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Effect of non-thermal plasma treatment on the bonding property of Bio-HPP post and core materials

Jun-hong Lin
Lei Jiang
Yu-shan Zhu
Yong-jin Guo
guoyongjin1975@fjmu.edu.cn

Research Article

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Abstract

Background: Biological high-performance polymer (Bio-HPP) may represent an ideal alternative material for post and core restorations. However, studies investigating the bonding property of Bio-HPP as a post and core material remain scarce.The purpose of this study was to evaluate the impact of different non-thermal plasma (NTP) treatments on the push-out bond strength of Bio-HPP post-and-core restorations.

Methods: Forty extracted human single-rooted teeth were endodontically treated, and standardized post spaces were prepared. Forty posts were milled from Bio-HPP disks using a computer-aided design and computer-aided manufacturing (CAD-CAM) system and randomly assigned to 4 groups (n = 10) according to the surface treatment applied: (A)control (no treatment); (B) air NTP; (C)oxygen NTP; and (D)nitrogen NTP. After cementation(DC CORE ONE)，a push-out bond strength test was performed. In addition, Bio-HPP disk-shaped specimens (r = 5 mm, h = 2 mm) were milled and subjected to surface characterization detection.

Results: All non-thermal plasma treatments significantly increased bond strength compared with the control (8.00 ± 2.08MPa) (P <0.05). Nitrogen plasma (18.19 ± 3.71MPa) resulted in significantly higher bond strength than air (13.35 ± 3.16MPa) or oxygen plasma (13.42 ± 3.92MPa) (P < 0.05). Bond strength decreased with increasing canal depth, with values highest in the coronal third, followed by the middle and apical thirds (P < 0.05).

Conclusions: Within the limitations of this study, non-thermal plasma treatments with air, oxygen, and nitrogen enhanced the push-out bond strength of CAD/CAM milled Bio-HPP posts, while nitrogen non-thermal plasma treatment being more effective. Bond strength decreased with increasing canal depth, with the coronal third exhibiting significantly higher values than the middle and apical thirds.

Background

When the remaining tooth structure is insufficient to retain a full crown, post and core restoration is required to support the definitive restoration. Previous research suggests that an ideal post and core material should possess sufficient elasticity to adapt to the natural flexure of the tooth during function.[1] Materials with mechanical properties like dentin may promote more favorable stress distribution and reduce the risk of root fracture.[2]

Biological high-performance polymer (Bio-HPP) is a variant of polyetheretherketone (PEEK). The incorporation of 20% ceramic filler with particles (0.3–0.5μm) enhances its physical performance.[3] Bio-HPP demonstrates excellent biocompatibility and chemical stability and can be combined with carbon fiber and glass fiber materials.[4,5] Root fracture in flared canals has been associated with cast post and core systems, primarily because of the mismatch in the elastic modulus between metallic alloys and dentin. Bio-HPP has a modulus of elasticity comparable to that of dentin, which may contribute to more favorable stress distribution and reduce the risk of root fracture.[6] In the event of fracture, the failure

pattern may be more favorable for subsequent restoration.[7] After post space preparation, most root canals exhibit a tapered configuration, with the coronal portion wider than the apical portion. Inadequate adaptation between the post and the root canal geometry may result in the formation of an excessively thick cement layer.[8] A thick cement layer may promote air entrapment and microcrack formation within the resin cement, thereby increasing the risk of post debonding under masticatory forces.[9] Clinically, Bio-HPP posts can be fabricated by CAD/CAM milling or injection molding. CAD/CAM–milled Bio-HPP posts provide improved adaptation to the canal walls and may reduce interfacial gaps.[10] A thinner cement layer may also decrease polymerization stress during adhesive curing.[11]

CAD/CAM technology enables the precise and reproducible fabrication of post-and-core restorations from standardized materials and has been increasingly applied in prosthodontics.[12]Although the performance of CAD/CAM–fabricated post-and-core systems has been investigated, limited evidence supports the clinical application of CAD/CAM–milled PEEK posts.[13,14] Limited adoption may be attributed to the low surface energy of PEEK-based materials and their chemical inertness and low surface energy, which inherently limit its adhesive potential.[15] Therefore, improving the bonding properties of Bio-HPP posts remains a key challenge for broader clinical application.

Various surface modification methods have been proposed, including concentrated sulfuric acid etching, airborne-particle abrasion, silica coating and plasma surface treatment.[16] Sulfuric acid etching has been shown to significantly enhance the bond strength of Bio-HPP posts.[17] However, the use of highly concentrated sulfuric acid raises concerns regarding clinical safety.[18] Moreover, prolonged exposure may alter the surface characteristics and adversely affect bonding durability.[19] Airborne-particle abrasion is widely used as a surface treatment for Bio-HPP posts because of its relative safety and efficiency. However, some studies have reported that this procedure may compromise the mechanical properties of PEEK-based materials.20 The silica coating technique is a airborne-particle abrasion technique, deposits silica-coated alumina particles onto the material surface. Nevertheless, previous studies have demonstrated that silica coating does not provide additional improvement in the adhesive properties of PEEK.[21]

Non-thermal plasma treatment has been shown to effectively modify polymer surfaces; however, its effects on PEEK depends on gas type and plasma parameters.[22] Systematic comparisons of different plasma gases on surface chemistry and bond strength remain limited. Consequently, the optimal surface modification strategy for Bio-HPP posts remains controversial.Non-thermal plasma treatment has been shown to enhance bonding performance while preserving surface integrity and is considered a promising strategy for the surface modification of Bio-HPP posts.[23] Previous studies evaluating plasma treatment of PEEK-based composites have primarily focused on materials with different filler compositions. Because the composition of Bio-HPP differs substantially from that of other PEEK-based composites, the effectiveness of this surface treatment cannot be directly comparable and requires further investigation.

Therefore,the purpose of this study was to evaluate the impact of different non-thermal plasma treatments on the push-out bond strength of Bio-HPP post-and-core restorations. The null hypothesis was that plasma treatment would not affect the bond strength of Bio-HPP posts.

Methods

Several methods are available for evaluating the bond strength of posts, including microtensile, pull-out, and push-out bond strength tests.[24] Among these, the push-out bond strength test applies shear stress simultaneously to the post-resin cement and resin cement-dentin interfaces, thereby more closely reproducing the functional loading conditions encountered in the oral environment and enabling a more clinically relevant assessment of post bond strength.[25] In addition, the push-out bond strength test is considered more reliable than the microtensile test, as premature failures frequently occur during specimen preparation in microtensile testing and the resulting stress distribution is more localized. For these reasons, the push-out bond strength test was selected in the present study to evaluate the bonding performance of CAD/CAM-milled Bio-HPP posts.

The composition of the post and core materials used in this study is presented in Table 1. The schematic diagram of the experimental process is shown in Fig. 1. Forty single-rooted mandibular premolars with a single canal were selected and randomly allocated to four groups (n = 10). All teeth were free of wedge-shaped defects, caries, cracks, and internal resorption, exhibited a favorable crown-to-root ratio, and had fully developed apices. After removal of calculus and periodontal ligament remnants, the specimens were stored in 0.9% saline solution at room temperature until use. The crowns were sectioned horizontally using a low-speed diamond saw under water cooling, with the cut made 2 mm coronal to the cemento-enamel junction, leaving a minimum root length of 12 mm. Root canal preparation was performed using a step-back technique with engine-driven instruments. The canals were alternately irrigated with 2.5% sodium hypochlorite and distilled water after each instrument change, then thoroughly dried with paper points before obturation. The canal orifices were sealed with glass ionomer cement (Glaslonomer FX-□; SHOFU), and the specimens were stored in distilled water at 37°C for one week. After this period, the teeth were removed from storage and standardized post space preparation was performed.Post space impressions were obtained using silicone rubber (betasil; Heraeus) and digitized with a digital scanner (3Shape TRIOS®; 3Shape). The resulting digital data were then processed using a CAD/CAM system, and customized posts were fabricated by milling dental PEEK blanks (breCAM.BioHPP; Bredent).

Table 1
Composition of the post and core materials used in this study

Material	Composition	Manufacture	Lot. no.
breCAM. BioHPP	PEEK, 20% weight titanium oxide	Bredent	482137
DC CORE ONE	surface-treated synthetic barium glass powder 1, light anhydrous silicate 1, and bisphenol A (2-hydroxypropoxy) dimethacrylate (commonly known as: Bis-GMA).surface-treated synthetic barium glass powder 2, surface-treated light anhydrous silicate, and alumina.	Kuraray	B43273
Tabel 2. Means ± standard deviations of push-out bond strength (MPa)			

In the present study, air, oxygen, and nitrogen were selected as the plasma supply gases to evaluate their effects on the bond strength between Bio-HPP and resin-based materials. Helium was excluded because previous findings by Stawarczyk et al. indicated that non-thermal helium plasma did not significantly improve the shear bond strength of PEEK-based materials.The use of these three gas types was intended to identify plasma treatment conditions with greater clinical relevance. Therefore, the Bio-HPP posts which were milled by using a CAD/CAM system were divided into 4 groups: group A had no treatment;group B was treated with air non-thermal plasma;group C was treated with oxygen non-thermal plasm;group D was treated with nitrogen non-thermal plasma.Preliminary experiments revealed that the bond strength of the specimens changed with treatment time. Therefore, in the push-out bond strength test, the plasma treatment time was standardized to the duration that produced the maximum bond strength in the preliminary evaluation.

Dual-cure resin luting agent(DC CORE ONE; Kuraray) was applied to the post space by using a conveyor. Each post was inserted immediately in the root canal by using finger pressure for 10 seconds to ensure complete seating of the post in the prepared post space, and excess resin cement was subsequently removed. Light curing was then performed for 40 seconds at an intensity of 1250 mW/cm² using a LED curing unit (Elipar DeepCure-S LED; 3M ESPE), followed by an additional setting period of 10 minutes. The adhesive specimens were subsequently embedded in acrylic resin (denture base resin; Shanghai New Century Dental Materials Co., Ltd.)and stored in an incubator at 37°C with 100% humidity for 7 days.

The specimens were sectioned perpendicular to the long axis of the post using a precision cutting machine (IsoMet HS; Buehler), beginning 1.0 mm below the coronal root surface and advancing toward the apex. At present, there is no consensus regarding the optimal thickness of dentin slices for push-out bond strength test. Recent studies have suggested the use of 1-mm-thick slices, as thinner specimens provide a more uniform interfacial stress distribution and reduce frictional effects during testing.[26] Therefore, six consecutive slices with a thickness of 1.0 ± 0.05 mm were obtained, excluding the first slice from the root surface. The remaining slices were numbered sequentially from 1 to 6, with slices 1 and 2 corresponding to the coronal third of the root, slices 3 and 4 to the middle third, and slices 5 and 6 to the apical third.The thickness and diameter of each specimen were measured using a digital vernier

caliper with an accuracy of 0.01 mm. Each specimen was positioned on a universal testing machine (AGS-X; Shimadzu), with the apical surface facing upward (Fig. 2). Loading was performed at a crosshead speed of 0.5 mm/min until post dislodgement or specimen failure, and the maximum load at failure (Fmax, N) was recorded. The bonding area (A, mm²) was calculated using the following formula: $A = \pi(R1 + R2) * \sqrt{(R2-R1)^2+h^2}$,where R1 = minimum post radius (mm), R2 = maximum post radius (mm), h = specimen thickness (mm), π = 3.14.

After the push-out bond strength test, the failure mode of the specimen was observed by stereomicroscope (MM-400/800 series, Nikon). The fracture mode was divided into three categories: (1) Interface fracture: the bonding surface between the post and the resin luting agentor the bonding surface between the resin luting agentand the dentin; (2) Cohesive fracture :inside the resin luting agentor the post; (3) Mixed(cohesive/adhesive)failure.

Bio-HPP disk-shaped specimens (r = 5 mm, h = 2 mm) were milled and subjected to the same surface treatment protocols for surface characterization. All specimens were sputter-coated with a thin layer of gold using a sputtering apparatus (GVC-1000, Microhertz) prior to morphological characterization. The surface microstructures were subsequently examined by field-emission scanning electron microscopy (Nova NanoSEM 230, FEI) operated at an accelerating voltage of 6 kV. Micrographs were acquired at magnifications of 2,000× and 30,000× (n = 5).The water contact angle of each group of specimens (n = 5) was measured using a water contact angle meter (SDC-100; Sindin). For each measurement, a 1 μL droplet of deionized water was carefully dispensed onto the specimen surface and allowed to equilibrate for approximately 3 s. After stabilization of the droplet profile, the contact angles on both the left and right sides were determined using the instrument's integrated analysis software, and the mean of the two values was calculated. Three measurements were performed at different locations on each specimen, and the average value was reported as the water contact angle for that specimen. Specimens subjected to different surface treatments were analyzed using X-ray photoelectron spectroscopy (ESCALAB QXi,Thermo Scientific). Survey spectra were acquired to determine the elemental composition of the surfaces, with particular focus on carbon (C), oxygen (O), and nitrogen (N). High-resolution spectra of the C 1s region were subsequently obtained and deconvoluted using XPSPEAK 4.1 software to evaluate variations in the chemical states and relative proportions of surface functional groups among the groups. Data analysis and graphical presentation were performed using Origin 2020 software (OriginLab, USA).

Statistical analysis were performed using a software program (IBM SPSS Statistics, v26.0; IBM Corp).The data were first assessed for normal distribution and homogeneity of variance. When the assumptions of normality and equal variances were met, a two-way analysis of variance (ANOVA) was conducted to evaluate the effects of treatment method and root canal depth on push-out bond strength (α = 0.05).Post hoc pairwise comparisons were performed using the least significant difference (LSD) test.The results of water contact angle were analyzed with one-way ANOVA (α = 0.05).

Tabel 2. Means ± standard deviations of push-out bond strength (MPa)

Experimental group	Coronal	Middle	Apical	Mean
A	10.06 ± 1.58^{Aa}	7.85 ± 1.40^{Ba}	6.12 ± 0.64^{Ca}	8.00±2.08
B	16.01 ± 2.57^{Ab}	13.70 ± 1.93^{Bb}	10.27 ± 1.22^{Cb}	13.35±3.16
C	16.94 ± 3.20^{Ab}	13.58 ± 2.24^{Bb}	9.69 ± 1.80^{Cb}	13.42±3.92
D	21.20 ± 3.60^{Ac}	17.90 ± 2.37^{Bc}	15.48 ± 1.65^{Cc}	18.19±3.71

Different superscript uppercase letters within same row and lowercase letters within same column indicate significant difference ($P < .05$). A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma.

Tabel 3. The fracture mode of each group

Experimental group	AP	AD	C	M
A	35	17	0	8
B	19	39	0	8
C	5	45	0	10
D	8	47	0	5

AP□adhesive-post interface; AD□adhesive-dentin interface; C□cohesive;M□mixed.A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma.

Results

The push-out bond strength results are presented in Table 2. Two-way ANOVA demonstrated that both surface treatment method and root canal depth had a significant effect on the push-out bond strength of the posts ($P < 0.05$). All non-thermal plasma-treated groups exhibited significantly higher bond strength than the control group ($P < 0.05$). Among the plasma treatment groups, the nitrogen plasma group showed significantly greater bond strength than the air and oxygen plasma groups ($P < 0.05$), whereas no significant difference was found between the latter two groups ($P > 0$ .05). In addition, root canal depth significantly influenced bond strength, with higher values observed in the coronal region, followed by the middle and apical regions ($P < 0.05$).

Figures 3. show schematic diagrams of the fracture modes and Table 3. shows the distribution of fracture modes in each group of specimens. No cohesive failure was observed in specimens from any of the experimental groups. In the control group, the predominant failure mode was adhesive failure at the adhesive-post interface. In contrast, specimens subjected to non-thermal plasma treatment mainly exhibited adhesive failure at the adhesive-dentin interface.

Representative SEM images are shown in Figs. 4. The surface of specimens in the control group exhibited a relatively smooth morphology, with randomly distributed inorganic filler particles and irregular scratches produced by sandpaper polishing. In contrast, specimens treated with air, oxygen, and nitrogen plasma displayed cleaner surfaces with etching-induced pits. Compared with the oxygen plasma-treated group, the air- and nitrogen-treated groups demonstrated more distinct small cavities on the surface.

The water contact angle measurements are shown in Table 4. Compared with the control group, the water contact angle of each non-thermal plasma treatment group was significantly decreased ($P < 0.05$). There were no statistically significant differences among the non-thermal plasma treatment groups ($P > 0.05$).

Table 4
Means ± standard deviations of water contact angle (degree)

Experimental group	**Contact angle**
A	73.83 ± 4.81^{a}
B	28.47 ± 3.65^{b}
C	30.11 ± 1.22^{b}
D	21.11 ± 3.21^{b}
Different lowercase letters within the same column indicate statistically significant differences among groups ($P < .05$).A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma.	

The surface elemental compositions of the specimens in each group are summarized in Table 5. Compared with the control group, specimens treated with air, oxygen, and nitrogen non-thermal plasma exhibited a marked decrease in the relative carbon content and a significant increase in the relative oxygen content, resulting in a substantially higher surface oxygen-to-carbon (O/C) ratio. Among the plasma-treated groups, the oxygen-treated specimens showed the highest relative oxygen content. The curve-fitting results of the C1s spectra are presented in Table 6 and Fig. 5. Compared with the control group, plasma-treated specimens showed a reduced proportion of C = C bonds and an increased proportion of polar functional groups, with the nitrogen plasma-treated group exhibiting the highest relative content of polar groups.

Table 5
XPS analysis of the percentage of elements on the surface of each group of specimens

Experimental group	C	O	N	O/C
A	86.80	12.54	<dl	0.14
B	79.52	17.67	1.97	0.22
C	74.67	24.83	0.20	0.33
D	70.23	19.42	9.02	0.28
A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma. <dl = below detectable limit.				

Table 6
Deconvolution of C1s and the concentration of relevant functional groups from XPS

Experimental group	C-C	C-O	C = O	O-C = O
A	73.61	15.82	8.56	2.01
B	69.46	14.31	13.33	2.90
C	64.62	16.75	6.25	12.38
D	58.67	19.76	15.67	5.90
A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma.				

Discussion

This in vitro study aimed to investigate the influence of plasma treatments using different gases on the bonding performance of Bio-HPP materials. The null hypothesis that plasma treatment would not affect the bond strength of Bio-HPP posts was rejected.

The experimental results showed that the control group exhibited relatively low mean push-out bond strength. This finding can be attributed to the low surface energy and hydrophobic nature of the material, which limit the adhesion between the post and the resin cement. These results are consistent with previous reports.[27,28] In current clinical practice, surface treatment of Bio-HPP posts is predominantly performed by sandblasting. Compared with sandblasting, non-thermal plasma treatment represents an environmentally friendly and efficient alternative that can increase surface roughness without compromising the mechanical properties of the material, while simultaneously enhancing surface energy and wettability and introducing chemical functional groups capable of interacting with resin-based materials.[29]

The results of this experiment show that air, oxygen and nitrogen non-thermal plasma treatment can effectively improve the push-out bond strength of Bio-HPP posts. In addition, the average push-out bond

strength of the nitrogen non thermal plasma treatment group is significantly higher than that of the oxygen and air non-thermal plasma treatment groups ($P < 0.05$). Currently used resin cements are primarily based on methacrylate chemistry, and Bio-HPP posts inherently lack functional groups capable of directly reacting with these materials. Plasma treatments using air, oxygen, and nitrogen can introduce reactive functional groups onto the post surface, thereby increasing the chemical affinity between Bio-HPP and methacrylate-based resin cements without compromising surface integrity.It has been reported that nitrogen non-thermal plasma improves the adhesive properties of polymers by forming special nitrogen functional groups such as amine (C-N) and imine groups. In addition to their ability to interact with functional monomers in resin cements, these nitrogen-containing groups appear to exhibit greater resistance to aging effects compared with oxygen-containing functional groups.This enhanced chemical stability may account for the significantly higher push-out bond strength observed in the nitrogen plasma-treated group relative to the air and oxygen plasma-treated groups.[30] The peak bonding performance in the nitrogen-treated group, despite its lower O/C ratio compared to the oxygen group, suggests that the type of functional groups is more critical than their sheer quantity. The introduced nitrogenous moieties, particularly primary amines (-NH2), can act as Lewis bases that interact strongly with the acidic phosphate groups in 10-MDP-containing resin cements.[31,32] This acid-base synergy, coupled with potential hydrogen bonding, likely facilitates a more robust chemical coupling at the Bio-HPP interface.

XPS analysis of the surface elements of each specimen showed that the chemical composition of the material surface changed significantly after treatment with air, oxygen and nitrogen non-thermal plasma. Specifically, plasma treatment resulted in a pronounced increase in surface oxygen content accompanied by a significant decrease in carbon content, leading to a substantial reduction in the carbon-to-oxygen (C/O) atomic ratio.These findings are consistent with results reported for other PEEK-based composite materials treated with non-thermal plasma.[33] In addition, specimens subjected to nitrogen plasma treatment exhibited a significant increase in the relative atomic percentage of nitrogen compared with untreated specimens, indicating that the majority of the additional nitrogen-containing functional groups were generated during the plasma treatment process.

When PEEK-based composite materials are exposed to a plasma environment, two major chemical processes are expected to occur at the surface. One process is chain scission, in which energetic ions bombard polymer chains at the material surface, resulting in the formation of small volatile degradation products. The other process involves the incorporation of functional groups onto the material surface.[34] Previous studies have demonstrated that enhancements in interfacial bonding performance are primarily attributed to chemical modification by surface-active functional groups, among which the formation of polar functional groups such as carboxyl and hydroxyl groups plays a critical role.[35] The results of the present study demonstrated that the content of polar functional groups on the specimen surfaces increased following low-temperature plasma treatment, indicating that polar groups may be grafted onto the material surface during plasma exposure. These changes promote stronger interactions between adhesive molecules and the substrate, thereby enhancing bonding performance. The high concentration

of carbon-oxygen functionalities observed on nitrogen plasma-treated specimens indicates pronounced post-treatment surface oxidation, likely resulting from the continued reactivity of plasma-generated surface-active sites with atmospheric species.Compared with air and oxygen cryogenic plasma treatment, the nitrogen cryogenic plasma treatment group had a higher C-O group content on the surface of the specimens. This indicates that when nitrogen plasma is used to treat materials, C-O groups are preferentially generated. These C-O groups may be introduced into the ether bond breaking sites or aromatic ring structures of the Bio-HPP post and core material.

Surface free energy, as a key parameter characterizing the surface properties of materials, is intrinsically related to the wettability of materials. Plasma treatment technology can significantly improve the surface free energy of materials by adjusting the chemical composition of the material surface and promoting the increase of the polar component of the material, thereby effectively improving the wettability of the material.[36] In this experiment, the water contact angle of each non-thermal plasma treatment group was significantly lower than that of the control group,while the water contact angle of the specimen in the nitrogen non-thermal plasma group was slightly lower than that in the air and oxygen plasma groups.Although all plasma treatments improved wettability to a similar extent ($P > 0$ .05), their impact on bond strength varied significantly. This indicates that while increased wettability is essential, it is not the sole determinant of success; rather, the specific chemical functionalization (XPS findings) in the nitrogen group plays a more decisive role in enhancing Bio-HPP bonding than physical wettability alone.[37,38]

Furthermore, significant differences in push-out bond strength were observed at different root canal levels ($P < 0.05$), with the coronal third exhibiting higher bond strength than the middle and apical thirds in all groups. This may be related to the distance from the light source, as adhesive curing becomes less complete toward the apical region. Some studieshave also suggested that variations in bond strength at different levels may be influenced by the C-factor, with higher values in deeper regions generating greater polymerization shrinkage stress at the bonded interface.[39] Furthermore, dentin tubules in the apical third are more irregular, with lower density and number, resulting in reduced resin tags and decreased micromechanical interlocking.[40] However, Monteiro et al. reported no significant differences in push-out bond strength across different levels of CAD/CAM-milled PEEK posts subjected to various surface treatments, which was attributed to the relatively large diameter of bovine root canals facilitating smear layer removal.[10] In addition, the choice of luting material can affect bond strength. Compared with other light-curable materials, fully acid-etch dual-cure cements exhibit less depth-dependent variation in degree of conversion. Their specialized fine-needle delivery tips allow precise placement to the apical end of the post space, ensuring uniform cement coverage along the canal walls and thereby enhancing bonding effectiveness.[41]

Fracture modes of bonded specimens are generally classified into three types: adhesive failure, cohesive failure, and mixed failure. In the present study, adhesive failure refers to debonding at the post-resin cement or resin cement-dentin interface, whereas cohesive failure indicates fracture within the post or

resin material itself, typically occurring when the bond strength exceeds the intrinsic material strength. Mixed failure involves a combination of adhesive and cohesive failures. The results showed that specimens in the untreated control group predominantly exhibited adhesive failure at the post-resin cement interface, indicating insufficient bond strength between the post and the resin cement. In contrast, specimens treated with various non-thermal plasma protocols primarily exhibited adhesive failure at the resin cement-dentin interface, suggesting that while some bonding was achieved between the post and resin cement, the interfacial strength between resin luting agentand dentin remained the limiting factor.This shift in failure mode from the post-interface to the dentin-interface suggests that plasma treatment elevated the Bio-HPP bond strength beyond the inherent capacity of the dentin-adhesive complex[42,43]. Consequently, the treated Bio-HPP surface no longer represents the weakest link in the restorative assembly, indicating a significant clinical safety margin.

A limitation of this in vitro study lies in the method used to fabricate CAD/CAM-milled Bio-HPP posts. The posts were produced by creating silicone molds of the post spaces followed by digital scanning to generate individualized posts. Although feasible, this approach increases chairside time and may compromise accuracy. Some manufacturers have proposed directly scanning the post space to generate a digital model for CAD/CAM milling, which could reduce chairside procedures; however, research on this approach remains limited. Another limitation of the present study is the absence of bond strength measurements after artificial aging. Given the complexity of the oral environment, future studies should assess the effects of intraoral stresses and thermal fluctuations on bond strength to evaluate the long-term performance of posts.

Conclusions

Within the limitations of this in vitro study, the following conclusion could be drawn:

1. Non-thermal plasma treatments,particularly with nitrogen gas,significantly enhance the push-out bond strength of CAD/CAM milled Bio-HPP posts compared with untreated controls.
2. The bonding effectiveness of Bio-HPP posts is site-specific, with the highest values achieved in the coronal third of the root canal, decreasing significantly toward the middle and apical regions.
3. Plasma treatment effectively shifts the primary failure site from the post-interface to the dentin-interface, indicating improved polymer surface receptivity.

Declarations

Ethics approval and consent to participate

This study protocol was reviewed and approved by the Ethics Committee of the Affiliated Stemmatological Hospital of Fujian Medical University (Approval No.: [2024] FYKLL No. 66). All experiments were conducted in accordance with the "Helsinki Declaration".This study has obtained the informed consent of all participants.

Consent for publication

Not applicable.

Competing 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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Author Contribution

Jun-hong Lin: Conceptualization, Methodology, Writing -Original Draft preparation, Data Curation, Statistical Analysis, Visualization.Lei Jiang: Writing - Review & Editing, Formal Analysis,Validation.Yu-shan Zhu:Software, Investigation.Yong-jin Guo: Project administration, Supervision, Writing - Review & Editing, Funding Acquisition.

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Authors' information

Jun-hong Lin: Resident dentist and MM, Fujian Key Laboratory of Oral Diseases & Fujian Provincial Engineering Research Center of Oral Biomaterial, School and Hospital of Stomatology, Fujian Medical University, China.

Lei Jiang:Attending dentist and Phd, Fujian Key Laboratory of Oral Diseases & Fujian Provincial Engineering Research Center of Oral Biomaterial, School and Hospital of Stomatology, Fujian Medical University, China.

Yu-shan Zhu: Postgraduate student,Fujian Key Laboratory of Oral Diseases & Fujian Provincial Engineering Research Center of Oral Biomaterial.

Yong-jin Guo:Associate Professor and Phd, Fujian Key Laboratory of Oral Diseases & Fujian Provincial Engineering Research Center of Oral Biomaterial, School and Hospital of Stomatology, Fujian Medical University, China.

Data Availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Figures

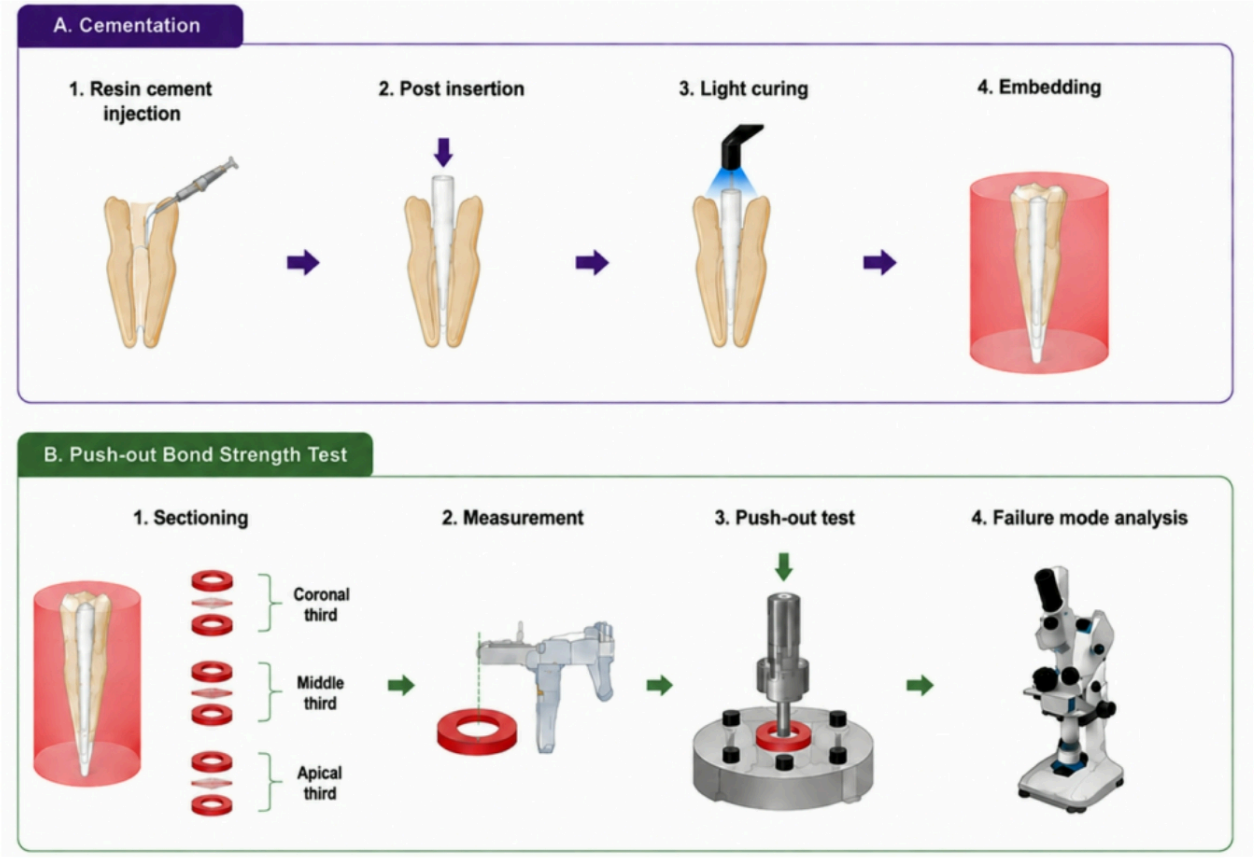


Figure 1

A diagram of the experimental process

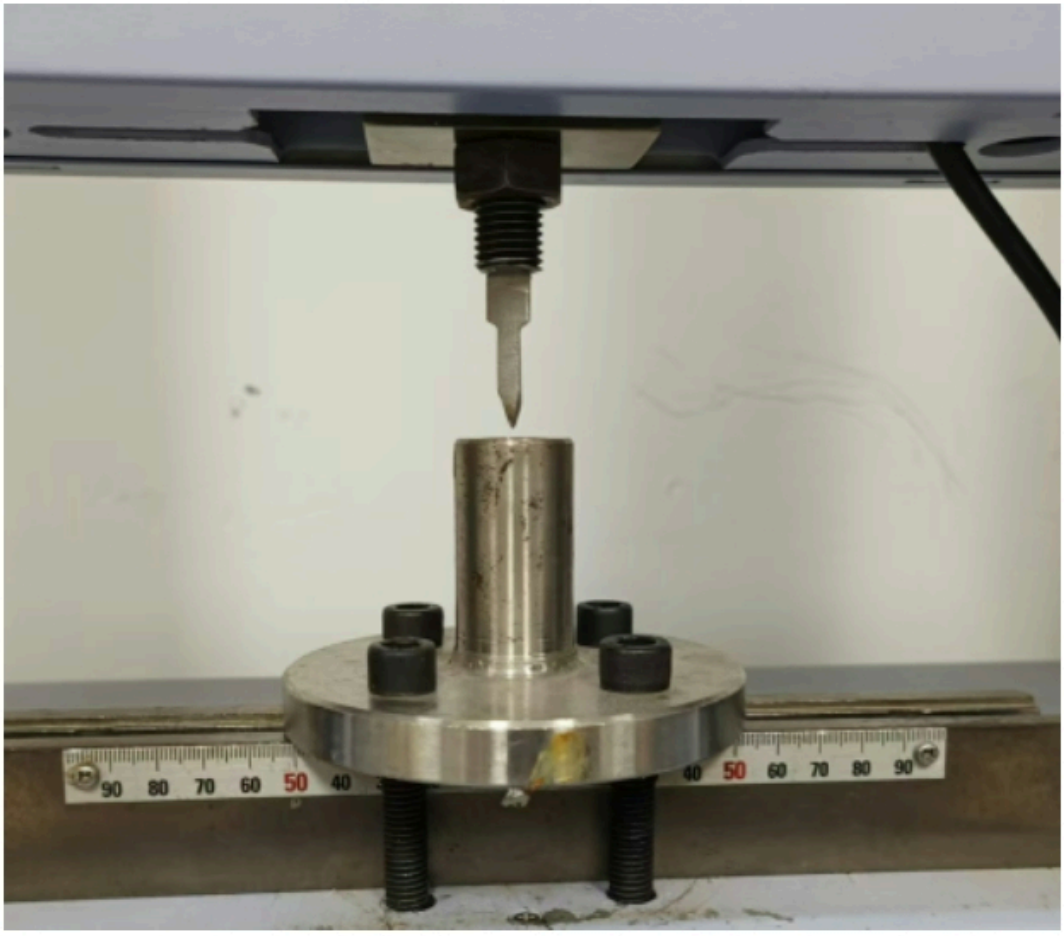


Figure 2

Push-out test

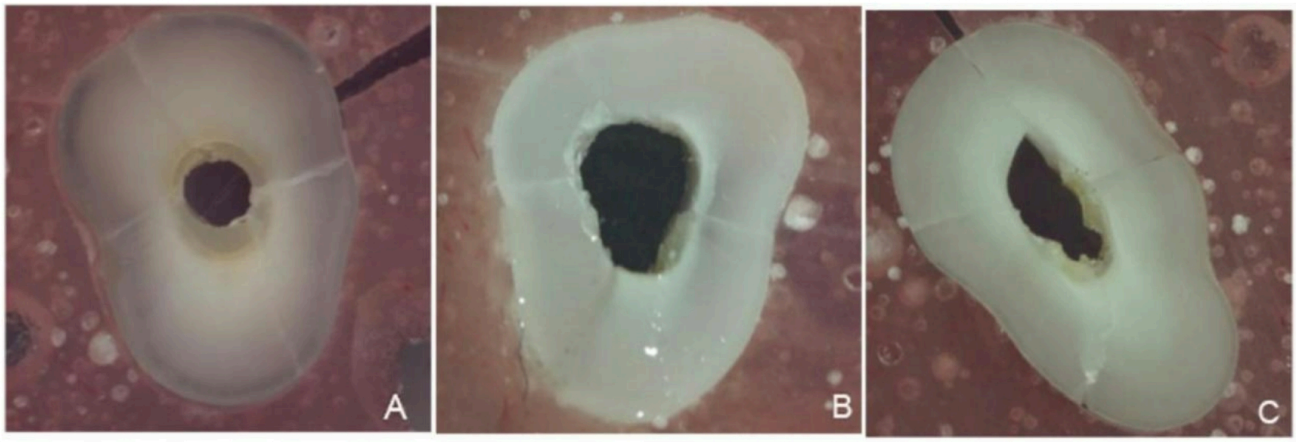


Figure 3

Schematic diagrams of the fracture modes (×40). A, adhesive failure between the adhesive and post. B, adhesive failure between the adhesive and dentin. C, mixed failure.

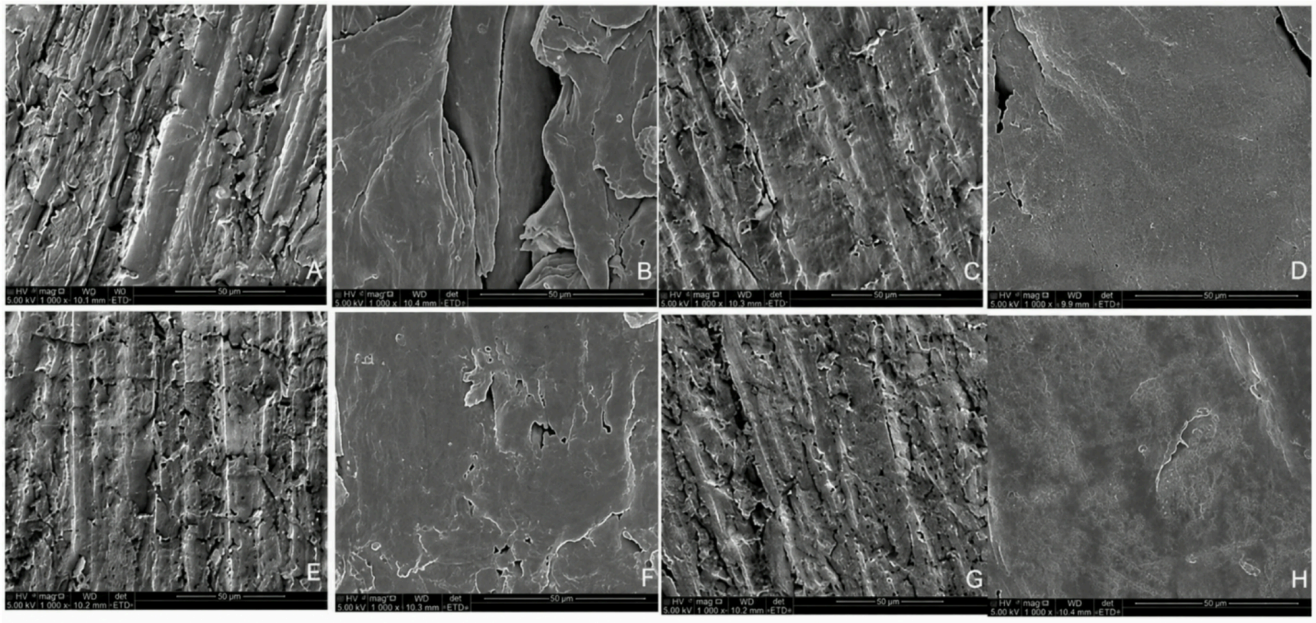


Figure 4

Scanning electron microscopy (SEM) images of specimens (original magnifications ×2000 and ×30000). A, B, no treatment(control). C, D, air plasma. E, F, oxygen plasma. G, H, nitrogen plasma.

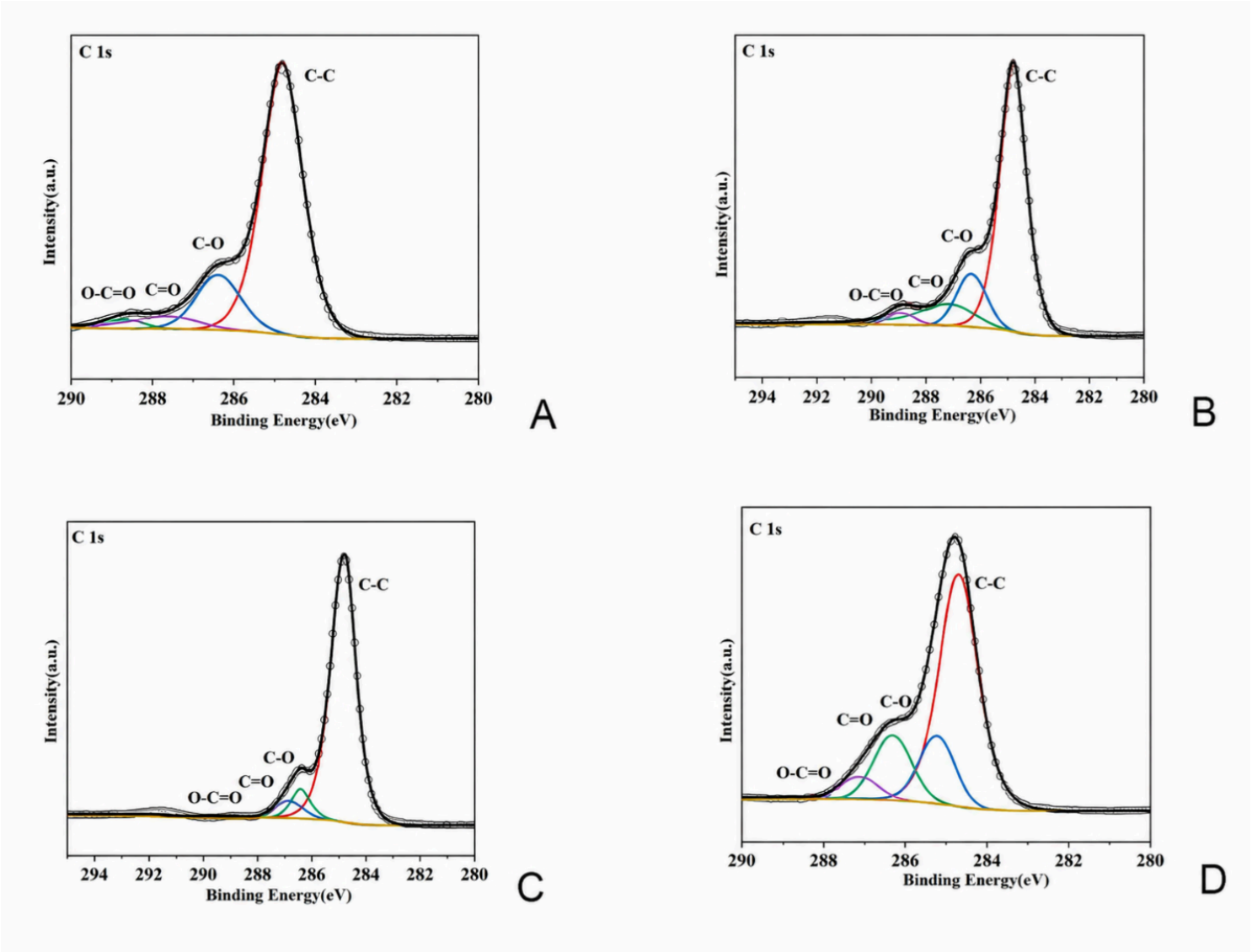


Figure 5

Peak spectra of C1s fitting on the specimens' surface under different surface treatments.A, no treatment(control). B, air plasma. C, oxygen plasma. D, nitrogen plasma.