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RESEARCH AND EDUCATION

Effect of different fiber post surface treatments on microtensile bond strength to composite resin

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ABSTRACT

Statement of problem. The interface of fiber post and composite resin is a site of potential failure of adhesion. Improving this interface adhesion through different pretreatments of the fiber post surface has been suggested, but the results are controversial.

Purpose. The purpose of this in vitro study was to evaluate the effect on the bond strength to composite resin of pretreating glass fiber post surfaces with hydrogen peroxide, phosphoric acid, and a silane coupling agent.

Material and methods. Glass fiber posts were treated for 1 or 5 minutes with 30% hydrogen peroxide or 35% phosphoric acid. Treated posts were divided into silanization and no silanization groups. Control groups included no treatment or treatment with silanization alone (total of 10 groups; n=14). Composite resin was bonded to the fiber posts, and the specimens were cut into beams with the fiber post in the middle and the composite resin at both sides. The beams were attached to a mechanical testing device, and microtensile bond strength was evaluated. Fracture modes were assessed using stereomicroscopy. Statistical analysis was done with 3- and 2-way ANOVA (α=.05). Additional specimens were evaluated with a scanning electron microscope (SEM) to evaluate the effect of treatments on the characteristics of fiber post surfaces.

Results. The highest bond strength values were found in the group treated with phosphoric acid for 5 minutes with silanization, followed by the group treated with silanization alone. ANOVA showed a statistically significant effect for silanization (P<.05), but no statistically significant effect for surface treatment. SEM evaluation revealed cracked and dislodged superficial fibers in all groups, with no obvious difference in fiber exposure among the groups.

Conclusions. The silane coupling agent had a significant effect on the bond strength of the tested glass fiber posts to composite resin, whereas 30% hydrogen peroxide or 35% phosphoric acid did not. (J Prosthet Dent 2016;■:■-■)

Most endodontically treated teeth have lost extensive coronal structure because of caries, fracture, attrition, or previous restorations. Restoring such teeth with composite resin may require the use of prefabricated posts to ensure retention of the foundation.[1-3] Fiber posts and composite resin are popular for coronal restoration because they can shorten the restoration procedure and possess adequate esthetic and mechanical properties.[4] Fiber posts have been shown to increase the fracture resistance of restored teeth and to conserve tooth structure.[5]

The clinical success of a post-and-core restoration depends on the materials selected and the quality of the interfaces, where materials of different compositions are in intimate contact.[6] Proper bonding at the post-composite resin interface is needed to dissipate occlusally generated stresses.[7] The effect of different pretreatments on the post surface on post-composite resin bond strength has been evaluated.[1,3,6-11] The purpose of these pretreatments has been to alter the resin surface of the fiber post to enhance its bond with composite resin through either chemical or mechanical bonding. Silane application has been shown to enhance the microtensile bond strength of resin core

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Clinical Implications

Applying a silane coupling agent to glass fiber posts before placing the composite resin foundation enhances bond strength. The surface treatment of fiber posts at chairside with 30% hydrogen peroxide or 35% phosphoric acid did not enhance bond strength to composite resin and is not recommended.

material to fiber posts,[11-13] but interfacial strength values have been found to be relatively low compared with the values achieved with dental substrates.[14] Hydrogen peroxide has been shown to selectively dissolve epoxy resin and expose glass fibers,[15] with reported increase in bond strength.[1,3,6-8] The effect of other chemical agents or procedures on the bond strength of fiber post to resin cements has been investigated,[6,8,10,16-22] but we have found few studies of the effect of clinically feasible procedures on the bond strength of fiber post to resin foundation. As the pretreatments should be done clinically, the availability of the treating agent, feasibility, and time consumed by the procedure at chairside should also be considered. Phosphoric acid is the material of choice for etching enamel and dentin during resin bonding procedure and is almost always at hand in the dental office.

The purpose of this in vitro study was to evaluate the effect of different surface treatments of epoxy resin-based glass fiber posts on its microtensile bond strength to composite resin foundation material and on the morphological aspects of the fiber post surface using the scanning electron microscope (SEM). The null hypothesis tested was that different fiber post surface treatments do not alter the bond strength of fiber post to composite resin.

MATERIAL AND METHODS

A total of 140 glass fiber posts (Whitepost DC #3; FGM) were used in this in vitro study. The coronal end of the post used was cylindrical in shape and 2 mm in diameter. The remaining apical 11 mm was conical in shape. The composition of the posts was 80.0% ±5.0% glass fibers and 20.0% ±5.0% epoxy resin (as stated by the manufacturer). Surface treatments and group codes are summarized in Table 1 comprising a total of 10 groups (n=14). Each post (including the control group) was rinsed with water for 30 seconds followed by air drying before surface treatment and, if applicable, after immersion in hydrogen peroxide or phosphoric acid (Condac 37; FGM). A silane coupling agent (Prosil; FGM) was applied with a microbrush and left to dry for 60 seconds according to the manufacturer's instruction. To bond the foundation composite resin, each post was placed horizontally on a glass slab, and composite resin (Clearfil Photo Core; Kuraray Dental) was added to the sides of the cylindrical part of the post in 2-mm increments up to 6 mm. The composite resin layer was filled up to the diameter of the post, and the thickness was controlled by placing another glass slab over the fiber post, stabilized by 2 other posts (Fig. 1). Each layer of composite resin was polymerized with a light-emitting diode polymerizing unit (LITEX 695; Dentamerica) with an output of 600 mW/cm^2 for 40 seconds. The set material was light-polymerized for another 40 seconds from the bottom side of the glass slab, and the specimen was gently removed.

Table 1. Study groups and respective surface treatments, followed by mean ±SD microtensile bond strength (MPa), of fiber post to composite resin

Group Code	Surface Treatment	Mean ±SD
C	None	16.8 3.3
S	1 min silanization	23.7 3.8
H1	1 min treatment with hydrogen peroxide	20.7 3.7
H1S	1 min treatment with hydrogen peroxide + 1 min silanization	21.9 3.6
H5	5 min treatment with hydrogen peroxide	19.6 2.8
H5S	5 min treatment with hydrogen peroxide + 1 min silanization	21.6 1.5
P1	1 min treatment with phosphoric acid	19.4 3.5
P1S	1 min treatment with phosphoric acid + 1 min silanization	22.0 2.7
P5	5 min treatment with phosphoric acid	21.1 3.2
P5S	5 min treatment with phosphoric acid + 1 min silanization	23.8 3.3

The sectioning and loading of the specimens began on completion of the composite resin bonding procedure to simulate the clinical situation of immediate preparation following core build-up. Each specimen was serially sectioned into 4 or 5 beams of about 1 mm in thickness with a rotary cutting disk (916D; Jota) under running water. Each beam contained a section of the fiber post with bonded composite resin at both sides (Fig. 1). The beam width was narrowed with the cutting disk to approximately 1.5 mm and bonded to the flat grips of a microtensile testing device (MTD-500; SD Mechatronik), using cyanoacrylate adhesive (Mitre Bond). Tensile load was applied to the specimen until failure at a crosshead speed of 0.5 mm/minute. The precise width and thickness of the beam at the failed interface was measured with a digital caliper (Instar) to the nearest 0.1 mm, and the strength was calculated using the mathematical formula described by Bouillaguet et al.[23] Three beams from each specimen were tested, and the mean strength was used for analysis.

Data were analyzed using 3- and 2-way ANOVA to examine the effects of surface treatment and silane application on microtensile bond strength. The Student

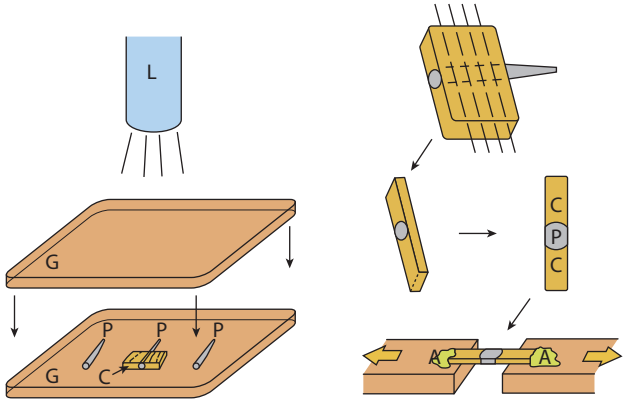

Figure 1. Schematic composite resin bonding procedure and specimen preparation. Refer to text for description. A, cyanoacrylate adhesive; C, composite resin; G, glass slab; L, light polymerizing unit; P, fiber post.

t test was used for pairwise comparison between groups (α=.05).

Failure mode was evaluated using stereomicroscopy (ZTX-20-W; Huaguang) at ×40 magnification. Failures were classified as adhesive between the post and composite resin, cohesive within the post, cohesive within the composite resin, or mixed failures.

Two additional fiber posts from groups C, H1, H5, P1 and P5 were examined with SEM (ProX; Phenom) to study the surface morphological characteristics of different surface treatments. Each fiber post was cut at the junctions of its cylindrical and tapered sections, and the cylindrical section was treated as mentioned for each group. Specimens were placed in an ultrasonic bath of distilled water (Elmasonic S; Elma Schmidbauer) for 3 minutes and washed with ethanol before SEM evaluation. Images of the specimens were obtained without gold sputtering.

RESULTS

Resulting microtensile bond strength values are summarized in Table 1. The highest values were obtained in group P5S, followed by group S. The 3-way ANOVA results showed no significant interaction among the 3 factors of silane application, surface treatment agent, and treatment time length (Table 2). Two-way ANOVA (including the control groups) showed a significant effect of silane application, but surface treatment with treating agents did not show any statistically significant effect (Table 3). Group S showed significantly higher bond strength than group C (P<.001).

Most of the specimens showed adhesive failure. Only 2 specimens from group S showed cohesive failure of the post.

SEM analysis revealed cracked and dislodged superficial fibers in all groups including group C. The cut edge of the fiber posts, however, showed intact internal fibers.

Table 2. Summary of 3-way ANOVA interaction analysis of effect of silane application, surface treatment agents, and treatment time on microtensile bond strength of fiber posts to composite resin in experimental groups

Source	Type III Sum of Squares	*df*	Mean Square	F	*P*
Corrected Model	196.628	7	28.090	2.895	.008
Intercept	50535.716	1	50535.716	5 207.905	<.001
Silane	127.716	1	127.716	13.162	<.001
Treatment agent	11.976	1	11.976	1.234	.269
Treatment time	6.938	1	6.938	0.715	.400
Silane×treatment agent	7.872	1	7.872	0.811	.370
Silane×treatment time	1.261	1	1.261	0.130	.719
Treatment agent×treatment time	40.114	1	40.114	4.134	.050
Silane×treatment agent×treatment time	0.750	1	0.750	0.077	.782

Table 3. Summary of 2-way ANOVA showing effect of different surface treatments on microtensile bond strength of fiber posts to composite resin

Source	Type III Sum of Squares	*df*	Mean Square	F	*P*
Corrected Model	559.736	9	62.193	6.067	<.001
Intercept	62116.973	1	62116.973	6 059.289	<.001
Silane	339.285	1	339.285	33.096	<.001
Surface treatment	76.719	4	19.180	1.871	.119
Silane×surface treatment	143.731	4	35.933	3.505	.009

The frequency of exposed glass fibers was not obviously different among the groups (Fig. 2).

DISCUSSION

Surface treatments affected the microtensile bond strength of glass fiber post to composite resin, so the null hypothesis was rejected. Silane application promoted bond strength, which is in accordance with previous reports.[11-13] The silane coupling agent used in this study was an ethanol solution of hydrolyzed 3-metacriloxipropil trimetoxysilane. Silane-coupling agent bridges the resin and OH-covered inorganic substances at the fiber post-composite interface. The highly cross-linked epoxy resin matrix of the fiber post does not have any functional groups available for reaction, so the chemical reaction is possible only between the composite resin and the exposed glass fibers of the post.[11]

The removal of the superficial layer of epoxy resin by means of chemical or mechanical treatment may leave more exposed fibers to react with the silane molecules. Previous studies have shown that hydrogen peroxide can effectively dissolve epoxy resin without damaging glass fibers.[1,3,8] These studies have shown a significant increase of bond strength between fiber post and composite resin after treatment with hydrogen peroxide. All of these studies used a silane-treated group as the control group. In the current study, silanization alone was found

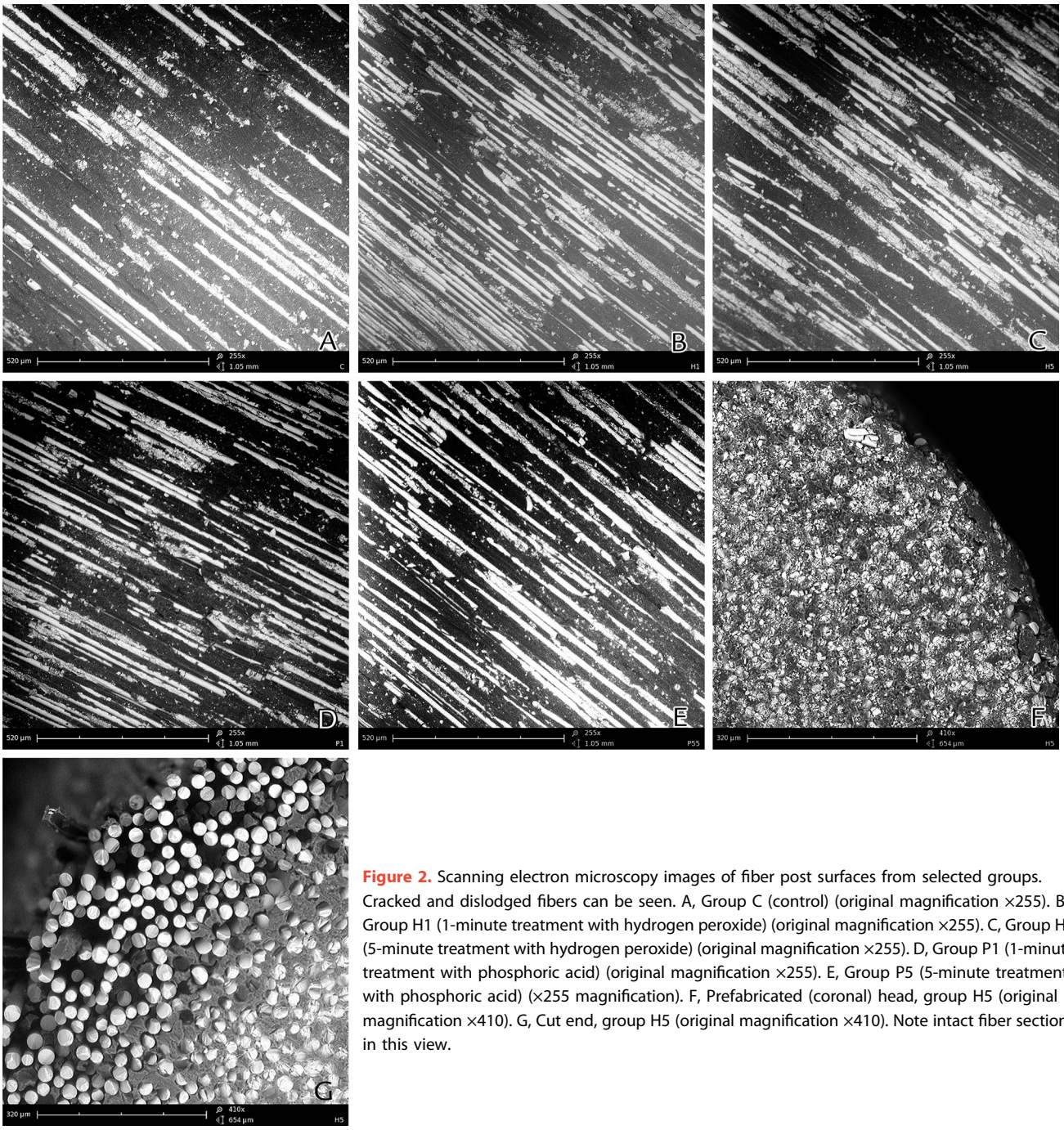

Figure 2. Scanning electron microscopy images of fiber post surfaces from selected groups. Cracked and dislodged fibers can be seen. A, Group C (control) (original magnification ×255). B, Group H1 (1-minute treatment with hydrogen peroxide) (original magnification ×255). C, Group H5 (5-minute treatment with hydrogen peroxide) (original magnification ×255). D, Group P1 (1-minute treatment with phosphoric acid) (original magnification ×255). E, Group P5 (5-minute treatment with phosphoric acid) (×255 magnification). F, Prefabricated (coronal) head, group H5 (original magnification ×410). G, Cut end, group H5 (original magnification ×410). Note intact fiber sections in this view.

to result in higher microtensile bond strength values than treatment with hydrogen peroxide. Results of the current study are in conflict with those of previous studies because it was found that hydrogen peroxide did not affect the bond strength achieved with silane. This conflict may be because of the different material used, as Vano et al[3] stated the bond strength between fiber post and composite resin depends on the materials selected. In a study on the effect of bleaching agents on composite-to-composite bond strength, Ferrari et al[24] found that 35% hydrogen peroxide did not significantly promote the composite-to-composite bond strength relative to that of the unbleached control group, whereas a mixture of sodium perborate and 3% hydrogen peroxide yielded better results. The authors suggested that composite resin might behave as a potential reservoir for residual hydrogen peroxide and oxygen byproducts as a consequence of its porosity and that residual

oxygen might have inhibited resin polymerization and reduced the composite-to-composite bond strength. In the present study, SEM analysis showed that the tested posts had a very rough surface, which may have similarly acted as a hydrogen peroxide reservoir, thus producing lower results in hydrogen peroxide groups. The results are also in accordance with those of the study of Mosharraf et al,[9] who reported higher microtensile bond strength between fiber posts and composite resin in an experimental group with silanization than another group with 24% hydrogen peroxide treatment without silanization.

Both hydrogen peroxide and phosphoric acid failed to show significantly higher bond strength results than the control group. Goncalves et al[18] reported that phosphoric acid could increase the bond strength between resin cements and glass fiber posts. They suggested that cleaning agents improved the bond strength either by removing debris and exposing glass fibers or by dissolving the epoxy resin and roughening the surface. Skupien et al[16] also indicated in a systematic review that cleaning the post before cementation with phosphoric acid improved retention compared with silane application without cleaning. The results of the current study also failed to show an increase in fiber post to composite bond strength by immersion in phosphoric acid when using silane coupling agent.

These results suggest that silanization alone can improve the bond strength between the fiber post and composite resin and that surface treatment with either hydrogen peroxide or phosphoric acid may not contribute to the bond strength. A possible explanation of this phenomenon may be the set of materials selected for this study. As revealed by the SEM images, the fiber post used in this study had a very rough surface, showing cracked and dislodged fibers. These characteristics resemble previous airborne-particle abrasion of the fiber post through the manufacturing process, similar to characteristics reported by Soares et al.[21] The authors of the current study were not able to find any documents provided by the manufacturer mentioning airborne-particle abrasion during the manufacturing process.

The microtensile bond strength test used in this study is regarded as a reliable method of bond strength evaluation.[11] Alternatively, a micro-push-out test could be adopted, in which the post is pushed out of a core cylinder by means of an appropriately sized punch. In both methods, the small size of the tested interface provides a uniform distribution of the load. The bond strength values were high relative to those of similar studies, which may be explained by the material selected or the roughness of the post surface.[3]

However, this experimental study has some limitations. The data of this in vitro study do not predict exactly the fiber posts performance in vivo. The results are also dependent on the composition of the materials. Analyzing other types of fiber posts and composite resins and comparing their performances would be of interest. The study of the effect of thermocycling and aging on the performance of fiber post-composite bond is also of interest.

CONCLUSIONS

Within the limitations of this in vitro study, it was concluded that the application of a silane-coupling agent on glass fiber posts before composite resin foundation placement enhanced the bond between fiber post and composite resin. Treating the fiber post surface with either 30% hydrogen peroxide or 35% phosphoric acid for 1 or 5 minutes did not contribute to the bond strength and thus is not recommended.

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