2O_2 for 10 min at RT, the posts were silanized for 60 sec. In group 4, after immersion in 10% H_2O_2 for 20 min at RT, the posts were silanized for 60 sec. In group 5 (control) the posts were silanized for 60 sec.

After the application of H_2O_2, the posts were rinsed with water and air-dried. A silane coupling agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein) was applied in a single layer on the post surfaces of groups 3 through 5 and gently air-dried after 60 sec, according to manufacturer's instructions. Monobond-S is a single-com-

ponent silanizing agent that contains 1 wt% of 3-methacryl-oxypropyltrimethoxysilane in an ethanol/water-based solvent.

Scanning Electron Microscopy (SEM)

After H_2O_2 treatment, two posts from groups 1 and 2 were examined with an SEM to study their surface conditions. One post from each group was observed longitudinally, while the other was cross-sectioned using a water-cooled diamond blade (Isomet, Buehler, Lake Bluff, IL). The prepared specimens were ultrasonicated for 5 min in deionized water, immersed in 96% ethanol and gently air-dried. Each post was sputter-coated with gold-palladium alloy (Polaron Range SC7620, Quorum Technology, Newhaven, England) and examined with an SEM (JSM 6060 LV, JEOL, Tokyo, Japan).

Core Build-up and Microtensile Testing

Core build-up was performed for the remaining ten posts in each group using either one of the two flowable composites ÆliteFlo (A) (Bisco Inc., Schaumburg, IL) and UniFil Flow (B) (GC Corporation, Tokyo, Japan) using an in vitro technique that was described in detail by Goracci et al. (15). Each post was positioned perpendicularly on a glass slab and secured with a needle holder at the apical end. A plastic matrix of 10 mm in diameter was placed around the cylindrical portion of the post (i.e., the part with uniform diameter) and adjusted so that the post would be exactly in the middle. An incremental technique was used for the core build-up with each composite increment cured for 20 sec using a halogen curing light with an output of 600 mW/cm^2 (VIP, Bisco Inc.). An additional 20 sec polymerization was subsequently performed from the bottom side of the cylinder previously in contact with the glass slab.

Two longitudinal cuts were made on the two opposite sides of the post/composite assembly with the Isomet saw under water cooling exposing 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. Beams of 1-mm in thickness were serially sectioned from each slab. Every slab yielded 4 to 5 beams for microtensile testing that was performed with a universal testing machine at a cross-head speed of 0.5 mm/min until failure. Interfacial strength was calculated using the mathematical formula previously described by Bouillaguet et al. (23).

The normally distributed data were analyzed with a two-way ANOVA design, to assess the effect of surface treatment and core build-up material on the interfacial strengths of the fiber posts. Post hoc comparisons were performed using the Tukey test. Statistical significance was set at $\alpha = 0.05$. The sample size in each group was checked to ensure a minimum power of 0.8 in the statistical analysis.

Results

SEM Evaluation

Pretreatment with 24% H_2O_2–10 min or 10% H_2O_2–20 min were comparable in their efficacy in modifying the fiber post surfaces. Both groups created rough surfaces along the entire post lengths (Fig. 1*A*). The exposed quartz fibers were not damaged or fractured by the oxidative action of H_2O_2 (Fig. 1*B*). Cross-sectional views of both groups revealed surface dissolution of the epoxy resin matrix to a depth of 50 μm (Fig. 2*A*), exposing approximately five layers of quartz fibers (*ca.* 10 μm in diameter) for micromechanical retention (Fig. 2*B*). The underlying epoxy resin remained intact and exhibited no signs of cracking or damage.

Interfacial Strength

The results of microtensile bond testing are shown in Table 1. The type of surface pretreatment had a significant influence on interfacial strength ($p < 0.0001$). The results achieved with 24% H_2O_2–10 min and 10% H_2O_2–20 min were comparable and significantly better than no treatment. Silane application in combination with H_2O_2 pretreatment produced the best overall results ($p < 0.0001$). The type of core material also had a significant influence ($p = 0.014$), as did the interaction of this factor with the type of pretreatment ($p < 0.01$). Post treatment with either 24% or 10% H_2O_2 enhanced the bond strength, particularly for UniFil Flow.

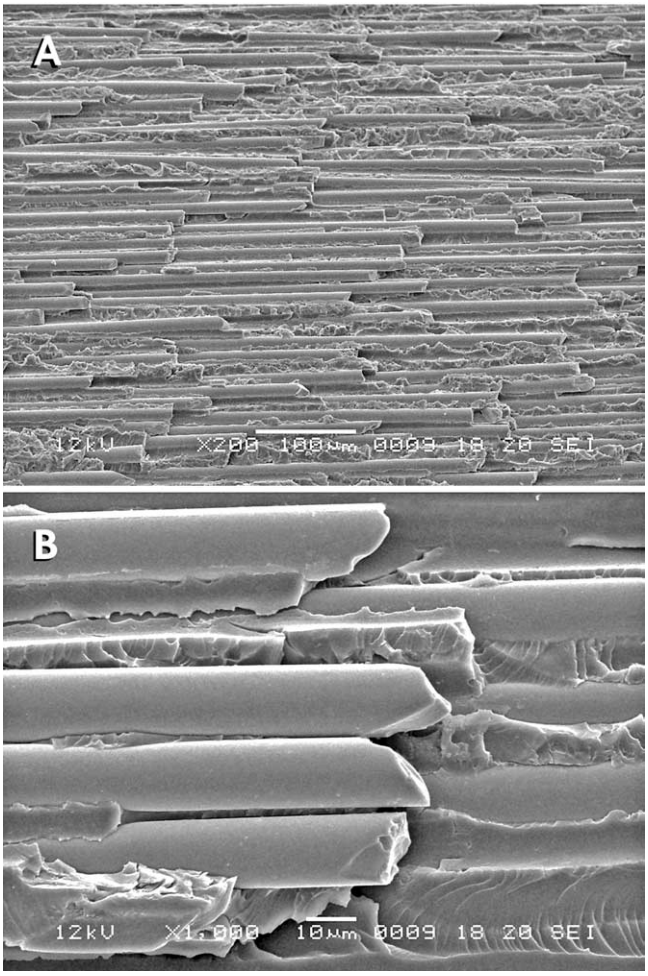

Figure 1. Representative SEM micrographs of a post surface after etching with 10% hydrogen peroxide for 20 min. (*A*) At a low magnification, exposure of the quartz fibers could be seen, as a result of the dissolution of the surface epoxy resin. (*B*) A high magnification view of the exposed quartz fibers, showing that they were not damaged by the etching treatment. Similar results were achieved when etching was performed with 24% hydrogen peroxide for 10 min (not shown).

Discussion

The use of H_2O_2 pretreatment and silane application significantly enhances interfacial strength between fiber posts and core materials. Interfacial strength was also influenced by the choice of flowable composites for core build-up. Thus, the null hypothesis tested in this study is rejected.

Unlike the use of more corrosive forms of industrial epoxy resin etching techniques such as sodium ethoxide or permanganate etching (24, 25), H_2O_2 etching provides an easy, effective and clinically feasible method for enhancement of interfacial strengths between fiber posts and resin composites, without the need of employing extremely corrosive liquids in a clinical setting. By removing a surface layer of epoxy

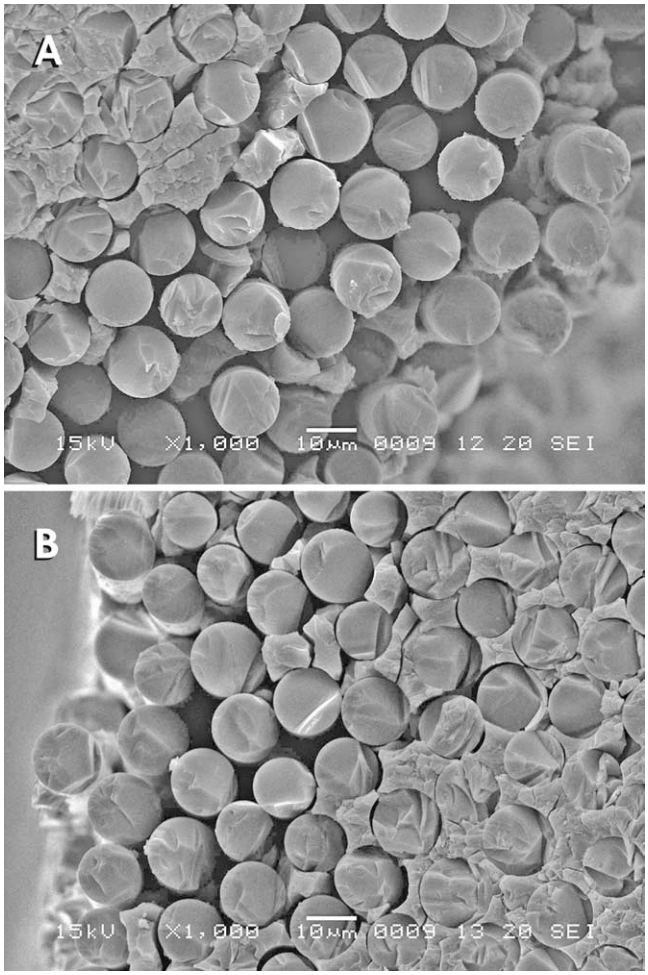

Figure 2. SEM images of the cross sections of fiber posts after treatment with 24% hydrogen peroxide for 10 min (*A*) and 10% hydrogen peroxide for 20 min (*B*). Dissolution of the epoxy resin from the post surfaces exposed the quartz fibers to a depth of 50 μm for silanization as well as created additional spaces for micromechanical retention of the flowable composites.

resin, a larger surface area of exposed quartz fibers is available for silanization. The spaces between these fibers provide additional sites for micromechanical retention of the flowable resin composites. This retention concept is reminiscent of the creation of hybrid layers in dentin and cementum (26), as the interface is contributed by both the quartz fibers from the fiber post and the methacrylate resin matrix.

A more aggressive etching procedure has recently been recommended that involves etching fiber posts with hydrofluoric acid (27). However, this technique considerably affected the integrity of the fiber posts. Microscopic analysis revealed uneven removal of the epoxy resin matrix and extensive damage to the quartz fibers as they were simultaneously attacked by the hydrofluoric acid. On the contrary, H_2O_2 etching is a considerably milder technique with the exposed quartz fibers remaining smooth and leaving the underlying epoxy resin matrix intact after the etching procedures.

The use of silane coupling agents improves surface wettability and creates a chemical union between the glass fibers and resin-based materials (28). In case of epoxy matrix glass-reinforced composites, amino-silanes are typically applied, since epoxy resin and glass can adhere through an amino-coupling reaction (29). The improved interfacial strength achieved following silanization may depend on the reaction between the silane molecules and the silica component in the quartz fibers (30, 31). In this study a prehydrolyzed silane coupling agent containing 3-methacryl-oxypropyltrimethoxysilane was used. The methacryloxyl group in this silane coupling agent probably enables a chemical union to be achieved between the silane-treated quartz fibers and the methacrylate-based resin composite matrix, thereby improving interfacial strength.

Although the mechanical properties of flowable composites are generally inferior to those of hybrid composites or core materials (32, 33), they were selected for this study by virtue of their previously reported adaptation to post surfaces (13–15). It could be seen from the interfacial strength results that the two flowable composites investigated did not differ significantly from one another in the absence of any surfaces pretreatment. Conversely, significant differences were observed between the two materials when post surfaces were pretreated with H_2O_2 etching and silane. The statistically significant interaction with the two factors "pretreatment" and "composite" suggests that these differences may depend on the variation in viscosity between the two flowable composites. It is possible that the lower viscosity of UniFil Flow enables a better penetration of this material within the 50 μm thick zone of denuded quartz fibers. Incomplete penetration of this zone may create a scenario that approximates the incompletely infiltrated hybrid layers that were observed when some dentin adhesives were applied to acid-etched dentin (34). Unlike denuded collagen fibrils within incompletely infiltrated dentin hybrid layers, quartz fibers cannot degrade via hydrolysis by endogenous metalloproteinases (35). Nevertheless, a weak zone may be created that could have accounted for the lower interfacial strength observed when the more viscous ÆliteFlo was applied to the surface-treated fiber posts. In the future, it would be necessary to examine whether the use of low viscosity hydrophobic resins such as those employed as pit-and-fissure sealants will further improve the novel concept of fiber post surface hybridization introduced in this study. The extent of resin impregnation within the zone of hybridized fiber post should also be examined with tracer penetration techniques. The use of hydrophobic resins in thin layers instead of dentin adhesives can pre-

TABLE 1. Interfacial strengths of the experimental groups after different types of fiber post-surface treatment

Core Material		Type of Treatment*		
		24% H_2O_2–10 min	10% H_2O_2–20 min	No Treatment
Æliteflo (A)	No silane	Group 1A (n = 24) 8.3 (4.2)^{def}	Group 2A (n = 24) 6.1 (4.8)^{ef}	
	Silane	Group 3A (n = 25) 11.7 (3.9)^{bc}	Group 4A (n = 26) 13.0 (4.6)^{ab}	Group 5A (n = 27) 6.7 (3.0)^{def}
UniFil Flow (B)	No silane	Group 1B (n = 26) 9.1 (3.2)^{cde}	Group 2B (n = 25) 9.5 (4.0)^{cd}	
	Silane	Group 5B (n = 24) 15.3 (3.2)^{a}	Group 4B (n = 25) 13.8 (3.3)^{ab}	Group 5B (n = 24) 5.3 (1.7)^{f}

*Values are mean (SD) in MPa. Groups connected with the same letter superscripts are not statistically significant ($p > 0.05$).

vent unnecessary water sorption and may alleviate the use of mechanically less favorable flowable composites as core build-up materials.

Acknowledgments

This study was based on a dissertation to be submitted by Dr. Francesca Monticelli for partial fulfillment of the requirements of the degree of Doctor of Philosophy in the University of Siena, Italy. The fiber posts examined in this study were generously sponsored by RTD. The flowable composites were generously sponsored by Ivoclar-Vivadent and Bisco Inc. The work was supported by the University of Siena, by grant CICYT/FEDER MAT 2004-06872-C03-02 from the University of Granada, Spain and by RGC CERG grant 10204604/07840/08004/324/01, Faculty of Dentistry, University of Hong Kong. The authors are grateful to Raquel Toledano, Anna Tay, and Cris Ferrari for secretarial support.

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