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ORIGINAL ARTICLE

Effect of surface treatments on the flexural properties and adhesion of glass fiber-reinforced composite post to self-adhesive luting agent and radicular dentin

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Abstract This study evaluated the effect of different surface treatments on the flexural properties and adhesion of glass fiber post to self-adhesive luting agent and radicular dentin. Seventy-five single-rooted human teeth were prepared to receive a glass fiber post (Reblida). The posts were divided into five groups according to the surface treatment: Gr C (control; no treatment), Gr S (silanization for 60 s), Gr AP (airborne-particle abrasion), Gr HF (etching with 9 % hydrofluoric acid for 1 min), and Gr M10 (etching with CH_2Cl_2 for 10 min). Dual-cure self-adhesive luting agent (Rely X Unicem) was applied to each group for testing the adhesion using micropush-out test. Failure types were examined with stereomicroscope and surface morphology of the posts was characterized using a scanning electron microscopy (SEM). Flexural properties of posts were assessed using a three-point bending test. Data were analyzed using ANOVA and Tukey's HSD test. Statistical significance was set at the 0.05 probability level. Groups treated with M10 showed significantly higher bond strength than those obtained with other surface treatments ($P < 0.05$). In general, improvements in bond strength (MPa) were found in the following order: M10 > C > S > AP > HF. Most failure modes were adhesive type of failures between dentin and luting agent (48.2%). SEM analysis revealed that the fiber post surfaces were modified after surface treatments. The surface treatments did not compromise the flexural properties of fiber posts. Application of M10 to the fiber post surfaces enhanced the adhesion to self-adhesive luting agent and radicular dentin.

Keywords Bond strength · Flexural properties · Glass fiber post · Methylene chloride · Self-adhesive

Introduction

In recent years, utilization of fiber-reinforced composite (FRC) root canal posts has increased with increasing the demand for esthetic and metal-free restorations [1, 2]. Tooth-colored FRC posts have several advantages, including improved light transmission throughout the root and gingival tissues [3], eradication of the corrosive reactions that happen with metal alloy prefabricated posts [4], and simple removal if endodontic retreatment is required [5]. The most important characteristic of FRC posts is their elastic modulus, which is closer to that of dentin compared to metal posts [6]. This biomimetic feature can provide the optimal stress distribution which reduces the possibility of root fracture, the most critical form of failure [7].

Reliable adhesion is considered to be a major problem with all types of posts, particularly glass fiber posts, as they are anticipated to be luted adhesively [8, 9]. The most common failure of FRC post restoration is dislodging of the posts from the root canals [10]. Several studies have been directed to improve the retention of the post because of this type of failure, including different pre-treatments of the posts and dentin or use of different luting agents [1, 3, 11–14]. Silanization is considered a reliable method to enhance the adhesion of luting fiber post as it is a fast chair-side procedure. However, it has been reported that the use of silane coupling agent alone [12] or in

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combination with sandblasting [15] did not increase the bond strengths when self-adhesive luting agents were used.

Different surface treatments have been applied for conditioning the post surface, including hydrofluoric acid etching, airborne-particle abrasion, hydrogen peroxide, silanization, and methylene chloride (CH_2Cl_2) with varying degrees of outcomes [15–21]. CH_2Cl_2 has been applied to epoxy resin-based fiber post for 5 s to improve the adhesion between fiber post and composite resin; however, the results showed that this treatment was not effective [21]. In a recent study, it was reported that application of CH_2Cl_2 for 10 min to the fiber post surfaces enhanced the adhesion to resin core materials [18].

Optimal post surface treatments that improve adhesion between fiber-reinforced posts and resin-based luting agents may probably enhance the bond strength [7]. The surface energy characteristics of dental posts, which can be modified using varying techniques, have been reported to influence the bonding of resin-based luting agents [22]. The present study aimed to evaluate the effect of different surface treatments on the morphological aspects and flexural properties of glass fiber post and micropush-out bond strength of fiber post to dual-cure self-adhesive luting agent. The null hypotheses tested were that the application of surface treatments would improve (i) the flexural properties of the post and (ii) the adhesion between the fiber post and the self-adhesive luting agent.

Materials and methods

Grouping of specimens

The specimens were divided into five groups according to the method of surface treatment applied as follows: control, no treatment (Group C); silanization, a silane coupling agent (Ceramic Bond; VOCO, Cuxhaven, Germany) was applied on the surface of the post with a brush and gently air dried for 60 s, according to the manufacturer's instructions (Group S); airborne-particle abrasion, the specimens were abraded with alumina particles (Al_2O_3) (50 μm) (Korox, Bego, Bremen, Germany) with a dental airborne-particle abrasion unit (Micro-Blaster; Daedong Industrial Co., Ltd., Daegu, Korea) at a pressure of 2 bar for 10 s and the distance was maintained at 15 mm between the nozzle tip and the specimen surface (Group AP); etching with hydrofluoric acid (Ultradent Porcelain Etch 9 % Buffered, Ultradent Products, South Jordan, UT, USA) for 1 min (Group HF); and etching with CH_2Cl_2 (Sigma, Aldrich) for 10 min, the specimens were immersed in CH_2Cl_2 for 10 min (Group M10). After the application of HF or CH_2Cl_2, all the posts were rinsed with deionized water for 3 min followed by air drying.

Tooth preparation

A total number of seventy-five extracted single-rooted human teeth (lower premolars, canines, and upper incisors) were selected for this study. The inclusion criteria were absence of caries or root cracks, absence of previous root canal treatments, posts or crowns, and a root length of 16 ± 1 mm. The teeth used in this study were obtained by protocols that were reviewed and approved by the appropriate institutional review board of the Faculty of Medicine and Dentistry, Mansoura University, Mansoura, Egypt. Teeth were decoronated perpendicularly to the longitudinal axis and beneath the cementoenamel junction using a low-speed diamond saw (Isomet, Buehler GmbH, Düsseldorf, Germany). All root canals were prepared by one trained operator. The canal patency was established and the root canals were prepared with the ProTaper system (Dentsply-Maillefer, Tulsa, OK, USA) and X-Smart-Endo-motor (Dentsply-Maillefer, Konstanz, Switzerland) using a crown-down technique with 3% sodium hypochlorite (NaOCl) irrigant. The canals were prepared to size 40, 0.06 taper with working length 1 mm short of the apex. The root canals were then irrigated with 5 mL of 17% ethylenediaminetetraacetic acid (EDTA) for 1 min [23]. Then, the root canals were irrigated with 10 mL of distilled water, dried with multiple paper points, and randomly divided into five groups ($n = 15$/group) on the basis of surface treatment to receive a glass fiber post (Rebilda post (RP); VOCO, Cuxhaven, Germany).

Canal filling

A non-standardized gutta-percha master cone (Lexicon, Dentsply Tulsa Dental, Tulsa, OK, USA) was fitted with tug-back to the working length of each root canal. AH Plus sealer (Dentsply, De Trey, Konstanz, Germany) was placed in the canal and spread with a # 45 K-file with a counterclockwise motion. The gutta-percha was compacted using the continuous wave technique up to 4–5 mm from the apex with a System B (SybronEndo, Orange, Calif, USA). Backfilling of gutta-percha was performed using thermoplastic gutta-percha and an Obtura II (Spartan, Fenton, MO, USA) at 185 °C. After the root canal filling, the access cavities of the teeth from all groups were restored with a non-eugenol temporary filling material (Coltosol, Coltène/Whaledent, Langenau, Germany) to accomplish an immediate coronal seal. Radiographs were taken with a #2-size RVG 6,100 digital sensor (Carestream Health, Rochester, NY, USA) to ensure adequate root canal filling. The specimens were stored at 37 °C and 100% humidity for 1 week to allow the sealer to set completely [23].

Then, the post space was prepared and part of the filling material was removed with the preshaping drill provided

with the post system. The canal walls of each specimen were enlarged with low-speed post drills provided by the manufacturer. To maintain the apical seal, at least 5 mm of the root canal filling was reserved at the apical level. The dual-cure self-adhesive luting agent Rely X Unicem (RXU; 3 M ESPE; Seefeld, Germany) was then placed on the post and into the canal space according to the manufacturer's instructions. Then, the coronary part of the exposed dentin was entirely restored with composite resin (Filtek P60; 3 M ESPE, St. Paul, MN, USA), and the specimens were stored at 37 °C and 100% humidity for 1 week [23].

Micropush-out assessment

After 1 week, the portion of each root that contained the fiber post was horizontally sectioned into six slices (2 coronal, 2 middle, and 2 apical slices) (1 ± 0.1 mm thick) using a low-speed diamond saw under water. Each slice was considered as a statistical unit. The thickness of each slice was measured using a digital caliper (Mitutoyo, Tokyo, Japan). Then, the apical aspect of the slice was loaded with a cylindrical plunger (diameter: 0.65, 0.98 and 1.25 mm for the apical, middle, and coronal slices) mounted on a universal testing machine (Model TT-B, Instron Co., Canton, MA, USA) at a cross-head speed of 0.5 mm/min until bond failure occurred [23]. The load at failure recorded in newtons (N) was divided by the area (mm^2) of the bonded interface for each specimen to calculate the bond strength in megapascals (MPa), according to the following equation [24]:

$$\text{Micropush} - \text{out bond strength (MPa)} = \frac{P}{A}, \tag{1}$$

where P is the maximum load (N) and A is the adhesion area of root canal filling (mm^2).

The adhesion area of each section was calculated by using the following equation:

$$L \times [\pi r_1 + \pi r_2], \tag{2}$$

where L was calculated as follows:

$$L = \left[h^2 + (r_1 - r_2)^2\right]^{1/2}, \tag{3}$$

In which r_1 is the coronal post radius, r_2 is the apical post radius, and h is the thickness of the slice. Debonded specimens were examined under a stereomicroscope (Olympus SZX-ILLB100-Olympus Optical Co. Ltd., Tokyo, Japan) at 50× magnification to evaluate the fracture pattern. The modes of failure were classified into four categories as follows: Type 1, adhesive failure between the post and luting agent; Type 2, adhesive failure between dentin and luting agent; Type 3, cohesive failure within the post; and Type 4, mixed failure [25].

Scanning electron microscopy (SEM) evaluation

The morphological aspects of fiber posts ($n = 3$/group) after treatment were examined using an SEM (JEOL, JSM-6510LV, JEOL Ltd., Tokyo, Japan). Each specimen was ultrasonically (Sonorex, Bandelin, Germany) cleansed for 3 min, immersed in 96% ethanol for 2 min, and then air dried. The specimens were sputter-coated with gold (SPI-Module Sputter Coater, Structure Probe Inc., West Chester, PA) and examined with SEM at 350× magnification [18].

Flexural strength (δ_f), flexural modulus (E_f), and stiffness (S)

A three-point bending test, in accordance with the ISO 10477 standard (10.0 mm span distance, 2 mm loading tip cross-sectional diameter, 1.0 mm/min crosshead speed), was used to measure the flexural strength (δ_f), flexural modulus (E_f), and stiffness (S) of post specimens. Fifteen specimens from each group were tested in a universal testing machine. The flexural strength (δ_f) (MPa) was calculated using the following equation [26–28]:

$$\delta_f = 8F_m L/\pi d^3. \tag{4}$$

The stiffness (S) (N/m) was calculated using the following equation [26, 27]:

$$S = F/D. \tag{5}$$

Flexural modulus (E_f) (GPa) was calculated using the following equation [26–28]:

$$E_f = S4L^3/\left(3\pi d^4\right), \tag{6}$$

where F_m is the applied load (N) at the highest point of the load–deflection curve, L is the span length (10.0 mm), d is the diameter (mm) of the specimen, S is the stiffness (N/m), and D is the deflection corresponding to the load F at a point in the straight-line portion of the trace. To eliminate the influence of the conical end of the posts, a short span length was used to get support for the post within the cylindrical part of the post. The parallel-sided cylindrical part of the post was used for measurement [26].

After the three-point bending test, representative post specimens from each group were ultrasonically cleansed for 3 min in deionized water followed by immersion in 96% ethanol for 2 min and air drying. Each specimen was sputter-coated with gold and examined with SEM in the fractured and intact regions of the posts at magnification of 500×.

Statistical analysis

Data of micropush-out bond strength were statistically analyzed (SPSS 13.0; Chicago, IL, USA) using two-way

analysis of variance (ANOVA) considering two factors (surface treatment and root level) and their interaction. The flexural properties mean values were compared with one-way ANOVA. Multiple comparisons were conducted by Tukey's HSD test and statistical significance was set at $P < 0.05$.

Results

Two-way ANOVA statistical analysis of the micropush-out bond strength testing data (surface treatment and root level) showed that the bond strength was significantly affected by the surface treatment and root level ($P < 0.001$). There was a significant interaction between the root level and surface treatment ($P < 0.001$).

The mean of the micropush-out bond strength (MPa) and standard deviations for the glass fiber post with the various treatments are presented in Table 1. The results achieved with M10 were significantly higher than those obtained with other surface treatments ($P < 0.05$). HF surface treatment showed significantly lower bond strength compared with the C group ($P < 0.05$). There were no significant differences between C and S surface treatments on the bond strength of glass fiber post ($P > 0.05$). On the other hand, AP treatment showed significantly lower bond strength compared with the C group in the coronal and middle levels of the root ($P < 0.05$). In general, improvements in bond strength (MPa) were found in the following order: M10 > C > S > AP > HF (Table 1).

For the root level, the coronal level of the root showed significantly higher bond strength compared with the middle and apical levels ($P < 0.05$) (Table 1). Most failure modes were adhesive type of failures between dentin and luting agent (48.2%), followed by mixed failures (38.5%). Furthermore, adhesive failures between post and luting agent (6.6%) and cohesive failures within the post (6.7%) were also observed (Fig. 1).

Table 1 Mean and standard deviation of the micropush-out bond strength (MPa) of post to self-adhesive luting agent and radicular dentin with different treatments

Post/Luting agent	Region of the canal	Surface treatments				
		C	S	AP	HF	M10
RP/RXU	Coronal	10.61 ± 2.01^{Aa}	9.57 ± 2.15^{ABa}	8.49 ± 2.09^{Ba}	5.49 ± 1.00^{Ca}	16.95 ± 2.29^{Da}
	Middle	8.15 ± 1.92^{Ab}	7.21 ± 1.47^{Ab}	5.26 ± 1.09^{Bb}	3.90 ± 1.13^{Bb}	10.88 ± 2.19^{Cb}
	Apical	4.17 ± 0.62^{Ac}	3.02 ± 0.96^{ABc}	2.92 ± 0.94^{ABc}	2.22 ± 0.68^{Bc}	6.64 ± 1.01^{Cc}

Mean values represented with common or same uppercase letters (row) are not significantly different ($P > 0.05$), while the mean values represented with different lowercase letter (column) are significantly different ($P < 0.05$) according to Tukey's HSD test

C control (no treatment), *S* silanization with a silane coupling agent, *AP* airborne-particle abrasion with alumina particles, *HF* etching with hydrofluoric acid, *M10* etching with CH_2Cl_2

Fig. 1 Failure pattern distribution of different groups tested (Type 1: adhesive failure between the post and luting agent; Type 2, adhesive failure between dentin and the luting agent; Type 3, cohesive failure within the post; and Type 4, mixed failure)
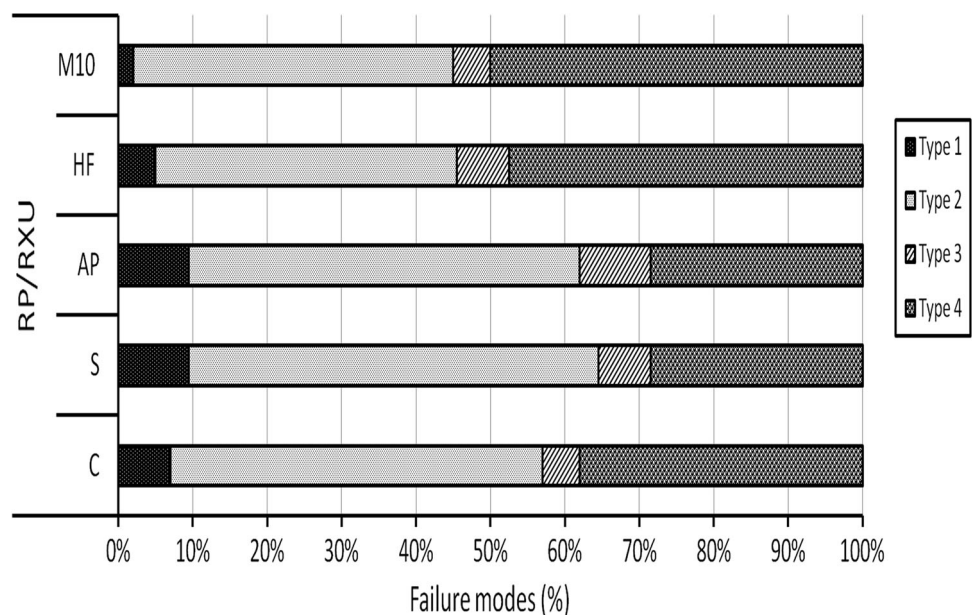
Fig. 2 Representative SEM photomicrographs of treated Riblida glass fiber post surfaces (350×) (**a–e**) and after flexure test (500×) (**f–j**) with *arrows* indicating fracture of superficial fibers and removal of resin on the surface opposite to load application: **a, f** control; **b, g** silane application for 60 s; **c** and **h** airborne-particle abrasion; **d** and **i** etching with hydrofluoric acid for 60 s; **e** and **j** etching with CH_2Cl_2 for 10 min, respectively
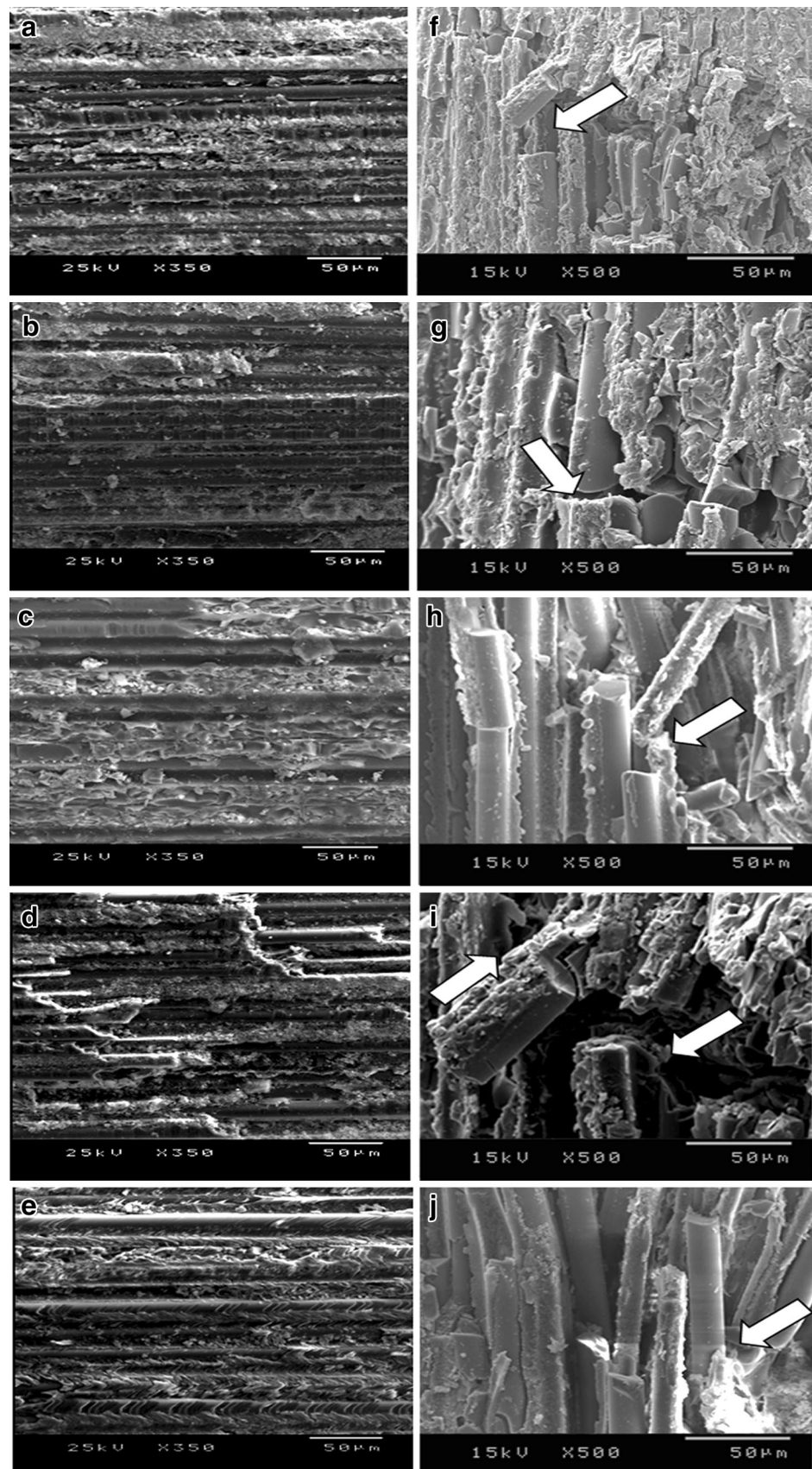
Table 2 Mean and standard deviation of the flexural strength (MPa), flexural modulus (GPa), and stiffness (N/m) values of the post with different treatments

Surface treatment	Flexural strength (δ_f)	Flexural modulus (E_f)	Stiffness (S)
C	894.91 ± 40.36	19.85 ± 1.83	287.94 ± 34.70
S	882.83 ± 47.59	18.69 ± 2.38	283.66 ± 24.74
AP	874.90 ± 41.15	18.56 ± 2.83	276.44 ± 33.12
HF	869.57 ± 38.25	18.09 ± 1.84	269.43 ± 37.78
M10	864.76 ± 42.64	18.31 ± 2.09	270.40 ± 40.39
P values[*]	0.322	0.239	0.512

C control (no treatment), *S* silanization with a silane coupling agent, *AP* airborne-particle abrasion with alumina particles, *HF* etching with hydrofluoric acid, *M10* etching with CH_2Cl_2

[*] There was no significant difference at $P > 0.05$

SEM images of the tested groups showed that the untreated RP posts revealed a rather rough surface with some glass fibers exposed (Fig. 2a). Posts treated with M10 showed changes in the surface topography (Fig. 2e). The resin matrix of the posts was dissolved, the glass fibers were exposed after surface treatments, and there was no damage to the exposed glass fibers. It appeared that the dissolution of the resin matrix formed retentive areas between the fibers. On the other hand, glass fibers were damaged after treatment with AP and HF (Fig. 2c, d). Alternatively, there were no changes on the surface topography for the posts treated with S compared with the C group (Fig. 2b).

The mean of the flexural strength (δ_f), flexural modulus (E_f), stiffness (S) values, and standard deviations are presented in Table 2. The flexural properties were not significantly affected by the surface treatments ($P > 0.05$). Following the bending test, SEM analysis of the fractured surfaces of the fiber posts revealed similar failure topography. Representative SEM analysis of the specimens showed that the fracture of superficial fibers of the posts and removal of resin from the surface opposing the load application (Fig. 2f–j).

Discussion

Based on the results of the present study, the null hypothesis that the surface treatments would improve the adhesion between fiber post and self-adhesive luting agent was accepted only for the M10 group. The null hypothesis that the application of surface treatments would improve the flexural properties of the post was rejected.

The adhesion between luting agents and post involves various factors, including fiber exposure due to the surface treatment of the post surface and the interlocking of the luting agents into microretentions of the post surface [29]. The bond strength of posts to dual-cure luting agent and root canal walls was evaluated using a micropush-out test. The bond strength was obtained at different levels of the root canal using thin slices of specimens. Consequently, the stresses formed in the bond interface were uniformly distributed. It has been shown that the micropush-out test is more appropriate than the microtensile method to evaluate the adhesion between fiber posts and post space dentin [30].

Dual-cure luting agents have been suggested to lute glass fiber posts, since light cannot adequately polymerize the luting agent in the deep areas of the root canal [31, 32]. Also, the simplicity of the clinical application of dual-cure self-adhesive luting agents to the post surface facilitates the luting procedure in the patient's mouth and, consequently, it is time saving [33, 34].

The interfacial bond strength between fiber posts and luting agent was significantly enhanced using M10 pretreatments compared with the other groups (Table 1). This could be attributed to the efficiency of the M10 surface treatment by removing the surface layer of the resin matrix of fiber posts. Consequently, additional surface areas of exposed glass fibers are provided which enhance the micromechanical retention of the luting agent [18, 19].

The application of a silane coupling agent did not enhance the interfacial bond strength. This could be due to the inadequate chemical bond between the posts that have little or no silanizable glass exposed and the silane functional monomer [18, 35]. This finding is in agreement with previous studies [18, 20, 35].

Surface treatments of glass fiber posts with AP and HF resulted in lower bond strength compared with the other groups (Table 1). This finding could be attributed to the disruption of the interface between the fibers and the resin matrix with removal of the matrix and damaging the fibers [36, 37]. This finding is supported by SEM results (Fig. 2c, d). Consequently, these treatments affect the adhesion of glass fiber posts due to the possibility of considerably modifying the shape and fit of the posts within the root canals [38].

For the root level, the highest bond strength was for the coronal region. The bond strength was lower in the middle and apical third [35, 39]. This could be attributed to various factors, including the complexity in visualization and access to the apical level, limitation in the flow and distribution of the material in this area of the canal that form additional bubbles and voids in the luting agent, and formation of thick smear layer throughout the post space preparation. This smear layer could not be conditioned by the adhesive luting agent or removed by EDTA/NaOCl irrigation for optimal bonding [25]. Failure modes analysis revealed that most of the failures occurred at the dentin and

luting agent, which is in accordance with the results of recently published studies [14, 39, 40].

Endodontic posts need to withstand the flexural loads applied to them during function, thus being helpful in retaining restorations [41]. The flexural strength property evaluates the resistance of a specimen to fracture. Specimens with higher values are more resistant to fracture, while those with lower values are less resistant. The flexural modulus property reveals the flexibility of a specimen. Specimens with higher values showed further stiffness, whereas lower values indicated more flexibility. High stiffness reveals a high flexural modulus and less strain capacity [36, 42]. In general, surface treatments did not compromise the flexural properties of posts in this study. This could be explained by the fact that as the stresses were apparently distributed homogenously, there were minimal alterations in the flexural properties after surface treatments [36]. This finding is in agreement with previous studies [28, 36]. Similarly, SEM analysis of the fiber posts after fracture showed comparable failure topography, including fracture of superficial fibers of the posts and removal of resin from the surface opposing the load application (Fig. 2f–j).

The present study has highlighted the possible potential for increasing the bond strength of methacrylate resin-based glass fiber post to the dual-cure self-adhesive luting agent and radicular dentin when using M10 solution as a surface treatment. Further in vivo studies are required for evaluation of the performance of the pre-treated posts during clinical service.

Based on the results presented and within the limitations of this study, the following conclusions can be drawn: The use of M10 as a surface treatment to methacrylate resin-based glass fiber post improved the adhesion between the fiber post and dual-cure self-adhesive luting agent. On the other hand, HF etching and AP treatments compromised the adhesion of fiber posts. The surface treatments performed did not compromise the flexural properties of the fiber posts.

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