

The impact of PEEK pretreatment using H₂SO₄, riboflavin, and aluminum trioxide on the extrusion bond strength to canal dentin luted with Polymethyl methacrylate and resin-based composite cement

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Abstract. – OBJECTIVE: To evaluate the effects of various surface pretreatment methods, including H₂SO₄, Riboflavin, and Al₂O₃, as well as different luting cement types, namely Methyl Methacrylate based Cement (MMBC) and composite-based cement (CBC), on the extrusion bond strength (EBS) of poly-ether-ether-ketone (PEEK) posts bonded to canal dentin.

MATERIALS AND METHODS: This study involved 120 single-rooted human premolar teeth that underwent endodontic treatment. Following root canal preparation, PEEK posts were fabricated from PEEK blanks using a CAD-CAM system, resulting in a total of 120 posts. The posts were randomly assigned to one of four groups based on their post-surface conditioning: Group A H₂SO₄, Group B RF, Group C Al₂O₃, and Group D (NC), each consisting of 30 posts. Within each group, there were two subgroups based on the type of luting cement used for bonding. Subgroups A1, B1, C1, and D1 (n=15 each) utilized CBC, while Subgroups A2, B2, C2, and D2 (n=15 each) used MMBC. The bond strength between the PEEK posts and root dentin was assessed using a universal testing machine, and the failure modes were examined under a stereomicroscope. Statistical analysis, including one-way analysis of variance (ANOVA) and Tukey's Post Hoc test with a significance level of $p=0.05$, was performed to analyze the data and evaluate the effects of surface treatment and luting cement type on the bond strength.

RESULTS: Group B2, which underwent RF conditioning followed by Super-Bond C&B cement application, exhibited the highest bond strength scores at the coronal section (9.57 ± 0.67 MPa). On the other hand, Group D1, which had no conditioning (NC) and used Panavia® V5 cement, showed the lowest EBS at the apical third (2.39 ± 0.72 MPa). The overall results indicate that the different conditioning regimens and luting cement types did not significantly influence the bond strength of PEEK posts to root dentin ($p>0.05$).

CONCLUSIONS: Riboflavin activated by photodynamic therapy (PDT) and H₂SO₄ can be effective surface conditioners for PEEK posts. These treatments have shown potential for enhancing the bond strength between PEEK and resin cement. Additionally, the study revealed that MMA-based cement outperformed composite-based cement in terms of bond integrity with PEEK posts.

Key Words:

Sulphuric acid, Riboflavin, Aluminium trioxide, Poly-ether-ether-ketone.

Introduction

The purpose of a post and core restoration is to strengthen the tooth structure, particularly when a significant portion of the clinical crown has been lost due to decay, fracture, or previous dental treatments. The post extends into the root canal, providing stability and anchorage for the core buildup material, which replaces the missing tooth structure and creates a suitable foundation for the final crown restoration¹. An optimal post material should be biocompatible, adhere to dentin effectively, meet aesthetic standards, and possess the same physical and mechanical properties as dentin². Prefabricated fiber posts have gained widespread usage in the field of dentistry for tooth restoration purposes³. Despite their popularity, prefabricated fiber post systems in dentistry have some limitations. These include issues such as debonding, difficulties in adapting to elliptical root canals, and a tendency for increased polymerization shrinkage^{4,5}.

The poly-ether-ether-ketone (PEEK) formed post system, which belongs to the PAEK (Poly-aryl-ether-ketone) resin family, has emerged as a popular and viable alternative to fiber posts in dentistry. One of its notable advantages is its lower elastic modulus of 3-4 GPa compared to that of root dentin^{5,6}. This property allows PEEK to act as a shock absorber, helping to distribute forces and reduce stress on the tooth structure⁶. PEEK posts are commonly manufactured using pressed or CAD/CAM technology, which enables them to closely mimic the shape and contours of the root canal⁷. However, achieving a strong bond between PEEK and dentin is challenging due to PEEK's resistance to surface changes and its low surface energy. This poses difficulties in achieving a successful push-out bond strength (PBS) at the interface between the PEEK post and dentin^{8,9}.

The use of concentrated sulfuric acid (H_2SO_4) as a pretreatment chemical to prepare the surface of PEEK posts has shown promising results in improving the extrusion bond strength (EBS) between the PEEK post and radicular dentin. However, it is important to note that the concentrated nature of sulfuric acid can have negative effects on the oral environment^{8,10}. While concentrated sulfuric acid may enhance the bonding properties of PEEK, its application should be performed with caution due to its corrosive nature. Contact with oral tissues or accidental leakage can lead to tissue irritation or damage⁷. Therefore, other alternatives have been identified to condition the post surface. Photodynamic therapy (PDT) is considered a progressive, modern, and unique surface treatment technique in dentistry^{9,11}.

Riboflavin (RF), an anionic photosensitizer, has been shown to possess antimicrobial properties at visible light wavelengths between 500 and 800 nm¹². However, the effect of using RF as a post-surface conditioner on EBS of PEEK posts bonded to root dentin has not been thoroughly investigated and more research is required^{13,14}. Similarly, it has been demonstrated that Al_2O_3 effectively modifies the surface morphology of various materials, including metals and polymers. Ourahmoune et al¹⁵ investigated the effects of Al_2O_3 on the surface morphology of polymeric substances¹⁶. Existing literature concerning the effect of these conditioners on the bond integrity of PEEK posts is insufficient and ambiguous.

The selection of luting cement is an important factor in determining the bond strength between PEEK posts and radicular dentin, in addition to the conditioning regime used. One effective adhesive

solution involves using an adhesive primer in combination with resin cement for bonding PEEK to the cement^{17,18}. Resin cement in contemporary dentistry can be categorized as either Methyl Methacrylate (MMA) based cement or composite-based cement. MMA-based cement typically consists of methyl methacrylate (MMA) and polymethyl methacrylate (PMMA) as key components, while composite-based cement is composed of a resin matrix and ceramic fillers^{13,19}. However, the precise impact of these luting cement on the bond strength of pretreated PEEK posts remains uncertain and requires further investigation. Current literature on the subject is limited, necessitating additional research to better understand the influence of MMA-based cement (MMBC) and composite-based cement (CBC) on the bond strength of PEEK posts following pretreatment.

Based on the existing literature, it can be concluded that there is a lack of sufficient data regarding the effect of PEEK post-conditioning on EBS on radicular dentin. In light of this, it was hypothesized that there would be no significant difference in the EBS of PEEK posts bonded to radicular dentin when using the latest surface conditioners (such as SA, RF, and Al_2O_3) compared to a control group with no conditioner (NC). Furthermore, it was also postulated that the use of MMBC and CBC would yield comparable bond strength results regardless of the type of conditioner employed. Therefore, the present study aimed to evaluate the impact of post-surface conditioners and different luting cement on the EBS of PEEK posts bonded to radicular dentin.

Materials and Methods

Root Preparation and Endodontic Treatment and Preparation of Post Space

120 human premolar teeth with a single root were collected for the investigation. After removing any detritus from the teeth with a periodontal scaler, the teeth were immersed in a 1% thymol solution at room temperature. The coronal portions of the teeth were then sectioned using a diamond disc and constant irrigation, resulting in 16 mm root lengths that were standardized. Cracked, fractured, and open-apex teeth were excluded from the study. Using a 10K file, the pulp chamber was accessed and a canal was initiated. Visually confirming the K file tip through the apex and then retracting it 1 mm from the anatomical apex yielded a working length (WL) of 15 mm. Using

the ProTaper Ni-Ti rotary system, the canals were prepared up to the F3 finishing file. Between each file, 5 ml of 2.5% sodium hypochlorite (NaOCl) was used to guarantee thorough canal disinfection. As a final disinfectant, the canal surfaces were irrigated for one minute with 5 ml of 17% ethylenediaminetetraacetic acid (EDTA). The canals were then rinsed with 10 ml of distilled water before being dried with paper cones. The canals were then filled with a mixture of gutta-percha (GP) and AH sealer^{14,20}.

To prepare the post space, a Gates Glidden drill #3 from Dentsply-Maillefer was used at a speed of 9,000 revolutions per minute (rpm). The drill was utilized to remove 11 mm of gutta-percha (GP) from the root canal. It is important to note that the apical seal was not compromised, as 4 mm of gutta-percha was intentionally retained in the apical region of all the roots. To maintain optimal cutting efficiency, a fresh drill was used for every five specimens to ensure consistent and accurate preparation of the post space⁹.

Poly-Ether-Ether-Ketone (PEEK) Posts Fabrication

In this study, a prefabricated acrylic pin (Pin-jet, Angelus, Waukesha, WI, USA) and the powder-liquid brush technique were utilized to create castings of each root using acrylic resin (Pattern Resin LS, GC America, Alsip, IL, USA). Any distortions or excess material on the casts were then removed using a mini-cutting drill. The casts were subjected to scanning and milling procedures using a CAD-CAM system (Amann Gyrbach, Vorarlberg, Austria) to generate 120 PEEK posts from a PEEK blank (Ceramill PEEK, Amann Gyrbach, Vorarlberg, Austria).

To ensure proper positioning within their respective root canals, the posts underwent a meticulous inspection using liquid carbon (Kota) and diamond burs (2135 and 2135FF, KG Sorensen). Subsequently, the posts were carefully cleaned to remove any carbon residues. Random allocation of the PEEK posts into four groups was performed based on the specific post-surface conditioning regimen employed (n=30 per group)²¹.

Group A: PEEK Post-Conditioned with SA

In this group, the milled PEEK posts underwent surface treatment using a 98% SA (sulfuric acid) solution for 60 seconds. After the treatment, the specimens were carefully rinsed with distilled water to remove any residual acid and then thoroughly dried to ensure complete desiccation.

Group B: PEEK Post-Conditioned with RF

The post surfaces in this group were conditioned using a 25 mol/L RF dye. Subsequently, the conditioned surfaces were irradiated using a green laser at a wavelength of 540 nm for 60 seconds. After the irradiation process, the specimens were washed with distilled water to remove any residual dye and then thoroughly dried to ensure complete desiccation.

Group C: PEEK Post-Conditioned with Al₂O₃

In this group, the PEEK specimens underwent a blasting process using 50 µm alumina oxide (Al₂O₃) particles. The blasting was performed from a distance of 10 mm with a pressure of 0.1 MPa for 10 seconds. Following the blasting procedure, the specimens were thoroughly washed with distilled water to remove any residual particles and then completely dried to ensure complete desiccation.

Group D: PEEK Post-Not Conditioned (NC)

In this group, no conditioning regime was applied. Each group was further divided into two subgroups based on the luting cement used (n=15)

Composite-Based Resin Cement (A1, B1, C1 and D1)

After the surface conditioning of the PEEK posts, they were cemented into their respective canals using a specific adhesive protocol. First, the PEEK posts were treated with Clearfil® Ceramic Primer Plus (Kuraray Medical, Tokyo, Japan). Then, Panavia® V5 adhesive was applied onto the surface of the PEEK post and post space using an applicator. The PEEK post was carefully positioned within the post space, and any excess cement was removed. Next, the post was dried using high air pressure for 60 seconds to ensure proper adhesion. Finally, the cured using an LED light (Woodpecker, Guangdong, China) for a duration of 90 seconds to achieve complete polymerization of the cement.

MMA-Based Resin Cement (A2, B2, C2 and D2)

Following the manufacturer's instructions, the PEEK post was cemented into its respective canal using Super-Bond C&B and M&C Primer (Sunmedical, Moriyama, Japan). The primer was applied to the PEEK surface, and then the cement

was applied to both the PEEK post surface and the post space using an applicator. The PEEK post was carefully inserted into the post space, and any excess cement was removed. Subsequently, the post was dried under high air pressure for 60 seconds to ensure proper adhesion. Finally, the cement was cured using an LED light (Woodpecker, Guangdong, China) for a duration of 90 seconds to achieve complete polymerization (Table I).

Thermocycling of the Experimented Specimens

After a 24-hour period, all the PEEK samples underwent a thermocycling protocol to simulate the effects of temperature changes in the oral environment. The thermocycling was performed using an Automatic Thermocycling Dipping Machine. The samples were subjected to 5000 cycles, with two water baths set at temperatures of 5°C and 55°C. Each dwell time and transfer time between the water baths was set to 20 seconds. This thermocycling process helps evaluate the stability and durability of the PEEK samples under temperature variations.

Assessment of EBS and Failure Analysis

For the EBS assessment, the root samples were sectioned into 1-mm thick sections, including both the coronal and middle apical regions. This sectioning was performed using a slow-speed saw with adequate irrigation to ensure precision and prevent damage. To measure the PBS values, the test sections were positioned horizontally beneath a metal blade attached to a universal testing machine (UTM). The metal blade had a thickness of 0.5 mm. During the testing, the metal blade was

moved in a vertical direction at a crosshead speed of 0.5 mm/min and at a 90° angle to the specimen. This process continued until fracture occurred, indicating the point of bond failure. The force exerted to debond the PEEK post from the radicular dentin was recorded and quantified in units of Megapascals (MPa). This measurement indicates the strength of the bond between the PEEK post and the radicular dentin. To determine the type of failure, the fractured surfaces of the specimens were carefully examined using a stereomicroscope. A preconfigured Olympus Stereo Microscope System, specifically the SZX7 model from Edmund Optics UK, was used for this purpose. The microscope provided a 40X magnification, enabling detailed observation of the fracture surfaces. The primary objective of this examination was to classify the type of failure that occurred at the interface between the PEEK post and the radicular dentin. The three possible types of failure are adhesive, cohesive, and admixed^{22,23}.

Statistical Analysis

The study's outcomes were analyzed using Statistical Packages for Social Sciences (IBM Corp., Armonk, NY, USA). To examine the effects of the surface treatment methods and different types of luting cement, a one-way analysis of variance (ANOVA) was performed. This statistical test allows for the comparison of means among multiple groups. Additionally, Tukey's post-hoc test was employed to further investigate and compare specific group differences. This test helps determine which groups significantly differ from each other in terms of the observed variables. A significance level (*p*-value) of 0.05 was chosen, indicating that

Table I. Composition of material used in the present study.

Material Type	Product Name	Manufacturer	Composition
MMA-based resin cement	Super-Bond EX	Sunmedical, Moriyama, Japa MMA, PMMA, 4-META, TBB-O MULTIBOND II Tokuyama Dental, Tokyo, Japan	MMA, PMMA, 4-META, TBB-O PMMA, co-activator, MMA, UDMA, HEMA, MTU-6, borate catalyst.
Primer	M&C Primer	Sunmedical, Moriyama, Japan	Primer A: MDP, VTD, MMA, acetone. Primer B: -MPTS, MMA. Primer A: MDP, MTU-6, Bis-GMA,
Composite-based resin cement	Panavia V5	Kuraray Noritake Dental, Tokyo, Japan	Bis-GMA, TEGDMA, titanium dioxide.
Primer	Clearfil® Ceramic Primer Plus	(Kuraray Medical)	Ethanol, -MPTS, MDP.

any observed differences with a probability of occurring by chance less than 5% would be considered statistically significant.

Results

Figure 1 presents the mean values and standard deviations (SD) of the extrusion bond strength (EBS) in megapascals (MPa) for different experimental groups at the cervical, middle, and apical levels of the root. The results indicate that Group B2, which underwent RF conditioning followed by Super-Bond C&B cement application, exhibited the highest bond strength scores at the coronal section (9.57 ± 0.67 MPa). On the other hand, Group D1, which had no conditioning (NC) and used Panavia[®]V5 cement, showed the lowest EBS at the apical third (2.39 ± 0.72 MPa).

The intergroup comparison analysis revealed that there were no significant differences in the bond scores among Group A2 (H_2SO_4 + Super-Bond C&B) and Group B2 (RF + Super-Bond C&B) at the coronal, middle, and apical levels. Both groups exhibited similar bond strength values: Group A2 (Coronal: 8.99 ± 0.44 MPa, Middle: 8.25 ± 0.69 MPa, Apical: 4.59 ± 0.41 MPa) and Group B2 (Coronal: 9.57 ± 0.67 MPa, Middle:

9.24 ± 0.62 MPa, Apical: 5.40 ± 0.94 MPa). Similarly, Group A1 (H_2SO_4 + Panavia[®]V5) and Group B1 (RF + Panavia[®]V5) also demonstrated comparable outcomes in terms of EBS of PEEK post to root dentin ($p > 0.05$).

The bond strength values for Group A1 were Coronal: 7.64 ± 0.74 MPa, Middle: 6.79 ± 0.39 MPa, Apical: 3.57 ± 0.45 MPa, while for Group B1, they were Coronal: 7.69 ± 0.23 MPa, Middle: 7.39 ± 0.71 MPa, Apical: 3.48 ± 0.54 MPa. Additionally, Group C2 (Al_2O_3 + Super-Bond C&B) and Group D2 (NC + Super-Bond C&B) also exhibited similar outcomes in terms of bond strength ($p > 0.05$). The bond strength values for Group C2 were Coronal: 6.18 ± 0.54 MPa, Middle: 5.74 ± 0.15 MPa, Apical: 3.12 ± 1.01 MPa, while for Group D2, they were Coronal: 6.57 ± 0.55 MPa, Middle: 5.61 ± 0.63 MPa, Apical: 3.12 ± 0.27 MPa.

In terms of intergroup comparison, Group C1 (Al_2O_3 + Panavia[®]V5) and Group D1 (NC + Panavia[®]V5) demonstrated comparable bond scores at the coronal, middle, and apical levels. The bond strength values for Group C1 were Coronal: 4.89 ± 0.23 MPa, Middle: 4.44 ± 0.74 MPa, Apical: 2.54 ± 0.63 MPa, while for Group D1, they were Coronal: 4.59 ± 0.22 MPa, Middle: 4.29 ± 0.12 MPa, Apical: 2.39 ± 0.72 MPa ($p > 0.05$). The overall results indicate that the different conditioning regi-

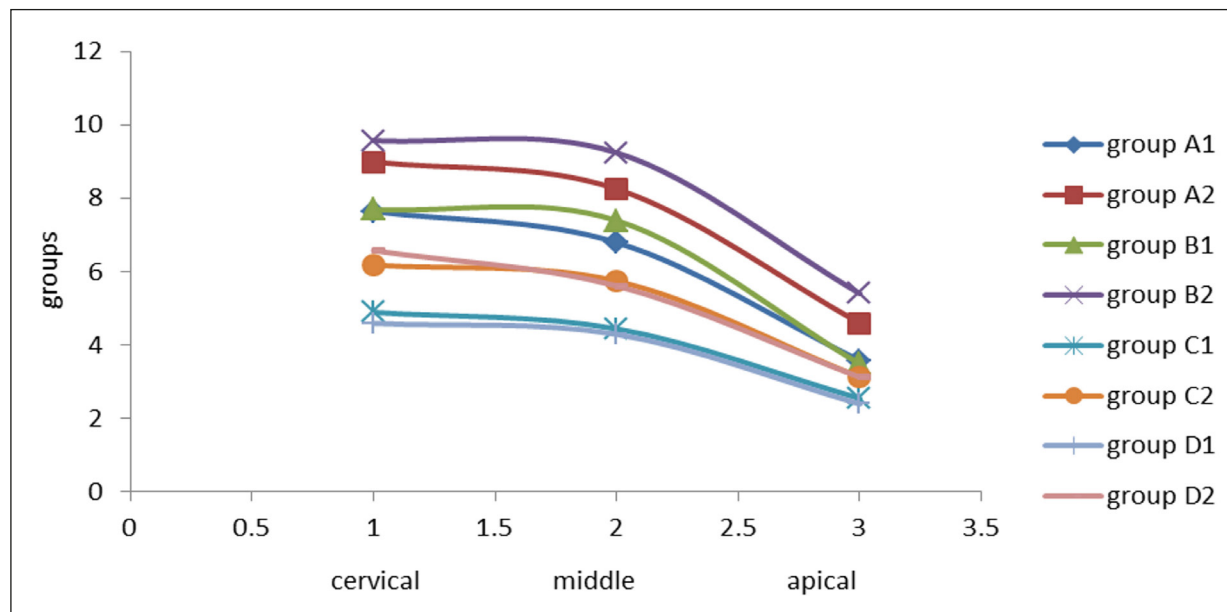


Figure 1. Means and Standard deviations (SD) of extrusion bond strength (MPa) values among experimental groups at cervical, middle, and apical levels of root. Group A1: H_2SO_4 + Panavia[®]V5, Group A2: H_2SO_4 + Super-Bond C&B, Group B1: RF + Panavia[®]V5, Group B2: RF + Super-Bond C&B, Group C1: Al_2O_3 + Panavia[®]V5, Group C2: Al_2O_3 + Super-Bond C&B, Group D1: NC + Panavia[®]V5, Group D2: NC + Super-Bond C&B.

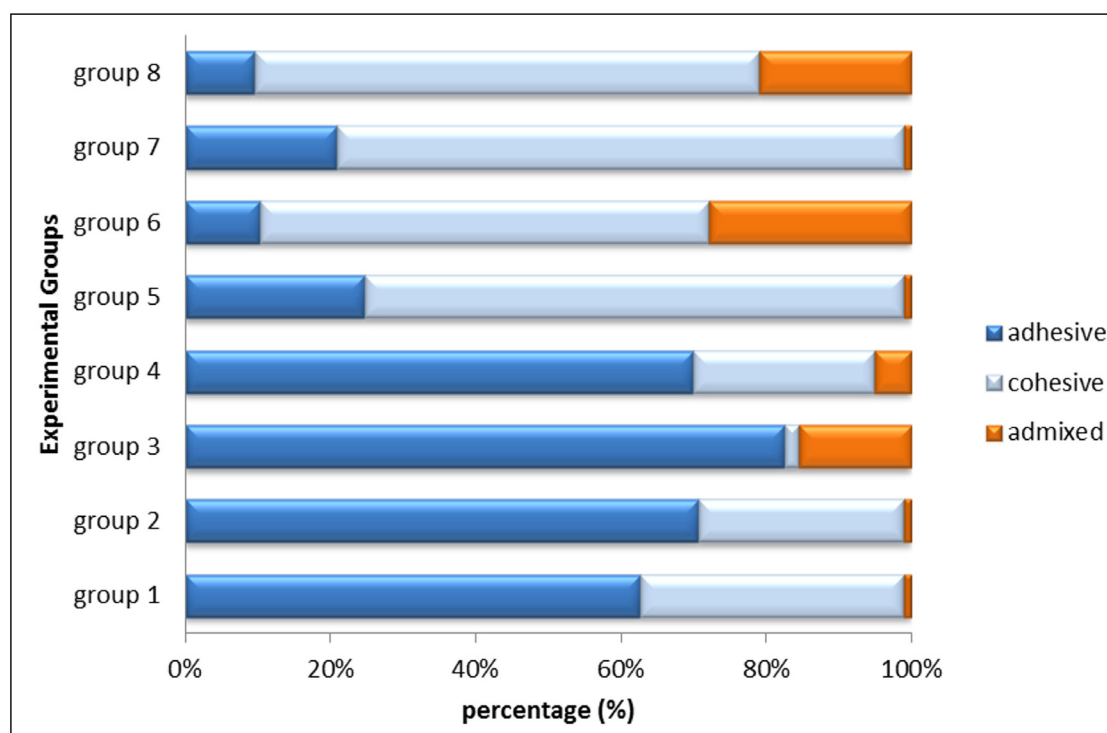


Figure 2. Percentage of failure analysis in different experimental groups.

mens and luting cement types did not significantly influence the bond strength of PEEK posts to root dentin ($p>0.05$). Regarding the failure mode analysis, it was observed that Group A1, A2, B1, and B2 predominantly exhibited adhesive failure patterns. On the other hand, Group C1, C2, D1, and D2 showed a higher occurrence of cohesive failures, followed by admixed failure patterns (Figure 2).

Discussion

The purpose of this laboratory-based study was to test two hypotheses. The first hypothesis suggested that using different methods of pretreatment (H_2SO_4 , RF, and Al_2O_3) would not significantly impact the EBS of PEEK posts bonded to radicular dentin compared to the control group (NC). However, the results partially rejected this hypothesis, as the SA and RB-treated groups showed better bond values compared to the sand-blasting and control groups.

The second hypothesis proposed that MMBC and CBC would exhibit similar bond strengths regardless of the surface conditioner used. However, the findings completely rejected this hypoth-

esis, as MMBC displayed superior bond integrity for PEEK posts compared to CBC.

Previous studies²⁴⁻²⁷ investigating the adhesion of PEEK to resin cement have consistently shown that without surface treatment, the bond strength is insufficient. Similarly, the results of our study supported this finding by demonstrating that surface pretreatment with H_2SO_4 and RF led to improved bond values of PEEK posts to root dentin. These findings align with a study conducted by S. Shabib²⁸, providing further support for the efficacy of H_2SO_4 and RF as surface pretreatment methods for enhancing the bond strength of PEEK posts to root dentin²⁸. It was revealed that the use of H_2SO_4 and Riboflavin photosensitizer positively influenced the bond strength of PEEK posts. These results can be explained by the mechanism of surface conditioning through photodynamic therapy (PDT), which involves the generation of reactive oxygen species (ROS) and oxygen free radicals. These chemical species contribute to the enhancement of the surface energy of the PEEK post, thereby improving its bond strength to the substrate²⁹. As a result, the PDT-induced anti-oxidative mechanism leads to increased surface roughness of the PEEK material. This surface roughness

promotes the formation of resin tags, which are microscopic extensions of resin cement, into the irregularities of the PEEK surface. These resin tags enhance the interlocking and adhesion between the PEEK and resin cement, thereby improving the overall bonding effectiveness between the two materials³⁰.

Similarly, the group that received H_2SO_4 pretreatment exhibited comparable bond strength values to the group treated with RF. One possible explanation for this finding is the generation of micro-porosities on the surface of the PEEK post. This can be attributed to the high corrosive capacity of H_2SO_4 , which dissolves the PEEK matrix through sulfonation action³¹. The chemical conditioning-induced oxidation of the PEEK surface also leads to the disruption of the aromatic ring structure, increasing the polarity of the surface³². This process introduces reactive functional groups that can form bonds with the cement used, resulting in enhanced bond strength. This observation is consistent with the findings reported by Zhou et al³³.

The low bond strength observed in the Al_2O_3 -treated group can be attributed to the formation of increased levels of porosity and uneven surfaces due to the abrasive particles impacting the PEEK surface at high speeds^{4,34}. This phenomenon may adversely affect the ability of PEEK to establish a strong bond with the resin cement, resulting in weakened interactions between the materials. This is consistent with previous studies^{4,34} that have highlighted the potential negative impact of abrasive treatments on the bond strength of PEEK³⁵.

Based on the classification of luting types of cement into MMA and CBC based on their material composition, it was observed that Super-Bond C&B, an MMA resin cement used for PEEK cementation, exhibited significantly higher bond integrity compared to CBC luting cement in all experimental groups³⁶. Two possible explanations can be given to justify this outcome. Firstly, the high bond strength may be attributed to the formation of a semi-interpenetrating polymer network (semi-IPN) structure at the interface between PEEK and the cement^{37,38}. This structure has been shown to greatly enhance the affinity between the two materials, as documented in various studies³⁶. The second possible explanation is related to the wettability of the resin cement. Pretreated PEEK surfaces exhibit various grooves of different depths and diameters. The low viscosity of MMA-based resin cement allows them to

penetrate the small grooves on the PEEK surface. When the penetrated MMA cures to polymethyl methacrylate (PMMA), it mechanically interlocks with the cement/PEEK contact³⁹. On the other hand, composite-based resin cement contains ceramic fillers that cannot enter the small grooves and roughness created on the PEEK surface after surface treatment, resulting in weak mechanical interlocking and, ultimately lower bond strength^{36,39}. However, it is important to note that further studies are still required to draw definitive conclusions and fully understand the outcomes of the existing studies.

The failure mode analysis revealed that samples treated with RF and H_2SO_4 predominantly exhibited adhesive failure patterns. This can be attributed to factors such as inadequate surface preparation, incompatible materials, insufficient adhesive application, and mechanical stress^{40,41}. On the other hand, the non-disinfection and Al_2O_3 groups displayed cohesive and admixed types of failure more frequently. Furthermore, it is important to acknowledge certain limitations of the present investigation. The concentration of PS and the specific chemical agent used for surface treatment may have influenced the results. Additionally, variations in dentinal structure among the samples could have also affected the outcomes. Moreover, as the study was conducted in a laboratory setting using *in vitro* methods, caution should be exercised in generalizing the findings to clinical scenarios. Future studies should consider conducting topographic analysis of PEEK posts using scanning electron microscopy (SEM) to further validate the results. Furthermore, investigating the effect of different conditioning methods on the mechanical properties of PEEK posts is essential for a comprehensive understanding of their performance.

Conclusions

The findings suggest that Riboflavin activated by Photodynamic therapy (PDT) and H_2SO_4 can be effective surface conditioners for PEEK posts. These treatments have shown potential for enhancing the bond strength between PEEK and resin cement. Additionally, the study revealed that MMA-based cement outperformed composite-based cement in terms of bond integrity with PEEK posts. This indicates that MMA-based cement may be a more suitable choice for cementing PEEK posts.

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Conflict of Interest

The authors of the present study have no conflict of interest.

Authors' Contributions

Conceptualization, methodology, software validation; formal analysis, investigation, resources; data curation, writing-original draft preparation, writing-review and editing; visualization was performed by Thamer Almohareb, Khold Alahdal, Ahmed Maawadh, Ahoud S Alshamrani, Laila Aldeeb, Ali Alrahlah. All the authors of the present study share contributions according to the author's sequence.

Ethics Approval

Specialist dental practice and research center (UDCRC-036-2022) (Saudi Arabia) approved the protocol.

Informed Consent

Not applicable.

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