Comparative evaluation of adhesion of glass fiber post to the root dentin after surface treatment of post and using universal bonding agent: An in vitro study

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Abstract
Objective: The purpose of the study is the evaluation and comparison of surface roughness and bond strength of glass fiber post (GFP) after different types of surface treatment and the application of a universal bonding agent.

Materials and Methods: Forty GFPs were divided into four groups based on surface treatment: Group I, silane coupling agent application for 60 s; Group II, air abrasion with 30 µm silicon dioxide powder particles in 2.5 bar pressure from 2 cm distance for 10 s, followed by silane coupling agent application; Group III, 9% hydrofluoric acid application for 10 s, rinsed and air-dried, followed by silane coupling agent application; and Group IV, silane coupling agent application, followed by universal bonding agent application. Surface roughness evaluation is done by a profilometer. All posts were cemented in the root of the maxillary central incisor with resin cement. After that, root was placed in an acrylic mold, and the external end part of the post was mounted on another acrylic mold. Pull-out bond strength was measured by a universal testing machine.

Results: Highest surface roughness and bond strength values were found in Group II.

Conclusion: Pretreatment of GFP increases the surface roughness of post as well as bond strength of post to root dentin. There is a correlation between surface roughness and bond strength. However, the use of only universal bonding agent also showed comparable pull-out bond strength of GFP, which means only use of universal bonding agent also a new alternative as pretreatment of GFP and helps in increase in bond strength.

Keywords: Bond strength; glass fiber post; pretreatment; surface roughness; universal bonding agent

INTRODUCTION
Glass fiber posts (GFPs) have been proposed as an alternative to metallic posts due to better masticatory stress distribution in the tooth-restoration structure and have optical properties similar to the natural dentition, which provides more favorable esthetic results. The elastic modulus of GFPs is similar to that of dentin, leading to a more homogeneous stress distribution compared to more rigid posts. The low modulus of elasticity of fiber-reinforced epoxy resin posts has been reported to reduce the risk of root fracture. Glass fiber posts are generally composed of reinforced glass fibers embedded in polymethyl methacrylate epoxy resin or, in some cases, urethane dimethacrylate, both of which have a similar flexural strength. The retention of GFPs in the root canal is dependent on proper adhesion between the resin cement and the intraradicular dentin and between the cement and post surface. GFP post retention depends on the strength of the chemical and micromechanical interaction among the post, dentin, and resin cement.

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The failure of this GFP often occurs because of debonding at the adhesive cement–dentin or the adhesive cement–post interface as a result of bond deficiencies. To increase the bond strength of posts to root dentin different types of surface treatments of posts are performed. In this in vitro study, we will compare the effect of three surface treatments named as, surface treatment with application of silane coupling agent, surface treatment with 30 µm SiO₂ air abrasion, surface treatment with 9% hydrofluoric acid on GFP, and also universal bonding agent application on GFP on adhesion with a resin-based luting agent.

Pull-out bond strength test is a type of shear bond test, it is mainly used to test the retentive ability of endodontic posts.[1] Hence, we have chosen a pull-out test for bond strength measurement of GFPs in root dentin.

The null hypothesis was that there was no difference in surface roughness of GFP after different types of surface treatment of GFP and no difference in bond strength of GFP after different types of surface treatment and application of universal bonding agent on GFP.

**MATERIALS AND METHODS**

Forty freshly extracted, intact, and noncarious human maxillary central incisors were collected from the Department of Oral and Maxillofacial Surgery, Guru Nanak Institute of Dental Sciences and Research, Kolkata, and were cleaned of calculus and soft-tissue remnants using an ultrasonic scaler. The teeth were disinfected using 3% sodium hypochlorite for 10 min and rinsed with distilled water according to OSHA guidelines. Inclusion criteria were freshly extracted maxillary central incisors; noncarious teeth, extracted from patients whose ages were between 20 and 40 years, and no morphologic variation of the tooth. Exclusion criteria were teeth with severe anatomic variation, any type of caries, noncarious lesions such as attrition, abrasion, erosion, abfraction, fracture, craze line, decalcification or fluorosis, or any kind of resorption.

**Sample preparation**

The teeth were stored in 0.1% Thymol solution at room temperature until the preparation for the study.

Decoronation of tooth done coronal to the cementoenamel junction of the tooth with a diamond disk in horizontal direction using a slow speed handpiece under cooling water to keep 15 mm root length. Then, pulp tissue was removed. Working length was established by passing the number 10K file, the tip of it was visible at the apex. Root canals were sequentially enlarged up to the size 50K file using the step-back technique. Irrigants were used: 5%–25% NaOCl and 17% EDTA alternatively. Enlarged canals were rinsed finally with distilled water, dried with paper points and the master cone was selected according to the master file (ISO size 50), and obturation was done using gutta-percha and AH Plus sealer by lateral compaction technique. The master cone was number 50. After obturation, tooth samples were stored in 0.1% thymol solution at room temperature for at least 72 h for a complete set of root canal sealers. Then, post space was prepared to a depth of 10 mm using peeso reamer up to size 3 leaving behind 5 mm of intact gutta-percha to preserve the apical seal of the tooth.

**Surface pretreatment of post**

Before post cementation, samples were divided into four groups according to surface treatment of post: [Figure 1].

Group I (n = 10): Only a silane coupling agent was applied on the post surface for 60 s and air-dried.

Group II (n = 10): Surface treatment of the post was done by air abrasion with 30 µm silicon dioxide powder particles at 2.5 bar pressure from a 2 cm distance for 10 s. Then, silane coupling agent was applied on the post surface and air-dried.

Group III (n = 10): 9% Hydrofluoric acid was applied to posts for 10 s; then rinsed with distilled water and air-dried the post. Then, silane coupling agent was applied on the post surface and air-dried.

Group IV (n = 10): Silane coupling agent was applied on the post surface. Then, universal bonding agent was applied on the post surface, followed by a light cure.

In Group I, the surface roughness of posts was measured after the application of a silane coupling agent on the post surface;
in Group II and Group III, the surface roughness of posts was measured before the application of a silane coupling agent on the post surface; and in Group IV, the surface roughness of posts was measured after application of bonding agent on the post by a digital profilometer [Figure 2].

After surface treatment cementation of the post for all the groups was done using a dual-cure resin luting cement (Multilink N, Ivoclar Vivadent). After that, mold (2 cm × 2 cm) is prepared with cold cure acrylic resin and during the dough stage root is placed inside the acrylic so that 2 mm of the coronal part of the tooth remains outside the mold surface. Then, the external end part of the post was mounted on another acrylic mold (2 cm × 2 cm) keeping a 2 mm exposed post at the junction of two blocks [Figure 3]. Then, specimens were stored in deionized water for 24 h at 37°C in an incubator. After that, pull-out bond strength was measured in all groups by a universal testing machine. The end part of the two blocks was cut according to the size of the holder of the universal testing machine. Then, the lower part of the sample was fixed in the lower holder of the machine and the upper part of the block was fixed in the upper holder of the movable machine [Figure 4].

The pull-out bond strength values were analyzed. Data of bond strength value of all groups were collected. Results were tabulated and means were obtained and statistical analysis was done.

**Statistical analysis**

Data were entered into a Microsoft Excel spreadsheet and then checked for any missing entries. It was analyzed using Statistical Package for Social Sciences version 21 (IBM SPSS Inc., Chicago, IL, USA). All the variables were continuous variables such as surface roughness and bond strength, which were summarized as mean and standard deviation. Graphs were prepared in Microsoft Excel.

The normality of the data was checked by the Shapiro–Wilk test. Data were found to be normal. Keeping in view the nature (continuous) and distribution (normal) of data, inferential statistics were performed using parametric tests of significance.

Inferential statistics were performed using the one-way analysis of variance test. One-way analysis of variance test was used to compare more than two independent means. *Post hoc* pairwise comparison was done using the *post hoc* Tukey’s test. The level of statistical significance was set at 0.05.

**RESULTS**

Table 1 intergroup comparison of mean surface roughness was done using one-way ANOVA test and the differences in mean surface roughness were found to be statistically significant. *Post hoc* pairwise comparison was done using Tukey’s test and it was found that surface roughness...
among Group 2 was found to be the highest and it was significantly more than that among Group 4, which was further significantly more than that among Group 1. The surface roughness of Group 3 samples was significantly more than that of Group 1 but did not show any significant difference with respect to Group 2 and Group 4.

Table 1: Intergroup comparison of mean surface roughness value

|          | n  | Mean       | SD    | 95% CI for mean Lower bound | Upper bound |
|----------|----|------------|-------|-----------------------------|-------------|
| Group 1  | 10 | 1.142      | 0.170 | 1.020                       | 1.263       |
| Group 2  | 10 | 1.592      | 0.185 | 1.459                       | 1.725       |
| Group 3  | 10 | 1.420      | 0.110 | 1.341                       | 1.498       |
| Group 4  | 10 | 1.347      | 0.081 | 1.289                       | 1.404       |

F, P
Post hoc pairwise comparison
- Group 1 $\times$ Group 2: 16.986, <0.001 (S)
- Group 1 $\times$ Group 3: 0.01 (S)
- Group 1 $\times$ Group 4: 0.01 (S)
- Group 2 $\times$ Group 3: 0.05 (NS)
- Group 2 $\times$ Group 4: 0.003 (S)
- Group 3 $\times$ Group 4: 0.666 (NS)

Table 2: Comparative evaluation of bond strength value between four groups measured in megapascal

|          | n  | Mean       | SD    | 95% CI for mean Lower bound | Upper bound |
|----------|----|------------|-------|-----------------------------|-------------|
| Group I  | 10 | 14.959     | 0.642 | 14.499                      | 15.419      |
| Group II | 10 | 23.549     | 0.775 | 22.995                      | 24.103      |
| Group III| 10 | 22.492     | 0.782 | 21.932                      | 23.052      |
| Group IV | 10 | 20.246     | 0.785 | 19.685                      | 20.807      |

F, P
Post hoc pairwise comparison
- Group 1 $\times$ Group 2: 261.198, <0.001 (S)
- Group 1 $\times$ Group 3: <0.001 (S)
- Group 1 $\times$ Group 4: <0.001 (S)
- Group 2 $\times$ Group 3: 0.03 (NS)
- Group 2 $\times$ Group 4: 0.003 (S)
- Group 3 $\times$ Group 4: 0.666 (NS)

Table 3: Correlation between surface roughness and bond strength in Group I, Group II, Group III, and Group IV

| Number of sample | Group I Surface roughness | Bond strength | Group II Surface roughness | Bond strength | Group III Surface roughness | Bond strength | Group IV Surface roughness | Bond strength |
|------------------|---------------------------|---------------|---------------------------|---------------|-----------------------------|---------------|---------------------------|---------------|
| 1                | 1.248                     | 14.51         | 1.363                     | 24.04         | 1.452                       | 23.91         | 1.377                     | 19.36         |
| 2                | 0.818                     | 14.90         | 1.302                     | 23.31         | 1.435                       | 22.67         | 1.485                     | 21.30         |
| 3                | 1.117                     | 14.58         | 1.489                     | 23.72         | 1.533                       | 22.42         | 1.278                     | 20.63         |
| 4                | 0.982                     | 15.15         | 1.687                     | 22.19         | 1.582                       | 21.91         | 1.339                     | 20.28         |
| 5                | 1.232                     | 16.17         | 1.829                     | 23.21         | 1.389                       | 21.81         | 1.252                     | 19.26         |
| 6                | 1.389                     | 14.26         | 1.782                     | 24.36         | 1.287                       | 22.13         | 1.268                     | 20.54         |
| 7                | 1.178                     | 15.19         | 1.688                     | 24.14         | 1.408                       | 22.54         | 1.345                     | 20.31         |
| 8                | 0.975                     | 15.79         | 1.775                     | 24.58         | 1.522                       | 21.56         | 1.472                     | 19.36         |
| 9                | 1.254                     | 14.87         | 1.473                     | 22.54         | 1.357                       | 23.75         | 1.292                     | 19.93         |
| 10               | 1.225                     | 14.17         | 1.532                     | 23.40         | 1.232                       | 22.22         | 1.358                     | 21.49         |

Correlation coefficient (P, 0.689 (NS))
- Correlation coefficient (P, 0.689 (NS))
- Correlation coefficient (P, 0.698 (NS))
- Correlation coefficient (P, 0.58 (NS))
- Correlation coefficient (P, 0.360 (NS))

NS: Not significant

Table 2 intergroup comparison of mean bond strength was done using one-way ANOVA test and the differences in mean bond strength were found to be statistically significant. Post hoc pairwise comparison was done using Tukey’s test and it was found that bond strength among Group 2 was found to be the highest and it was significantly more than that among Group 3, which was further significantly more than that among Group 4, which was further significantly higher than that among Gr 1.

**DISCUSSION**

Fiber posts are producing a favorable stress distribution and providing more esthetic outcomes for endodontically treated anterior teeth.[5] The pull-out test shows a better stress distribution and is capable of accurately measuring bond strength between fiber posts and root dentin. The pull-out test is mainly used to test the retentive ability of endodontic posts.[3] In the pull-out test, stresses are far more homogeneous across the interface than in shear and, therefore, maximum principal stress values are much closer to the nominal strength.

Silane coupling agents are hybrids of organic–inorganic compounds that arbitrate adhesion between organic and inorganic matrices by an intrinsic dual reactivity. Silanes form a large group of organic compounds that essentially contain a silicon (Si) atom or atoms. Silane resemble ortho esters, and they can be bifunctional, i.e., they have a dual reactivity. The organic functional part (e.g., vinyl–CH=CH2, allyl–CH2CH=CH2, amino–NH, isocyanato–N=C=O) can polymerize with an organic matrix. The alkoxy groups (e.g., methoxy–O–CH, ethoxy–O–CH2CH) can react with an inorganic substrate, in both cases forming covalent bonds between the matrices. GFPs are silanized to improve the adhesion between the epoxy resin matrix of fiber post and methacrylate-based resin cement. Silane application would promote adhesion by increasing the post surface wettability, as well as by chemically bridging methacrylate groups of the resin and hydroxyl groups of glass fibers.[4]
About 99% of all glass fibers are made from E-glass. Electrical glass (E-glass) is resistant to chemical attacks and has special electric properties. Pretreatment of E-glass fibers by monofunctional silane, γ-methacryloxypropyl trimethoxysilane leads to a chemical bond. The main contribution to the values obtained was made not by the solely mechanical interlocking of the resin composite, but also by the formation of covalent siloxane bonds through silane treatment.

Surface roughness of GFP is increased with pretreatment such as air abrasion, and etching with hydrofluoric acid which helps increase micromechanical retention. The highest mean roughness value was observed in Group II, as well as the highest bond strength value is in the same group. There is a correlation between surface roughness and bond strength. Increasing the surface roughness increases the bond strength.

In Group I, only a silane coupling agent was applied to the GFP, which showed the lowest mean surface roughness value and also showed the lowest mean value of bond strength. This result is in accordance with a similar study done by Singh et al.

The particle size is considered to be a factor contributing to the volume loss and the surface distortion of the fiber post. Magni et al. demonstrated that air abrasion with 30 µm silicon oxide did not affect the flexural strength of fiber posts and it may be questioned whether the use of particles larger than 110 µm could weaken the post and impair its mechanical properties. So for this study, we choose 30 µm silicon oxide particles for air abrasion. Group II showed the highest mean surface roughness value and also showed the highest mean value of bond strength. This result is in accordance with a study done by D’Arcangelo et al.

In Group III, 9% hydrofluoric acid was applied, followed by silane coupling agent application on GFP. Hydrofluoric acid is intended to create a rough pattern on the surface, which allows for micromechanical interlocking with the resin cement and GFP. In this study, the mean bond strength value of Group III is 22.492 Mpa [Table 2]. This result is similar to a study done by D’Arcangelo et al.

In Group IV, a silane coupling agent was used, followed by the application of a universal bonding agent (G-Premio BOND) on a GFP. These universal adhesives differ from the current self-etching system by incorporating monomers capable of producing a chemical bond with the dental tissue. The results showed that silane coupling agent application with the use of a universal bonding agent as surface treatment could influence the bond strength of GFP to root dentin. This result may be because of its unique combination of three functional monomers (4-META, MDP, and MDTP), notably excluding HEMA, which ensures excellent stability and increased bond strength of GFP to root dentin. It contains a high amount of nano-sized cross-linking silica fillers and photoinitiators which help to obtain a strong bonding layer. Dalitz et al. also found the silane layer before the universal adhesive application on GFP seems to have improved bond strength.

In the present study, the mean surface roughness value of Group I is significantly lower than Group II, Group III, and Group IV. Surface pretreatment significantly enhanced the surface roughness. The mean value of bond strength of Group I is significantly lower than all the experimental groups. These results are due to the efficacy of the air abrasion with silica particles and HF treatment in the fiber post surfaces. The partial removal of the resin matrix from GFP due to sandblasting in conjunction with silica particles and hydrofluoric acid treatment increased the number of exposed glass fibers and consequently the surface area available for reacting with the silane, allowing for higher bond strengths than untreated posts. Micromechanical retention produced by airborne-particle abrasion and chemical bonding resulting from silane coupling agent application. This technique depends on the penetration depth of the silica particles into the glass fiber material. Atsu et al. stated that this type of pretreatment increases the silica content of post surfaces which may facilitate siloxane bond formation with resin cement.

**CONCLUSION**

Within the limitation of the present study, it can be concluded that silicon oxide particles with air abrasion on GFP cause the highest amount of surface roughness and also showed the highest pull-out bond strength. Same way hydrofluoric acid also creates microporosities on GFP thereby increasing surface roughness and increase in pull-out bond strength. Hence, pretreatment of GFP increases the surface roughness of post as well as bond strength of post to root dentin. There is a correlation between surface roughness and bond strength. However, the use of only universal bonding agent also showed comparable pull-out bond strength of GFP, which means only use of universal bonding agent is also a new alternative as pretreatment of GFP and helps in increase in bond strength. The benefit of it reduces the use of special equipment and reduces the number of steps followed during the procedure and saves the clinician’s time.

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**Conflicts of interest**
There are no conflicts of interest.
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