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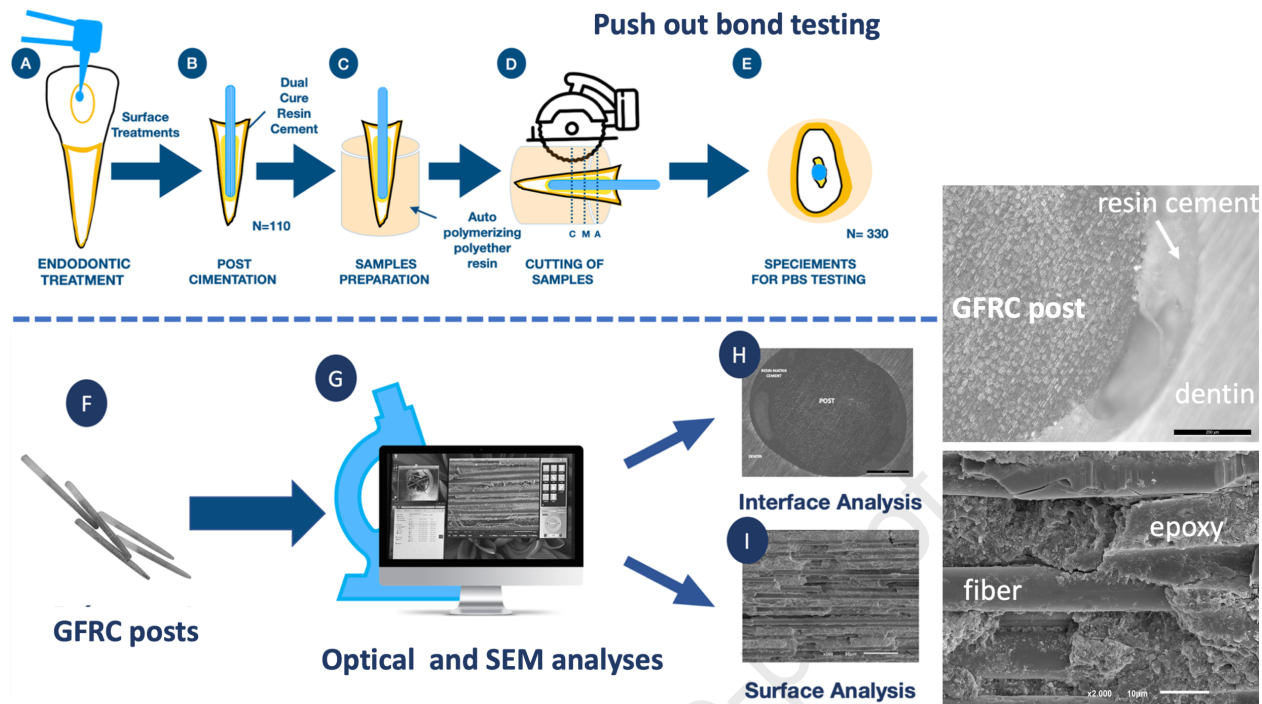
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Laser-texturing and traditional surface modification to improve the adhesion of glass fiber-reinforced composite posts to resin cements.

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Abstract

Objectives: The aim of this study was to perform experimental evaluation of the synergistic effects of laser-texturing and different traditional surface modification approaches to improve the push-out bond strength of glass fiber-reinforced composite (GFRC) posts to resin-matrix cements used in endodontically treated teeth rehabilitation.

Methods: One hundred and ten freshly extracted mandibular single-rooted premolars were endodontically treated and groups of specimens were divided according to the GFRC cementation after different surface treatment, as follow ($n = 10$): silane-based conditioning (SIL); 9.7 % HF acid-etching (HF); 35% H_2O_2 etching (H_2O_2); grit-blasting (GB); HF plus H_2O_2 etching (HFH_2O_2); 6 W Nd:YAG laser-texturing (L6W); 4.5 W Nd:YAG laser-texturing (L4.5W); 3 W Nd:YAG laser-texturing (L3W); 3 W Nd:YAG plus 35% H_2O_2 (L3WH $_2O_2$) ; 3 W Nd:YAG plus SIL (L3WSIL); and no-treatment (C). GFRC posts were cemented into the tooth root canals using a dual-cured resin cement. Then, specimens were cross-sectioned and mechanically assessed by push-out bond strength tests. Specimens were inspected by optical microscopy and scanning electron microscopy at magnification from $\times 30$ up to $\times 2000$. Data were analyzed using one-way analysis of variance and Tukey post hoc test ($p = 0.05$).

Results: Surface analyses of the GFRC posts showed a rough and retentive morphological aspect with a removal of the outer epoxy matrix layer and exposure of glass fibers after laser-texturing, grit-blasting or etching under 35% H_2O_2 . The highest bond strength values at 21.8 MPa was recorded for GFRC posts after laser-texturing on 3W plus silane-based conditioning followed by the group etched with 35% H_2O_2 (20.5 MPa). The failure mode was characterized as cohesive and mixed pathways. The lowest

bond strength values around 5 and 9 MPa were recorded for untreated GFRC surfaces or specimens etched with HF that was noticeable by adhesive failure pathways.

Conclusions: The combination of acidic etching and silane conditioning with laser-texturing at medium intensity promoted an adequate surface modification of GFRC posts and increased adhesion to a resin-matrix cement. Such combination of physicochemical approaches can enhance the long-term mechanical behavior of the restorative interface at endodontically treated teeth.

Clinical Relevance: Combining traditional and novel physicochemical approaches can provide promising adhesion pathways for glass fiber-reinforced composite posts to resin-matrix cements. A high mechanical interlocking of the resin-matrix cements and stable retention of the teeth root intracanal posts can decrease the risks of clinical failures by fracture and detachment of the adhesive interface.

Keywords: Intracanal post; resin cement; surface modification; adhesion; bond strength.

Introduction

Glass fiber-reinforced composite (GFRC) posts are widely utilized in rehabilitation of endodontically-treated teeth with extensive loss of coronal structure, mainly in anterior and pre-molars [1–3]. Cementation with customized GFRC posts can be carried out in chair-side procedures within a single-step providing the repairing of the loss tooth structures [4] [2,5]. Glass fiber-reinforced composite (GFRC) GFRC posts possess optical properties which can mimic the shade and translucence of the dentin required for aesthetic outcomes. Additionally, the mechanical properties of GFRC posts such as elastic modulus and strength are quite similar to those of enamel and dentin

leading to a proper stress distribution through the restorative interface and tooth tissues [6–8] [1,9,10]. Nevertheless, some limitations are still reported in the literature concerning the surface modification of GFRC posts and their bond strength to resin-matrix cements [11,12] [13] [14]. The success of endodontically-treated teeth with GFRC posts depends on a strong bond between resin-post and resin-dentin interface with establishments of reliable bonds at the interfaces between different materials [1–3] [6–8]. The lack in adhesion causes the propagation of cracks at the restorative interfaces leading to catastrophic fractures and the detachment of the GFRC posts [11,12] [13] [14].

GFRC posts are composed of around 30-50 vol. % micro-scale glass fibers embedded in an organic matrix. The short inorganic fillers can include quartz, glass-ceramics, or amorphous silica fibers with diameter at around 5 μm and length at approximately 15 μm [13,15]. The organic matrix comprises around 40 and 60 vol.% epoxy resin depending on the manufacturer [14]. The mechanical properties of the GFRC posts depend on the type and percentage of fibers. The elastic modulus of GFRC posts can range from approximately 18 up to 30 GPa while the flexural strength at approximately 400 MPa and fracture toughness at around 1-3 $\text{MPa}\cdot\text{cm}^{-2}$ [1,16]. Additionally, the fitting and the mechanical integrity of the interface between the GFRC posts to the resin-matrix cement enhances the stress distribution through the restorative interface towards the tooth tissues [17]. An appropriate bonding improves stress distribution from the occlusal loads and therefore that is a major factor for the long-term success of the restorative structures and endodontically treated teeth [17] .

Several physicochemical methods for surface modification have been proposed for improving the adhesion of GFRC posts to resin-matrix cements [17–19]. One

standard approach involves the conditioning of the surfaces using a silane-based compound such as 3-methacryloxypropyltrimethoxysilane. It establishes a chemical bonding between the inorganic fibers and the organic components from the resin-matrix cements [19,20]. Previously, the roughness can be increased by etching with reactive compounds such as hydrofluoric acid (HF), hydrogen peroxide (H₂O₂), hydrochloric acid, or potassium permanganate [19,21,22]. The increase in roughness provides higher surface area for wettability and mechanical interlocking of the adhesive systems and resin-matrix cements [19,21,23]. Otherwise, the surface can be treated by traditional grit-blasting with abrasive alumina or silica particles leading to increased roughness. Novel laser-assisted methods have been studied for surface treatment regarding the type of laser (i.e., Er:YAG, Nd:YAG, Er,Cr:YSGG laser, diode laser) and mode of irradiation involving irradiance, exposure time, and operative mode. Laser-texturing causes the surface modification by thermomechanical ablation depending on the laser parameters [4,13,24–26]. The combination of laser-texturing with the traditional physicochemical methods can enhance the morphological aspects of the GFRC surfaces and their adhesion to resin-matrix cements [18,27,28].

The purpose of the present study was to perform experimental evaluation of the synergistic effects of laser-texturing and traditional surface modification approaches to improve the push-out bond strength of glass fiber-reinforced posts (GFRC) to resin-matrix cements used in endodontically treated teeth rehabilitation. It was hypothesized that the combination of physical and chemical methods of surface modification promotes an enhancement of the bond strength of GFRC post surface to resin-matrix cements.

2. Materials and Methods

2.1. Preparation of specimens

The present study laboratory study has been written according to Preferred Reporting Items for Laboratory studies in Endodontology 2021 guidelines [29]. The research work plan was previously reviewed and approved by an institutional review board from the University Institute of Health Sciences (IUCS), CESPU, Portugal, with the ethics committee reference namely CE/IUCS/CESPU-14CE-IUCS2022. All procedures performed involving human participants followed the ethics standards of the IUCS ethics committee and therefore with the 1964 Helsinki declaration and its later amendments or comparable Ethics Standards. The need for informed consent was waived by the ethics committee/Institutional Review Board of IUCS at CESPU, Portugal.

One hundred and ten single-rooted human mandibular premolar teeth (mean root length at 15 mm) with completely formed apex were selected for this study considering root sizes and absence of caries, visible fracture lines, or cracks [30]. Teeth were previously extracted concerning orthodontic reasons and immediately placed in 6% NaOCl (CanalPro™, Coltene/Whaledent Altstätten, Switzerland) for 5 min followed by immersion in 5% formalin at room temperature for 7 days and lately immersed in distilled water for 7 days. The anatomic crowns were initially sectioned, and all teeth were endodontically treated teeth, as seen in Figure 1.

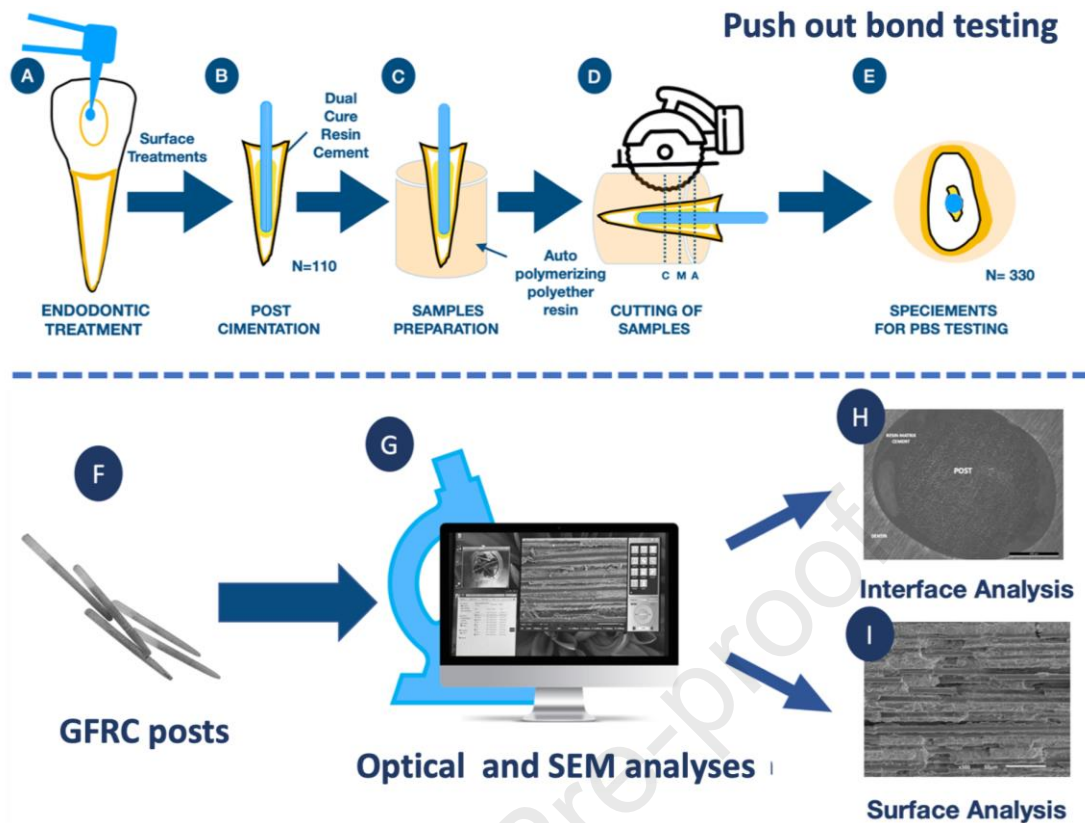


Figure 1. Schematics of the preparation of specimens and microstructural analyses. (A) Endodontic treatment. (B) Cementation procedure. (C) Samples preparation. (D-E) Cross-section of specimens for bond strength tests (F) Fiber posts with surface treatment (G) Microscopical evaluation (H) Optical interface analysis (I) Scanning electron microscopy

The working length was determined using an endodontic file type K-flexofile™ ISO # 10 (Dentsply-Maillefer, Switzerland) until it is visible through the apical foramen and then 1 mm was subtracted. Physically assisted instrumentation was carried out by reciprocating friction motion using #25.08 primary files (25-mm length) (Wave One™, Dentsply-Maillefer, Switzerland)(Figure 1A). The tooth root canals were disinfected using 3% NaOCl solutions between each 3 reciprocating movements using a syringe with lateral irrigation needle (30G). The canals of the teeth were dried with calibrated paper

cones (Dentsply-Maillefer, Switzerland). At last, the canals were filled using typical primary gutta-percha cones (Dentsply Maillefer, Switzerland) plus single-cone technique and vertical compaction with gutta-percha points which was embedded within resin-matrix cement (AH-Plus™, Dentsply-Maillefer, Switzerland). Periapical X-Ray analyses were performed for confirmation of the filling integrity after gutta percha compaction. At last, specimens were stored in 100% humidity at 37°C for 2 weeks to allow a complete setting of the root canal sealer [20]. Afterwards, intracanal space was initially shaped using a heated metallic tip (BeeFill, VDW, Germany) and gutta-percha was removed using reamers sizes 2, 3, 4 (Largo Peeso reamers™; Dentsply Intl, USA). Specific parallel-sided twist drills with 1.5-mm ø (Parapost nº 6 Black P-42™; Coltène/Whaledent Inc, USA) were used to ensure the gutta-percha removal and to achieve the fitting of the GFRC post at low speed. Thus, the gutta-percha removal was ensured using parallel-sided twist drills recommended for the GFRC post with diameter higher than the tooth root canal. No attempt to generate pressure on instruments against the intracanal dentin surfaces was made when using either Largo reamers or drills. Silicone stops (Dentsply Intl, USA) were placed on each drill to ensure that the canal shaping was achieved as previously determined lengths. X-ray images of tooth roots were acquired using a X-ray clinical apparatus (Corix 70 Plus KVP X-ray™, CORAMEX S.A, Mexico) to inspect the gutta-percha removal. X-ray analyses were performed on triangular scanning technique at 70 kVp and 8 mA for 53 s.

One hundred GFRC posts were randomly divided into eleven groups ($n = 10$), according to the surface treatment as seen in Table 1.

Table 1. Groups of GFRC posts and surface treatment.

Groups	Surface treatment
Control (C)	Rinsing in 70% propanol
SIL	Conditioning with 3-methacyloxy-propyltrimethoxysilane [3-MPS] (Monobond™, Ivoclar-Vivadent, Schaan, Liechtenstein) under reciprocating motion for 60 s and then gently oil-free air dried for 5 s following the manufacturer's instructions [17]
HF	Etching in 9.7% HF (Ultradent Products, Inc, South Jordan, UT) for 20 s, rinsing in distilled water for 30s and gentle air drying for 5 s [17]
H ₂ O ₂	Etching in 35 % H ₂ O ₂ (Whiteness HP Max, FGM) for 1 min and gentle air drying for 5 s [31]
GB	Grit-blasting with 50-µm Al ₂ O ₃ particles at 2 bar for 20 s at distance of 10 mm away from the surface (Dento-prep™, Ronviga, Dagaard, Denmark)(7) under rotatory motion. Then, rinsing in 70% propanol
HF-H2O2	First, etching in 9.7% HF (Ultradent Products, Inc, South Jordan, UT) for 20 s, rinsing in distilled water for 30s and gentle air drying for 5s. Then, etching in 35 % H ₂ O ₂ (Whiteness HP Max, FGM) for 1min . [17]
6W 4.5W 3W	Ablation under rotatory motion under Nd-YAG laser 1064-nm wavelength (OEM Plus 100 W, Italy) on 3, 4.5, or 6 W intensity at repetition rate of 20khz, maximum pulse energy of 0.3 mJ, and pulse duration of 0.36 µs.[32]
3W-H ₂ O ₂	Ablation under rotatory motion under Nd-YAG laser 1064-nm wavelength (OEM Plus 100 W, Italy) on 3, 4.5, or 6 W intensity at repetition rate of 20 khz, maximum pulse energy of 0.3 mJ, and pulse duration of 0.36 µs [32]. Then, etching in 35 % H ₂ O ₂ (Whiteness HP Max, FGM) for 1 min
3W-SIL	Ablation under rotatory motion under Nd-YAG laser 1064-nm wavelength (OEM Plus 100 W, Italy) on 3, 4.5, or 6 W intensity at repetition rate of 20 khz, maximum pulse energy of 0.3 mJ, and pulse duration of 0.36 µs [32]. Then, surface conditioning with silane (SIL) and gentle air drying [17]

For the cementation of the GFRC posts, intraradicular dentin was previously conditioned by a universal adhesive system (Futura bond Universal™, VOCO, Germany) according to the manufacturer's instructions. The universal bonding agent was applied inside the root canals using a fine microbrush at reciprocating friction movement for 30 s. Following, the resin-matrix cement material (ParaCore Automix™, Coltene Whaledent, USA) was applied directly into the intracanal space using a syringe tip. The

GFRC post was also coated with the cement and then inserted into the tooth root canal on slight pressure. The dental inspector apparatus (Ney surveyor, Germany) was used to align the post space with the long axis of the tooth. The excessive cement layer was removed and the cement was then light-cured using a light curing unit (LCU) with irradiance at $800\text{mW}/\text{cm}^2$ and wavelength at 420-480 nm (LY- A180TM, Anyang Zongyan Dental Material Co, Ltd, China) for 40 s. After cementation, periapical X-Ray analyses were performed for evaluation of the filling integrity. Specimens were then assembled with a self-curing polyether modified resin (Technovit 400TM, Kulzer GmbH) in a short length of polyvinyl chloride mold. Each root was previously scratched on the buccal and lingual surfaces with a tungsten carbide bur to increase root retention in the acrylic resin block [32–35]. Specimens were cross-sectioned at 90 degrees relative to the plane of the GFRC post to resin-matrix cement interface using a precision cutting-machine (IsometTM, Buhler), as shown in Figure 1 D . Cross-sections were carried out into 1-mm-thick slices at three different regions (cervical, coronal and apical) [33]. For interfaces analysis, surfaces were wet ground down to 2400 Mesh using SiC abrasive papers and then polished with $1\text{-}\mu\text{m}$ Al_2O_3 particles. Surfaces were ultrasonically cleaned in propanol (Sigma-Aldrich, USA) for 10 min and then in distilled water for 10 min.

2.2. Microscopic analyses

GFRC specimens from each group were separated before cementation for initial surface analyses by scanning electron microscopy (SEM) Figure 1I. Also, groups of cross-sectioned specimens were inspected by optical microscopy and SEM. The microstructural analyses of cross-sectioned specimens started by optical

microscopy and then further details were inspected by scanning electron microscopy (SEM).

Groups of cross-sectioned specimens were inspected by optical microscopy (Figure 1H) within magnification ranging from $\times 10$ up to $\times 500$. Microstructural analyses were performed using an optical microscope (Stemi DV4TM; Carl Zeiss, Germany) connected to a computer for image processing, using Leica Application SuiteTM software (Leica Microsystems, Germany). A number of six micrographs were acquired at $\times 500$ magnification, for each specimen ($n = 18$). The software Adobe PhotoshopTM (Adobe Systems Software, Ireland) was used to analyze black and white images, with the black regions representing the organic matrix and the white regions representing the inorganic fibers or particles. Image JTM software (National Institutes of Health, USA) was used to quantify the porosity percentage of the cross-sections. The fracture zone was also evaluated after push-out bond strength tests to characterize the failure mode. The failure at the GFRC post to resin cement interface or between resin cement and dentin without tooth structure damage was classified as adhesive failure mode. The cohesive failure was considered when the fracture occurred through the GFRC post itself or tooth structures. Mixed failure mode was considered involving both adhesive and cohesive previous conditions [11].

For SEM, surfaces were sputter coated with a AgPd thin layer (MED 010, Bal-Tec AG, Balzers, Liechtenstein) and inspected using a SEM unit (TM-3030TM, Hitachi, Krefeld, Germany) coupled to energy dispersive spectroscopy (EDX, Swift 2000, Hitachi, Japan). On GFRC posts, SEM analyses were carried out at 10 kV and magnifications ranging from $\times 500$ to $\times 2000$ at 20 kV under secondary electrons (SE) mode to provide the topographic information of the GFRC fibers and organic matrix. Mechanical integrity and distribution

of fibers was evaluated along the entire post surface extension. On cross-sectioned specimens, SEM analyses were carried out at 15 kV under SE and backscattered electrons (BSE) mode to provide microstructural details of the materials, interface zone, inorganic fillers, and fibers [31,34–36]. On each specimen, a total of three micrographs were acquired at x1000 magnification ($n = 9$). EDX was performed at 15 kV under backscattered electrons (BSE) following standard guidelines for chemical analyses of the inorganic fillers. The chemical analysis of the materials was performed by EDX under BSE mode using a silicon drift detector energy-dispersive spectrometer (SDD-EDS). The characteristic X-Rays emitted from the atoms are harvested by the SSD, that evaluate the signal considering standard computational data to estimate the atomic fraction.

2.3. Push out bond assays

Push-out bond tests were performed using a universal testing machine (Lloyd LF Plus™, Ametek Inc, Lloyd Instruments, UK). As seen in Figure 1, loading was applied onto the cross-sectioned specimens at crosshead speed of 0.5 mm/min from apical to coronal direction until adhesion failure [11,37]. The fracture load was recorded in N while the strength values were recorded in MPa following the equation 1:

$$(N/A) A = \pi(r_1 + r_2) \times \sqrt{(r_1 + r_2)^2 + h^2} \quad (1)$$

where r_1 was the radius of the cervical area, r_2 was the radius of the apical area, and h was the thickness of each specimen.

The failure at the GFRC post to resin cement interface or between resin cement and dentin without tooth structure damage was classified as adhesive failure mode.

The cohesive failure was considered when the fracture occurred through the GFRC post itself or tooth structures. Mixed failure mode was considered involving both adhesive and cohesive previous conditions [11].

The statistical analysis of the data was carried out using OriginLab statistical software program (OriginPro 2023b™, Origin Lab, Northampton, MA, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were assessed to determine whether a sample can be included from a population with a particular distribution. Data were statistically evaluated by two-way ANOVA and Shapiro-Wilk to determine statistical relationship or differences in values of push-out bond strength values [32]. Levene's test was also used to correct the Kolmogorov-Smirnov test within a normal distribution, thus increasing the power of the test. A probability (p) value less than 0.05 (significance level at 5%) was considered statistically significant while a p value below 0.001 (significance level at 0.1%) was considered highly significant. In this study, the normal distribution of the data was not noticed when using the Kolmogorov—Smirnov test followed by Levene's test. Then, Tukey test was applied to compare the results between the groups. A power analysis was performed by the t-student test and ANOVA to determine the number of specimens for each group (n) to validate the push out bond strength results, and therefore to disclose a test power of 100% in this study [32].

3.Results

3.1. Surface analyses

SEM images of the GFRC posts free of surface modification and after surface treatment are shown in Figure 2 and 3. As seen in Figure 2B and C, glass fibers were

embedded in the epoxy matrix and therefore the epoxy matrix area was exposed to the luting materials.

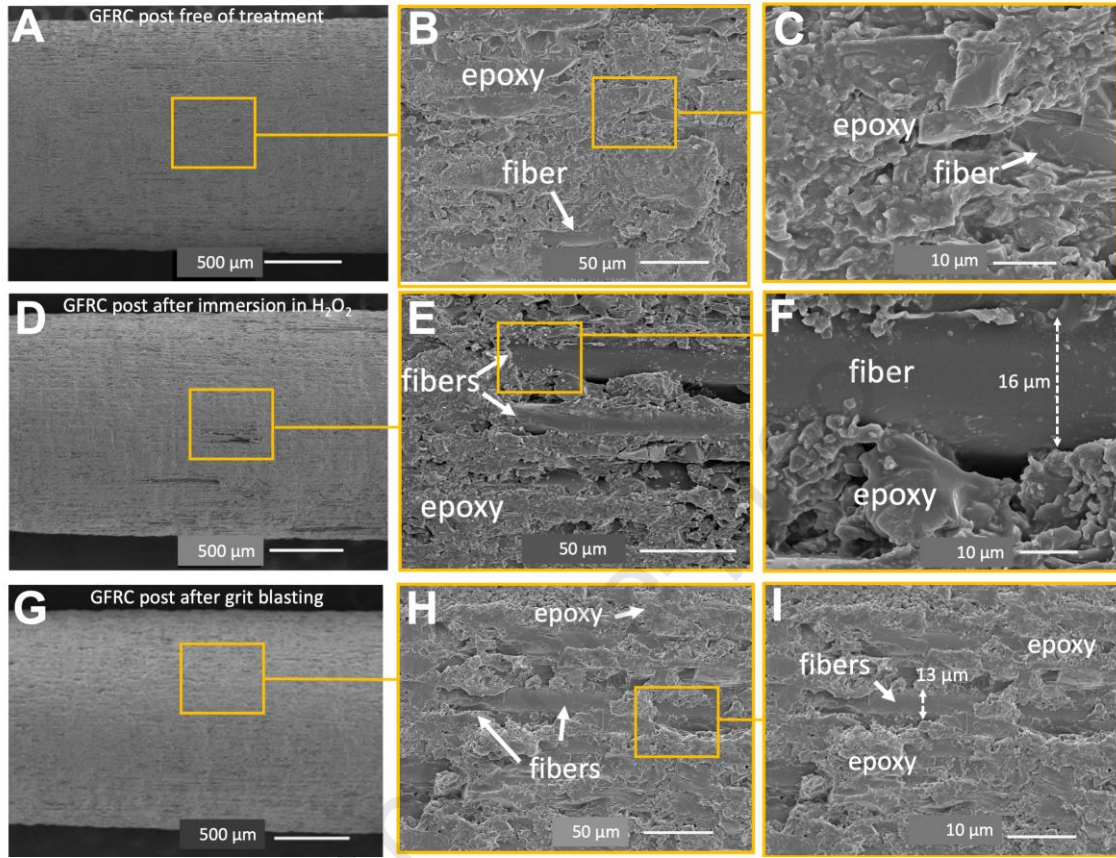


Figure 2. SEM images of GFRC posts (A-C) free of surface modification and (D-F) after immersion in H_2O_2 solution for 1 min or (G-I) grit-blasting with Al_2O_3 particles. Images acquired at 15kV on SE mode at magnification at (A,D,G) X50, (B,E,H) $\times 500$, and (C,F,I) $\times 2000$.

On the other hand, glass fibers were gradually uncovered after surface treatment by immersion in H_2O_2 solution (Fig. 2D-F) or grit-blasting with alumina particles (Fig. 2G-I). The removal of epoxy matrix arisen randomly, and fibers were noticed at some spots. Furthermore, a higher removal of epoxy matrix and exposure of glass fibers was detected after Nd:YAG laser irradiation on 3W followed by silane conditioning or

immersion in H_2O_2 solution (Figure 3). A rough morphological aspect was also evidenced that increases the surface area for further mechanical interlocking with adhesive systems and resin-matrix cements. The effect of the laser ablation at different portions of the GFRC post surface is noticeable even on SEM images at low magnification (Figure 3A). The removal of epoxy matrix increased in function of the laser power augmentation up to 6W, as seen in Figure 3A-C.

Nevertheless, micro-cracks and detachment of glass fibers were detected on GFRC posts after high power laser ablation (Figure 3A-C). Surface of the GFRC fibers showed smooth morphological aspects on SEM images at high magnification (Figure 3 C and F). Nevertheless, fracture of fibers occurred that can be detrimental for further bonding to adhesive systems and resin-matrix cements. There were no differences in the morphological aspects on GFRC posts after the silane deposition (Figure 3G-I) although it can improve the physicochemical adhesion to adhesive and resin-matrix cements acquired at 15kV on SE mode at $\times 2000$ magnification. For instance, the combination of H_2O_2 etching for 1 min followed by HF etching did not reveal morphological aspects for mechanical interlocking.

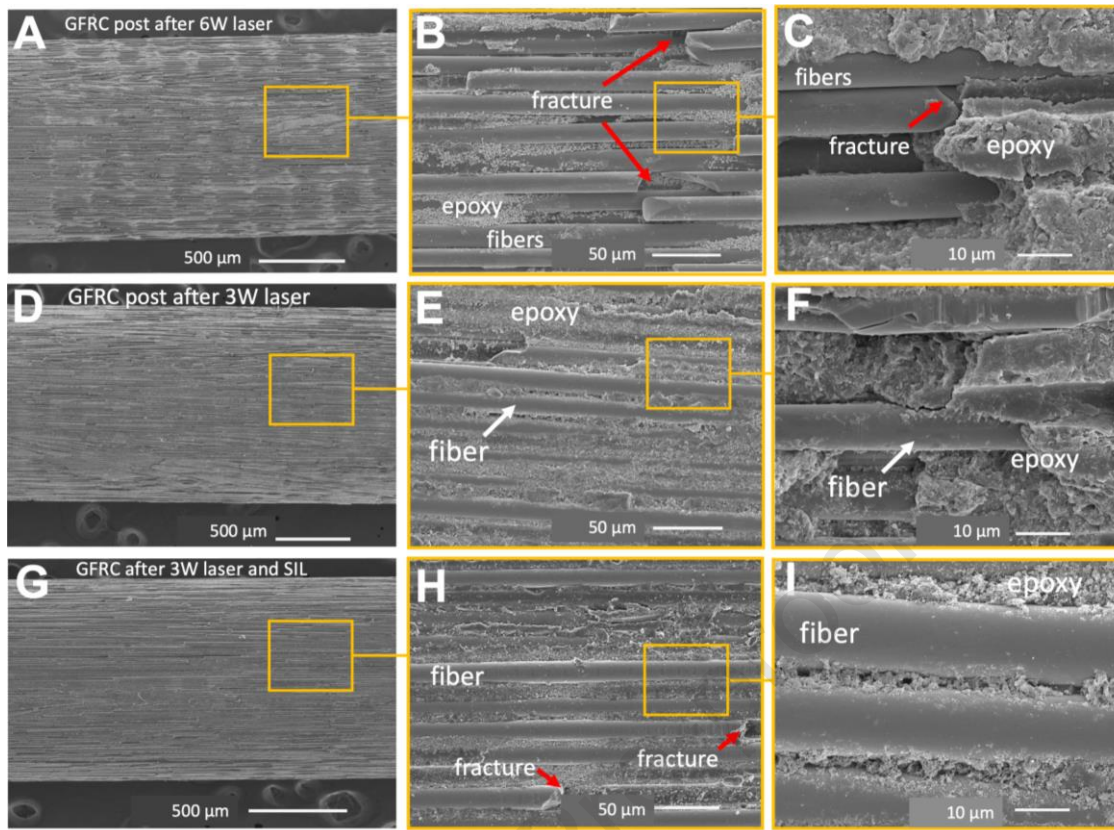


Figure 3. SEM images of GFRC posts after surface modification under Nd:YAG laser irradiation on (A-C) 6 W, (D-F) 3 W or (G-I) 3 W plus silane conditioning. Images acquired at 15kV on SE mode at magnification at (A,D,G) $\times 50$, (B,E,H) $\times 500$, and (C,F,I) $\times 2000$.

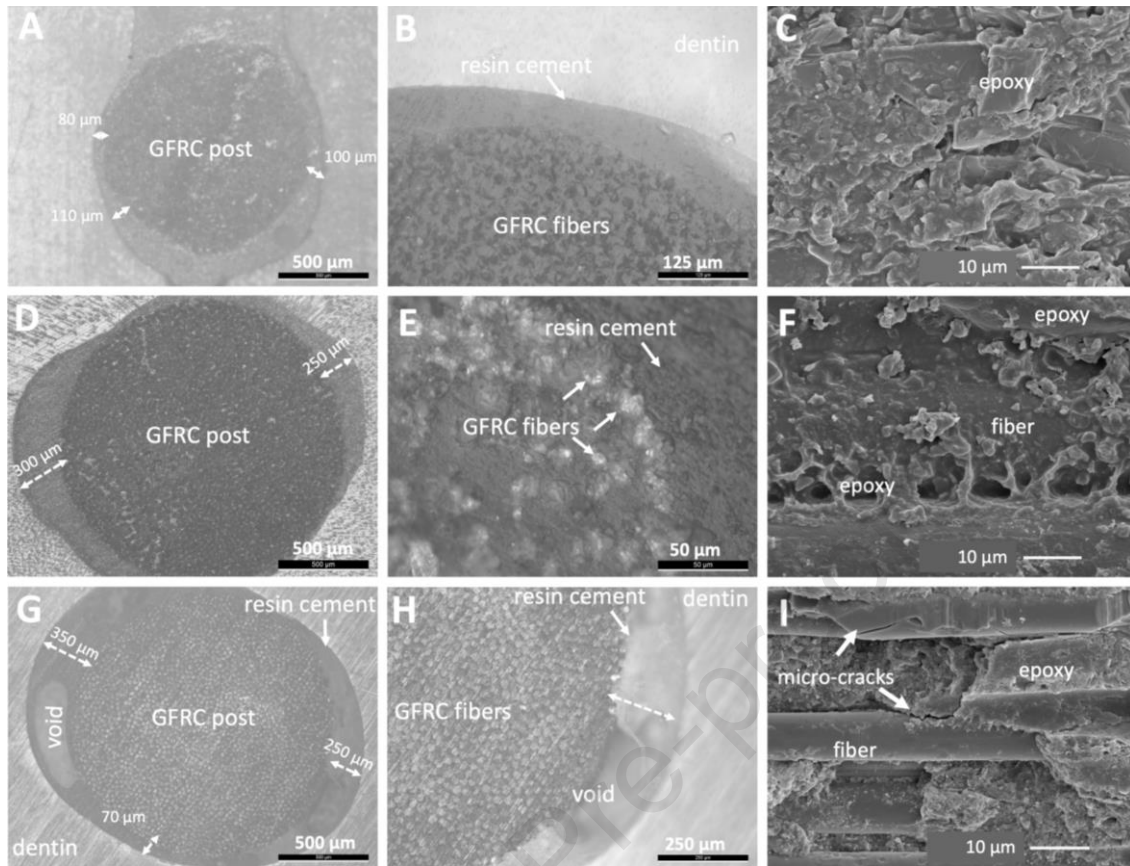


Figure 4. Optical microscopy images of the interfaces after cross-sectioning GFRC post to resin-matrix cement and tooth for the following groups: (A,B) control group; (D,E) H_2O_2 ; (G,H) 3 W. SEM images acquired at 15 kV on SE mode at $\times 2000$ magnification of the interfaces after cross-sectioning GFRC post to resin-matrix cement and tooth for the following groups: (C) control, (E) H_2O_2 ; (H) 3W.

As seen in Figure 4, a variation of resin-matrix cement layer is noticed in the microstructure of the interfaces after cross-sectioning GFRC post to resin-matrix cement and tooth by optical microscopic analyses. The thickness of the resin-matrix cement layer ranged from approximately $30\ \mu\text{m}$ (Figure 4A) up to $350\ \mu\text{m}$ (Fig. 4G). Also, some defects were detected such as micro-scale voids (Fig. 4G and H) after cementation.

3.2. Push-out bond strength

In Figure 5, the mean values and standard deviation of the push out bond strength values (MPa) are shown for each group at different regions of the GFRC post to resin-matrix cement and tooth specimens.

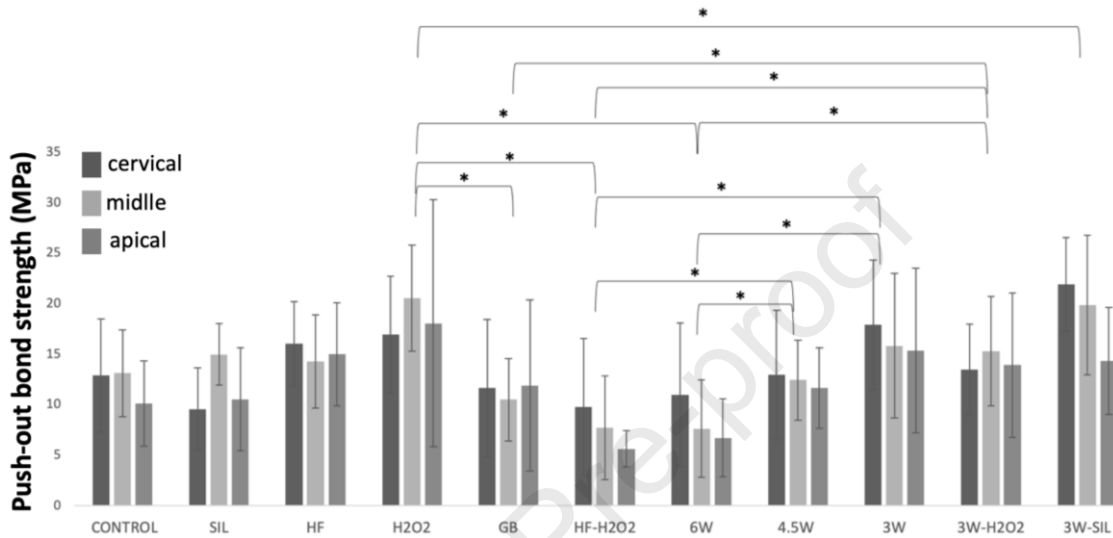


Figure 5. Mean values and standard deviation of push out bond strength values (MPa) per group at different regions of the GFRC post to resin-matrix cement and tooth specimens. *Represents statistic differences between groups ($p < 0.05$)

Regarding the type of GFRC surface, the highest mean values of push out bond strength for GFRC post to resin-matrix cement and tooth were recorded for the groups etched with 35% H_2O_2 solution or laser-textured under 3 W power plus conditioning with silane agent. There were not statistical differences in push out bond strength values between those groups ($p < 0.05$). The third highest strength mean values were recorded for the group treated under laser irradiation on 3W with or without 35% H_2O_2 etching. The lowest mean values of push out bond strength for GFRC post to resin-matrix cement and tooth were recorded for groups treated under laser irradiation at high power (6 W)

or by a combination of H₂O₂ plus HF etching solutions. Considering the cross-section region, higher push out bond strength values were measured at the cervical and middle thirds when compared to the values measured at the apical third for all the groups. Statistical differences ($p < 0.05$) were recorded between groups as shown in Table 2 (supplementary data). The distribution of push out bond strength values for each group of specimens is given in Figure 6.

The type of fracture was recorded for each group of specimens, as seen in Figure 6. On the control group, the most common failure was the type I and II, namely adhesive and cohesive, respectively (Fig. 6). The highest number of failures type I was recorded for groups of specimens treated only with silane, HF or H₂O₂/HF solutions. The group treated with grit-blasting revealed failures of type I and III (tooth fracture) while the combination of laser irradiation on 3W followed by 35% H₂O₂ etching resulted in cohesive failure (type II). The group treated under laser irradiation on 3 W followed by silane conditioning revealed adhesive (type I) and cohesive (type II) failures.

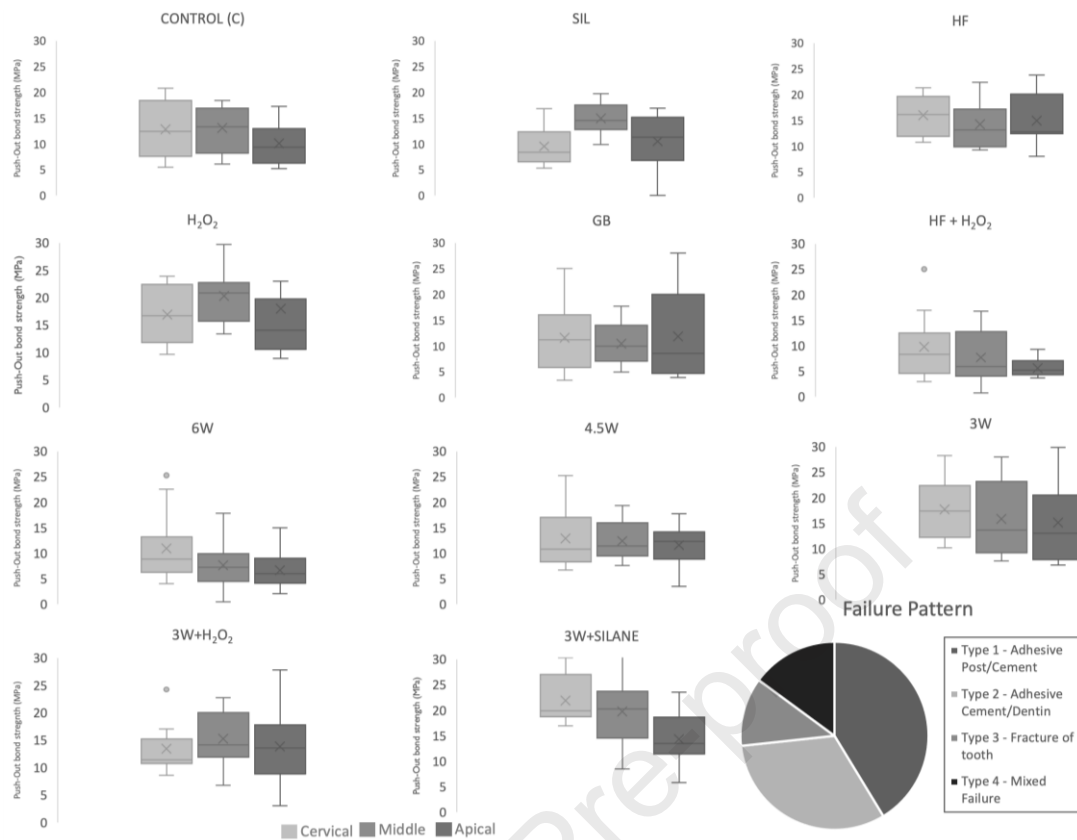


Figure 6. Distribution of push out bond strength values (MPa) per group in different regions of the GFRC post to resin-matrix cement and tooth assemblies.

4.DISCUSSION

The use of GFRC posts cemented with resin-matrix cements depends on a high adhesion among the GFRC surfaces, adhesive, resin-matrix cement, and intraradicular dentin. The challenge to achieve an effective bonding between GFRC post and resin-matrix cement has received increasing attention from the scientific community. The present study reported the effects of different physicochemical surface treatment of GFRC posts on their push out bond strength to resin-matrix cements after cementation in endodontically treated teeth. The novelty of this study deals with the use of laser irradiation at different powers and the combination with traditional physicochemical

approaches including silane conditioning, acidic etching, and grit-blasting with alumina particles. The findings revealed the exposure of glass fibers from the epoxy matrix of the GFRC posts after laser irradiation, grit-blasting, or immersion in H_2O_2 . The rough aspects of the surfaces after laser irradiation increased the push out bond strength to a resin-matrix cement. Furthermore, the combination between laser irradiation and silane conditioning or immersion in H_2O_2 solution provides the highest push out bond strength to the resin-matrix cement and tooth. The difference between groups was statistically significant the synergistic effects of laser irradiation and traditional physicochemical surface modification. Such findings validate the hypothesis raised in the present study.

In our study, GFRC posts free of surface treatment (control group) had relatively continuous smooth surface without the exposure of glass fibers which were covered into the epoxy matrix as also found in previous studies [17,18,22]. In fact, the roughness and morphological aspects of untreated GFRC surfaces of the control group did not provide the mechanical interlocking required for the adhesive system and resin-matrix cement [27]. Also, the polymer-based matrix in the structure of GFRC involves a high ratio of crosslinks connections which are unable to be reactivated, and so unable to react with the monomers of adhesive systems and resin-matrix cements. The same morphological aspects were noted for GFRC surfaces which were solely immersed in 9.7% HF solution that indicate no chemical reactivity with the epoxy matrix. Hydrofluoric acid (HF) has been as etching approach of glass ceramics such feldspar-based porcelain or lithium disilicate reinforced glass ceramics to increase the roughness for cementation of prosthetic structures [38,39]. However, the immersion GFRC posts in 9.7% HF did not reveal any surface alteration to increase the bonding to adhesive system or resin-matrix cement. The surface integrity maintaining the smooth surface topographic aspects of

commercially available GFRC posts promoted the lowest push out bond strength values, as seen in Figure 5 and 6. Also, the dominant failure pathways were characterized by adhesive failures which occurred at the GFRC post and resin-matrix cement interfaces. It means a low retention of the resin-matrix cement throughout the GFRC post surfaces.

The application of chemical agents, including hydrofluoric acid (HF), hydrogen peroxide (H_2O_2), hydrochloric acid ($\text{H}_2\text{O}:\text{HCl}$), and potassium permanganate results in modifications on both the morphological aspects and chemical composition of GFRC surfaces [23,40,41]. Nevertheless, such compounds are extraordinarily strong oxidizers and their use requires specific attention and care. In this study, the epoxy matrix of GFRC posts was partially removed after immersion in H_2O_2 solution for 1 min uncovering the glass fibers leading to a hybrid and rough surface with aimed morphological aspects for mechanical interlocking of the luting materials. Indeed, hydrogen peroxide has the capability to expose the glass fibers by selectively dissolving the epoxy resin bonds without the damage of the glass fibers [31,39]. The etching effect of HF solutions at different proportions depends on their capability to break the polymeric bonds through the substrate oxidation pathway [31,39]. The surface treatment with H_2O_2 also depends on the concentration and immersion time as reported in literature [21,27,31]. In this way, the commercial availability of 35 % H_2O_2 solution shows the feasibility and chair side potential for clinicians and therefore clinical guidelines can be improved. The selectively surface modification by removing the epoxy matrix of GFRC posts has been suggested to roughen the surface and enhancing the mechanical retention to luting materials [21,31]. The increase in surface irregularities leads to an increase in the surface area and consequent high bond strength to flowable polymer-based materials such as adhesive systems and resin-matrix cements [18,19] [42]. The surface modification of

GFRC posts in H_2O_2 solution promoted high mean push out bond strength values (Figure 5 and 6) to resin-matrix cement after cementation in tooth root canals. The failure mode was characterized by cohesive pathway since the high adhesion of the resin-matrix cement to the treated GFRC posts. According to different studies, the association surface treatment with H_2O_2 with other selective mechanical surface treatment technique could enhance the bond strength results of GFRC posts to resin cements [21,31,43]. Nevertheless, the combination of H_2O_2 and HF had a negative effect on the H_2O_2 chemical reactivity with the epoxy matrix as revealed by morphological aspects of the surfaces and push out bond strength values as well as by an adhesive failure mode. The results were corroborated with those found in previous studies [33,44,45].

The conditioning of GFRC posts with the silane compound did not promote any visible topographical alteration although the silane coating establishes a chemical bonding to OH^- groups over the glass fibers and to the organic components of the epoxy and luting materials [20,46]. Silane coupling agent is a hybrid organic-inorganic compound that can intervene the bonding between organic and inorganic molecules via intrinsic dual chemical reactivity [20,27,46]. Silane increases the GFRC surface wettability and bonding energy to adhesive systems and resin-matrix cements. Several factors influence the effectiveness of the silane layer, such as its chemical composition, pH, presence of solvent, molecular size, and the application procedure [23,47]. However studies reported that chemical bonding of the silane is higher on the glass fibers than that to the epoxy matrix [22,31,44,48]. Thus, a previous surface treatment is recommended to uncover the glass fibers by grit-blasting with Al_2O_3 particles, H_2O_2 etching, or laser-texturing [9,38] [49]. Grit-blasting with Al_2O_3 particles is a well-known physical surface modification to providing a micro-scale texturized and retentive

surface. Grit-blasting leads to an increase in the roughness and therefore in the total surface area for adhesion to other materials. resin-matrix cements. Consequently, it provides an enhanced mechanical interlocking of adhesive systems and resin-matrix cements onto GFRC posts. Previous studies reported variable roughness values related to several parameters such as particles type and size, air pressure, surface type, abrasion distance, and time [50,51]. In this study, Al_2O_3 particle with size at 50 μm were grit-blasted at 10-mm distance away from the surface leading to randomly rough surfaces quite similar to that found in previous studies [38,43,50]. The push out bond strength values were quite similar to those values measured on the untreated surfaces. Also, the failure mode was characterized by adhesive pathways revealing low retention of the resin-matrix cement. The roughness can be balanced by the careful selection of the size of the abrasive particles and the appropriate adjustment of air-abrasion parameters such as distance, pressure, and time [13]. Nevertheless, extreme pressure or prolonged exposure time of the air-abrasion process can cause excessive removal of the organic matrix and glass fibers, leading to the development of micro-cracks and accumulation of debris, that negatively affect the bond strength of GFRC posts to resin-matrix cements [43]. Thus, the grit-blasting procedure could be enhanced using 110- μm Al_2O_3 particles to provide surfaces with higher roughness and adhesion to resin-matrix cements. Additionally, the conditioning of the surface with a silane agent can improve the physicochemical bonding to adhesive systems and resin-matrix cements.

The physical modification of surfaces by traditional surface treatment result in the exposure of underlying fibers within the outer layer of the substrate, that can subsequently be subjected to additional physicochemical surface approaches. Regarding surface treatment by laser irradiation, the location and surface patterns can

be previously designed increasing the adhesion of GFRC posts at certain spots. The laser-assisted approach involves melting and removal of the epoxy matrix by a thermomechanical ablation regarding the interaction between the laser irradiation and surface. Different laser parameters should be considered such as laser type, wavelength, power, mode, and irradiance time [25,52,53]. The most common lasers are the following: Nd:YAG, Er,Cr:YSGG laser, diode laser [25,54,55]. In this study, Nd:YAG (1064-nm wavelength) at three different power were assessed, namely 3, 4.5, or 6 W, on 0.36 μ s pulse duration, at 20 kHz and maximum pulse energy of 0.3 mJ followed by surface modification in H₂O₂ solution or after silane-based conditioning. The present findings revealed a removal of the outer epoxy matrix layer by laser irradiance exposing the glass fibers although the fibers were partially destroyed on high power (6 W) irradiance. Conversely, previous studies demonstrated that application of high power settings of lasers can result in significant damage of the GFRC post surfaces [4,24,25]. On low-irradiance laser (3 W), the outer epoxy matrix layer was appropriately removed uncovering the glass fibers without the formation of micro-cracks and fracture. The rough morphological aspects of GFRC posts after laser irradiation at 3 W increased their push bond strength to resin-matrix cement. Also, the mean push out bond strength values increased (17.8 MPa) after the Nd:YAG laser with a power setting of 3 W (L3W) when compared to the untreated surfaces (5.6 MPa). The Nd:YAG laser irradiation on 4.5 W (L4.5W) had the second-strongest effect on the push out bond strength at 12.9 MPa, while the Nd:YAG laser with a power setting of 6W (L6W) had the weakest effect on the push out bond strength (10.9 MPa). Differences between laser groups, were statistically significant ($p<0.05$), namely between the group with laser a maximum power 6 W and other laser-textured groups.

Accordingly, the surface modification with H₂O₂ or silane conditioning enhanced the adhesion of the laser-treated GFRC surfaces to the resin-matrix cements as represented by cohesive failure mode. The removal of the outer epoxy layer by different physicochemical methods such as laser plays also key role for the effectiveness of silane deposition, as it exposes the glass fibers according to previous studies [18,38]. The rough and retentive morphological aspects of a hybrid surface composed of glass fibers and epoxy matrix can enhance the bond strength of glass fiber-reinforced composite posts to adhesive systems and resin-matrix cements. The present findings revealed that the combination of low-irradiance Nd:YAG laser at 3 W in combination with traditional physicochemical methods promoted a high adhesion of GFRC surfaces to resin-matrix cements. In previous studies, promising results were also reported on Er:YAG lasers or diode lasers at 980-nm wavelength [13] [54,55]. A previous study also performed laser-texturing of GFRC posts using Er,Cr:YSGG (2780-nm wavelength) at 1.5 or 2 W power and SEM analysis revealed the removal of outer epoxy matrix uncovering glass fibers without damage [56]. On the other hand, some previous studies have claimed that laser irradiation might not improve the push out bond strength of GFRC posts to resin-matrix cements [9,53]. Nevertheless, different laser settings should be evaluated on the surface modification of GFRC posts including laser type, wavelength, power, emission mode, pulse energy, frequency, pulse duration, air/water spray cooling [4,26,28,57]. Also, recent commercially glass fiber-reinforced composite posts should be assessed regarding the surface modification on traditional and laser-assisted methods to enhance the bond strength of their surfaces to resin-matrix cements. Future studies can support the development of clinical guidelines for using laser-assisted pathways in combination with traditional surface modification methods. Indeed, laser-assisted approaches have

increasingly been developed that can become a feasible approach in clinical practice including the modification of prosthetic surfaces for enhanced adhesion to resin-matrix cements.

5. Conclusions

The partial dissolution of the epoxy matrix on glass fiber-reinforced composite posts was achieved by traditional methods such as immersion in 35% hydrogen peroxide solution for 1 min or grit-blasting with alumina particle. However, etching in 9.7% hydrofluoric acid solution did not reveal any surface change and the neither the combination between hydrofluoric acid and hydrogen peroxide solutions. The surface modification of glass fiber-reinforced composite posts deals with a partial removal of the outer epoxy matrix layer exposing the glass fibers. Also, the partial removal of the outer epoxy matrix layer was successfully achieved using a novel laser-assisted approach with Nd:YAG laser on medium intensity level. The glass fibers were uncovered from the epoxy matrix without damage such as micro-cracks or fracture. Additionally, the combination of the laser-texturing and surface conditioning silane immersion in hydrogen peroxide promoted a high adhesion of glass fiber-reinforced composite posts to resin-matrix cements. Such combined approach between traditional and novel technological methods can be recommended to increase the retention of glass fiber-reinforced composite posts into endodontically treated teeth.

Conflict of interest: The authors declare no conflict of interest.

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Declarations

Ethics approval and consent to participate: All procedures performed involving human participants followed the ethics standards of the research committee of the University Institute of Health Sciences (IUCS) at Cooperativa Ensino Superior Politécnico Universitário (CESPU), Portugal, and therefore with the 1964 Helsinki declaration and its later amendments or comparable ethics standards. The project for the present study was previously reviewed and approved by the IUCS Ethics committee with the following Ethics Committee Reference number: CE/IUCS/CESPU-18/2022. Informed consent was unnecessary following the national regulations and since all data were processed anonymously. The need for informed consent was waived by the ethics committee/Institutional Review Board of IUCS at CESPU, Portugal.

Consent for publication: Not applicable.

Availability of data and materials: All data generated or analyzed during this study are included in this published article. The datasets used and/or analyzed during the current study available from the corresponding author on reasonable request.

Code Availability: Not applicable.

Authors' contributions: Conceptualization, J.C.M.S.; B.H.; V.F.; methodology, V.F.; O.C., and J.C.M.S.; investigation, V.F.; O.C.; writing—original draft preparation, V.F.; writing—review and editing, J.C.M.S.; M.O.; F.S.. Supervision, F.S., J.C.M.S., and M.O. All authors participated in the writing process, read and approved the final manuscript.

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Clinical Relevance

Combining traditional and novel physicochemical approaches can provide promising adhesion pathways for glass fiber-reinforced composite posts to resin-matrix cements. A stable retention of teeth root intracanal posts can decrease the risks of clinical failures by fracture of the adhesive interface.

Declaration of interests

☐ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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