

The effects of non-thermal plasma and conventional treatments on the bond strength of fiber posts to resin cement

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Objectives: This study compared the effect of hexamethyldisiloxane (HMDSO) and ammonia (NH₃) plasmas on the bond strength of resin cement to fiber posts with conventional treatments. **Materials and Methods:** Sixty-five fiber posts were divided into 5 groups: Control (no surface treatment); H₂O₂ (24% hydrogen peroxide for 1 min); Blasting (blasting with aluminum oxide for 30 sec); NH₃ (NH₃ plasma treatment for 3 min); HMDSO (HMDSO plasma treatment for 15 min). After the treatments, the Ambar adhesive (FGM Dental Products) was applied to the post surface ($n = 10$). The fiber post was inserted into a silicon matrix that was filled with the conventional resin cement Allcem Core (FGM). Afterwards, the post/cement specimens were cut into discs and subjected to a push-out bond strength (POBS) test. Additionally, 3 posts in each group were evaluated using scanning electron microscopy. The POBS data were analyzed by one-way analysis of variance and the Tukey's honest significant difference *post hoc* test ($\alpha = 0.05$). **Results:** The Blasting and NH₃ groups showed the highest POBS values. The HMDSO group showed intermediate POBS values, whereas the Control and H₂O₂ groups showed the lowest POBS values. **Conclusion:** Blasting and NH₃ plasma treatments were associated with stronger bonding of the conventional resin cement Allcem to fiber posts, in a procedure in which the Ambar adhesive was used. (*Restor Dent Endod* 2017;42(2):125-133)

Key words: Bonding; Fiber post; Plasma gas; Surface treatment

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Introduction

Aiming to prevent vertical root fractures and coronal microleakage, modern endodontics advocates a conservative preparation followed by immediate adhesive restoration of the teeth. In endodontic practice, the use of fiber posts has gained popularity and is perceived as a promising alternative to cast posts.¹⁻³ Among other advantages, fiber posts have a similar modulus of elasticity to dentin, excellent aesthetic behavior, and the ability to form a bond using an adhesive protocol, thereby creating the so-called 'monobloc' (dentin adhered to cement that has adhered to the fiber post).⁴⁻⁶ This restorative adhesive technique plays an essential role in the long-term success of the restoration and endodontic treatment. Thus, reliable bonding among the fiber post, resin cement, and dentin should be regarded as crucial to the success of the restoration of endodontically treated teeth.⁴

Due to differences in composition, a poor chemical bond is expected between the fiber post and the resin cement that is used to adhere it to the surrounding dentin

inside the root canal. To overcome this problem, several surface modifications of the fiber post, including chemical and mechanical treatments, have been proposed. Among them, silanization, hydrofluoric acid and phosphoric acid etching, hydrogen peroxide application, and blasting with aluminum oxide particles are well documented.⁷⁻¹² Recently, however, plasma technology has also been used for this purpose.^{4,13}

Plasma treatment is an effective and clean technology, since the bulk properties of the materials may remain unaltered or maintained well after the treatment. It consists of partially ionized gases that contain highly reactive particles, including electronically excited atoms, molecules, ions, and free radical species. Depending on the plasma chemistry or gas composition, these highly reactive plasma species can react with clean and etched surface materials, bond to various substrates, combine to form a thin layer of plasma coating, and consequently alter the characteristics of a surface.^{14,15} This technology can improve the hydrophilicity or hydrophobicity and surface free energy of a substrate, thereby increasing its reactivity.¹³⁻¹⁷ Hexamethyldisiloxane (HMDSO) plasma is commonly used in the treatment of metals for dental implants, due to its anti-corrosive behavior.¹⁸⁻²⁰ Additionally, HMDSO-coated titanium surfaces improve the degree of cell activity at the implant-tissue interface.¹⁸ HMDSO plasma presents additional advantages for industrial applications, including low toxicity, easy manipulation, and commercial availability.^{19,20} Nevertheless, HMDSO treatment renders a surface hydrophobic.¹⁶ Contrarily, ammonia (NH₃) plasma has been employed in the field of materials science and engineering to improve surface hydrophilicity, adhesion, and the durability of bond strength.^{21,22} In addition, this treatment can promote cell attachment and growth.²³

The aim of the present study was to compare the effect of HMDSO and NH₃ plasmas on the bond strength of resin cement to the fiber post with conventional surface treatments. The null hypothesis tested was that these treatments had no influence on the bond strength of resin cement to fiber posts.

Materials and Methods

Sample preparation

Sixty-five epoxy resin fiber posts (White Post DC3, FGM, Joinville, SC, Brazil) 2 mm in diameter and 20 mm in length were used. Posts underwent an ultrasonic bath for 10 minutes in 70% alcohol so that any superficial contaminant would be removed.¹³ Afterwards, the posts were divided into 5 groups ($n = 13$) according to the surface treatment (Table 1).

Table 1. Fiber post surface treatments

Group	Treatment
Control	No surface treatment
H ₂ O ₂	24% hydrogen peroxide for 1 min
Blasting	Blasting with aluminum oxide for 30 sec
NH ₃	NH ₃ plasma treatment for 3 min
HMDSO	HMDSO plasma treatment for 15 min

HMDSO, hexamethyldisiloxane.

Conventional treatments

In the Control group, no surface treatment was applied to the fiber posts. For the H₂O₂ group, the fiber posts were immersed in 24% H₂O₂ at room temperature for 1 minute.⁹ In the blasting group, the fiber posts were sandblasted with 50 μm aluminum oxide particles (Microetcher II, Danville Engineering, San Ramon, CA, USA) for 30 seconds at a distance of 20 mm perpendicular to the post surface at a pressure of 0.4 MPa. Afterwards, in all groups, the fiber posts were rinsed with 10 mL of distilled water and air-dried.

Plasma treatments

Plasma treatment was performed by plasma-enhanced chemical vapor deposition (PECVD) using a glow-discharge reactor operating at 13.56 MHz. The vacuum chamber was pumped down to 0.1 Pa, and vaporized monomer gas was allowed to fill the reactor up to 15 Pa.¹³ Surfaces were modified using 2 different substances: (1) HMDSO for 15 minutes (Sigma-Aldrich, Rio de Janeiro, RJ, Brazil) and (2) NH₃ (Sigma-Aldrich) for 3 minutes. No other gas was allowed in the reactor during deposition. Both surface modifications were accomplished using a radiofrequency power supply and a self-bias of -280 V was obtained from the voltmeter (Figure 1).¹³ At the end of the process, the radiofrequency power supply was turned off and the system allowed to cool down before exposure of the samples to air.

Push-out bond strength test

The setup of this step is depicted in Figure 2. Immediately after the treatments, the Ambar adhesive (FGM Dental Products) was applied on the post surface and cured for 20 seconds with an irradiance of 500 mW/cm² (Optilight LD MAX, Gnatus, Ribeirão Preto, SP, Brazil). The cylindrical portions of 10 fiber posts for each group ($n = 10$) were

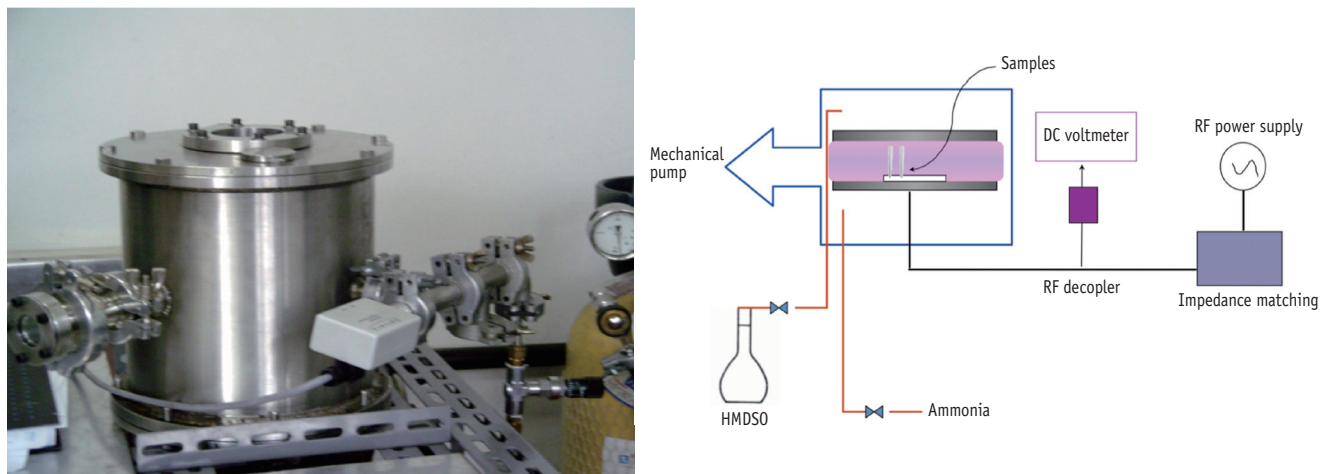


Figure 1. Plasma equipment and schematic drawing showing its components. DC, direct current; RF, radiofrequency; HMDSO, hexamethyldisiloxane.

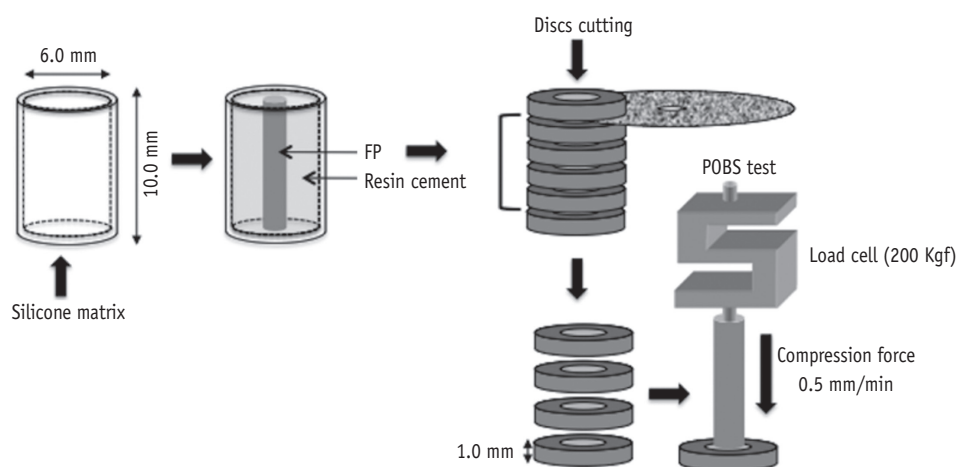


Figure 2. Schematic setup of the push-out bond strength test. FP, fiber post; POBS, push-out bond strength.

inserted into a silicone matrix (10 mm in height and 6 mm in internal diameter) and positioned on transparent adhesive tape, with the upper cylindrical face positioned in the center of the matrix. Afterwards, the silicon matrix was fully filled with conventional resin cement (Allcem Core, FGM) in order to build up a cylindrical sample of resin cement matrix around the fiber post. The resin cement was cured for 40 seconds with an irradiance of 500 mW/cm² in 4 positions spaced at 90° about the silicone matrix diameter and through the top of the fiber posts.

After the silicone mold was removed, the resin cement-fiber post blocks were cut perpendicular to the long axis

of the posts into six 1.0 mm thick discs using a diamond saw under water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA). The first and the last discs of each block were discarded. A total of 4 discs were analyzed per sample, totally 40 discs per group. The exact thickness of each disc was checked with a digital caliper (MPI/E-101, Mitutoyo, Tokyo, Japan).

Bond strength was evaluated with the push-out bond strength (POBS) test 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 was

used to test the specimens. To express the bond strength (MPa) for each specimen, the failure load (N) was divided by the area (A , mm²) of the bonded interface, which was calculated as follows: $A = 2\pi rh$, where r is the post radius, and h is the thickness of the disc in millimeters. The POBS data were analyzed using one-way analysis of variance (ANOVA) and Tukey's honest significant difference *post hoc* tests ($\alpha = 0.05$).

The fractured specimens were analyzed under a stereomicroscope (SMZ800, Nikon Instruments, São Paulo, SP, Brazil) equipped with the AxioVision 4.8 software (Carl Zeiss Microscopy GmbH, Oberkochen, Germany) and the failure patterns were classified as adhesive at the interface between the fiber post and resin cement, cohesive within resin cement, and mixed.

Topographical analysis

To evaluate qualitatively the effects of treatments on post surfaces, scanning electron microscopy (SEM, JSM 6460 LV, JEOL, Tokyo, Japan) was used. Fiber posts were evaluated before and after applying the adhesive. For adhesive application, 3 fiber posts of each group had half of their length treated with Ambar adhesive (FGM) and cured with a light-curing unit (Optilight LD MAX) employing an irradiance of 500 mW/cm² for 20 seconds. The specimens were then mounted on stubs, sputter-coated with gold-palladium alloy, and analyzed at $\times 80$ and $\times 500$ magnifications. Five SEM micrographs were obtained for each specimen (for portions with and without adhesive).

Fourier transform infrared spectroscopy analysis

In order to analyze the chemical modifications of the fiber post surface by the plasma treatment, Fourier transform infrared spectroscopy (FTIR) was used. Infrared analysis was performed on a Nicolet 6700 spectrometer (Thermo Scientific, Waltham, MA, USA) in the attenuated total reflectance mode, and all spectra were acquired in absorbance mode in the range of 650 - 4,000 cm⁻¹. Analyses with FTIR were performed before and after Ambar adhesive application. The absorbance peak of the aromatic C=C double bond located at 1,608 cm⁻¹ was used as the reference peak for both analyses (post surface and post/adhesive). The control samples (without plasma treatment) were compared with the samples after plasma deposition.

After the polymerization reaction of the adhesive, the aliphatic peak located at 1,638 cm⁻¹ was evaluated in relation to the aromatic double-bond peak. The degree of conversion (DC) of the plasma-treated sample was compared to that of the control group according to the equation: $DC = 1 - \left\{ \frac{[\text{abs}(\text{aliphatic C=C}) / \text{abs}(\text{aromatic C=C})]_{\text{plasma}}}{[\text{abs}(\text{aliphatic C=C}) / \text{abs}(\text{aromatic C=C})]_{\text{control}}} \right\} \times 100$.²⁴

Results

Table 2 shows the bond strength values and failure patterns of the groups evaluated. The Blasting group and the NH₃ group showed the highest POBS values. The HMDSO group showed an intermediate POBS value, whereas the lowest POBS values were found for the H₂O₂ and Control groups ($p < 0.05$). Most of the failures showed mixed failure patterns.

Figure 3 shows representative SEM micrographs of fiber posts for each experimental group before (a, b, c, d, and e) and after (a', b', c', d', and e') adhesive application: (a - a') Control; (b - b') H₂O₂; (c - c') Blasting; (d - d') NH₃; (e - e') HMDSO. Before the adhesive application, a visual inspection of the images demonstrated that the Control, Blasting, and HMDSO groups showed similarly rougher topography, characterized by the removal of the surface layer of the epoxy resin without fiber damage (red arrow). Contrarily, the H₂O₂ and NH₃ groups presented smoother surfaces without remarkable changes. After adhesive application, all groups showed a smooth appearance, suggesting that the adhesive monomers penetrated into the irregularities produced by the treatments.

Chemical modifications on the fiber post surface after plasma treatments were evaluated in relation to the control (fiber posts without plasma treatment). Table 3 shows the intensity of different functional groups normalized in relation to C=C in aromatic ring. For ammonia plasma, a clear reduction of the aliphatic C=C bond was observed. This reduction can be attributed to the better wetting of the adhesive to the post. Furthermore, peaks at 1,370 and 1,550 cm⁻¹ indicate the presence of symmetrical and asymmetrical stretch in the NO₂ bonded to either an alkyl or aromatic ring.²⁵ However, with HMDSO plasma the presence of peaks related to Si-O, Si-OH, and Si-C

Table 2. Push-out bond strength values (mean \pm standard deviation) and failure patterns of the different groups

Group	Push-out bond strength value (MPa)	Failure pattern (%)		
		Adhesive	Cohesive	Mixed
Control	9.41 \pm 2.6 ^{c*}	0	0	100
H ₂ O ₂	10.33 \pm 2.6 ^c	0	0	100
Blasting	13.71 \pm 3.9 ^a	0	0	100
NH ₃	12.35 \pm 2.9 ^{ab}	0	0	100
HMDSO	11.10 \pm 3.3 ^{bc}	10	0	90

HMDSO, hexamethyldisiloxane.

*Different letters indicate statistically significant differences (the Tukey honest significant difference *post hoc* test, $p < 0.05$).

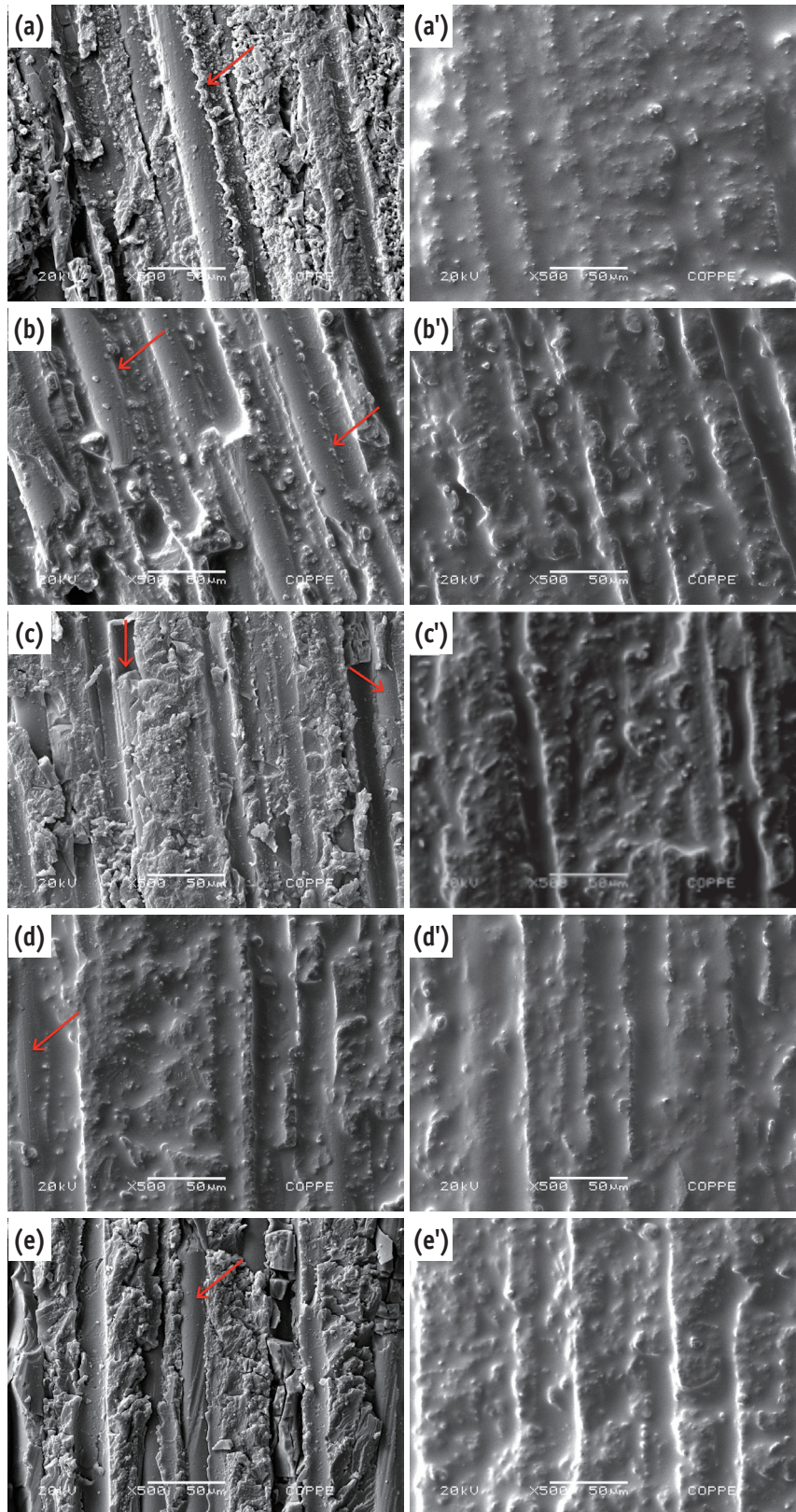


Figure 3. Photomicrography of the surface of fiber posts in different groups: (a) Control; (b) H_2O_2 ; (c) blasting; (d) NH_3 ; (e) HMDSO. Images (a - e) show the state before adhesive application and images (a' - e') after adhesive application. HMDSO, hexamethyldisiloxane.

Table 3. Intensity of different functional groups normalized to that of the aromatic C=C double bond

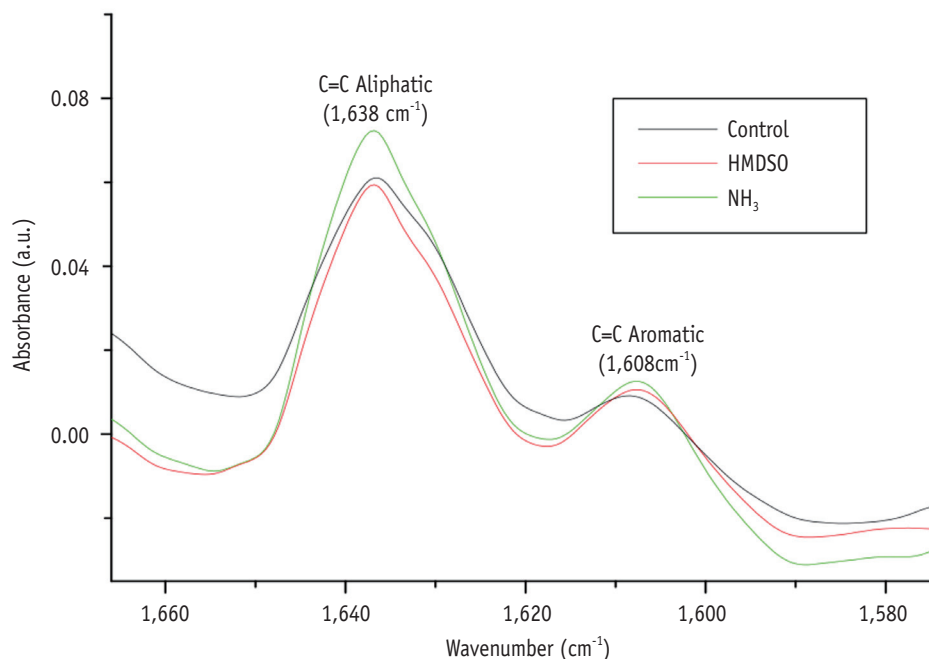
	C=O ketone (1,715 cm ⁻¹)	C=C aliphatic (1,638 cm ⁻¹)	C=C aromatic (1,608 cm ⁻¹)	C-C aromatic (1,514 cm ⁻¹)	CH ₂ /CH ₃ (1,450 cm ⁻¹)	C-O-C oxirane or SiOH (824 cm ⁻¹)
Control	12.87	1.08	1	2.94	6.27	3.60
Ammonia	6.18	0.29	1	2.82	3.49	3.69
HMDSO	9.20	0.80	1	3.99	4.74	5.79

HMDSO, hexamethyldisiloxane.

Table 4. Increase in the degree of conversion of the adhesive applied after plasma treatments

Treatment	I _{1,638 cm⁻¹} (Aliphatic)	I _{1,608 cm⁻¹} (Aromatic)	I _{Aliphatic} / I _{Aromatic}	Increase in the degree of conversion (%)
Control	0.871	0.144	6.1	-
Ammonia	1.183	0.254	4.7	23.0
HMDSO	1.002	0.226	4.4	28.0

HMDSO, hexamethyldisiloxane.

**Figure 4.** Fourier transform infrared spectroscopy spectra of the fiber post surface after adhesive application. HMDSO, hexamethyldisiloxane.

(800 - 950 cm⁻¹)²⁵ was difficult to distinguish, as there was a strong pattern of superposition in the resin peaks with a strong increase in peak intensity. After adhesive application, the DCs in the control and plasma groups were

evaluated. As can be observed in Table 4 and Figure 4, the HMDSO and ammonia plasma treatments improved the DC of the adhesive by 28 and 23%, respectively.

Discussion

The present study compared the effect of conventional and plasma treatment in the adhesion of resin cement to fiber posts associated with the use of an adhesive containing methacryloyloxydecyl dihydrogen phosphate (MDP). The MDP molecule has a hydrophobic alkylene group and a hydrophilic phosphate group to permit adhesion to hydrophilic and hydrophobic surfaces.²⁶

Plasma treatment modifies a surface with a substance, gas, or a combination of gases. It can be performed in high vacuum or atmospheric conditions (at low pressure).^{14,16,17,27} In dentistry, it has been proposed to increase the adhesion to dental substrate and composites,^{27,28} between polyethylene fiber and composite,^{14,17} and between fiber posts and resin cements.¹³ It has also been used in the treatment of metals for dental implants¹⁸ and disinfection of root canals.²⁹ Different substances are used to generate plasma, including argon, ethylene diamine, HMDSO, helium, and oxygen.^{13,14,17,18,27-29}

In the present study, an Ambar adhesive containing MDP was used. To evaluate the impact of the hydrophobic and hydrophilic groups of MDP on the adhesion between post and cement, ammonia and HMDSO were chosen to promote the hydrophilicity and hydrophobicity of the substrate surface, respectively.^{16,21,22}

Immersion in H₂O₂ and sandblasting were proposed in the present study as conventional treatments. According to de Sousa Menezes *et al.*⁹, the immersion of the post in 24% H₂O₂ for 1 minute generated fiber exposure without damage and an improvement of microtensile bond strength. Previous studies also reported that blasting treatment permitted the removal of epoxy resin (the superficial layer) without fiber damage and with the improvement of bond strength.^{10,12}

Regarding the bond strength analysis, the null hypothesis was rejected. The treatments influenced the bond strength of conventional resin cement associated with the use of adhesive containing MDP to the fiber post. The higher bond strength values were founded in the Blasting group and the NH₃ plasma group. The HMDSO group showed intermediate values, similar to those of the NH₃, 24% H₂O₂, and Control groups. The Control group showed lower values, statistically similar to those of the 24% H₂O₂ group.

Comparing the conventional treatments, the positive results regarding the Blasting group can be attributed to the morphology observed by SEM. In this group, a surface layer of the resin matrix of the fiber post was removed, but the remaining resin matrix was rougher than in other groups, as can be observed visually in the SEM images. Blasting can create a larger surface area associated with the exposed glass fibers, which provides additional sites for micromechanical retention of the adhesive material.^{7,11} The positive result of blasting was in accordance with previous

studies.^{11,12}

In the present study, the control group showed statistically similar results with 24% H₂O₂ group. It can be associated with the cleaning process of posts, with ultrasonic bath in 70% alcohol for 10 minutes, to remove superficial contaminant, proposed previously by Costa Dantas *et al.*¹³ This protocol removed partially the epoxy resin of post without fiber damage (Figure 1a, red arrow). This result differs from De Sousa Menezes *et al.*⁹ who verified an improvement of microtensile bond strength using 24% hydrogen peroxide for 1 minute. The differences in the results can be associated with the cleaning process of the posts used here.

Blasting and NH₃ plasma showed similar bond strength results with different topographies. In this case, micromechanical retention was predominant in the Blasting group in comparison with the NH₃ plasma group. NH₃ plasma promoted a mild etching on the surface, creating a smoother surface. The positive result in the NH₃ plasma group is associated with chemical modifications on the post surface and the improvement of the DC in the adhesive, as demonstrated in the present study by FTIR analysis. Additionally, previous studies report that NH₃ plasma changes the surface chemically, including amino groups, and producing a hydrophilic surface to improve the adhesion process.^{22,30}

Depending on the plasma chemistry or gas composition, highly reactive plasma species can react with surfaces in different ways. They can etch the surface, bond to substrates, or combine to form a thin layer of plasma coating.^{14,15} In the present study, different plasmas interacted differently on the same surface. HMDSO plasma resulted in no topographic modifications on the surface, while ammonia plasma resulted in mild etching on the surface, creating a smoother surface. Both plasmas led to chemical modifications at the surface, but only ammonia plasma was able to facilitate better bonding of the adhesive to the fiber post.

Conclusion

The present study showed that blasting and NH₃ plasma were associated with stronger bonding of the conventional resin cement Allcem to fiber posts, in a procedure in which Ambar adhesive was used.

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Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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