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Original Article

Comparison of Different Surface Treatments on the Push-Out Bond Strength of Glass Fiber Reinforced Posts to Root Dentin

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Abstract

Introduction: Successful post retention within the root canal is a prerequisite in the post endodontic restoration of a structurally compromised tooth. Apart from the formation of surface roughness on the glass fiber post (GFP), the surface treatment strategies should generate a strong chemical bond of the exposed epoxy resin matrix in the post with the root dentin through a resin cement.

Purpose: To compare the influence of different surface treatments on the push-out bond strength (PBS) of GFPs to root dentin and to determine the modes of failure between root dentin, resin luting cement, and the post surface.

Methods: Sixty extracted mandibular premolars were divided into six groups (n = 10). Rotary instrumentation followed by obturation was done with a single cone technique. Sixty GFPs (6 groups; n = 10) were treated with one of the following: No surface treatment, Silane coupling agent, Bonding agent, Sandblasting, 9.6% Hydrofluoric acid, and 100 mL alkaline solution of Potassium permanganate. Treated posts were luted to the prepared post space using self-adhesive resin cement. Coronal, middle, and apical portions obtained after sectioning the roots embedded in resin mold were subjected to a PBS test, and the "peak force" at bond failure was measured. The modes of failure between the interfaces were assessed using Scanning Electron Microscopy. Statistical analysis was performed using the Kruskal–Wallis test and Bonferroni multiple comparison test.

Results: Group III (Bonding) and Group IV (Sandblasting) showed the highest bond strength in the coronal and apical sections.

Conclusion: The surface treatment of GFPs using either a universal adhesive or sandblasting was reliable. PBS for coronal and apical root sections was superior to the middle sections. A mixed mode of failure was predominant among tested specimens.

Key words: Glass fiber post, Push-out bond strength, Sandblasting, Surface treatment, Tribochemical silica coating

INTRODUCTION

Post and cores rehabilitate root canal-treated teeth with excessive coronal tooth loss. Posts provide adequate retention for the core but will not strengthen the root.[1] Various *in-vitro* studies have illustrated the superior qualities of using glass fiber posts (GFPs). The prevalence of unfavorable root fractures using fiber posts has been drastically reduced compared to prefabricated conventional cast posts.[2] This is because forces acting on the structure but absorbed by the tooth are not distributed to the underlying root, the fiber post, and the core. Properties of esthetic fiber posts, such as their elastic modulus resembling that of root dentin, and the adhesive technique for post-cementation are added advantages.[3,4] The type of luting cement and the bonding strategy used for cementing the post within the dentin is one of the main factors deciding the successful post retention. The multistep process of luting fiber post through total-etch or self-etch adhesive systems and a low-viscosity resin is complex and hence technique sensitive.

Recently, 10-methacryloyloxydecyl dihydrogen phosphate (10 MDP), a dual adhesive monomer-based self-adhesive resin cement, was introduced to eliminate the requirement for pretreatment of either the tooth or on the surface of the

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posts. It owes its adhesive property to the acidic monomers, which can infiltrate and demineralize the tooth substance, providing micromechanical retention.[4] The basic adhesion potential of a material will be improved by following different surface treatment regimens. The three main categories of surface treatments include Rough surface promotion, Chemical adhesive optimization, and a mixture of the above two methods.[5] An increased micromechanical bonding results from either mechanical (sandblasting) or chemical treatments (etching with hydrogen peroxides or hydrofluoric acid [HF]), which improves the penetration of the adhesive on the surface of the post. The approach aims to increase surface roughness and optimize the chemical bonding to fiber posts.[6]

Silanization of the post before cementation refines the bond strength of GFPs with resin cement, as observed in the previous studies. In contrast, others reported that using a silanizing agent alone showed no increase in bond strength; thus, controversy exists regarding the effectiveness of silanization.[3,7,8] Further studies are needed to prove its potential for improving the GFPs to resin cement interfacial adhesion. Therefore, the current study compared the influence of a variety of surface treatments on the push-out bond strength (PBS) of GFPs to root dentin and determined the modes of failure between the root dentin, resin luting cement, and the surface of the GFP.

MATERIALS AND METHODS

The sample collection and specimen preparation were done at the department of Nanosciences, Amrita Institute of Medical Sciences and Research Centre, and the Department of Conservative Dentistry and Endodontics, Amrita School of Dentistry, Edappally, Ernakulam. The study was conducted with the approval of the institutional ethical committee (IRB-AIMS-2019-308). A single investigator performed the laboratory procedures to avoid bias.

The sample size calculation was performed from the Department of Biostatistics, AIMS, Kochi. Sixty single-rooted mandibular premolars (extracted for periodontal or orthodontic reasons) were divided into ten teeth each (n = 10) in all the six groups tested in the study [Figure 1a].

Preparation of Samples

An ultrasonic scaler was used to clean the external debris on the specimens initially stored in distilled water at room temperature. Diamond bur at slow speed with enough water cooling was used to decoronate each tooth till the cementoenamel junction to standardize root canal length to 18 mm. Rotary instrumentation of the roots was performed using Protaper universal system till file size F2 (Dentsply, Maillefer, Switzerland) was 1mm short of apex. After the change of each drill during canal shaping, the canal was irrigated with 1 mL of 5.25% solution of sodium hypochlorite. After a final rinse using distilled water, paper points were used to dry the canal, followed by obturation with Protaper F2 single cone gutta-percha and AH plus sealer.

Post-Space Preparation, Surface Treatment of the Post, and Luting Procedures

Post-space preparation was done using peeso reamers sequentially from #1 to #3, sparing 4mm gutta percha at the root apex.[9] For canal irrigation, 5.25% NaOCl and 17% EDTA were used initially, followed by a final rinse using distilled water. The root canals were then dried using paper points. The total number of posts was distributed equally into six groups (n = 10). These posts were then surface treated using one of the respective methods [Table 1].

Specimen Preparation and Push-Out Test

A 10-MDP containing self-adhesive resin cement (SpeedCEM Plus, IvoclarVivadent, US) was used for luting the surface-treated GFPs into the prepared post space, as stated by the manufacturer. After 1 week of storage at room temperature under humid conditions, the specimens were mounted on an acrylic resin mold. These were then sectioned perpendicular to the long axis with Isomet (Isomet, Buehler, USA) under sufficient water cooling. After sectioning, one section each from the apical, middle, and coronal thirds (0.06–1 mm thickness) was obtained [Figure 1b]. The PBS of the cut

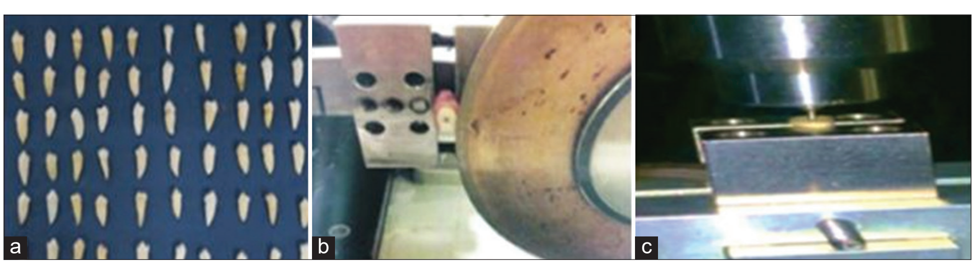

Figure 1: (a) Mandibular premolars selected for the study. (b) Specimens sectioned using an Isomet device (c) Push out bond strength tested on a universal testing machine

Table 1: Post surface treatments

Group I	No surface treatment was performed on post surface
Group II	Post treated with a silane coupling agent (Ultradent, 505 West Ultradent Drive, South Jordan) for 60 s using a disposable brush and then dried
Group III	Post surface treated using a self-etching bonding agent for 20 s (Futura Bond DC, Dual curing self-etching bond, Cuxhaven, Germany)
Group IV	Post surface treated using 50 µm silica coated alumina particles using Cojet system (3M ESPE, Seefeld, Germany) at 2–3 bar for 15 s from 10mm distance
Group V	Post surface were treated with 9.6% HF (Ultradent, 505 West Ultradent Drive, South Jordan) for 15 s
Group VI	Post surface were treated with 100 mL alkaline solution of $KMnO_4$ (Dept. of Biochemistry, AIMS, Kochi) for 10 min

HF: Hydrofluoric acid, $KMnO_4$: Potassium permanganate

sections was tested using a Universal testing machine (Shimadzu Corporation AutoGraph AGS-X Series, Kyoto, Japan) at a crosshead speed of 1 mm/min [Figure 1c]. The peak force at which the post segment extrudes from the test specimen was recorded in Newtons (N) and considered the point of bond failure. This value was then changed to Mpa. The fractured specimen from each group was submitted for scanning electron microscopic (SEM) analysis to evaluate the various failure modes as either adhesive (resin cement-dentin or resin cement-post interface), cohesive (either within resin cement, dentin, or post surface), or mixed (between resin cement, dentin, and post surface). The specimens were fixed on aluminum stubs, sputter coated with gold, and observed under SEM (X25).

Statistical analysis was performed using SPSS Version 21.0 (SPSS, Chicago, IL, USA). The statistical significance of the difference in averages among the six groups with various surface treatments on the GFPs was tested using the Kruskal-Wallis test. Bonferroni's multiple comparison tests were performed to test the statistically significant pairs or groups. The Kruskal-Wallis test was applied to test the statistically significant pairs or groups ($P < 0.05$).

RESULTS

The mean and standard deviation of PBS at the coronal, middle, and apical sections in each of the six groups is given in Table 2. For the coronal sections of the respective groups, a significant difference in bond strength was noted (P = 0.001). The highest mean PBS was observed for the application of bonding agent (Group III [42 ± 13.3]) followed by Sandblasting (Group IV [40.10 ± 27.7]) and HF (Group V [40.3 ± 31.1]). There seems to be no statistically significant difference between the three groups. The control group (Group I [11.40±4.16]) with no surface treatment presented the lowest bond strength.

Table 2: Mean (Mpa) and SD values of push out bond strength using the Kruskal Wallis H test in all the groups at coronal, middle and apical segments

Groups	Apical	Push out bond strength (Mpa)		
		Coronal	Middle	Apical
I	Control	8.81±4.16	14.31±11.39	12.99±16.62
II	Silanization	25.10±42.21	36.70±83.24	11.41±4.11
III	Bonding agent	37.71±33.86	43.19±46.73	38.44±62.39
IV	Sandblasting	38.97±27.79	31.03±21.75	31.24±18.15
V	HF	35.97±31.15	26.84±29.61	29.73±25.63
VI	Potassium permanganate	14.30±3.27	9.70±3.60	14.09±21.82

Data are shown as mean±standard deviation, HF: Hydrofluoric acid, SD: Standard deviations

All the middle root sections presented a statistically significant difference in bond strength ($P = 0.006$), with the highest mean bond strength for the bonding agent group (Group III [44.20 ± 46.7]) and lowest for Potassium permanganate (KMnO4) group (Group VI [18.45 ± 3.60]).

The apical sections showed a statistically significant difference in bond strength between the different groups ($P = 0.001$), with the highest mean values observed for sandblasting Group (Group IV [43.0 ± 18.15]) followed by the bonding group (Group III [40.7 ± 62.3]). In contrast, the lowest value was evident for the potassium permanganate group (Group VI [16.80 ± 21.8]), which had a statistically significant difference compared to other groups except for the Control group (Group I [18.40 ± 16.62]) [Tables 3-5]. Regarding the association between modes of failures and different surface treatments on GFPs, there was no statistical significance for the presence of bond failures in any of the coronal, middle, or apical root sections for the respective surface treatments evaluated [Table 6].

The SEM analysis of all the root sections that underwent the bond strength test revealed that of the three types of bond failures, mixed failures were predominant between the GFPs, root dentin, and resin cement [Figure 2].

DISCUSSION

Among the various methods available, the push-out test is the most suitable method to study the adhesion between posts and root canal dentin.[10] Here, an indenter pushes a small fiber diameter into a 1mm thick specimen, uniformly distributing the applied load throughout the bonded interface.[10,11] Fiber posts are cemented widely using resin-based adhesive cement. The introduction of self-adhesive or all-in-one adhesive cement simplified the luting procedure and eliminated the need for the pretreatment of teeth.[12]

Table 3: Statistical significance of the difference in the averages among six groups calculated using Kruskal Wallis test and intergroup comparison using Bon Ferroni test at coronal third

Groups	Group I	Group II	Group III	Group IV	Group V	Group VI	Mean bond strength (Mpa)	X²	P
Group I	1	−12.35	−30.6*	−28.7*	−28.9*	−14.05	11.40	24.798	0.001*
Group II	12.35	1	−18.25	−16.35	−16.55	−1.7	23.75		
Group III	30.6*	18.25	1	−1.90	−1.7	−16.55	42.00		
Group IV	28.7*	16.35	1.90	1	−0.20	−14.65	40.10		
Group V	28.9*	16.55	1.7	0.20	1	−14.85	40.30		
Group VI	14.05	1.7	16.55	14.65	14.85	1	25.45		

Kruskal Wallis test, *: $P<0.05$, Bon Ferroni *post-hoc*, *: $P<0.05$

Table 4: Statistical significance of the difference in the averages among six groups calculated using Kruskal Wallis test and intergroup comparison using Bon Ferroni test at middle third

Groups	Group I	Group II	Group III	Group IV	Group V	Group VI	Mean bond strength (Mpa)	X²	P
Group I	1	−0.70	−20.60	−15.70	−9.55	−5.15	23.60	16.56	0.006*
Group II	0.70	1	−19.90	15.00	−8.85	−5.85	24.30		
Group III	20.60	19.90	1	−4.90	−11.05	−25.75*	44.20		
Group IV	15.70	15.00	4.90	1	−6.15	−20.85	39.30		
Group V	9.55	8.85	11.5	6.15	1	−14.7	33.15		
Group VI	5.15	5.85	25.75*	20.85	14.7	1	18.45		

Kruskal Wallis test, *: $P<0.05$, Bon Ferroni *post-hoc*, *: $P<0.05$

Table 5: Statistical significance of the difference in the averages among six groups calculated using Kruskal Wallis test and intergroup comparison using Bon Ferroni test at apical third

Groups	Group I	Group II	Group III	Group IV	Group V	Group VI	Mean bond strength (Mpa)	X²	P
Group I	1	−6.80	−22.30	−24.60*	−20.50	−1.60	18.40	22.72	0.001*
Group II	6.80	1	−15.50	−17.80	−13.70	−8.40	25.20		
Group III	22.30	15.50	1	−2.30	−1.80	−23.90*	40.70		
Group IV	24.60*	17.80	2.30	1	−4.10	−26.20*	43.00		
Group V	20.50	13.70	1.80	4.10	1	−22.10	38.90		
Group VI	1.60	8.40	23.90*	26.20*	22.10	1	16.80		

Kruskal Wallis test, *: $P<0.05$, Bon Ferroni *post-hoc*, *$P<0.05$

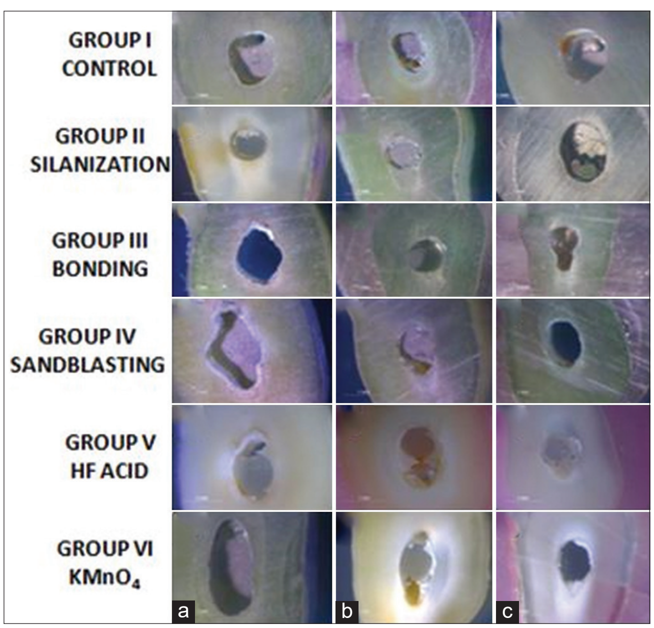

Figure 2: Stereomicroscopic analysis of (a) Coronal (b) Middle and (c) Apical third from all six group

The present study infers that the mode of surface treatment can influence the bond strength of fiber post-system to dentin. From the results of the current study, it was observed that compared to the middle root sections, PBS for the coronal and apical root sections was much higher in most of the specimens.[13,14]

Among the different surface treatments, the highest bond strength was presented by the bonding agent group (Group III), followed by sandblasting (group IV) in the coronal and apical root sections compared to all other groups, as stated by Spicciarelli *et al.*[15,16] Using a dual-cure bonding agent over the epoxy resin-based fiber post surface enhanced the adhesion between the dual-cure resin composite and the posts, as stated by Aksornmuang *et al.*[17] However, contradictory to the above findings, Balbosh and Kern in 2006 and Radovic *et al.* in 2007 reported reduced bond strength values.[18,19]

Tribochemistry relates to forming chemical bonds by applying kinetic energy in the form of sandblasting without

Table 6: Statistical significance of the association between modes of failures and different root sections of all six groups measured using Kruskal Wallis test

Group	*n* (Sample)	Mean rank	X²	*P*
Coronal	10	99.75	3.12	0.21
Middle	10	88.42		
Apical	10	83.33		

Kruskal Wallis test *: $P<0.05$

any application of additional heat or light.[20] Tribochemical silica is those silica particles coated with aluminum particles. The CoJet system employed in the present study uses airborne micro-blasting sand composed of silica-modified aluminum trioxide. This modifies the GFPs with a reactive silica-rich outer surface liable for silanization.[21] The five criteria in airborne particle abrasion using tribochemical silica are the angle to the substrate surface, the distance of the nozzle from the substrate surface, impact, working time, coverage area, an air pressure.[22] Spraying with high velocity over the substrate helps penetrate these particles to a depth of about 15 microns with increased surface area improving resin cement to fiber post-bond strength.[23,24] Furthermore, the bond strength between GFPs and resin cement improved with sandblasting compared to HA or phosphoric acid etching.[25] Despite obtaining sufficient bond strength with this method, this type of pretreatment may possess the risk of modifying the shape and fit of the posts in the root canals and hence is considered a more aggressive option.[26]

9.6 % HF (group V) treatment of posts showed better bond strength results than treatment with a 1.03% alkaline solution of potassium permanganate. This finding may be attributed to the mechanism of action of HF acid that dissolves the epoxy resin matrix, exposing the fibers and creating micro spaces between them. But the efficiency of HF acid is considered a dispute. HF acid application may be aggressive for post fibers causing dissolution of the resin matrix, even though the bond strength received with it is satisfactory.[18]

Lower bond strength was noted in all sections except for the middle sections with the silanizing agent (Group II) application, as supported by Gencoglu *et al*. and Wrbas *et al*.'s findings.[27] The silanizing agent acts through chemical optimization of the post surface, thus linking the inorganic phase of the GFPs to the organic matrix of the adhesive system or resin cement owing to its bifunctional properties.[19] No change in adhesion between fiber posts and dentin was observed, except between the post surface and composite core build-up when a silane coupling agent was used.[28] Yet, in other studies, silane treatment shows unassured results.[25,26,29,30] The fiber post composition comprising of an epoxy resin matrix, inorganic particles, and fiberglass particles, preventing an intimate interaction between the dental elements from adhesive systems and fiber posts, may have caused this insufficiency.[26]

Potassium permanganate ($KMnO_4$) treatment was conventionally used for industrial procedures, and the same is applied for the pretreatment of fiber posts. This involves a sequential treatment using three different chemicals (swelling, etching, and neutralizing) on the GFP surface. This improves the surface area available for bonding due to the partial removal of the epoxy resin matrix, thus creating micro-retentive spaces. This epoxy resin matrix modification is ensured by the manganese oxide present in the composition. $KMnO_4$ chemical treatment generally gave noteworthy results.[31] However, the present study showed drastically reduced PBS compared to all other test groups except for the control group. According to Belwalkar *et al*., the interfacial adhesion between GFP, resin cement, and dentin is influenced by the type and method of application of resin luting cement, adhesive strategy, quality, quantity, and surface treatments of root dentin.[32]

SEM studies regarding root sections of all the teeth revealed three main types of bond failures,namely;(1) Adhesive failure either at the post/resin cement interface or the resin cement/root dentin interface(2) Cohesive failure occurring within the resin cement and (3) Mixed mode of failure (Both cohesive and adhesive failures exists).[33] Among these, mixed types of bond failure predominated over cohesive or adhesive types. This indicated a good interaction of the resin cement with the post and root dentin after surface treatments.

However, the present study failed to conduct a thermo-mechanical aging of the specimen. Therefore, the obtained results cannot be considered a complete clinical simulation. The study also lacks an evaluation of the effectiveness of newer bonding agents on the PBS of GFPs to root dentin.

CONCLUSION

The surface treatment of GFPs using either universal adhesive application or sandblasting with tribochemical silica showed promising results. It was also found that the PBS for coronal and apical root sections was superior to those of the middle sections in the bonding agent group. A mixed mode of failure was found to be predominant when compared to adhesive and cohesive among tested specimens.

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