Fiber Post Etching with Hydrogen Peroxide: Effect of Concentration and Application Time

Murilo de Sousa Menezes, PhD, MD, DDS, Ellyne Cavalcanti Queiroz, MD, DDS,† Paulo Vinícius Soares, PhD, MD, DDS,* André Luis Faria-e-Silva, PhD, MD, DDS,‡ Carlos José Soares, PhD, MD, DDS,* and Luis Roberto Marcondes Martins, PhD, MD, DDS§*

Abstract

Introduction: Etching is necessary to expose the fibers and enable both mechanical and chemical bonding of the resin core to the fiber post. This study evaluated the effect of concentration and application time of hydrogen peroxide on the surface topography and bond strength of glass fiber posts to resin cores. **Methods:** Fiber posts were etched with 24% or 50% hydrogen peroxide for 1, 5, or 10 min (n = 10). Posts without any treatment were used as a control. After etching, the posts were silanated and adhesive resin was applied. The posts were positioned into a mold to allow a self-cured resin core to be inserted. The post/resin assembly was serially sectioned into five beams that were subjected to a tensile bond strength test. Data were subjected to two-way ANOVA and Tukey test (α = 0.05). The surface topography was analyzed using scanning electronic microscopy. **Results:** Non-etched post presents a relatively smooth surface without fiber exposure. Application of hydrogen peroxide increased the surface roughness and exposed the fibers. All experimental conditions yielded similar bond strength values that were higher than those obtained in the control group. **Conclusion:** Both 24% and 50% hydrogen peroxide exposure increased the bond strength of resin to the posts, irrespective of the application time. *(J Endod 2011;37:398–402)*

Key Words

Bond strength, fiber post, hydrogen peroxide etching

From the *Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil; †Private Clinic, Uberlândia, Minas Gerais, Brazil; ‡Department of Dentistry, School of Dentistry, Federal University of Sergipe, Aracaju, Sergipe, Brazil; and §Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, São Paulo, Brazil.

Address requests for reprints to Dr Murilo de Sousa Menezes, Department of Operative Dentistry and Dental Materials, Federal University of Uberlândia, Av Pará 1720, Bloco 2B, Campus Umuarama, Uberlândia, MG 38400-902, Brazil. E-mail address: murilomenezes@foufu.ufu.br
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The similar elastic modulus of fiber posts, resin cements, resin composite, and dentin is considered to be advantageous for improving the performance of restorations in endodontically treated teeth (1, 2). In addition to the elastic modulus, the bond among the materials, as well as the bond of the materials to the dental substrate, may generate a homogeneous structure known as "monoblock" (3, 4). A proper bonding at the dentin/cement, post/resin cement, and post/composite interfaces is needed for dissipation of stresses generated by occlusal loads. Failure related to any of these interfaces might impair the formation of the monoblock. Although the most frequent cause of failure in post-retained restorations is debonding at the cement/dentin interface (5, 6), the interface between the cement/composite with the post also plays a role in the performance of the restoration.

It has been suggested that resin cements bond to fiber posts via micromechanical and chemical mechanisms (7–10). The organic component of fiber posts is generally epoxy resin with a high degree of conversion and highly crosslinked (11). This polymer matrix is virtually unable to react with the monomers of resin cements. Silane coupling agents commonly used in dentistry react with the glass fibers and may not bond well to the organic component (12). Therefore, it has been suggested to treat the post in order to roughen the surface and expose the glass fibers, allowing micromechanical interlocking of the adhesive/cement with the post (8). In addition, a chemical bonding may be established by using silane (12, 13).

Sandblasting and hydrofluoric acid etching are techniques used to improve the bonding of adhesive/cement to fiber posts (9, 14, 15). Because these techniques can sometimes damage the glass fibers and affect the integrity of the posts (9), substances that selectively dissolve the epoxy matrix without interfering with the fibers have been studied (10, 12, 13, 16). Potassium permanganate, sodium ethoxide, and hydrogen peroxide (H_2O_2) may effectively remove the epoxy resin and expose the fibers, which are then available to be silanated (8, 10, 12, 16). H_2O_2 at concentrations of 10% and 24% effectively removes the surface layer of the epoxy resin (13). However, application periods of 10 or 20 minutes used in previous studies are clinically impractical (13, 17). Thus, the aim of this study was to evaluate the effect of higher concentrations of H_2O_2 and shorter application times on the bond strength between resin composite and glass fiber post. In addition, the surface of fiber posts was evaluated using scanning electronic microscopy (SEM). The null hypothesis tested was that neither the concentration of H_2O_2 nor the application time would affect the bond strength.

Materials and Methods

Microtensile Bond Strength Test

Materials used in this study are described in Table 1. Fiber posts, each with a maximum diameter of 2.1 mm, were used in this study. Polyvinylsiloxane impression material (Aquasil; Dentsply DeTrey, Konstanz, Germany) molds were obtained to standardize the core buildup on the posts. Two plastic plates (10 mm long × 4 mm wide × 1 mm thick) were attached along the post surface, one plate opposite to the other and both in the same plan, using cyanoacrylate adhesive. The post attached to the plates was centrally positioned into a plastic tube (20-mm inner diameter × 15 mm high), and the impression material was placed into the tube. The post attached to the plates was

TABLE 1. Description, Manufacturer, and Composition of the Materials Used in the Study

Material	Description	Manufacturer	Composition*
Aestheti-Plus	Fiber post	Bisco Inc, Schaumburg, IL	Quartz fibers embedded in an epoxy resin matrix
Core-Flo	Self-cured composite resin	Bisco Inc, Schaumburg, IL	Base: Ethoxylated Bis-GMA, glass filler, TEGDMA, silica Catalyst: Bis-GMA, glass filler, TEGDMA
Porcelain primer	Silane agent	Bisco Inc, Schaumburg, IL	MPS, ethanol, water
All-Bond 2	Adhesive system	Bisco Inc, Schaumburg, IL	Primer A: NTG-GMA, acetone, ethanol, water Primer B: BPDM, photo-initiator, acetone Bonding: Bis-GMA, UDMA, HEMA Prebond: Bis-GMA, TEGDMA, BPO, HEMA

Bis-GMA, bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; MPS, methacryl-oxypropyltrimethoxysilane; NTG-GMA, N-tolylglycine glycidyl methacrylate or N-(2-hydroxy-3-[(2-methyl-1-oxo-2-propenyl)oxy]propyl)-N-tolyl glycine; BPDM, biphenyl dimethacrylate; UDMA, urethane dimethacrylate; BPO, benzoylperoxide; HEMA, 2-hydroxyethyl methacrylate.
*As informed by the manufacturers.

removed after polymerization of the polyvinylsiloxane, leaving a space to insert the post and composite resin.

The fiber posts were immersed in 24% or 50% H_2O_2 at room temperature for 1, 5, or 10 minutes ($n = 10$). After immersion in solutions of H_2O_2, the posts were rinsed with distilled water and air dried. Ten posts were rinsed only with water and used as a control. A silane coupling agent was applied in a single layer on the post surfaces and gently air dried after 60 seconds. The nonsolvated adhesive All-Bond 2 was applied over the post surface and light cured for 20 seconds. Light activation was performed using a halogen lamp (VIP Jr; Bisco Inc, Schaumburg, IL) with 600-mW/cm^2 irradiance. The post was inserted into the corresponding space of the mold. The self-cured resin composite Core-Flo was mixed and inserted into the space created by the plastic plates in the mold using a Centrix syringe (DFL, Rio de Janeiro, RJ, Brazil). After 30 minutes, the mold was sectioned with a scalpel blade to remove the specimens, which were stored under 100% humidity conditions for 24 hours.

The specimens were serially sectioned using a low-speed saw (Extec, Enfield, CT) to obtain five 1-mm-thick sections. The setup for preparation is shown in Figure 1. The beams were attached to the flat grips of a microtensile testing device with cyanoacrylate adhesive and tested in a mechanical testing machine (DL 2000; EMIC, São José dos Pinhais,

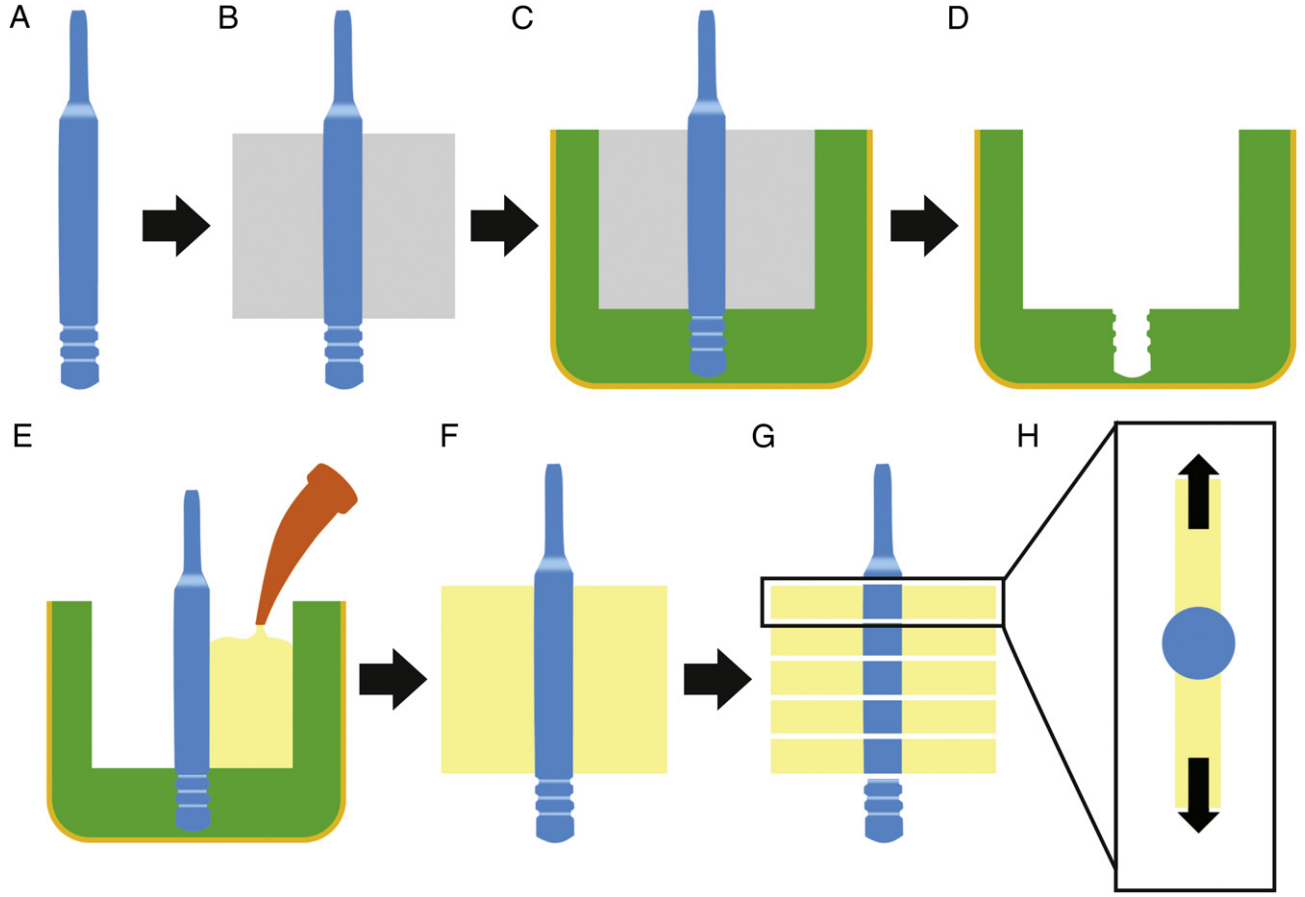

Figure 1. A schematic illustration of mold and sample preparation. (*A*) Intact fiber post, (*B*) plastic plates attached to the post, (*C*) post impression made with polyvinylsiloxane impression material, (*D*) mold created, (*E*) insertion of resin composite into the mold using a syringe, (*F*) resin composite attached to the fiber post, (*F*) sectioned specimen, (*G*) bonded specimens, and (*H*) indication of the load application.

PR, Brazil) at a cross-head speed of 0.5 mm/min until failure. After the test, the specimens were carefully removed from the fixtures with a scalpel blade, and the cross-sectional area at the fracture site was measured to the nearest 0.01 mm with a digital caliper to calculate the tensile bond strength values.

The average value of the five beams in the same specimen was recorded as the microtensile bond strength (MPa) for that specimen. Statistical analysis was performed by applying a two-way analysis of variance followed by a Tukey post hoc test at a 95% confidence level. The factors evaluated were "concentration of H_2O_2" and "application time."

Surface Topography

Two additional fiber posts per group were used for the analysis of the surface topography using SEM. The posts were immersed into a solution of H_2O_2 (24% or 50%) for 1, 5, or 10 minutes following the same procedures described previously. After etching (the control did not receive any treatment), the specimens were ultrasonically cleansed for 5 minutes using deionized water followed by immersion in 96% ethanol for 2 minutes and air drying. The posts were coated with gold (SCD 050; Baltec, Vaduz, Liechtenstein) and evaluated by SEM (JSM-5600LV; JEOL, Tokyo, Japan).

Results

Microtensile Bond Strength Test

Results are shown in Figure 2. The statistical analysis did not show significant differences for the factor "concentration of H_2O_2" (P = 0.25), "application time" (P = 0.06), or the interaction between the factors (P = 0.3). The Tukey test showed that the control group presented the lowest means, whereas there was no significant difference among the groups treated with hydrogen peroxide. All failures were adhesive between the fiber post and resin core.

Surface Topography

SEM pictures are shown in Figure 3. The glass fibers were almost entirely covered by epoxy resin in the nonetched posts. A relatively smooth surface with poor retention was also observed. Etching with H_2O_2 increased the surface roughness along the entire post length for all concentrations and application times. Exposure to 24% H_2O_2 for 1 minute generated the lowest fiber exposure, whereas the other experimental conditions showed similar etching patterns. The exposed glass fibers were not damaged or fractured by any etching protocol.

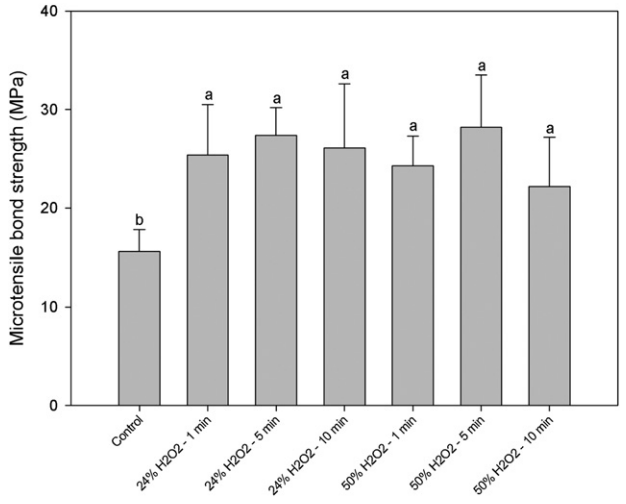

Figure 2. Results for microtensile bond strength. Distinct letters indicate statistical differences (Tukey test, α = 0.05).

Discussion

Etching the fiber post with H_2O_2 before the adhesive procedure and silane application improved the bonding of the resin core to the glass fiber posts. However, the concentration of H_2O_2 did not affect the bond strengths. Both concentrations used in this study (24% and 50%) generated similar values of bond strength of the resin core to the fiber post. Likewise, the application time did not influence the bonding to the fiber posts. Thus, the null hypothesis tested was accepted.

Most of the fiber posts are covered by epoxy resin, which has a high degree of conversion and few reactive sites to chemically bond to the adhesive resin (11). This weak bond can be compensated by micromechanical retention to spaces over the post surface and/or by using a silane agent (9, 13, 16). In the present study, the SEM analysis showed that the intact fiber post presents a relatively smooth surface, which may impair mechanical retention. On the other hand, a silane coupling agent containing methacryloxypropyl trimethoxysilane (MPS) was used in this study. It has been shown that this MPS silane is unable to chemically bond to the epoxy resin (12). However, MPS silanes are able to couple OH-covered substrates (such as glass fibers) and to the organic matrix of resin adhesives (7, 18, 19). Thus, exposure of glass fibers by etching is necessary to obtain both mechanical retention and chemical bonding (10, 13, 16).

Both 24% and 50% H_2O_2 were able to partially dissolve the epoxy resin and expose the glass fibers after a 1-minute exposure. Despite the slight etching obtained by 24% H_2O_2 after 1-minute exposure, it was sufficient to produce bond strength similar to that obtained with higher concentrations or longer application times. It is important to note that all treatments with H_2O_2 exposed the fibers without damaging them. Dissolution of the epoxy resin probably relies on an electrophilic attack of the H_2O_2 to the cured secondary amine (20). Thus, the spaces created between the fibers provide conditions for the micromechanical interlocking of the resin adhesive with the post. Furthermore, the exposed fibers become available to chemically bond to the adhesive through the silane agent.

It has been documented that the use of peroxides during endodontic procedures might compromise the adhesive cementation of posts (21). This effect is attributed to the presence of residual oxygen into dentinal tubules interfering with the polymerization of the adhesive resin (22). However, the use of peroxide over the fiber post increased the bond strengths. The deleterious effect of the peroxide was probably not observed because of the absence of residual oxygen into the post structure. Another important observation was the absence of cohesive failures within the resin composite during the microtensile test. The high flow of the resin used in this study probably allowed a close contact between the resin and the post, reducing the presence of voids (23).

It is reasonable to expect that higher peroxide concentrations require shorter times to properly etch the fiber posts. However, the present results show that a relatively low concentration of H_2O_2 (24%) used in a feasible clinical time (1 minute) generated bond strength similar to that obtained with a higher concentration (50%) applied for longer times (5 and 10 minutes). H_2O_2 is frequently used in dental practice, mainly for dental bleaching, and is easy and safe to use. Based on the results of this study, the lower concentration (24%) of H_2O_2 used for only 1 minute is preferable in clinical use.

Acknowledgments

The authors deny any conflicts of interest related to this study.

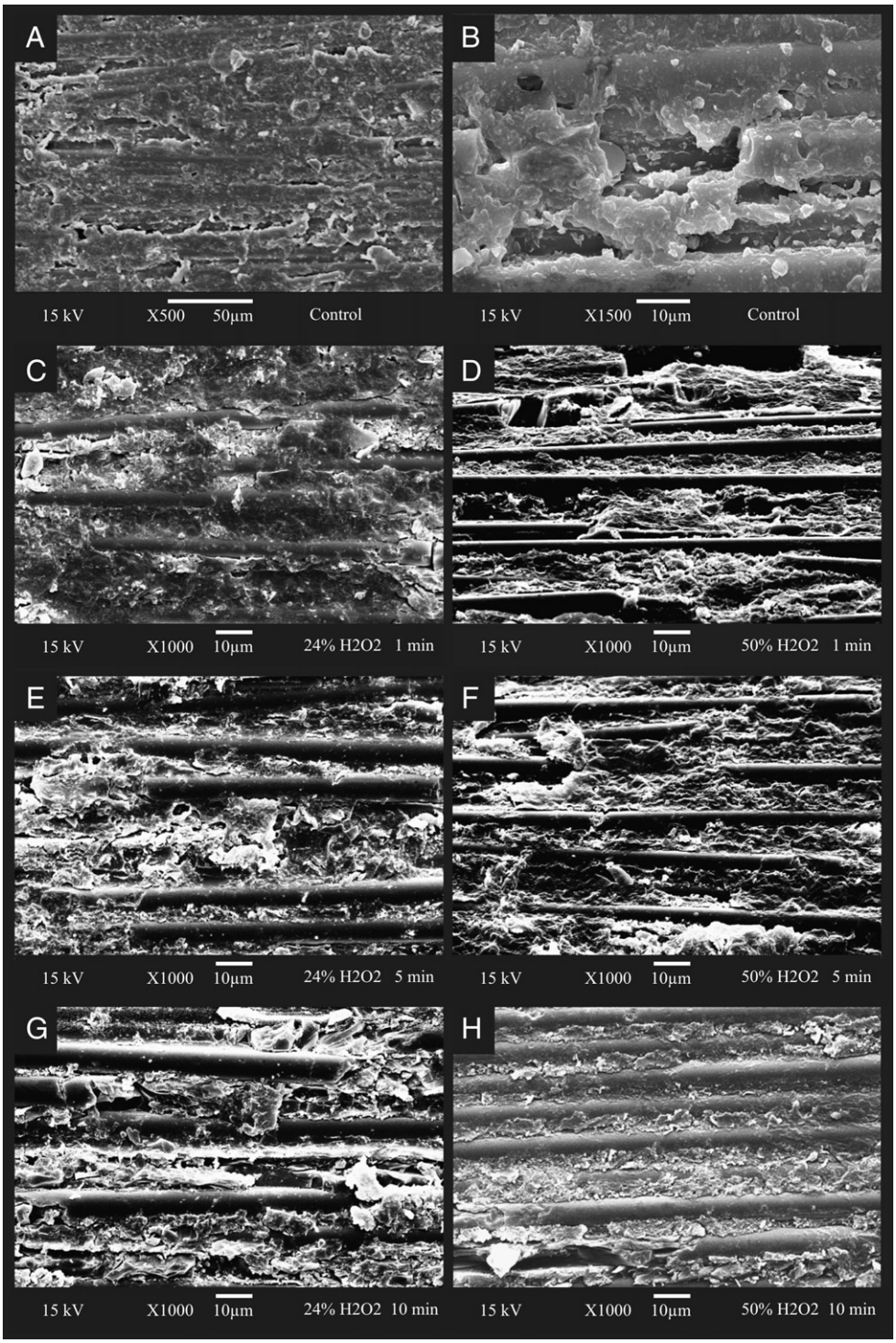

Figure 3. The analysis of the surface topography by SEM. (*A* and *B*) Without treatment, (*C*) 24% H_2O_2 for 1 minute, (*D*) 50% H_2O_2 for 1 minute, (*E*) 24% H_2O_2 for 5 minutes, (*F*) 50% H_2O_2 for 5 minutes, (*G*) 24% H_2O_2 for 10 minutes, and (*H*) 50% H_2O_2 for 10 minutes.

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