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Flexural properties, morphology and bond strength of fiber-reinforced posts: influence of post pretreatment

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ABSTRACT

The aim of this study was to assess the influence of surface pretreatments of fiber-reinforced posts on flexural strength (FS), modulus of elasticity (ME) and morphology of these posts, as well as the bond strength (BS) between posts and core material. Fifty-two fiber posts (smooth and serrated) were assigned to 4 groups (n=13): no treatment (control), 10% hydrogen peroxide (HP) for 10 min (HP-10), 24% HP for 1 min (HP-24) and airborne-particle abrasion (Al_2O_3). To evaluate FS and ME, a 3-point bending test was performed. Three posts of each group were examined by scanning electron microscopy. Composite resin was used as the core build-up and samples were sectioned to obtain microtensile sticks. Data were analyzed by ANOVA and Tukey's test (α=0.05). For FS, significant differences were observed between posts type and surface pretreatment ($p<0.05$), with the highest means for the smooth posts. Al_2O_3 provided higher FS than HP-24. Al_2O_3 promoted higher ME than HP-24 and control. SEM images revealed partial dissolution of the resin matrix in all treated groups. The smooth posts had higher BS and FS than serrated posts ($p<0.05$). Mechanical properties of the glass fiber posts and the bond strength between posts and composite material were not altered by the surface treatments, except for airborne-particle abrasion that increased the post elastic modulus.

Keywords: aesthetics, dental practice, fiber post.

RESUMO

O objetivo deste estudo foi avaliar a influência do pré-tratamento de superfície dos pinos de fibra de vidro na resistência à flexão (RF), módulo de elasticidade (ME) e morfologia, bem como a resistência de união (RU) entre os pinos e o núcleo de preenchimento. Cinqüenta e dois pinos de fibra de vidro (lisos e serrilhados) foram divididos em 4 grupos (n=13): sem tratamento (controle), peróxido de hidrogênio a 10% por 10 min (HP-10), peróxido de hidrogênio 24% por 1 min (HP-24) e jato de óxido de alumínio (Al_2O_3). Para avaliar a RF e ME, o ensaio de flexão de 3 pontos foi realizado. Três pinos de cada grupo foram examinados em MEV. Resina composta foi utilizada como núcleo de preenchimento e as amostras foram seccionadas para obter palitos de microtração. Os dados foram analisados por ANOVA e teste de Tukey (α=0,05). Na RF, observou-se diferença estatisticamente significante entre os pinos e tipo de pré-tratamento de superfície (p<0,05), com as maiores médias para os pinos lisos. Al_2O_3 proporcionou maior RF que HP-24. Al_2O_3 promoveu maior ME que HP-24 e grupo controle. MEV revelou dissolução parcial da matriz de resina em todos os grupos tratados. Os pinos lisos tiveram a maior RU (p<0,05). Os pinos lisos apresentaram RF e RU superior aos pinos serrilhados (p<0,05). As propriedades mecânicas dos pinos de fibra de vidro e a resistência de união entre os pinos e o material resinoso não foram alterados pelos tratamentos de superfície, com exceção do jato de óxido de alumínio que aumentou o módulo de elasticidade dos pinos.

INTRODUCTION

Glass fiber-reinforced posts have been proposed for the restoration of endodontically treated teeth due to the esthetic coloration and favorable biomechanical properties (1-3). The modulus of elasticity of glass fiber posts is almost similar to that of tooth structure and resinous materials (3), thus it can provide optimal stress distribution and reduce the risk of root fracture.

Chemical and mechanical treatments of the post surface as well as changes in the matrix of the posts appear to influence the bond strength between resin materials and fiber posts (4-10). The post matrix, consisting of epoxy resin, has no functional group able of reacting with the silane and the effectiveness of the adhesion may be compromised (8,11). Surface post treatments with subsequent silanization are proposed to allow better interaction with the resin cements and enhance chemical and micromechanical bonding (12).

Some techniques are proposed for conditioning the fiber post surface, such as hydrogen peroxide (HP) solution (12-14) and airborne-particle abrasion (7,15-17). It is speculated that HP is able to degrade the epoxy resin matrix and expose the glass fibers. The use of more concentrated products with less exposure time could improve the adhesion and optimize the clinical time (12). Airborne-particle abrasion produces a roughened surface as a result of the high-speed impact of abrasive particles, enabling better interaction with the resin cement, but the roughened surface may produce surface damage which, in turn, reduces the strength of the post (7).

Surface pretreatment of posts is expected to increase the chemical and mechanical bond between the composite core material and post, but the best method has not yet been established. Furthermore, few studies evaluated the effect of conditioning methods on the morphology and mechanical properties of posts. The aims of this study were 1. to assess *in vitro* the effect of surface treatments of two glass fiber posts (smooth and serrated) with 10% HP for 10 min (HP-10), 24% HP for 1 min (HP-24) and airborne-particle abrasion (Al_2O_3) on flexural strength (FS), modulus of elasticity (ME) and morphology of these posts; 2. to evaluate the bond strength (BS) between fiber posts and composite core material.

MATERIAL AND METHODS

Two types of glass fiber-reinforced posts were selected: 1) Reforpost (Angelus, Londrina, PR, Brazil) and 2) White Post DC (FGM, Joinville, SC, Brazil). Posts from Reforpost system are parallel, serrated and have a 1.5-mm diameter (corresponding to the #3). Posts from White Post DC system have dual shape and diameter of 1.6 mm in the cylindrical part (corresponding to the #1).

Fifty-two posts of each type were cleaned with 70% ethanol, dried and distributed randomly into 4 groups (n=13), according to the type of surface treatment: A - no treatment (control, ST); B - 10% HP (Dynamics, São Paulo, SP, Brazil) for 10 min (HP-10); C - 24% HP (Dynamics) for 1 min (HP-24) and D - airborne particles abrasion (Polidental LTDA, Cotia, SP, Brazil) (Al_2O_3).

In groups 1B and 2B, the posts were immersed in 2 mL of 10% HP for 1 min and then immersed in four baths with

3 mL of distilled water, remaining 1 min in each. They were then subjected to the final wash with 3 mL of distilled water and air-dried.

In groups 1C and 2C, the posts were immersed in 2 mL of 24% HP for 10 min, immersed in four baths with 3 mL of distilled water for 1 min each, subjected to a final wash with 3 mL of water distilled and air-dried.

In 1D and 2D groups, the posts were abraded with aluminum oxide particles (50 µm), using a microjet system (Bio-Art, São Carlos, SP, Brazil) at a distance of 10 mm from the specimen, 90° inclination and 2 bar pressure. The posts were demarcated into 4 parts and each was sprayed for 3 s, a total of 12 s. Subsequently, the posts were washed with distilled water and air-dried in the same sequence of groups B and C.

Three-Point Bending Flexural Test

For this test, the standards suggested by DIN-EN 843-1 were followed. Five posts of each group were stored at 37° C for 48 h. To standardize the post shape, only the cylindrical portion of White Post DC system (smooth) was used. Each post diameter was measured at 5 different points with digital caliper (Digimess; Precision Gaging Shiko Ltd., China). The mean measures of each post were recorded and tabulated for subsequent calculations. The flexural test was carried out in universal testing machine (Instron 4444), following ISO 178 standard specifications. A metallic device attached to Instron machine was developed for this test. This device fixed the distance between the center support points in 8 mm and fits the dimensions of the sample. The load cell was applied perpendicular to the post long axis with a crosshead speed of 0.5 mm/min until fracture. Post flexural strength (σ_f) was calculated using the following equation: $\sigma_f = 3F_{max} L/2d^2$, where, F_{max} is the maximum load in N, L is the distance between the support points in mm (standardized in 8 mm) and d is the post diameter.

Modulus of Elasticity

From the values obtained by 3-point bending test, the modulus of elasticity (E_f) were calculated by the following equation: $E_f = S4L^3/3d^4$, where S is the stiffness, L is the distance between the support points in mm (8 mm) and d is the diameter of the post. Therefore, to obtain the E_f value, it was previously calculated value of *S* by the equation: F=S/D, where F is the maximum load value in Newton and D is the deflection value (in mm).

SEM Analysis of Posts Surface

Three posts of each groups were cleaned in a ultrasonic bath (3L Alpha Plus; Ecel, Ribeirão Preto, SP, Brazil), immersed in 95% ethanol, air-dried and stored at 37°C for 1 h. Gold-palladium sputter coating was carried out (Denton Desk II; Denton Vacuum LLC, Moorestown, NJ, USA) and the posts were examined with a scanning electron microscope (JSM-5410; JEOL Ltd., Tokyo, Japan). SEM micrographs were done with standardized magnifictaion of × 500 and were analyzed qualitatively.

Bond Strength Test

The core was filled with self-curing resin (Core-Flo; Bisco Inc., Schaumburg, IL, USA) using a silicone mold (Aquasil Ultra LV; Dentsply, Milford, DE, USA) obtained from matrix formed by post fiber and acetate plates. Five posts of each group were prepared for microtensile test. A silane layer (Bis-Silane; Bisco Inc.) was applied, after 60 s, as recommended by the manufacturer. The following was done applying a layer of the adhesive system All Bond 2 (D/E Resin; Bisco Inc.) and light-cured for 20 s. The post was inserted inside the silicon mold in the space provided. The composite resin (Core-Flo; Bisco Inc.) was mixed for 15 and inserted in the spaces corresponding to the core using a Centrix syringe (DFL, Rio de Janeiro, RJ, Brazil). After 30 min, the molds were cut and the samples removed and stored in distilled water at 37°C.

After 48 h, the specimens were fixed in a precision cutting machine (Minitom; Struers, Copenhagen, Denmark). Serial sections were made perpendicular to the long axis of the post using a water-cooled diamond disc (Extec, Enfield, CT, USA). The sections distanced themselves from each other about 1 mm, which allowed obtaining 5 to 6 slices *per* sample, resulting in 1 x 1 x 10 mm sticks-shaped, consisting of resin/post/resin. The sticks (almost 28 for each group) were evaluated in a stereomicroscope (Leica microsytem LTD, Wetzlar, Germany) (×40) and the 15 most homogeneous ones were selected. Those with defects or close to the adhesive interface were discarded.

On each stick, height and width were measured with digital caliper (Digimess; Precision Gaging Shiko Ltd.). The samples were fixed individually in a metal device with cyanoacrylate adhesive (Loctite Super Bonder, São Paulo, SP, Brazil). The set was attached to the universal testing machine and were tested with load cell of 50 kgf and crosshead speed of 0.5 mm/min until breaking. The tensile strength values were transformed into MPa. The samples were analyzed in the stereomicroscope (×40) and classified according to the fracture mode: cohesive of the post; cohesive of the resin; adhesive between post and resin; and mixed (presence of cohesive and adhesive fracture in the same specimen).

Statistical Analysis

Data of flexural strength (MPa), modulus of elasticity (GPa) and the microtensile bond strength (MPa) were analyzed by ANOVA and Tukey test with significance level of 5%, using SPSS (Statistical Package for the Social Sciences; SPSS Inc., Chicago, IL, USA).

RESULTS

Three-Point Bending Flexural Test

Statistical analysis revealed a significant effect on both factors: *post type* and *post pretreatment* ($p<0.05$). No significant difference were verified in the interaction of these factors ($p>0.05$). Smooth posts (White Post DC) had higher mean values than the serrated posts (Reforpost) ($p<0.05$).

Pretreatment with Al_2O_3 provided highest values of flexural strength and was statistically similar ($p>0.05$) to HP-10 and ST, which were statistically similar ($p>0.05$). Fiber posts pretreated with HP-24 showed the lowest flexural strength and did not differ from HP-10 and ST ($p>0.05$). There were significant differences between HP-24 and Al_2O3 groups ($p<0.05$). None of the proposed pretreatments differed from the control group ($p>0.05$) (Table 1).

Table 1. Mean and standard deviation of flexural bond strength (MPa), modulus of elasticity (GPa) and microtensile bond strength to resin (MPa) of fiber-reinforced posts after different surface pretreatments.

Surface pretreatment	Flexural strength	Modulus of elasticity	Bond strength
No treatment (control)	687.68 ± 54.29 ab	11.36 ± 0.84 A	10.55 ± 6.97 ●
10% HP/10 min	681.18 ± 78.19 ab	11.62 ± 2.19 AB	16.12 ± 7.17 ♣
24% HP/1 min	652.23 ± 83.92 a	10.14 ± 1.14 A	19.76 ± 7.58 ♣
Airborne-particle abrasion	730.29 ± 53.09 b	13.43 ± 1.98 B	18.88 ± 10.93 ♣

Flexural strength: Same lowercase letters indicate statistical similarity ($p>0.05$; Tukey's critical value=60.31). Modulus of elasticity: Same uppercase letters indicate statistical similarity ($p>0.05$; Tukey critical value=2.01). Bond strength: Same symbols indicate statistical similarity ($p>0.05$; Tukey's critical value=5.46).

Modulus of Elasticity

Analysis of variance showed a statistically significant difference ($p<0.05$) between the surface treatments of the posts. Factor type of interaction between post and type of treatment showed no statistically significant difference ($p>0.05$). Al_2O_3 blasting resulted in higher values of modulus of elasticity and was statistically similar to group HP-10 ($p>0.05$). HP-24 treatment gave the lowest modulus of elasticity and was not significantly different from ST-10 and HP, which were statistically similar ($p>0.05$). Only Al_2O_3 increased the modulus of elasticity of the posts in the control group ($p<0.05$) (Table 1).

SEM Analysis

SEM micrographs of serrated and smooth posts revealed that those pretreated with HP (10% for 10 min or 24% for 1 min) and airborne-particles abrasion were different from the untreated posts (control). On untreated posts, for both the smooth and serrated groups, the fibers were shown to be continuous and covered by epoxy resin matrix (Figs. 1A and 1B). Pretreatment with 10% HP for 10 min, promoted a slight exposure of the post fibers (arrows), a more evident aspect for the smooth type (Figs. 2A and 2B). Application of 24% HP for 1 min provided more areas with dissolution of resin matrix and subsequent exposure of the post fibers (arrows). In these groups, some areas with discontinuity of the fibers (circles) were verified (Figs. 2C and 2D). Posts abraded with airborne-particles provided exposed fiber areas (arrows) and discontinuity (circles) post fibers. Some probable remnants of the abrasive particles used during air-abrasion were noted in this group (Figs. 2E and 2F).

Bond Strength Test

Statistical analysis showed significant difference on both factors: post type and post pretreatment ($p<0.05$). There was no significant statistical difference ($p>0.05$) in the interaction of factors. Smooth posts (White Post DC) had higher mean values than the serrated posts (Reforpost) ($p<0.05$).

ST had the lowest mean bond strength and was statistically different from the other groups ($p<0.05$). Overall, all surface pretreatments increased the microtensile bond strength of the fiber-reinforced posts to resin core material (Table 1). The analysis of failures after showed a predominance of adhesive mode in control group and mixed in treated groups of both posts, regardless of the surface pretreatment (Table 2).

Table 2. Failure types (%) after microtensile bond strength test.

Failure	Serrated posts				Smooth posts			
	NT	10% HP	24% HP	Al_2O_3	NT	10% HP	24% HP	Al_2O_3
Adhesive	66	40	40	33	53	40	40	33
Cohesive (post)	7	7	0	14	7	0	7	0
Cohesive (resin)	0	7	7	0	7	0	0	7
Mixed	27	46	53	53	33	60	53	60

NT: no treatment; 10% HP - 10% hydrogen peroxide after 10 min; 24% HP: 24% hydrogen peroxide after 1 min; and Al_2O_3: Airborne-particle abrasion.

DISCUSSION

Fiber-reinforced posts are preferred than cast posts due to higher post retention, reduced risk of root fracture and esthetics (18). However, the longevity of fiber-reinforced posts restorations depends mainly on the strong bond between resin core material, dentin and post (4,12,14). Different treatments have been proposed to modify the surface of fiber posts in order to improve the adhesive capability as well as the clinical performance of these materials (19).

The results of this study showed that smooth posts had higher flexural strength than serrated posts, although the elastic modulus was similar for both posts, regardless of surface pretreatment they have undergone. A possible explanation for the lower flexural strength obtained by serrated posts can be the configuration of these posts, which promote disruption of longitudinal glass fibers close to the additional retentions (20) and also provide stress concentration in these regions (21). The fact that the post resistance is strongly influenced by the resin matrix fibers, justifies the decrease in flexural strength of serrated posts (7,22).

Regarding the bond strength, it is important to highlight that the methacrylate-based luting agent used in this study has no chemical affinity for the epoxy resin matrix of posts, but has affinity for the glass fibers (5,11,13,14). Therefore, superficial cracks found in the fibers of serrated posts can be a critical point on the post/resin interface and probably reflected in lower adhesion (20). Authors have investigated different procedures for improving the interfacial bond strength between posts and resin-based materials using chemical and mechanical surface treatments and the benefit of using silane on post surface was confirmed (6,8). Monticelli et al. (6) reported an increased wettability of the fiber post surface after silanization.

In the present study, the flexural strength of fiber-reinforced posts abraded with airborne-particles was superior to those pretreated with 24% HP for 1 min, but both treatments were not different from 10% HP for 10 min and for the untreated posts (control). HP has the ability to dissolve the resin matrix, breaking the epoxy resin bonds and, consequently, exposing the fibers for silanization (5,16). In this process, only the resin matrix is partially dissolved, which reflects the selectivity of HP (12,14). As the fibers remain unchanged (15), there is no interference on the mechanical properties of the posts, hence resulting in similar values of flexural resistance, regardless the surface treatment. The SEM micrographs of groups pretreated with HP showed areas of partial degradation of the resin matrix.

Overall, the outcome of this study is corroborated by Soares et al. (7) that reported that the airborne-particle abrasion did not influence the flexural resistance of posts. However, in our study, aluminum oxide increased the modulus of elasticity of glass fiber posts. According to Schmage et al. (8), the post diameter was reduced after pretreatment with airborne-particle abrasion. In this study, the protocol used for air-abrasion (particles of 50 μm, pressure of 2.0 bars for 15 s and a distance of 10 mm from the surface) produced undesirable changes in the post shape, reducing its diameter, which were confirmed by measurements in made in the posts treated with airborne-particle abrasion. The reduction of the post diameter is related to the modulus of elasticity, since it is calculated by the equation $E_f = S4L3/3\pi d4$, which shows that this property is inversely proportional to diameter. Thus, the decrease in post diameter would increase the modulus of elasticity (2). HP did not alter the post diameter and therefore did not affect its elastic modulus.

When analyzing the bond strength, it was found that all protocols used in this study increased the bond strength of the post/resin interface. This fact can be confirmed by the analysis of the fractures after mechanical test, which revealed a predominance of adhesive failures in the control and treated groups of in both posts, regardless of the pretreatment. Non-treated fiber-reinforced posts have a smooth surface, which limits mechanical interlocking with resin cements, and purely adhesive failure modes are commonly found at the post-composite resin interfaces (16,23). The increased bond strength of posts treated with HP as compared to untreated corroborates the previous findings (6,12-14). In this study, SEM analysis confirmed the removal of epoxy resin surface, creating microgaps that enabled the retention of the adhesive system.

The increased bond strength by airborne-particles abrasion can be ascribed to the irregularities in the post surface, which increases the contact area and create potential spaces for micromechanical retention of the adhesive system, as observed in other studies (8,15,16).

Among all treatments tested in this study, airborne-particle abrasion was the only method that changed the elastic modulus of posts. Additionally, it is a sensitive technique and requires greater control of the operator. Further studies should be conducted to confirm the non-interference of airborne-particles in the adhesive strength.

The use of HP at high concentration may be a suitable alternative for surface pretreatment of the post (12). It was observed, in this study, that the mechanical properties and adhesion of resin to post were not affected by increasing the concentration of HP (from 10% to 24%) and decreasing the exposure time of the post to the solution (from 10 min to 1 min), which could be interesting to reduce the clinical time. However, protocols for the application of HP at different concentrations and exposure times need to be investigated.

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