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ORIGINAL ARTICLE

Effect of polydopamine surface pre-treatment on push-out bond strength of customised short fiber reinforced post

ABSTRACT

Aim: *To evaluate and compare the effect of silane and polydopamine functionalized surface treatment on the push out bond strength of customised fiber reinforced post system cemented to root dentin.*
Methodology: *Thirty single rooted premolar teeth were endodontically treated followed by post -space preparation. Fiber reinforced posts systems (EverStick-POST Stick, ESP) were randomly assigned (n=10) for surface treatment with Polydopamine (group 1), silane (group 2) and control (no treatment) (group 3), following which posts were cemented using resin cement. Following 24 hours, each sample was subdivided into four slices (2 mm thick) to determine the push out bond strength in the coronal and middle thirds and expressed in megapascals (MPa). Data was statistically analyzed using Post hoc Tukey HSD and Student t tests using one-way ANOVA (p=0.05). Failure modes were investigated using a stereomicroscope. Surface treated posts from each group were also analysed under SEM.*
Results: *Polydopamine surface treatment showed significantly higher push out bond strength than silane and control groups in the middle third (p<0.05) with more of mixed type of failures in both the experimental groups. SEM images revealed good homogenization and surface deposition of polydopamine molecules on the post surface.*
Conclusion: *Polydopamine surface treatment showed positive effect than silane on the adhesion of customised fiber reinforced post to root dentin.*

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Received 2022, June 12

Accepted 2022, October 16

KEYWORDS *E-glass fiber post, Polydopamine, Silane-coupling agent, Surface pre-treatment*

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Peer review under responsibility of Società Italiana di Endodonzia
10.32067GIE.2023.37.01.03

Introduction

Restoration of endodontically treated teeth with extensive tooth loss requires the placement of intra-canal post to rehabilitate the lost structure. This helps to preserve the structural integrity of the crown and root to increase the durability of the tooth. The placement of post aids in sufficient build-up to retain the core and to reinforce the roots by resisting occlusal loads (1). Over the decades, fiber reinforced posts (FRP) have become the material of choice, due to their modulus of elasticity that is almost equivalent to dentin. This enables better resistance to catastrophic failures under extensive fracture loads. Moreover, they provide esthetic life-like appearance and negligible corrosion (2). The durability of FRP lies in a strong bond between the residual dentin/ core material and post, enabling the interface to dissipate stresses under functional loading. Debonding of post system is the most common failure attributing to ineffective polymerization, weak cementation interfaces and inadequate post-canal fit (3). In order to increase the bond strength and durability, various surface pre-treatment strategies of FRP have been proposed in literature ranging from silane-coupling agents, airborne particle abrasion, hydrofluoric acid, laser, hydrogen peroxide that have proven to significantly increase their retention and bonding (4). The most common surface treatment method is silanization which helps in enhancing chemical bond at the interface. Silane coupling agents (SCA) contain methacryloxyproyl trimethoxysilane that possesses catenation property and vacant orbitals which make it reactive. However, SCA produces a non-homogenous layer which results in a weak chemical bond (5).Various studies have evaluated the effect of silane on bond strengths of fiber posts and proved that SCA treatment alone could not prevent dislocation of FRC post. Thus, debonding of FRP remains the main challenge that needs to be focussed (6-8).

Also, several studies investigated the influence of voids within the cement-post interface on the bond strength of FRP (9-11) Micro-CT analysis on post-cement interface restored either with oval or circular showed that the volume of cement was greater with oval posts compared to circular posts (12). This leads to more volume of cement at the interface that contributes to increased solubility and subsequent microleakage. In such cases, use of a customised FRP will result in conservation of tooth structure and the use of minimal cements. Individually formed posts using strips of polyethylene or glass fibres as an alternative to prefabricated fiber posts provided higher fracture resistance than preformed FRP in oval-shaped canals (13). EverStick-POST Stick (GC, Europe, NV (ESP)) is one such post system which is more pliable, mouldable and flexible post comprising of continuous and unidirectional embedded E glass fibers within a polymer matrix that can be anatomically customised. The matrix contains linear phase polymers, polymethacrylathe (PMMA), and a cross-linked polymer 2,2-bis[(4-2-hydroxy-3-methacryloloxy-propoxy) phenylpropane] (poly bis-GMA). These fibers are manufactured using IPN (Interpenetrating Polymer Network) technology which facilitates penetration of resin cement into the post thereby increasing the bonding and a well-sealed resin cement-dowel interface (14). Anna et al showed no post-cement failures with the ESP suggesting better interfacial adhesion of cement to these posts (15). However, several studies also mentioned the need for various other surface treatment methods for ESP, as surface treatment with hydrofluoric acid or sandblasting swith aluminium oxide particles were found to be ineffective for improving the bonding of resin core materials to ESP (16-18).

Recently, polydopamine pre-treatment of conventional FRP has proven a novel strategy for surface functionalization. PDA is a mussel-derived surface protein which can be easily deposited on all types of organic and inorganic substrates with uniform film thickness and durability. In addition, it possesses various functional groups such as catechol, amine and imine which make it suitable for promoting adhesion on various surfaces (19). PDA exhibits hydrophillicity and biocompatibility onto coated materials such as polyethylene, silicone rubber and glass particles (20). PDA forms an ultrathin active layer and chemical structure that can support the matrix surface and cause a significant rise in the bond strength (21). Kanyılmaz et al have evaluated PDA functionalization significantly increased the adhesion strength of fiber posts (22). However there is no current literature evidence on the performance of customisable FRP on PDA surface treatment. Hence the aim of this study was to evaluate and compare the effect of silane and PDA functionalized surface treatments on the push out bond strength of ESP cemented to root dentin. The null hypothesis was PDA does not have any effect on the push out bond strength of EFP system.

Materials and Methods

Specimen preparation

Freshly extracted, thirty, single rooted mandibular premolars without cracks, caries, restoration or root-filling were selected for this study. The teeth were cleaned using ultrasonic scalars to remove the calculus and decoronated at the level of cementoenamel junction (CEJ) using a low speed diamond disc under water cooling, to obtain 14mm long roots. The working length was determined using a size 10-K file 1 mm short of the apical foramen. The roots were then embedded in self-cured acrylic resin. Root canals were prepared using ProTaper Gold rotary files in crown-down manner by a single operator and irrigated with 5mL of saline and 5 mL of sodium hypochlorite. Final irrigation was done using 17% EDTA solution and dried using absorbent paper points. All thirty teeth were sectionally obturated till 4mm of apical third of root and post-space preparation was done using Peeso-reamer no. 3 to a depth of 9 mm. The most suitable size of Everstick post was selected according to canal morphology. The depth of the prepared canal was measured using a periodontal probe and the post was cut accordingly using sharp scissors. The end of the post was cut obliquely in order to make the post tapered and fit into the canal. The post was adapted to the canal and light cured for 20 seconds. Post was then removed and light-cured for 40 sec and a layer of enamel resin was applied to the post surface to activate the IPN feature. These posts were kept in a dark container temporarily in order to protect them from light.

Preparation of PDA

PDA was prepared according to the protocol given by Chen et al (23) 0.08 gm of dopamine hydrochloride and 0.04 gm of tris-buffer (oxidizing agent) were measured using an electronic balance and added to a beaker containing 40 mL of distilled water. The solution was subjected to sonication using a magnetic stirrer for 10 min and kept aside for use.

Post surface treatment

The samples were randomly divided into three groups as per the surface treatment protocol (n=10).

Group 1 – PDA. The posts were immersed into this PDA solution for 14 hours for functionalization. After this procedure, the posts were removed and dried.

Group 2 – Silanization. The surface of the posts were coated with silane-coupling agent (Monobond N, IvoclarVivadent, Liechtenstein) for 5 min. and then air-dried.

Group 3 – Control group did not receive any surface treatment.

Two post of each group was prepared separately and subjected to Field emission-scanning electron microscopy (FE-SEM) (Zeiss Leo 440 QEMSCAN SEM, Carl Zeiss AG, Germany) analysis after surface pre-treatment to evaluate the surface morphological changes.

Post cementation

The adhesive strategy recommended by the manufacturer was followed. Briefly, self-etch adhesive (Variolink N, Ivoclar Vivadent, Schaan, Liechtenstein) was applied and left for 10 seconds and dried. The post surfaces and the root canal were coated with resin cement (Variolink dual cure, Ivoclar-Vivadent, Schaan, Liechtenstein) that was mixed according to the man ufacturer's instructions. The posts were gently seated into the post spaces with finger pressure. The excess cement was removed and the samples were then cured from the coronal side for 20s with a light curing device (Bluephase G2, Ivoclar-Vivadent, Schaan Liechtenstein) using 1.200 mW/cm^2 power.

Evaluation of Push-out bond strength [PBS]
The roots were sectioned horizontally using a diamond disc to obtain dentin post assembly slices of 2 mm thickness, obtaining total of 20 slices from coronal and middle third for each group. Each of these slices was transferred to a specially designed metal apparatus with 2 mm diameter holes. PBS was evaluated in Instron Testing Machine using a punch pin of 1mm diameter, with a load of 450 N at a crosshead speed of 0.5mm/s until failure was used. The values of maximum force applied were obtained in Newtons (N) and the PBS value of coronal and middle slices were calculated using the formula:

$$\text{Push out bond strength} = \frac{\text{Maximum force}}{\text{Area } (2\pi rh)}$$

where
r means radius of the dentin slices
h height of the dentin slice

The mean bond strength value of the 20 slices each for the coronal and middle third was calculated as megapascals (MPa). Statistical analysis of the PBS data was performed using SPSS statistics software. Normal distribution of data was confirmed using Shapiro Wilk's test. The data were statistically analyzed using Post hoc Tukey HSD and Student t tests using one-way ANOVA. The significance level was set at 0.05 (P=0.05).

Analysis of failure mode
All the failed specimens were analyzed using a stereomicroscope (Leica M205, Leica-Microsystems, Germany) at 25x magnification to find out the type of failure. Adhesive, cohesive and mixed types of failure were categorized for all the specimens and percentage of each failure pertaining to each group was calculated. The interfaces at the root surface were also visualized under SEM (Zeiss Leo 440 QEMSCAN SEM, Carl Zeiss AG, Germany) for evaluation of surface topography of dentinal wall and resin penetration.

Results

PBS Evaluation
Group 1 showed significantly higher mean PBS as compared to groups 2 and 3 in the middle third of the root. However, mean PBS values obtained at coronal third did not show much significant difference between them. On intragroup comparison, coronal third showed significantly higher mean bond strength values than those of the middle third for all the groups ($p<0.05$).

Table 1
Mean comparison of push-out bond strength values following surface treatment of EFP in coronal third

Group	N	Mean	Std Deviation	95% Confidence Interval for Mean		Minimum	Maximum
				Lower Bound	Upper Bound		
Pda coronal	20	5.64	0.59	5.22	6.07	4.91	6.52
Silane coronal	20	5.04	1.00	4.33	5.76	3.98	7.03
Control coronal	20	3.94	0.61	3.51	4.38	2.93	4.55

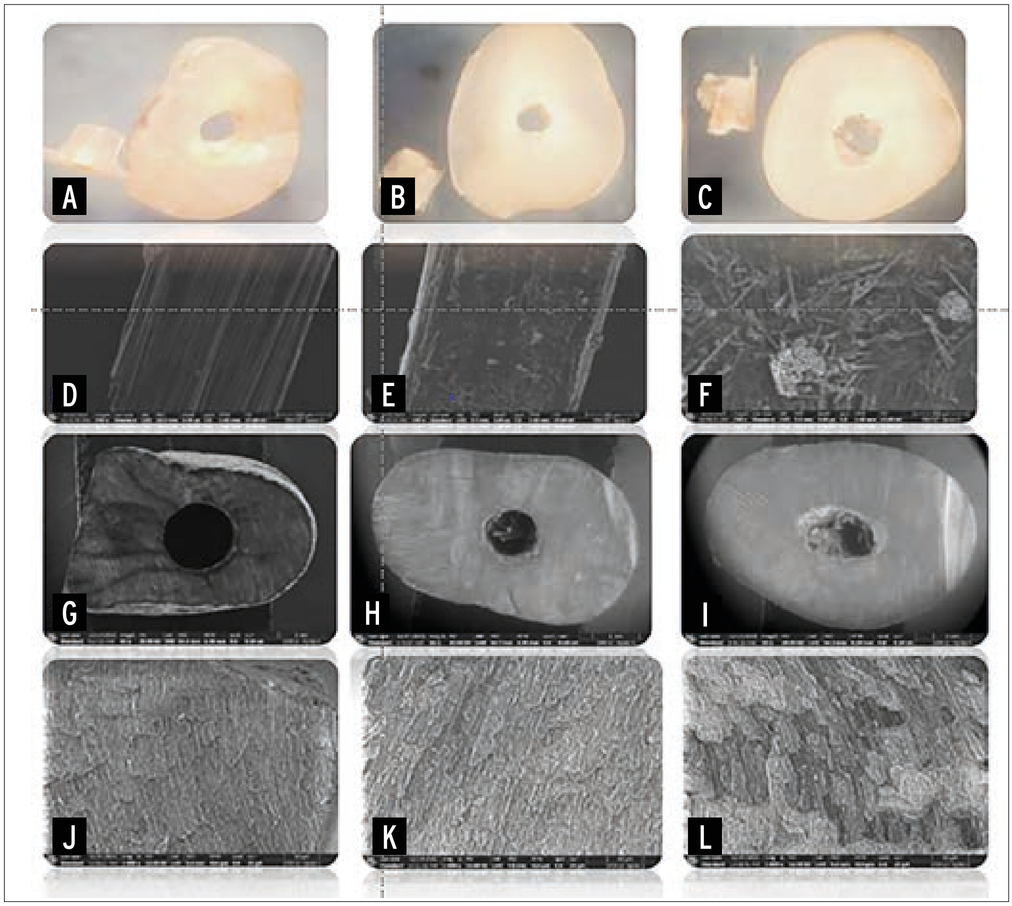

Figure 1
(A-C) Stereomicroscopic images of EFP samples after push-out bond strength testing under stereomicroscope (50x). SEM images of EFP surfaces after different surface treatments (250x); **(D-F)** SEM images of EFP surfaces after different surface treatments (250x); **(D)** control sample showing no surface alteration on EFP surface; **(E)** SCA sample shows non-homogenous deposition on EFP surface; **(F)** PDA sample shows a rough surface with random orientation of E-glass fibers increasing micro-retention. **(G-I)** Representative EFP samples after push-out bond strength tests (50x). SEM images confirm a mixed type of failure in PDA group with remnants of resin cement, dentin and EFP. **(J-L)** Cement-dentin inter-diffusion zone of various samples (1000x). **(J)** The smoother surface of control post, **(K)** well-defined hybrid layer or formation of resin tags was not discernible in SCA treated EFP. **(L)** PDA treated EFP surfaces showed long and continuous resin tags.

Stereomicroscopic analysis (Fig 1A-C) revealed that the numbers of dentin-cement interface failure were significantly more of mixed type in both the groups. There was no significant difference in the number of adhesive failure between silane and PDA treated groups.

Sem analysis of surface treated post
SEM image of untreated post (Fig 1D) shows revealed smooth surface without any irregularities. However, SCA and PDA surface treated posts (Fig 1E, F) showed a rough surface, creating spaces for micromechanical retention. Comparatively PDA treated EFP resulted in relatively higher irregularities.
As shown in Fig. 1K-L, SEM images of SCA and PDA functionalized EFP samples showed cement-dentin inter-diffusion zone with long and continuous tags. PDA treated samples showed more homogenously functionalized surface creating spaces for micromechanical retention with residual adhesive cement depicting superior bonding ability. However, adhesive interface of SCA treated samples showed smooth and regular orientation of resin tags.

Table 2
Mean comparison of push-out bond strength values following surface treatment of EFP in middle third

Group	N	Mean	Std Deviation	95% Confidence Interval for Mean		Minimum	Maximum
				Lower Bound	Upper Bound		
Pda middle	20	4.44	0.66	3.97	4.91	3.48	5.47
Silane middle	20	2.75	0.39	2.47	3.03	1.99	3.16
Control middle	20	1.82	0.44	1.50	2.13	1.05	2.39

Table 3
Failure patterns of tested specimens (%)

	Cohesive (%)	Adhesive (%)	Mixed (%)
Pda	10	40	50
Silane	25	35	40
Control	15	50	35

Discussion

The results of the present study shows that surface treatment of posts either with SCA or PDA had enhanced the PBS of post/dentin interface significantly compared to the control group. Moreover PDA showed significantly higher PBS than other experimental groups thus rejecting the null hypothesis. EPS with 48% of its volume with fiber in its composition has different cross section, which was not always symmetrical and round, as distinct from the other two posts. This was supported by the SEM observation of the present investigation. The pre-treated post removes the resin matrix from the surface of the posts, resulting in an enhancement of the surface roughness and in a greater exposure of the fibres when compared to comparison to the untreated post. This attributes the lower PBS seen in control group.
Despite an abundance of literature evidence on the various pre-treatment strategies, no surface treatment agent has proven to show effective adhesion (1). SCA is one of the most common pre-treatment strategies and it is used to cement indirect ceramic and metallic crown, ceramic laminates and repair E-glass fiber reinforced resin composites, and filler reinforced resin composites (24). Prado et al reported greater performance of SCA compared to 24% H_2O_2 pre-treatment on fiber posts surfaces based on push-out bond strength (25). A systematic review of 178 studies concluded that SCA with post surface pre-treatment alone cannot improve the retention and clinical survival of fiber posts (26). Two studies reported that the silane treatment of fiber posts did not prevent dislocation (27,28). Therefore, SCA alone is insufficient for attaching to non-silica based materials.
When considering the PBS, middle third of the root was significantly lesser than the coronal third. This can be attributed to the less degree of conversion which leads to incomplete polymerization due to lesser accessibility of the curing light (29). Also, the reduced dentinal surface area for adhesion compared to coronal third accounts for lesser PBS. Moreover, SCA have proven to favour methacrylate based systems rather than epoxy based systems (30). Gradually overtime the volatility of the solvent in SCA could also influence the bond performance of the same. Furthermore, the key step in the action of SCA is hydrolysis which is strongly pH dependent (31).
Moreover, the silanization performance is affected by its silane concentration, similarity in their functional groups with the resin monomer functional groups, blending with cross-linking silanes, their pH,

nature of the solvent mixture (24). Also, surface treatment with hydrofluoric acid or sandblasting with aluminium oxide particles were not found to be effective methods for improving the bonding of resin core materials to EPS (1).
PDA is a recently emerging bio-coating material generated from the catechol derivative dopamine which has been devised with the capacity to bind to universal substrates (19). Dopamine oxidation produces a polymer that contains indole and dopamine units in various oxidation states, as well as pyrroles to a lesser amount. The chemical properties of PDA is attributed to the presence of oxidised o-quinone and o-hydroquinone groups in its chemical structure (32,33). In addition, PDA possesses functional groups like imine, amine and thiol which enhance the hydrophilicity and wet adhesion. PDA coatings have been shown to be highly effective and biocompatible adhesives with enhanced action especially in protein bonding. Moreover, it has been used to increase the interfacial adhesion in a range of different fiber-reinforced composites, achieving significant improvements in dry strength and modulus for carbon, glass and polymer fibers. Chen et al. designed a novel PDA pre-treatment approach involving dopamine functionalization to increase the adhesion of glass fiber post surfaces to luting cements. He also demonstrated that hydrogen peroxide and PDA combination of surface treatment of prefabricated FPS resulted in higher bond strength (23). However, this was the first kind of comparative study between PDA and SCA coated EFP system on PBS. The higher PBS contributed by PDA surface treatment compared to the SCA group can thus be attributed to the presence of various functional groups and superior wet-adhesion ability of PDA (34, 35). The stronger adhesive ability with the substrate could be due to the higher the dopamine content and immersion time. Fiber posts treated with dopamine solution for longer time might have resulted in a surface modification of post with various functional groups such as hydroxyl, carboxyl and amino groups. This could have reacted with organic functional monomers improving hydrophillicity and chemical combination attributing to higher bond strength of this group.
On PDA intragroup comparison, middle third and coronal did not show any statistical significant difference on PBS due to the wet adhesion ability, increases in the hydrophillicity of the post surface thus providing a better chemical bond to the resin cement (19, 20). PDA does not destroy the structures or material properties of the substrate at the same time by stabilizing the bond strength. Shari et al showed that PDA incorporated self-etch and total etch adhesives resulted in higher degree of conversion, bond strength and durability due to its biomimetic remineralization and MMP inhibition activity (36). This can be attributed to the effective resin-dentin bond in both the coronal and middle thirds of the root.
On SEM analysis, the most common type of failure observed was mixed. In general, considering the post-cement-dentin interface, mixed failure is considered more favourable when compared to adhesive failure. The relationship between surface treatment and failure modes agrees with previous studies (26, 30).This could be due to various factors such as incompatibility between the adhesive and resin, inadequate mechanical properties of EFP or cementation protocol. PDA group showed more percentage of mixed failure than SCA group. Also, SEM images confirmed the presence of an irregular and rough surface accounting for better micro-mechanical retention thereby enhancing the better bond with resin cement.
PDA functionalization on EFP also causes random orientation of fibers which can provide an isotropic reinforcement effect on post system (37). On the other hand, SCA treated post samples showed comparatively smoother surface, which might be the cause for more adhesive failures. Control group also showed comparatively more mixed failures with much smoother surface on SEM findings. This could be the reason for the debonding failure seen in fiber post system.
The limitations of the study include high-

er immersion time in PDA affecting the clinical simulation, and a mild staining of the PDA treated EFP surface from opaque to light brown compromising aesthetic requirement (32). Hence, further evaluation on the effect of PDA on long term resin dentin bond strength after aging protocol and longevity of EFP system can also be evaluated in future studies . Moreover, it has been demonstrated in various studies that PDA produces bactericidal action against both Gram-positive and Gram-negative bacteria so which antibacterial studies can also be analysed for long term durability of EFP systems (38, 39).

Conclusions

Within the limitations of this study, it can be concluded that PDA surface pre-treatment resulted in significantly higher PBS values than SCA on the middle third of the root. SEM images also revealed good homogenization and surface deposition of PDA on the EFP surface causing random orientation of the E-glass fibers. Hence, PDA can be an effective surface pre-treatment strategy to prevent the debonding of flexible fiber post systems thereby improving the success rate of post endodontic restoration.

Clinical Relevance

The surface treatment with PDA on flexible fiber post systems can prevent debonding of post thereby improving the success rate of post-endodontic restoration.

Conflict of Interest

No potential conflict of interest was reported by the author(s).

Acknowledgments

All the authors have equally contributed to conception, design, data acquisition and interpretation, drafted and critically revised the manuscript. All authors gave their final approval and agree to be accountable for all aspects of the work.
There is no any financial support or relationships that may pose conflict of interest by disclosing any financial arrangements have with a company whose product figures prominently in the submitted manuscript or with a company making a competing product, or any conflict relating to technology or methodology.

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