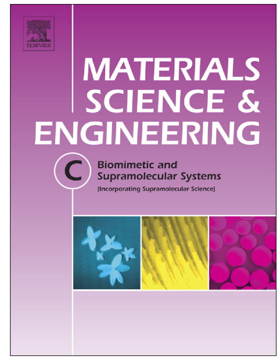

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Maíra Prado, Juliana N. Marques, Gisele Damiana Pereira, Eduardo M. da Silva, Renata A. Simão

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Evaluation of different surface treatments on fiber post cemented with a self-adhesive system

Maíra Prado[1], Juliana N. Marques[1,2], Gisele Damiana Pereira[2], Eduardo M. da Silva[3], Renata A. Simão [1]

[1] Department of Metallurgic and Materials Engineering, Federal University of Rio de Janeiro – UFRJ, Rio de Janeiro, RJ, Brazil.

[2] Department of Clinical Dentistry, Endodontic Division, Federal University of Rio de Janeiro – UFRJ, Rio de Janeiro, RJ, Brazil.

[3] Analytical Laboratory of Restorative Biomaterials – LABiom-R, School of Dentistry, Federal Fluminense University. Niterói, RJ, Brazil.

*Corresponding author: Dr Maíra do Prado

Cidade Universitária - Centro de Tecnologia – Bloco F, sala F-201- Ilha do Fundão – Rio de Janeiro, RJ - CEP 21941-972 – Brazil

Phone/ Fax: 0055 (21) 3938- 8526

E-mail: maira@metalmat.ufrj.br

ABSTRACT

Surface treatment of fiber-reinforced posts can increase adhesion, especially on the post/resin cement interface. The purpose of this in vitro study was to evaluate the effect of different surface treatments on fiber post cemented with a self-adhesive system. Sixty fiberglass epoxy resin posts were cleaned, dried and divided into 6 groups (n = 10): Control (no surface treatment), silane (silane coupling agent was applied homogeneously on surface), 24% hydrogen peroxide (H_2O_2) (immersion during 1 min), blasting (blasting with aluminum oxide for 30 sec), NH_3 plasma (plasma treatment for 3 min) and HMDSO plasma (plasma treatment for 15 min). After the treatments, posts were inserted into a silicon matrix that was filled with the resin cement RelyX U200. Afterwards, the post/cement specimens were cut perpendicularly to the long axis of the posts into six 1.0 mm thick discs and submitted to a push-out bond strength (POBS) test. Failure pattern was classified in 5 types: Type I: Cohesive in post; Type II: Cohesive in cement; Type III: Cohesive post and cement; Type IV: Adhesive post/cement; and Type V: Mixed (association between cohesive and adhesive). Data were analyzed by one-way ANOVA and Tukey HSD' post hoc test ($\alpha = 0.05$). Silane (15.94 ± 6.5), blasting (13.13 ± 3.6), NH_3 plasma (14.44 ± 4.0) and HMDSO plasma (13.23± 5.3) showed higher POBS when compared to control ($p < 0.05$) and similar among them. H_2O_2 (9.40 ± 4.0) treatment showed POBS values statistically similar to control (9.65 ± 3.6). Failures were predominantly cohesive post and cement, type III, in all groups. In conclusion, surface treatments influenced in the adhesion of fiberglass post with the self-adhesive cement RelyX U200. Silane, blasting with aluminum oxide and plasmas (NH_3 and HMDSO) showed results superior to 24% hydrogen peroxide.

Key words: blasting; bond strength; hydrogen peroxide; plasma; silane.

1. Introduction

For the restoration of endodontically treated teeth, the use of fiber posts luted with resin cement and combined with composite core build-up materials is becoming very frequent [1].

Self-adhesive cement was developed a decade ago, with the purpose of simplifying the cementation process by assembling all the components into a single product. This combination has resulted in a material that self-adheres to dentin, does not require pretreatment of the surface of the tooth, is simple to implement, and which application can be performed in a single step. Given that the removal of the smear layer is not recommended with most self-adhesive cements, there is increased tolerance to moisture and the release of fluoride ions [2-5].

An important aspect of adhesive procedure for fiber post cementation is that two interfaces are involved, namely, resin cement/root dentin and fiber post/resin cement. The adhesion in both interfaces is crucial for the long-term success of restoration and to the endodontic treatment [6]. Regarding the first interface (root dentine/ resin cement), it has been widely investigated using conventional and self-adhesive cements [5,7,8].

Several surface post modifications, including chemical and mechanical treatments, have been proposed to improve adhesion in the fiber post/resin cement interface [9]. Surface treatments include sandblasting using Al_2O_3 particles [10], etching with acidic solution [10,11], hydrogen peroxide [12], silane coupling agents [10,11,13], plasma irradiation [11,14], Er:YAG laser irradiation [15], and ultraviolet irradiation [6].

Concerning the association between fiber posts and the self-adhesive resin cement, fewer studies evaluated this interaction [6,11,16,17]. Regarding RelyX U200, Reza et al. [6] evaluated the influence of ultraviolet irradiation on fiber post surface. The influence of conventional (silane, peroxide and blasting) and plasma treatments on adhesion of fiber post to this resin cement is unknown. This way, the aim of the present study was to evaluate the effect of different surface treatments on fiber post cemented with a self-adhesive system. The null hypothesis tested was that surface treatments, including sandblasting treatment using Al_2O_3 particles, hydrogen peroxide, silane coupling agent and ammonia (NH_3) and hexamethyldisiloxane (HMDSO) plasmas had no influence in the bond strength of fiber posts to the self-adhesive resin cement RelyX U200.

2. Materials and Methods

2.1 Push-out bond strength analysis

2.1.1 Sample preparation

Sixty fiber epoxy resin posts (White Post DC3, FGM, Joinville, SC, Brazil) of 2 mm in diameter and 20 mm in length were used. Posts were submitted to an ultrasonic bath for 10 min in 70% alcohol so that any superficial contaminant would be removed. Afterwards, the posts were divided into 6 groups (n = 10) according to the surface treatment:

Control group: no surface treatment was applied to the fiber posts.

Silane group: a thin layer of silane (Prosil, FGM, Joinville, SC, Brazil) was applied homogeneously on fiber post surface using a microapplicator (Cavibrush, FGM, Joinville, SC, Brazil), waiting 1 minute. Then, the surface was dried with air jet for 5 seconds.

H_2O_2 group: fiber posts were immersed in 24% hydrogen peroxide at room temperature for 1 min, rinsed with 10 mL of distilled water and dried with air jet.

Blasting group: fiber posts were sandblasted with 50 µm aluminum oxide particles (Microetcher II; Danville Engineering, San Ramon, CA, USA) for 30 sec at a distance of 20 mm perpendicular to the post surface at the pressure of 0.4 MPa. Afterwards, the fiber posts were rinsed with 10 mL of distilled water and dried with air jet.

HMDSO and NH_3 groups: Plasma treatment was performed on the cathode of a diode glow-discharge plasma reactor operating at 13.56 MHz. The vacuum chamber was pumped down to 0.1 Pa, and monomer vapor or gas was allowed to fill the reactor up to 15 Pa. Surfaces were then modified using (i) HMDSO (Sigma Chemicals by Tedia, Rio de Janeiro, Brazil) for 15 min and (ii) NH_3 (Sigma Chemicals by Tedia, Rio de Janeiro, Brazil) for 3 min. Both surface modifications were accomplished at -280 V self-bias voltage (VB). At the end of the process, radiofrequency was turned off and the system allowed to cool down before exposure of the samples to air.

After the treatments, the cylindrical portion of fiber posts for each group were inserted into a silicone matrix (10 mm height and 6mm of internal diameter), positioned upon a transparent adhesive tape, with their upper cylindrical faces positioned in the center of the matrix (n=10). Then, the silicon matrix was fully filled with the resin cement (RelyX U200, 3M ESPE, St. Paul, MN, USA) in order to build up a core around the fiber post. The resin cement was cured for 40 sec with an irradiance of 500 mW/cm^2 (Optilight LD MAX, Gnatus, Ribeirão Preto, SP, Brazil) in four positions spaced of 90˚ in the silicone matrix diameter and through the top of the fiber posts.

2.1.2 Push-out bond strength test

After the matrix was removed, the resin cement-fiber post blocks were cut perpendicularly to the long axis of the posts into 1.0 mm thick discs using a diamond saw under water cooling (Isomet 1000, Buëhler, Lake Bluff, IL, USA). The first and the last slice of each block were discarded. A total of four discs were analyzed per sample, totaling 40 discs per group. The exact thickness of each disc was checked with a digital caliper (MPI/E-101, Mytutoyo, Tokyo, Japan).

Bond strength was evaluated through push-out test by using a universal testing machine (DL 1000, Emic, São José dos Pinhais, PR, Brazil), with a 200-kgf load cell, at a crosshead speed of 0.5 mm/min. A cylindrical steel post with a diameter of 1.2-mm diameter was used to test the specimens. Fig. 1 summarizes the steps taken for the setup.

To express the bond strength for each specimen (MPa), the failure load (N) was divided by the area A (mm^2) of the bonded interface. It was calculated as follows: $A = 2\ \pi rh$, where $\pi = 3.14$, r is the post radius, and h is the thickness of the disc in mm.

The fractured specimens were analyzed under a stereomicroscope (SMZ800, Nikon Instruments, São Paulo, SP, Brazil) and the failure pattern was classified in 5 types: Type I: Cohesive in post; Type II: Cohesive in cement; Type III: Cohesive post and cement; Type IV: Adhesive post/cement; and Type V: Mixed (association between cohesive and adhesive).

2.1.3 Statistical analysis

Kolmogorov-Smirnov as well as Levene tests have been used to evaluate normality and heteroscedasticity in the data. The data were then analyzed statistically using one-way ANOVA and the Tukey HSD post hoc test for pairwise multiple comparisons ($\alpha = 0.05$). IBM SPSS version 15.0 was used to perform the analyses.

2.2 Chemical and topographical analyses

Eighteen posts were selected and used in this phase. They were cleaned as described previously and divided into 6 groups (n = 3) according to the surface treatment showed in Table I. The same posts were used for chemical and topographical analyses.

2.2.1 FTIR Analysis

Fourier transform infrared spectroscopy (FTIR) was used to analyze the chemical modifications on fiber post surface after treatments. Infrared analysis was performed on a Nicolet 6700 spectrometer (Thermo Scientific, Waltham, MA, USA) in ATR mode. All spectra were acquired in absorbance mode in the 650-4000 cm^{-1} range. The total intensity of the peaks were normalized in 100%, for comparison of the relative intensity of the bands in each spectrum.

2.2.2 Topographical analysis

Scanning electron microscopy was used to evaluate qualitatively the effect of treatments on post surface. The specimens were mounted on stubs, sputter-coated with Au-Pd and analyzed at x500 (JSM 6460 LV (JEOL, Tokyo, Japan). Five SEM micrographs were obtained for each specimen.

3. Results

3.1 Push-out bond strength results

Statistical analysis showed difference among treatments (ANOVA; $F = 12.73$; $p < 0.001$). Table II shows mean, median and standard deviation POBS values. Descriptive statistics for all experimental groups is described in Table III. Regarding the surface treatments evaluated, silane, blasting and plasmas treatments showed higher POBS when compared to control ($p < 0.05$) and similar among them ($p > 0.05$). H_2O_2 showed POBS values statistically similar to control ($p > 0.05$). Regarding failure pattern, Fig. 2 shows representative images of the groups. Failures were predominantly cohesive post and cement, type III, in all groups (Fig. 3).

3.2 Chemical analysis

Fig. 4 shows the FTIR spectra of control group (fiber post without treatment) and the peaks of interest evaluated. They were: C=O Ketone (1715 cm^{-1}), C=C Aliphatic (1638 cm^{-1}), C=C Aromatic (1608 cm^{-1}), C-H in aromatic ring (between 1595 and 1472 cm^{-1}), CH_2 /CH_3 (1450 cm^{-1}), CH_3 in Si-$(CH_3)_x$ (1230 cm^{-1}), siloxane bonds (–Si–O–Si–O–) (between 1230 and 1100 cm^{-1}), C-O-C oxirane/ SiOH (824 cm^{-1}). The peaks intensity of experimental groups was evaluated in relation to control (Table IV). In the silane group, there is an improvement in the peaks 1230-1100 cm^{-1} and 824 cm^{-1} correspondents to –Si–O–Si–O–and C-O-C oxirane or SiOH bonds. In H_2O_2 group, there is a reduction in the Si-$(CH_3)_x$ bond (1230 cm^{-1}) due to the removal of epoxy resin in the interface with fiber. It was also observed for the blasting group, where a reduction of C=C Aromatic (1608 cm^{-1}) was also observed due to the epoxy resin removal on post surface.

For ammonia plasma, a clear reduction of the C=C aliphatic bonds was observed. The improvement in the 1595 - 1472 cm^{-1} region can be associated with the stretch of ammide II (1516 cm^{-1}). For HMDSO plasma, there is an increase of C=C (1608 cm^{-1}), C-H in aromatic ring (1472-1595 cm^{-1}) peak and C-O-C oxirane/ SiOH (824 cm^{-1}).

3.3 Topographical results

Fig. 5 shows representative photomicrographs of fiber posts surface of the groups evaluated. Control, silane and HMDSO groups evidenced similar topography, with slightly removal of epoxy resin after treatment. Blasting and H_2O_2 showed the degradation of the epoxy resin matrix and exposed fibers with no apparent fiber damage. NH_3 group presented smoother surfaces without remarkable change in the fiber exposition.

4. Discussion

Although the bonding between the fiber post and root canal dentin plays a pivotal role in the long-term success of a restoration, reliable bonding between the post and resin cement is also indispensable [14]. If the bonding of this interface is poor, debonding and/or fracture can occur. Therefore, good adaptation and reliable bonding between the post surface and resin cement must be achieved. To improve the adhesion of this interface, different superficial treatments are proposed on the fiber post surface [6,9-15]. The present study evaluated the effect of different surface treatments in the adhesion of fiber posts to the self-adhesive resin cement RelyX U200.

The null hypothesis tested was rejected. The different surface treatments had influenced in the bond strength of fiber posts to the RelyX U200 cement. Silane, blasting and plasmas showed results superior to control and H_2O_2 groups. Control group presented the lower bond strength values, statistically similar with 24% H_2O_2 group.

The positive results regarding silane can be associated with the chemical bond between the core resin matrix and the exposed glass fibers of the post. Also, silane is known to increase surface wettability resulting in chemical bridges formation with OH-covered substrates (fiberglass) [16-18]. FTIR analysis showed an improvement in the –Si–O–Si–O– bond (peak 1230 - 1100 cm^{-1}) and SiOH bond (peak 824 cm^{-1}). The improvement in the siloxane bonds can be associated with the positive results of bond strength found here. Previous studies evaluated the effect of silane in the adhesion of fiber posts luted with the self-adhesive resin cement RelyX U100, a precursor of the cement used in the present study. In this aspect, Leme et al. [19] and Zicari et al. [16] observed that the use of silane for post treatment improved the adhesion of post to RelyX U100. However, Mazzitelli et al. [20] did not found positive results when Silicate/silane coating was used in association with RelyX U100.

Positive results of blasting group can be associated with the surface created. Blasting removed a surface layer of the epoxy resin of fiber post without fiber damage [12,16,21], as observed in Fig. 5. It creates a larger surface area, associated with the exposed glass fibers, which provide additional sites for micromechanical retention of the resin cement [22,23]. No study evaluated the blasting effect on fiber post in association with the self-adhesive cement RelyX U200. However, Zicari et al. [16] associated blasting with silane, using RelyX U100, and observed favorable results. Also, Senyilmaz et al. [24] observed that the blasting pre-treatment of a zirconium-based ceramic improved the bond strength of the resin cements RelyX U100.

For plasma treatments, two substances were used. NH_3 plasma has been proposed to chemically change the surface, including amino grouping, and producing a hydrophilic surface to improve the adhesion process [25,26]. HMDSO has low toxicity, easy manipulation, and creates a hydrophobic surface [27]. The positive results for the plasma groups can be associated with chemical modifications observed in the FTIR analysis. Costa Dantas el al. [11], evaluating the self-adhesive cement RelyX U100, and Yavirach et al. [14], evaluating a conventional cement, observed positive results of plasma on adhesion of fiber post to resin cement. However, the gases used, as well as the cement, were different from the present study.

H_2O_2 treatment removes the epoxy resin layer of post exposing the fibers [28]. It can be clearly observed in the topographical analysis (Fig. 5). The inferior results of H_2O_2 when compared with other treatments can be due to morphology of surface and the interaction of H_2O_2 with resin cement. Regarding chemical analysis, both blasting as well as H_2O_2 led to the removal of the epoxy resin on the post surface. However, different from blasting, H_2O_2 treatment acts also on the interface, reducing the Si-$(CH_3)_x$ bond (1250 cm^{-1}), an interfacial bond, that link the fiber with epoxy resin.

Regarding the method, in the present study the authors opted to evaluate only the post/cement interface. This option was based in previous studies [11,29,30] that verified a predominance of adhesive failure patterns between dentin and cement. If the two interfaces were evaluated simultaneously, probably the failure would occur in the dentin/ cement interface. It would be therefore impossible to evaluate the cement / post interface.

5. Conclusion

Within the limitations of this in vitro study of the bond strength between the post system and the resin cement RelyX U200, the following conclusions were drawn:

1. Surface treatments influenced the adhesion of fiber post to cement.

2. Silane, blasting and plasmas (NH_3 and HMDSO) showed results superior to 24% hydrogen peroxide.

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FIGURE LEGENDS

Fig. 1. The setup of push-out bond strength steps.

Fig. 2. Representative images of failure partner: control group - type III (A); group - type III (B); H_2O_2 group - type III (C); Blasting group - type II (D), type III (E), type V (F); NH_3 group - type III (G), type V(H); HMDSO group type III (I), type IV(J).

Fig. 3. Failure partner (%) of the groups evaluated.

Fig. 4 FTIR spectra and the peaks of interest evaluated.

Fig. 5 Representative micrographs of fiber posts surface: (A) Control, (B) silane, (C) H_2O_2, (D) Blasting, (E) NH_3, and (F) HMDSO.

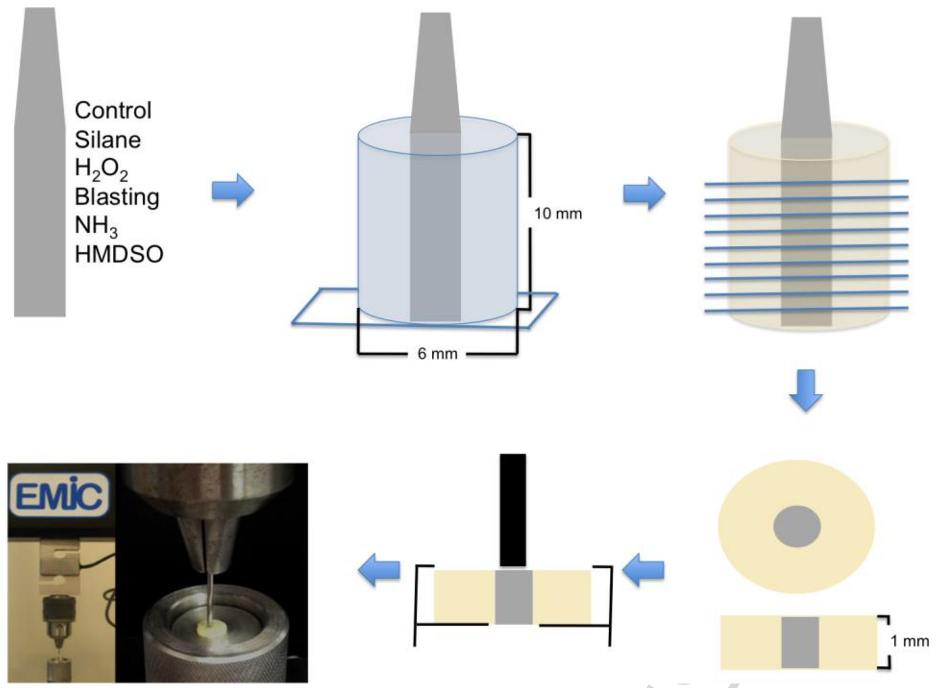


Figure 1

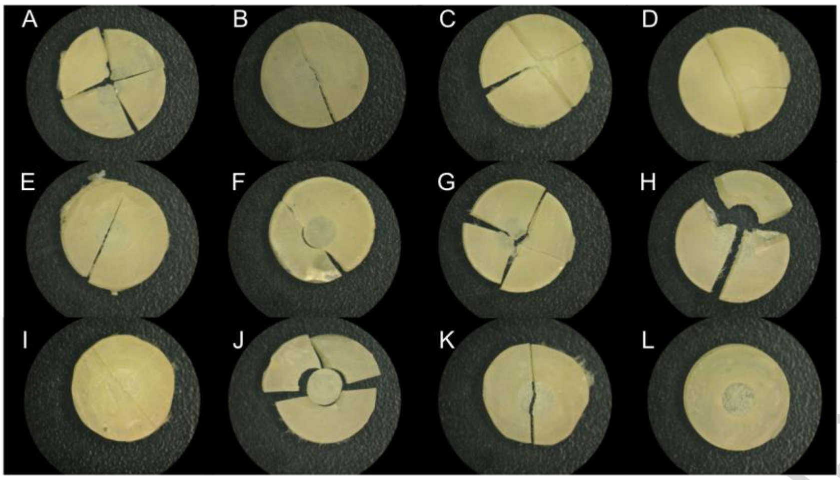
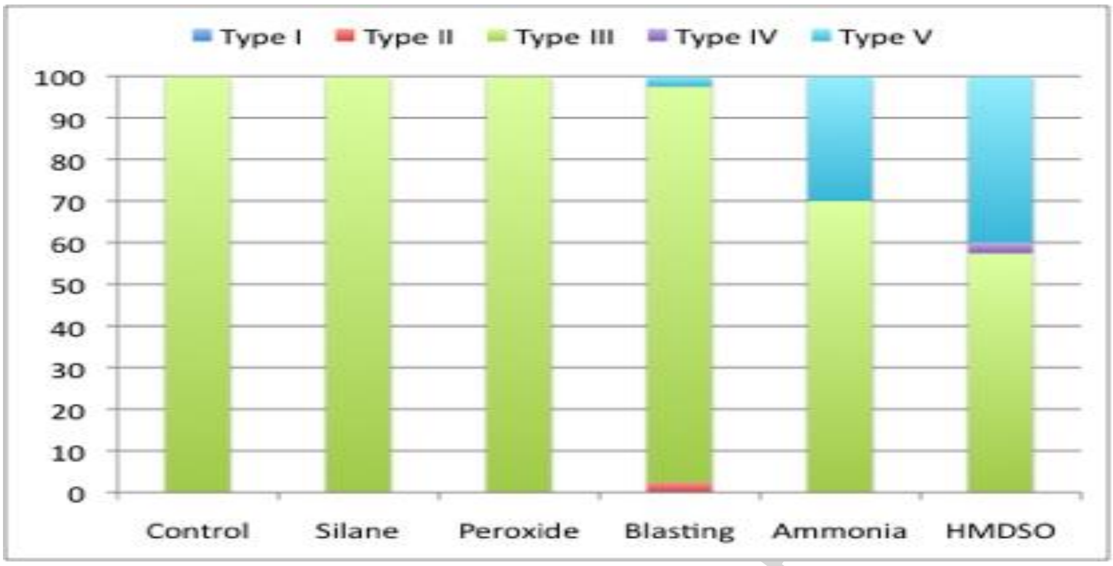


Figure 2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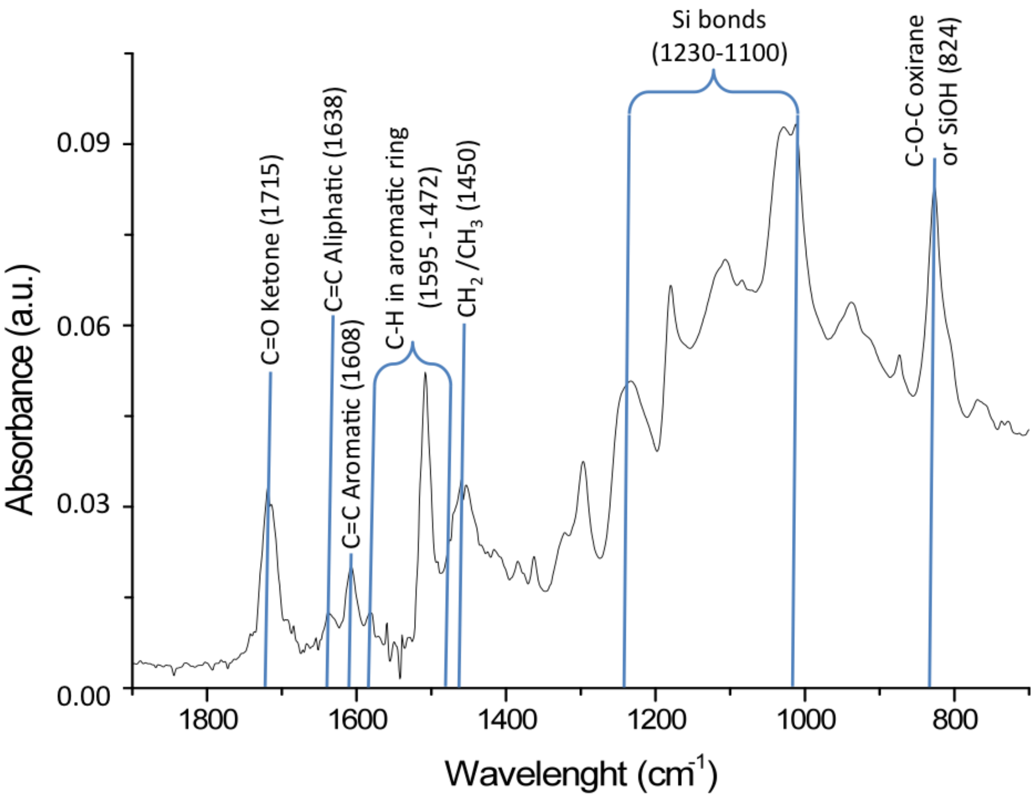
Blasting
Ammonia
HMDSO

Figure 3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C-O-C oxirane
or SiOH (824)
C=O Ketone (1715)
C=C Aliphatic (1638)
C=C Aromatic (1608)
C-H in aromatic ring
(1595 -1472)
CH2 /CH3 (1450)
0.09
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Absorbance (a.u.)
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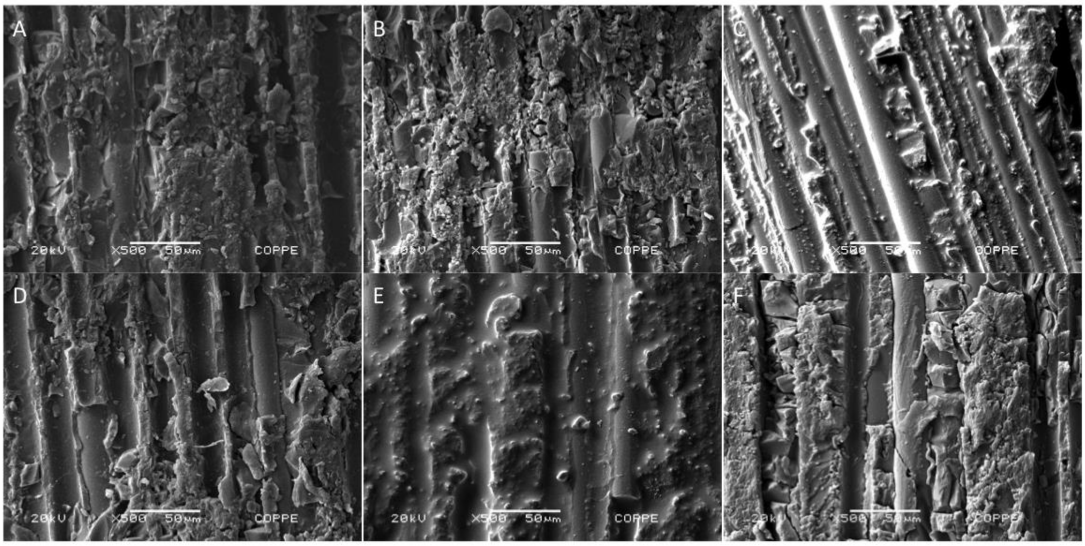
1200
1000
800
Wavelenght (cm-1)

Figure 4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20kV X500 50μm COPPE
E
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F
20kV X500 50μm COPPE

Figure 5

Table I. Treatments on fiber post surface.

Groups	Treatment
Control	Control - no surface treatment
Silane	silane coupling agent was applied homogeneously on surface
H_2O_2	24% hydrogen peroxide for 1min
Blasting	Blasting with aluminum oxide for 30 s
NH_3	NH_3 plasma treatment for 3 min
HMDSO	HMDSO plasma treatment for 15 min

Table II. Mean, median and standard deviation values (MPa) of the different groups.

Groups	Mean	Median	Std. Deviation
Control	9.648415[b]	8.589172	3.6378658
Silane	15.935862[a]	14.267902	6.5315797
H_2O_2	9.400132[b]	8.190005	3.9823654
Blasting	13.133998[a]	13.295602	3.5748676
NH_3	14.441980[a]	14.518392	4.0386371
HMDSO	13.235348[a]	11.455415	5.2826789

[a,b]Different letters indicate statistically significant differences (Tukey HSD test; $p < 0.05$).

Table III. Descriptive statistics for all groups.

Multiple Comparisons

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Control	Silane	-6.2874464*	1.1821125	.000	-9.889089	-2.685804
	Peroxide	.2482829	.8528379	1.000	-2.328137	2.824703
	Blasting	-3.4855826*	.8064389	.001	-5.921230	-1.049935
	NH_3	-4.7935648*	.8594280	.000	-7.390114	-2.197015
	HMDSO	-3.5869329*	1.0141593	.011	-6.662229	-.511637
Silane	Control	6.2874464*	1.1821125	.000	2.685804	9.889089
	Peroxide	6.5357293*	1.2095533	.000	2.858342	10.213117
	Blasting	2.8018639	1.1772979	.267	-.786626	6.390354
	NH_3	1.4938816	1.2142088	.977	-2.196481	5.184244
	HMDSO	2.7005136	1.3282341	.503	-1.316576	6.717603
Peroxide	Control	-.2482829	.8528379	1.000	-2.824703	2.328137
	Silane	-6.5357293*	1.2095533	.000	-10.213117	-2.858342
	Blasting	-3.7338654*	.8461518	.000	-6.290362	-1.177369
	NH_3	-5.0418477*	.8967974	.000	-7.750392	-2.333304
	HMDSO	-3.8352158*	1.0460154	.007	-7.001967	-.668465
Blasting	Control	3.4855826*	.8064389	.001	1.049935	5.921230
	Silane	-2.8018639	1.1772979	.267	-6.390354	.786626
	Peroxide	3.7338654*	.8461518	.000	1.177369	6.290362
	NH_3	-1.3079823	.8527935	.874	-3.884803	1.268838
	HMDSO	-.1013503	1.0085432	1.000	-3.160682	2.957982
NH_3	Control	4.7935648*	.8594280	.000	2.197015	7.390114
	Silane	-1.4938816	1.2142088	.977	-5.184244	2.196481
	Peroxide	5.0418477*	.8967974	.000	2.333304	7.750392
	Blasting	1.3079823	.8527935	.874	-1.268838	3.884803
	HMDSO	1.2066320	1.0513953	.988	-1.975704	4.388968
HMDSO	Control	3.5869329*	1.0141593	.011	.511637	6.662229
	Silane	-2.7005136	1.3282341	.503	-6.717603	1.316576
	Peroxide	3.8352158*	1.0460154	.007	.668465	7.001967
	Blasting	.1013503	1.0085432	1.000	-2.957982	3.160682
	NH_3	-1.2066320	1.0513953	.988	-4.388968	1.975704

*. The mean difference is significant at the 0.05 level.

Table IV. Intensity of absorbance in the different functional groups in relation to control.

	C=O Ketone (1715 cm^{-1})	C=C Aliphatic (1638 cm^{-1})	C=C Aromatic (1608 cm^{-1})	C-H in aromatic ring (1595 – 1472 cm^{-1})	CH_2 /CH_3 (1450 cm^{-1})	Si-$(CH_3)_x$ (1230 cm^{-1})	Siloxane bonds –Si–O–Si–O– (1230 - 1100 cm^{-1})	C-O-C oxirane/ SiOH (824 cm^{-1})
Silane	0.95	1.05	1.06	1.04	0.97	0.84	1.04	1.90
H_2O_2	0.96	1.09	1.04	1.02	1.04	0.80	1.08	1.00
Blasting	0.98	1.02	0.75	1.04	1.02	0.85	1.06	0.94
NH_3	0.73	0.41	1.02	1.38	0.77	1.60	1.00	1.65
HMDSO	0.74	0.77	1.53	1.39	0.85	1.68	1.00	1.56

Highlights

- The present study evaluated the effect of different surface treatments on fiber post cemented with a self-adhesive system.
- Surface treatments influenced in the adhesion of fiberglass post with the self-adhesive cement RelyX U200.
- Silane, blasting with aluminum oxide and plasmas (NH_3 and HMDSO) showed results superior to 24% hydrogen peroxide.