Surface characterization and bonding properties of milled polyetheretherketone dental posts

Benli, Merve ; Eker Gümüş, Beril ; Kahraman, Yusuf ; Huck, Olivier ; Özcan, Mutlu

Abstract: PEEK has been used in many dental applications except intra-radicular post. The aim of this study is to test polyetheretherketone (PEEK) as a dental post material through tensile bond strength (TBS) and surface roughness (SR), and to compare it with glass-fiber and cast-metal posts. Thus, 60 human maxillary central incisors with a single root were endodontically treated and divided into three groups (n = 20) according to the type of post (Group P: PEEK, Group F: Glass-fiber, Group M: Cast-metal). Appropriate surface treatment was employed for each group and SR was determined by a three-dimensional non-contact profilometer before cementation. All posts were luted to the canal dentin using self-etch resin cement (Panavia F2.0). Pull-out test was performed on a universal testing machine at a speed of 0.5 mm/min crosshead speed until failure, and TBS were calculated. One-way ANOVA, Tukey's HSD, and Pearson chi-squared tests were performed for statistical analyses (= 0.05). According to the results, group F demonstrated the highest SR (2.93 ± 0.18 µm) and lowest TBS values (10.05 ± 0.53 MPa), while group P exhibited lowest SR (1.37 ± 0.11 µm) and highest TBS values (14.33 ± 0.58 MPa) ($p < 0.001$). No significant differences in failure modes were identified among groups, mostly adhesive (p = 0.243). As conclusion, PEEK may be a reliable and contemporary option for dental post systems when used with appropriate surface treatment and luting agent. This high-performance polymer may be a novel candidate as a contemporary dental post system due to its superior mechanical, chemical, thermal, and esthetical properties with low risk of fracture.

DOI: https://doi.org/10.1007/s10266-020-00484-1

Originally published at:
Benli, Merve; Eker Gümüş, Beril; Kahraman, Yusuf; Huck, Olivier; Özcan, Mutlu (2020). Surface characterization and bonding properties of milled polyetheretherketone dental posts. Odontology / the Society of the Nippon Dental University, 108(4):596-606.
DOI: https://doi.org/10.1007/s10266-020-00484-1

Surface Characterization and Bonding Properties of Milled Polyetheretherketone Dental Posts: An In Vitro-Study

Merve Benli● Beril Eker Gümüş● Yusuf Kahraman● Olivier Huck● Mutlu Özcan

Ass. Prof. Dr. Merve Benli

Istanbul University, Faculty of Dentistry, Department of Prosthodontics, Istanbul, Turkey

Dr. Beril Eker Gümüş

Yıldız Technical University, Science and Technology Application and Research Center, Istanbul, Turkey

Dr. Yusuf Kahraman

Yıldız Technical University, Science and Technology Application and Research Center, Istanbul, Turkey

Prof. Dr. Olivier Huck

INSERM, UMR 1260 'Osteoarticular and Dental Regenerative Nanomedicine', Faculté de Médecine, Fédération de Médecine Translationnelle de Strasbourg (FMTS), France

Pôle de Médecine et de Chirurgie Bucco-Dentaire, Hôpitaux Universitaires de Strasbourg, France

Faculté de Chirurgie Dentaire, Université de Strasbourg, France

Prof. Dr. Mutlu Özcan

University of Zürich, Dental Materials Unit, Center for Dental and Oral Medicine Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zürich, Switzerland

Corresponding to:

Ass. Prof. Dr. Merve Benli

Istanbul University, Faculty of Dentistry, Department of Prosthodontics, Istanbul, Turkey

Turgut Özal Millet Cd, 34093 Fatih/İstanbul

Tel: +90 212 414 20 20-30362

E-mail: benlimerve@hotmail.com

Acknowledgements

The authors would like to acknowledge Benan İlayda Tellioğlu Karaman, Şeyma İbiçoğlu and the research assistants of the Department of Maxillofacial Prosthodontics at Istanbul University for their assistance.

Disclosure of Statement

The authors do not have any financial interest in the companies whose materials included in this article.

Surface Characterization and Bonding Properties of Milled Polyetheretherketone Dental Posts: An In Vitro-Study

Abstract

Objectives: To test polyetheretherketone(PEEK) as a dental post material through tensile bond strength (TBS) and surface roughness (SR), and to compare it with glass-fiber and cast- metal posts.

Materials and Methods: Sixty human maxillary central incisors with a single root were endodontically treated and divided into 3 groups (n=20) according to the type of post (Group P: PEEK, Group F: Glass-fiber, Group M: Cast-metal). Appropriate surface treatment was employed for each group and SR was determined by a three-dimensional non-contact profilometer before cementation. All posts were luted to the canal dentin by using self-etch resin cement (Panavia F2.0). Pull-out test was performed on a universal testing machine at a speed of 0.5 mm/min crosshead speed until failure, and TBS were calculated. One-Way ANOVA, Tukey's HSD, Pearson chi-squared tests were performed for statistical analyzes (α = 0.05).

Results: Group F demonstrated the highest SR (2.93±0.18 µm) and lowest TBS values (10.05±0.53 MPa), while group P exhibited lowest SR (1.37±0.11 µm) and highest TBS values (14.33±0.58 MPa) (p<0.001). No significant differences in failure modes were identified among groups, mostly adhesive (p=0.243).

Conclusions: PEEK may be a reliable and contemporary option for dental post systems when used with appropriate surface treatment and luting agent.

Clinical significance: PEEK has been used in many dental applications except intraradicular post. This high-performance polymer may be a novel candidate as a contemporary dental post system due to its superior mechanical, chemical, thermal and esthetical properties with low risk of fracture.

Keywords: Polyetheretherketone, dental post, tensile bond, surface roughness, post failure.

Introduction

Endodontic treatment, or root canal treatment, is a very common treatment in dentistry.[1] Such an effective treatment procedure leads to reduced mechanical properties of the endodontically treated teeth (ETT) due to decreased tooth resistance to fracture in comparison with vital teeth. Such changes are mainly due to extensive tissue removal during endodontic access.[2-4]

The restoration of ETT with more than 50% of coronal tooth loss often requires the placement of a dental post and core.[5-7] Dental post design and post material play an important role in the survival probability of ETT and affect the stability of the restoration.[8-10] It has been reported that ETT have been reinforced with the use of dental posts and increase of the fracture strength has been demonstrated clinically (4,5-50 y) emphasizing the need of new biomaterials development. Contemporary dentistry adopted two types of dental post systems, prefabricated and cast metal ones, to provide the crown retention and to restore the ETT.[11] Although cast metal dental posts have been widely used for years, drawbacks and disadvantages such as metallic color, corrosion, decementation,[12] non-adhesive bonding, loss of retention, high modulus of elasticity,[13] stress concentration, root fractures and necessity of removal of extensive root structure have driven dental practitioners and manufacturers to investigate for new options such as fiber post systems.[14-16] Fiber-reinforced resin or glass-fiber posts are considered as advantageous options for the rehabilitation of ETT in comparison with metal posts due to their sufficient esthetics, uniform stress distribution, biocompatibility, corrosion resistance, easy and rapid handling and adhesion to restorative resinous materials.[11,17-21] Despite such interesting properties, fiber posts induce mechanical stress in cervical dentine and restoration margin and do not strengthen the tooth structure.[22] Besides, even though they have a lower elasticity modulus (45.7-53.8 GPa) than those of metal posts (95.0- 110.0 GPa), this modulus is still almost three times that of dentin (18.6 GPa).[23,24] Their main disadvantage is low bond strength to root canal wall or the dislodgement of the post, consequently leading to the micro-leakage and the failure of the dental post.[2]

Polyetheretherketone (PEEK) is a semi-crystalline high-performance thermoplastic polymer which is becoming more popular in dentistry due to its interesting mechanical, thermal, and chemical properties, fatigue resistance, low water absorption and excellent biocompatibility.[25-28] Its mechanical properties, like elastic modulus, can also be set by changing the filler content and incorporating inorganic fillers.[25,29] The elastic modulus of PEEK similar to dentin is a key advantage that reduces forces transferred to the restorations and enables this material to function as a stress breaker, as uniformly distributed stress has been reported when dental materials have an elastic modulus similar to dentin. [25,27,30] Furthermore, its radiolucency renders possible the step-by-step evaluation of the treatment procedures.[31,32] PEEK has been proposed as an esthetic (whitish color), metal free and functional material in various dental applications, such as fixed prostheses, removable prostheses and its components, dental implants, individual abutments, maxillary obturator prostheses and orthodontic wires.[33,34] This material, which exhibits high-fracture resistance with a fracture load of 1383 N, has become an alternative to glass ceramics and metal with its shock-absorbing ability.[28,30,35] However, there has been no attempt to evaluate this notable, high-performance polymer PEEK as a dental post material by using appropriate surface treatments and resin bonding systems in human teeth.

Therefore, due to the lack of consensus regarding the ideal post material for restoring ETT to date, this study aims to make further development about this deficiency by evaluating PEEK as a potential dental post material. It is aimed to assess the long-term safety and biomechanical behavior of PEEK as a post material in comparison with other contemporary dental posts. The first hypothesis tested was that the PEEK post system would have a higher surface roughness (SR) and tensile bond strength (TBS) than glass-fiber and metal posts. The second hypothesis is that PEEK would represent a good alternative for dental post materials.

Materials and methods

This study was approved by the Ethics Committee of the Faculty of Dentistry (Process number 2019/19) and teeth were donated by anonymous patients who applied for dental treatment.

Sample materials

In the present study, three types of materials were evaluated as dental post systems by preparing post samples, including base metal, glass-reinforced fiber and PEEK. The details and compositions of the materials employed in the present study were displayed in **Table 1.**

Sample selection and preparation

The sample size for posts (n=20 per group) was determined based on an initial power analysis (PS software; Dupont and Plummer, 1997) which was estimated according to similar previous studies (mean gamma count with 80% power and 5% significance level).[39,72,73] Therefore, 60 human permanent maxillary central incisors with a single and patent root canal, mature apex, straight root, no caries involving the root, no previous endodontic treatment and no resorption/internal calcifications were selected based on radiographic and clinical examinations. All teeth were cleaned of both soft tissues and calculus and stored for 3 months in 0.1% thymol solution (Thymol; Supelco®, Sigma_Aldrich Chemie GmbH, St. Louis, Missouri, USA) at room temperature. To provide roots with a standard 15 mm length, the crown portions of the teeth were removed by cutting close to the cementoenamel junction and perpendicular to their long axis.

Root canal preparation strategy

Following the removal of the dental pulp, a 10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) was used to confirm the patency and to obtain a standardized working length (14 mm) by passing 1 mm less beyond the apex of each canal. All root canal preparations were performed manually by a single operator using a step-back technique with the final file number of 55 (Dentisply Maillefer, Ballaigues, Switzerland). During root canal preparation, 2 ml of 5.25% NaOCl irrigation was performed after each instrument, followed by distilled water rinsing. After drying canals with paper points (Ocean Dental, Istanbul, Turkey), endodontic obturation was achieved with gutta-percha cones (Diadent, Chungcheongbuk-do, Korea) and sealed (AH-Plus; Dentsply DeTrey GmbH, Konstanz, Germany) by performing the lateral condensation technique with a controlled force of 3 kg. Cervical portions of root canals were obturated with a temporary filling material (Coltosol-F; Coltène/Whaledent AG, Altstätten, Switzerland), and teeth were left in distilled water at 37°C for one week.

Post space preparation was performed following the standard technique with a length of 10 mm and leaving 4 mm of gutta percha as the apical seal. Removal of gutta-percha up to the specified depth was fulfilled with a warm plugger (D-PERFECT; Guangdong, China) and enlargement of the root canals was performed with drills as supplied by the manufacturer of the fiber post system (Angelus Dental Products Industry, Paraná, Brasil).

Intra-radicular post system preparation

The specimens with prepared root canals were randomly divided into 3 groups according to the selected intra-radicular post system:

Group F: Glass-fiber post (Angelus Reforpost, Exacto #3; Angelus Dental Products Industry, Paraná, Brasil), 10 mm length

Group M: Cast-metal post (Wiron 99; BEGO Bremer Goldschlägerei Wilh, Bremen, Germany), 10 mm length

Group P: Polyetheretherketone (PEEK) (PEEK-OPTIMA®; Juvora Ltd., Wyre, Lancashire, UK), 10 mm length

In group F, glass fiber post system of 1.5 mm diameter (Angelus Reforpost, Exacto #3) was selected. In group M, cast-metal posts made of Ni-Cr alloy (Wiron 99) were used. Metal posts of the same shape and size as the posts of group F were obtained by molding the root canals with acrylic resin (Palavit G; Heraus Kulzer, Hanau, Germany) as per standard dental casting procedure and smoothed with 320,400 and 600 grits of silicon carbide paper respectively (3M, Campinas, São Paulo, Brazil).[36] All casting procedure was performed by the same dental technician.

In group P, PEEK posts were produced by a CAD-CAM system (Amann Girrbach AG, Koblach, Austria). Virtual images were obtained with the laboratory scanner of the digital system (Ceramill® map 200+; Amann Girrbach AG, Koblach, Austria) by scanning a prefabricated glass fiber post from the group F. After developing milling plan, 20 posts were milled from filled PEEK disc (PEEK-OPTIMA Dental Disc) in the milling unit of the selected digital system (Ceramill® mikro 5X; Amann Girrbach AG, Koblach, Austria). Produced PEEK posts were polished to obtain a standard surface under abundant water for 60 s using silicon carbide papers from P400 up to P2000 in an automatic polishing device (SAPHIR 550; ATM GmbH, Mammelzen, Germany) at 25 N vertical force.

Surface treatment

Before surface treatment of the posts to achieve cementation, all of them were cleaned with 70% ethanol, water and air-dried. According to the instructions from the manufacturer, group F was conditioned with phosphoric acid (K-Etchant GEL; Kuraray, Umeda, Osaka, Japan) treatment for surface alteration. Acid was left for 5 s and washed with water and dried with airflow. Airborne particle abrasion with 50 µm Al_2O_3 particles (Cobra; Renfert, Hilzingen, Germany) was applied to group M for 20 s at a pressure of 35 psi conditions from 4 sides and 10 mm from the post surface.[37] For group P, post surfaces were etched with sulfuric acid (98%) (sulfuric acid; BRTR Kimya, İzmir, Turkey) for 1 min and then rinsed with deionized water for 1 min carefully and dried (**Figure 1**).[35,52]

Surface roughness measurement and surface observation

After surface treatments, the surface roughness (Ra) of all the posts was determined by a 3D non-contact profilometer (AEP Nanomap-1000WLI; AEP Technology, Santa Clara, CA, USA) with an optical resolution of 550 nm. Average of Ra values were calculated with SPIP software (Image Metrology A/S, Lyngby, Denmark) by using three single individual measurements, according to ISO 4287.[38]

The structural surface topography of each group after surface treatment was observed under a scanning electron microscope (SEM) (Carl Zeiss EVO LS 10; Carl Zeiss NTS, Germany) at 250x magnification after sputter-coating with gold.

Luting posts

Before luting, all root canals were cleaned by 96% ethanol, irrigated by saline solution and dried with paper points. Self-etch resin cement (Panavia F2.0; Kuraray Noritake Dental Inc. Okayama, Japan) was used for luting process. After applying ED Primer A and B properly, pastes (A and B) were prepared by mixing and implemented to the posts to insert them into the post spaces. During the insertion of the posts, a loading apparatus (custom-made) was utilized to provide the standardized force of 49 N for 60 s to stabilize them. Resin cement was light polymerized (DEMI™ light-curing unit; Kerr Corporation, Orange, CA, USA) for 40 s. All luting procedure was performed according to the instructions from the manufacturer.

Aging process and pull-out test

Samples were left in distilled water at 37 °C for 30 days and then underwent 6000 thermocycles (5°C-55°C) with a 30 s dwell and 6 s transition time before pull-out test (Automated thermocycling machine; Gökçeler Machines, Sivas, Turkey).

Pull-out test was carried out with a universal testing machine (Model 8872; Instron Corp., Norwood, MA) at a speed of 0.5 mm/min crosshead speed until failure. The samples were set in the jig of the machine with the post lateral surface parallel to the loading direction. The peak force that induced debonding of the post from the root canal was recorded in Newton (N) and taken as the point of failure. The bonding area of each specimen was calculated by considering the conical shape of the root canal to determine the bond strength.[39] TBS in MPa was calculated by following the formula (TBS: fracture load (N)/ bonding area (mm^2)=N/mm^2=MPa) and the method described by Quitero et al.[39,40]

Failure mode analysis

Failure modes were determined under an optical microscope (Axio Zoom.V16; Carl Zeiss Meditec, Oberkochen, Germany) at a 150x magnification after debonding by one calibrated examiner who was unaware of the group distribution. Modes were categorized as follows: (1) adhesive (between the resin cement and post), (2) cohesive (within the post or resin cement material), (3) mixed (both cohesive and adhesive).[41] Determination of the failure mode was carried out by dividing the bonding area into 4 sections as described by Renata et al.[42] Additionally, SEM (Carl Zeiss EVO LS 10; Carl Zeiss NTS, Germany) at 33x and 101x magnifications was used to evaluate the surface topography and further assess the fractures by obtaining at least three images of each post group.

Statistical analysis

Statistical analyses were made using standard statistical software (SPSS V23; IBM Armonk, NY, USA). All data were submitted to the Shapiro-Wilk test to verify the normality of data and one-way ANOVA was used to compare normally distributed data of Ra and TBS between groups. Multiple comparisons were carried out with Tukey's HSD and Pearson chi-square test was used to compare failure rates by groups. Analysis results were shown as means and standard deviation for quantitative data and as frequency (%) for categorical data. Significance level was considered for $p<0.05$.

Results

Surface roughness and SEM evaluation

The mean and standard deviation of Ra values of the post surfaces with surface treatments were listed in **Table 2**. The Ra values were significantly different among tested posts materials ($p<0.001$). Group F showed the greatest surface roughness (2.93±0.18 µm), while the group P exhibited the lowest values (1.37±0.11 µm).

SEM demonstrated a characterization of PEEK by a sponge-like porous surface with pitting and wide and deep pores from sulfuric acid etching that proposed a considerable effect on the adhesion between PEEK and resin cement. Chemical etching resulted in micropores and filler particles on the PEEK which increased surface roughness. Group F exhibited visual cues like inward rhombic traces performed by the manufacturer for mechanical retention of the post. Additional surface treatment with phosphoric acid resulted in a cleft-like appearance in some parts of the surface. The irregular structured surface and amorphous cavities with some porosities were observed in the metal posts. Surfaces of the metal posts were also covered with crystalline deposits due to airborne abrasion **(Figure 2).**

TBS analysis

Results of TBS values for all post specimens to resin cement were shown in **Table 3.** The Shapiro-Wilk test displayed no violation of the assumption of normality for 75% of the tested groups, providing parametric analysis of the data. The type of post material showed a significant effect on the TBS results between groups($p<0,001$). The TBS values of the groups ranged from 10.05±0.53 to 14.33±0.58 MPa. The PEEK posts treated with sulfuric acid showed the highest TBS mean values (14.33±0.58 MPa), while the lowest values were found on fiber posts (10.05±0.53 MPa).

Failure analysis

The distribution of failure modes was displayed in **Table 4.** Failure modes between the tested groups were not significantly different ($p=0.243$) and all groups displayed predominant adhesive failures between luting agent and the post material. Additionally, mixed failures were observed for group F (35%), followed by group M (30%) and group P (20%). No cohesive failure was observed within the luting agent or the material bulk in any group, except from the group M within resin cement (10%).

The SEM and optical microscope analysis of the specimen surfaces after TBS test were shown in **Figure 3.** Almost no resin cement was observed on the surface of the post specimens of groups P and F, consisting with the predominant failure mode of adhesive. The root canals of these two groups had resin cement remnants on their inner walls, even though some mix failure areas can be found. Unlike the specimens of group P and F, group M also exhibited cohesive failure, with remnant resin cement attached to the metal post and the canal wall.

Discussion

The present study evaluated the performance of PEEK as a potential dental post material by comparing it with contemporary fiber and metal dental posts. The overall results of this study showed the highest TBS values for PEEK specimens, while SR values of PEEK did not exceed the values of fiber and metal group. Therefore, PEEK may represent a good alternative for dental post materials according to the tested parameters.

Sufficient elasticity is an expected property of an ideal dental post material to be compatible with the natural flexing movements of the tooth structure that cannot be achieved by rigid post systems.[43] Relative rigidity differences between dental post and root cause tensions at different intensities, leading to fractures and debonding.[44] In the treatment of ETT, the use of high elastic modulus metal posts has been found to induce vertical root fractures, leading to the extraction of the tooth.[45] This disadvantage is of paramount importance as root fracture and loss of post retention are considered the main biomechanical reasons for final restoration failure.[11] In the present study, a novel high performance polymer PEEK with an elastic modulus similar to that of dentine has been presented as an alternative post material due to its high fracture resistance and biocompatibility.[46-48] Based on the obtained results, PEEK post seems to be a suitable alternative system for clinical applications and consistent with the findings of the study from Lee et al., who evaluated PolyEtherKetoneKetone (a member of the PolyArylEtherKetone (PAEK) family) with same objective.[44] However, the used methodology was different as they performed the finite element analysis and did not use natural teeth with post-treatment, as in this study. Therefore, to our knowledge, the current paper is the first study to evaluate PEEK as a new dental post system with a set-up similar to clinical conditions.

TBS value has been reported to be correlated with the surface topography of the material being treated, increasing the contact area between the two materials with irregularities.[53] In this study, the SEM images of group P confirmed these data by displaying that the rough surface with some irregular holes and bulges resulted from sulfuric-acid etching was suitable for bonding resin cement with PEEK. According to the results, acid-treated PEEK (14.33±0.58 MPa) specimens have displayed the highest TBS values compared with groups F (10.05±0.53 MPa) and M (12.79±0.39 MPa). The TBS result for the group P of the present study was in agreement with the only comparable study which reported 13.37±2.38 MPa TBS.[54] This slightly lower TBS value can be attributed to more thermal cycling applied than our study with different surface treatment methods (sandblasting) and different types of adhesive materials used. Comparison for PEEK has been performed partially with literature due to limited data, as remaining studies evaluated the adhesion of PEEK to veneering resins with various bond strength tests. Several studies have focused on the bond strength (tensile or shear) of the adhesive systems or veneering resins to PEEK and showed various results ranging from 3.6 MPa to 30.9 MPa.[30,40,55] These wide range results can be due to the applied pretreatment methods, the content of the tested PEEK material (unfilled, filled), adhesive system, thermal aging, and various testing methodologies. More specifically, TBS results of the groups M and F agreed with a previous study that had a similar study design with the present study.[56] Another point worth noting is that metal and fiber posts have been shown to generate higher stress distribution profiles than PEKK cases in a recent study.[44] Taken together, these results pointed out that PEEK post seems to be advantageous in bond strength and stress distribution profile over other available dental post options. However, this study investigated only the bond strength of the PEEK posts and even though PEEK exhibits a high-fracture load, it is not possible to comment directly on the fracture resistance of these post system. [28,30,35] Therefore, the fracture resistance should be evaluated in further studies under an appropriate set-up to reveal whether this post system is clinically feasible or not. [71]

In adhesive procedures, SR is a significant factor, and it requires various surface treatment methods to increase the bonding area and microroughness of the dental material.[57] The quantitative Ra data measured corresponded to the qualitative investigation by SEM shown in **Figure 2** that exhibit different surface appearances for the groups. Confirmed by SEM images, PEEK demonstrated pits and pores distributed with filler particles on the surface, as demonstrated by other previous investigations.[49,50] Although group P showed the lowest Ra (1.37±0.11 µm) value, it resulted in the highest TBS. It seems that chemical and micro-topographical alterations of the PEEK surface created by sulfuric acid etching improved the surface polarity and penetration of the resin cement.[50] In this study, PEEK posts showed higher Ra values than previous studies with the same pretreatment conditions.[28,30,35,40] This discrepancy can be attributed to the difference in the contents of the PEEK materials, fabrication process, and adhesive systems used. As expected, the outcomes of the present study for fiber and metal posts coincide with previous studies by revealing similar results in surface roughness.[58,59,60] However, one can expect that different surface treatment modalities for PEEK may change results, warranting further studies. Additionally, interpenetrating polymer networks can be employed to contribute to a fruitful adhesion between the PEEK and resin-based materials.[61]

The failure mode of all groups was predominantly adhesive. A small amount of resin cement remnant has been shown on the PEEK specimens after the TBS test with cohesive failure areas in the luting agent **(Figures 3a, d, and g)**. Even though some resin remnant can be found on the group P, a substantial portion of luting agent was found to be attached to the PEEK surface with the presence of mechanical and adhesive bonding during the bonding process.[27,40,54,62,63] This can be due to the content of the resin cement like 10-methacryloyloxydecyldihydrogenphosphate (MDP), as MDP has a binding effect on the roughened PEEK surface due to the impact of bifunctional adhesive monomer.[44] For group F, some resin remnants were seen to be attached to the post surface, but this group mainly expressed adhesive failure mode, as was in the previous studies.[64-67] For group M, very few cohesive failures were observed in addition to the main failure mode of adhesive differently. This result was also consistent with the previous studies that applied the same luting agent to metal surfaces as in the current study.[68] Although the high TBS values provided good adhesion between post material and luting agent, the bonding process, especially for PEEK, should be improved because of the ratio of the adhesive failures.

In previous studies, higher bonding values have been obtained by applying the acid etching to PEEK surface.[40,49,50] Treating PEEK with sulfuric acid increases the intensity of the functional carbon-oxygen groups on the superficial layer of the material, thus resulting in more functional elements available for the adhesive system.[30,35,50] Furthermore, most of the previous studies used sulfuric acid (98%) for surface treatment of PEEK[35,51,52] and it was shown to increase bond strength value.[26] Thus, in the current study, sulfuric acid pre-treatment has been performed for PEEK surface conditioning due to its extremely corrosive nature.[51] Due to its dangerous nature not compatible with a chair-side application, alternatives to such conditioning should be proposed including pre-treatment by the manufacturer, and other surface treatment modalities such as silica coating, air abrasion, applying adhesive systems, lasers, plasma, or their combinations.[26,30,53] These procedures need to be evaluated in future studies.

PEEK material may be a good option as a dental post when considering the cost-effectivity of the treatment, as PEEK framework designs have been shown to reduce cost.[74] Among the three types of post systems evaluated in the present study, base metal ones are the least costly option and may be considered in strict cost containment. [74,75] Metal posts are cost-effective if they are in preformed shape, as they can either be cast and laboratory procedures for cast metal posts will cause the patient to make additional payments.[75] According to this, the PEEK post is a moderate option in cost, whereas the glass-fiber post is found to be the costliest. However, the production of PEEK posts by using a CAD/CAM system includes special training, scanning equipment and waste of material due to the subtractive method, and is more expensive than conventional milling techniques. [76] Thus, it is recommended to benefit conventional milling methods in obtaining PEEK posts, and these results are only valid for initial treatment costs for ETT. Because, it is difficult to evaluate the long-term cost-effectiveness of PEEK posts due to scarce data and further studies should be performed considering the complications of this system to better decide the effectivity when compared with present post systems.

From a methodological point of view, a limitation of the present study is the lack of reproducing the oral environment as individual variations can change the results. However, as this was the first study to evaluate PEEK as a dental post, the main goal was to evaluate the overall feasibility and establish a bonding between the root canal and PEEK. Other limitations were applying one type of surface treatment modality for PEEK, using one type of luting agent and tooth (anterior). However, the findings of the present study can be used for clinical decisions in dental post applications integrating with the patient's need and the consideration of the dental practitioner. Further studies should be performed to confirm the present findings and elucidate the long-term feasibility of use of the PEEK as a dental post material.

Conclusions

Within the limitations of the present study, it could be concluded that sulfuric acid treatment seemed suitable to bond the PEEK post system to root dentine. According to the results, PEEK showed the highest TBS and lowest Ra values as a dental post system than those from metal and fiberglass posts. Thus, the findings of the present study and lower elastic modulus may point out that PEEK may be a reliable and contemporary option for dental post systems.

References

1. Manfredi M, Figini L, Gagliani M, Lodi G. Single versus multiple visits for endodontic treatment of permanent teeth. Cochrane Database Syst Rev. 2016;12:CD005296.

2. Dietschi D, Duc O, Krejci I, Sadan A. Biomechanical considerations for the restoration of endodontically treated teeth: a systematic review of the literature, Part II (Evaluation of fatigue behavior, interfaces, and in vivo studies). Quintessence Int. 2008; 39:117-29.

3. Dietschi D, Duc O, Krejci, Sadan A. Biomechanical considerations for the restoration of endodontically treated teeth: a systematic review of the literature--Part 1. Composition and micro- and macrostructure alterations. Quintessence Int. 2007;38:733-43.

4. Saker S, Özcan M. Retentive strength of fiber-reinforced composite posts with composite resin cores: Effect of remaining coronal structure and root canal dentin conditioning protocols. J Prosthet Dent. 2015;114:856-61.

5. Ferrari M, Vichi A, Grandini S, Goracci C. Efficacy of a self-curing adhesive-resin cement system on luting glass-fiber posts into root canals: an SEM investigation. Int J Prosthodont. 2001;14:543-9.

6. Kimmel SS. Restoration and reinforcement of endodontically treated teeth with a polyethylene ribbon and prefabricated fiberglass post. Gen Dent. 2000;48:700-6.

7. Durmuş G, Oyar P. Effects of post core materials on stress distribution in the restoration of mandibular second premolars: a finite element analysis. J Prosthet Dent. 2014;112:547-54.

8. Yang A, Lamichhane A, Xu C. Remaining coronal dentin and risk of fiber-reinforced composite post-core restoration failure: a meta-analysis. Int J Prosthodont. 2015;28:258-64.

9. Ukon S, Moroi H, Okimoto K, et al. Influence of different elastic moduli of dowel and core on stress distribution in root. Dent Mater J. 2000;19:50-64.

10. Madfa AA, Kadir MR, Kashani J, et al. Stress distributions in maxillary central incisors restored with various types of post materials and designs. Med Eng Phys. 2014;36:962-7.

11. Madfa AA, Al-Hamzi MA, Al-Sanabani FA, et al. 3D FEA of cemented glass fiber and cast posts with various dental cements in a maxillary central incisor. Springerplus. 2015;4:598.

12. Drummond JL, Bapna MS. Static and cyclic loading of fiber reinforced dental resin. Dent Mater. 2003; 19:226-3.

13. Tay FR, Pashley DH. Monoblocks in root canals: a hypothetical or a tangible goal. J Endod. 2007;33:391-8.

14. Fredriksson M, Astbäck J, Pamenius M, Arvidson K. A retrospective study of 236 patients with teeth restored by carbon fiber-reinforced epoxy resin posts. J Prosthet Dent. 1998;80:151-7.

15. Dikbas I, Tanalp J. An overview of clinical studies on fiber post systems. ScientificWorldJournal. 2013;2013:171380.

16. Komada W, Miura H, Okada D, Yoshida K. Study on the fracture strength of root reconstructed with post and core: alveolar bone resorbed case. Dent Mater J. 2006; 25:177-82.

17. Goracci C, Ferrari M. Current perspectives on post systems: a literature review. Aust Dent J. 2011;56:77-83.

18. Santos-Filho PC, Veríssimo C, Soares PV, et al. Influence of ferrule, post system, and length on biomechanical behavior of endodontically treated anterior teeth. J Endod. 2014;40:119-23.

19. Schmitter M, Rammelsberg P, Gabbert O, Ohlmann B. Influence of clinical baseline findings on the survival of 2 post systems: a randomized clinical trial. Int J Prosthodont. 2007; 20:173-8.

20. Naumann M, Blankenstein F, Dietrich T. Survival of glass fibre reinforced composite post restorations after 2 years-an observational clinical study. J Dent. 2005; 33:305-12.

21. Naumann M, Sterzenbac G, Alexandra F, Dietrich T. Randomized controlled clinical pilot trial of titanium vs. glass fiber prefabricated posts: preliminary results after up to 3 years. Int J Prosthodont. 2007; 20:499-503.

22. Schmitter M, Rammelsberg P, Lenz J, et al. Teeth restored using fiber-reinforced posts: in vitro fracture tests and finite element analysis. Acta Biomater. 2010;6:3747-54.

23. Cheleux N, Sharrock PJ. Mechanical properties of glass fiber-reinforced endodontic posts. Acta Biomater. 2009; 5:3224-30.

24. Sakaguchi RL, Powers JM. Craig's Restorative Dental Materials.13th ed. Philadelphia, Elsevier Mosby; 2012.

25. Kurtz SM, Devine JN. PEEK biomaterials in trauma, orthopedic, and spinal implants. Biomaterials. 2007; 28: 4845-4869.

26. Silthamitag P, Chaijareenont P, Tattakorn K, et al. Effect of surface pretreatments on resin composite bonding to PEEK. Dent Mater J. 2016;35:668-74.

27. Henriques B, Fabris D, Mesquita-Guimarães J, et al. Influence of laser structuring of PEEK, PEEK-GF30 and PEEK-CF30 surfaces on the shear bond strength to a resin cement. J Mech Behav Biomed Mater. 2018;84:225-234.

28. Chaijareenont P, Prakhamsai S, Silthampitag P, et al. Effects of different sulfuric acid etching concentrations on PEEK surface bonding to resin composite. Dent Mater J. 2018;37:385-392.

29. Díez-Pascual AM, Díez-Vicente AL. Nano-TiO2 reinforced PEEK/PEI blends as biomaterials for load-bearing implant applications. ACS Appl Mater Interfaces. 2015; 7: 5561-5573.

30. Stawarczyk B, Jordan P, Schmidlin PR, et al. PEEK surface treatment effects on tensile bond strength to veneering resins. J Prosthet Dent. 2014; 112:1278-1288.

31. Feerick EM, Kennedy J, Mullett H, FitzPatrick D, et al. Investigation of metallic and carbon fibre PEEK fracture fixation devices for three-part proximal humeral fractures. Med Eng Phys. 2013; 35: 712-722.

32. Toth JM, Wang M, Estes BT, et al. Polyetheretherketone as a biomaterial for spinal applications. Biomaterials. 2006; 27: 324-334.

33. Skirbutis G, Dzingute A, Masiliunaite V, Sulcaite G, Zilinskas J. PEEK polymer's properties and its use in prosthodontics. A review. Stomato. 2018; 20:54-58.

34. Maekawa M, Kanno Z, Wada T, et al. Mechanical properties of orthodontic wires made of super engineering plastic. Dent Mater J. 2015; 34: 114-9.

35. Stawarczyk B, Beuer F, Wimmer T, et al. Polyetheretherketone-a suitable material for fixed dental prostheses? J Biomed Mater Res B Appl Biomater. 2013; 101:1209-16.

36. Haralur SB, Al Ahmari MA, AlQarni SA, Althobati MK. The effect of intraradicular multiple fiber and cast posts on the fracture resistance of endodontically treated teeth with wide root canals. Biomed Res Int. 2018; 2018:1671498.

37. Fonseca RG, de Almeida JG, Haneda IG, Adabo GL. Effect of metal primers on bond strength of resin cements to base metals. J Prosthet Dent. 2009; 101: 262-8.

38. ISO, 2009. Geometrical Product Specifications (GPS) – Surface texture: Profile Method – Terms, Definitions and Surface Texture Parameters (ISO 4287:1997). International Organization for Standardization, (https://www.iso.org/iso/en/prods-services/ ISO store/store.htm).

39. Zamboni Quitero MF, Garone-Netto N, de Freitas PM, de Cerqueira Luz MA. Effect of post translucency on bond strength of different resin luting agents to root dentin. J Prosthet Dent. 2014; 111:35-41.

40. Keul C, Liebermann A, Schmidlin PR, et al. Influence of PEEK surface modification on surface properties and bond strength to veneering resin composites. J Adhes Dent. 2014; 16:383-92.

41. Tsuka H, Morita K, Kato K, et al. Evaluation of shear bond strength between PEEK and resin-based luting material. J Oral Biosci. 2017; 59:231-6.

42. Renata GF, Santos PA, Isabella GH, Gelson LA. Effect of metal primers on bond strength of resin cements to base metals. J Prosthet Dent. 2009; 101:262-8.

43. Lanza A, Aversa R, Rengo S, et al. 3D FEA of cemented steel, glass and carbon posts in a maxillary incisor. Dent Mater. 2005; 21:709-15.

44. Lee KS, Shin JH, Kim JE, et al. Biomechanical Evaluation of a Tooth Restored with High Performance Polymer PEKK Post-Core System: A 3D Finite Element Analysis. Biomed Res Int. 2017; 2017:1373127.

45. Komada W, Miura H, Okada D, Yoshida K. Study on the fracture strength of root reconstructed with post and core: alveolar bone resorbed case. Dent Mater J. 2006;25:177-82.

46. Stawarczyk B, Sener B, Trottmann A, et al. Discoloration of manually fabricated resins and industrially fabricated CAD/CAM blocks versus glass-ceramic: effect of storage media, duration, and subsequent polishing. Dent Mater J. 2012; 31:377-83.

47. Beuer F, Steff B, Naumann M, Sorensen JA. Load-bearing capacity of all-ceramic three-unit fixed partial dentures with different computer-aided design (CAD)/computer-aided manufacturing (CAM) fabricated framework materials. Eur J Oral Sci. 2008; 11:381-6.

48. Zok FW, Miserez A. Property maps for abrasion resistance of materials. Acta Mater. 2007; 55:6365-71.

49. Schmidlin PR, Stawarczyk B, Wieland M, et al. Effect of different surface pre-treatments and luting materials on shear bond strength to PEEK. Dent Mater. 2010; 26:553-9.

50. Hallmann L, Mehl A, Sereno N, Hammerle CHF. The improvement of adhesive properties of PEEK through different pretreatments. Appl Sur Sci. 2012; 258:7213-8.

51. Zhou L, Qian Y, Zhu Y, et al. The effect of different surface treatments on the bond strength of PEEK composite materials. Dent Mater. 2014; 30: 209-15.

52. Sproesser O, Schmidlin PR, Uhrenbacher J, et al. Effect of sulfuric acid etching of polyetheretherketone on the shear bond strength to resin cements. J Adhes Dent. 2014; 16: 465-72.

53. Caglar I, Ates SM, Yesil Duymus Z. An in vitro evaluation of the effect of various adhesives and surface treatments on bond strength of resin cement to polyetheretherketone. J Prosthodont. 2019; 28:e342-e349.

54. Rikitoku S, Otake S, Nozaki K, et al. Influence of SiO2 content of polyetheretherketone (PEEK) on flexural properties and tensile bond strength to resin cement. Dent Mater J. 2019; 38:464-470.

55. Stawarczyk B, Taufall S, Roos M, et al. Bonding of composite resins to PEEK: the influence of adhesive systems and air-abrasion parameters. Clin Oral Investig. 2018 Mar;22(2):763-771.

56. Strefezza C, Amaral MM, Quinto J Jr, et al. Effect of 830 nm diode laser irradiation of root canal on bond strength of metal and fiber post. Photomed Laser Surg. 2018; 36:439-44.

57. Rosentritt M, Preis V, Behr M, et al. Shear bond strength between veneering composite and PEEK after different surface modifications. Clin Oral Investig. 2015; 19:739-44.

58. Ruschel GH, Gomes ÉA, Silva-Sousa YT, et al. Mechanical properties and superficial characterization of a milled CAD-CAM glass fiber post. J Mech Behav Biomed Mater. 2018; 82:187-92.

59. Maroulakos G, Wanserski MW, Wanserski MM, et al. Effect of airborne-particle abrasion on 3-dimensional surface roughness and characteristic failure load of fiber-reinforced posts. J Prosthet Dent. 2019; 121:461-69.

60. Bezzon OL, Pedrazzi H, Zaniquelli O, da Silva TB. Effect of casting technique on surface roughness and consequent mass loss after polishing of NiCr and CoCr base metal alloys: a comparative study with titanium. J Prosthet Dent. 2004; 92:274-7.

61. Deng S, Djukic L, Paton R, Ye L. Thermoplastic–epoxy interactions and their potential applications in joining composite structures – A review. Compos. Part A. Appl. Sci. Manuf. 2015,68: 121-32.

62. Younis M, Unkovskiy A, ElAyouti A, et al. The effect of various plasma gases on the shear bond strength between unfilled polyetheretherketone (PEEK) and veneering composite following artificial aging. Materials (Basel). 2019;12. pii: E1447.

63. Fuhrmann G, Steiner M, Freitag-Wolf S, Kern M. Resin bonding to three types of polyaryletherketones (PAEKs)-durability and influence of surface conditioning. Dent Mater. 2014; 30:357-63.

64. Balbosh A, Kern M. Effect of surface treatment on retention of glass-fiber endodontic posts. J Prosthet Dent. 2006; 95:218-23.

65. Kermanshah H, Bolhari B, Sedaghat F, Omrani LR. Effect of intracanal post space treatments on push-out bond strength of fiber posts to root dentin. J Dent (Tehran). 2017; 14:55-61.

66. Oguz Ahmet BS, Egilmez F, Ergun G, Cekic Nagas I. Surface treatment effects on bond strength of CAD/CAM fabricated posts to root canal dentin. Am J Dent. 2019; 32:113-7.

67. Silva NRD, Rodrigues MP, Bicalho AA, et al. Effect of resin cement mixing and insertion method into the root canal on cement porosity and fiberglass post bond strength. J Adhes Dent. 2019; 21:37-46.

68. Shafiei F, Behroozibakhsh M, Abbasian A, Shahnavazi S. Bond strength of self-adhesive resin cement to base metal alloys having different surface treatments. Dent Res J (Isfahan). 2018; 15:63-70.

69. Soares CJ1, Valdivia AD, da Silva GR, et al. Longitudinal clinical evaluation of post systems: a literature review. Braz Dent J. 2012; 23:135-740.

70. Ferrari M, Cagidiaco MC, Goracci C, et al. Long-term retrospective study of the clinical performance of fiber posts. Am J Dent. 2007; 20:287-91.

71. Makade CS, Meshram GK, Warhadpande M, Patil PG. A comparative evaluation of fracture resistance of endodontically treated teeth restored with different post core systems - an in-vitro study. J Adv Prosthodont. 2011; 3:90-5.

72. Aleisa K, Al-Dwairi ZN, Alghabban R, Goodacre CJ. Effect of luting agents on the tensile bond strength of glass fiber posts: An in vitro study. J Prosthet Dent. 2013; 110:216-22.

73. Bonfante G, Kaizer OB, Pegoraro LF, do Valle AL. Tensile bond strength of glass fiber posts luted with different cements. Braz Oral Res. 2007; 21:159-64.

74. Zoidis P. Polyetheretherketone overlay prosthesis over high noble ball attachments to overcome base metal sensitivity: A Clinical Report. J Prosthodont. 2018; 27:688-93.

75. Schwendicke F, Stolpe M. Cost-effectiveness of different post-retained restorations. J Endod. 2017; 43:709-14.

76. Harb IE, Abdel-Khalek EA, Hegazy SA. CAD/CAM constructed Poly(etheretherketone) (PEEK) framework of Kennedy Class I removable partial denture: A Clinical Report. J Prosthodont. 2019;2 8: e595-e598.

Table and figure legends

Table 1. List of materials used

Table 2. Comparison of mean surface roughness (Ra) values of all groups before post cementation

Table 3. Comparison of mean tensile bond strength (TBS) values of tested groups

Table 4. The percentages of failure modes in each experimental group

Figure 1(a-b). (a) Wear characteristics of PEEK posts after sulfuric acid etching. (b) SEM image of acid-etched PEEK surface.

Figure 2. SEM images of each study group after surface treatment. **(a)** PEEK(Polyetheretherketone); **(b)** Metal post; **(c)** Glass-fiber post.

Figure 3. Optical microscope images **(a, b, c)** of the roots and SEM images **(d, e, f, g, h, i)** of the post surfaces following tensile bond test. **(a, d, g)** PEEK(Polyetheretherketone);**(b, e, h)** Metal post; **(c, f, i)** Glass-fiber post.

Table 1. List of materials used*.

Material	***Product Name***	***Composition***	***Manufacturer***	***Batch Number***
Surface treatment	Air abrasion (Cobra Blast Aluminum oxide-Renfert)	Al_2O_3 particles (50 µm)	Cobra; Renfert GmbH, Hilzingen, Germany	15941112
	H_2SO_4 (%95-98) (Tekkim-Sülfürik Asit)	H_2S0_4, (Fe)≤ 0.005%, (Pb) ≤ 0.0005%, (Cr) ≤0.0005%	Tekkim Kimya; Nilüfer, Bursa,Turkey	260419171001
	Phosphoric Acid (K-Etchant GEL)	40% phosphoric acid	Kuraray CO.LTD., Umeda, Osaka, Japan	011204
Post materials	Ni-Cr alloy (Wiron 99)	Ni 65.6%, Cr 22.5%, Mo 9.5%, Si 1.0%, Ce, Mn, Nb.	BEGO Bremer Goldschlägerei Wilh, Bremen, Germany	3038
	Glass-fiber post (Angelus Reforpost, Exacto #3)	Glass fiber (80%); pigmented resin (19%), stainless steel filament (1%).	Angelus Dental Products Industry, Paraná, Brasil	12640

	PEEK (PEEK-OPTIMA®)	Polyetheretherketone, glass-fiber filler.	Juvora Ltd., Wyre, Lancashire, UK	3723847
Luting agent	Adhesive resin cement (Panavia F2.0)	*Paste A*: MDP, dimetacrylates,silanated slica filer, silanated colloidal silica, dl-camphorqunone, catalysts,iniators *Paste B*: Dimethacrylates, silanated barium glass filler, surface-treated sodium floride, catalysts, accelerators, pigments	Kuraray Noritake Dental Inc. Okayama, Japan	51217

* Written followed by the information of the manufacturers.

Table 2. Comparison of mean surface roughness (Ra) values of all groups before post cementation.

Group	***Ra(µm)***	***p******
P	1.37±0.11[a]	<0.001
M	2.52±0.25[b]	
F	2.93±0.18[c]	

*One-way ANOVA, a-c: Identical letters indicate no significant differences (*p*>0.05).

Table 3. Comparison of mean tensile bond strength (TBS) values of tested groups.

Groups	***TBS (MPa)***	***p****
P	14.33±0.58[a]	<0.001
M	12.79±0.39[b]	
F	10.05±0.53[c]	

*One-way ANOVA, a-c: Identical letters indicate no significant differences (p>0.05).

Table 4. The percentages of failure modes in each experimental group.

	Groups				
Failure mode	P(n=20)	M(n=20)	F (n=20)	N=60	***p****
Adhesive	16 (80%)	12 (60%)	13 (65%)	41 (68.3%)	
Cohesive (within resin)	0 (0%)	2 (10%)	0 (0%)	2 (3.3%)	0.243
Mixed	4 (20%)	6 (30%)	7 (35%)	17 (28.3%)	

* Pearson's chi-square test

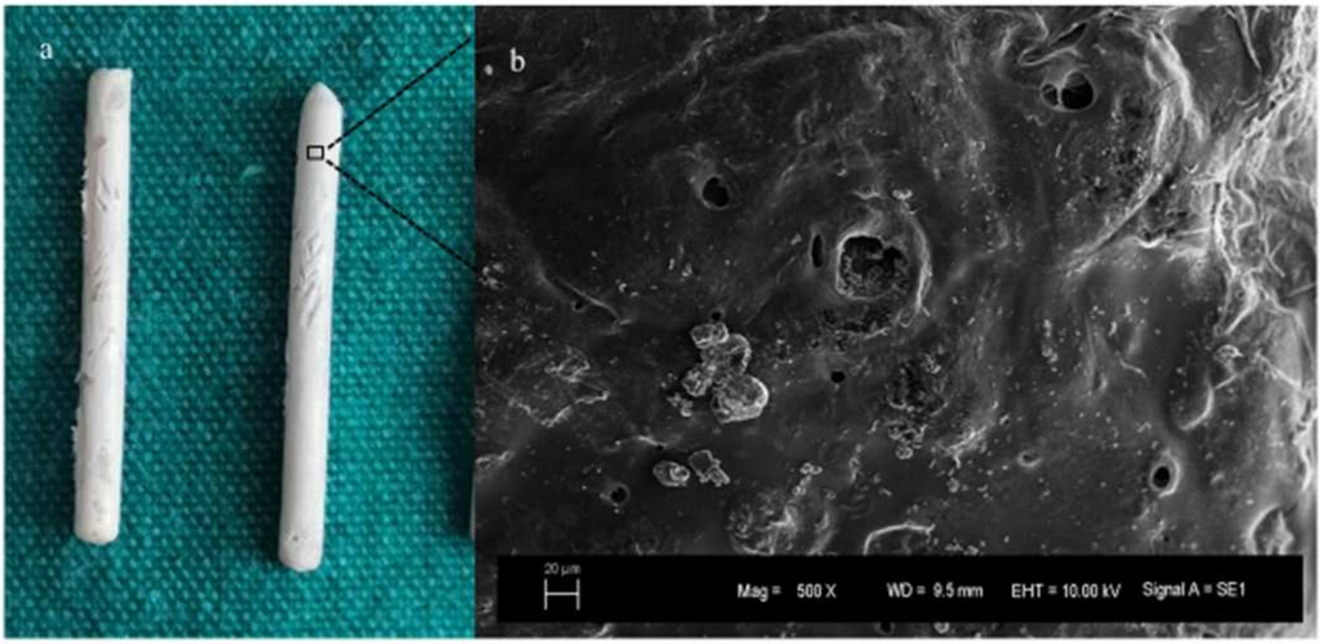

Figure 1(a-b). **(a)** Wear characteristics of PEEK posts after sulfuric acid etching. **(b)** SEM image of acid-etched PEEK surface.

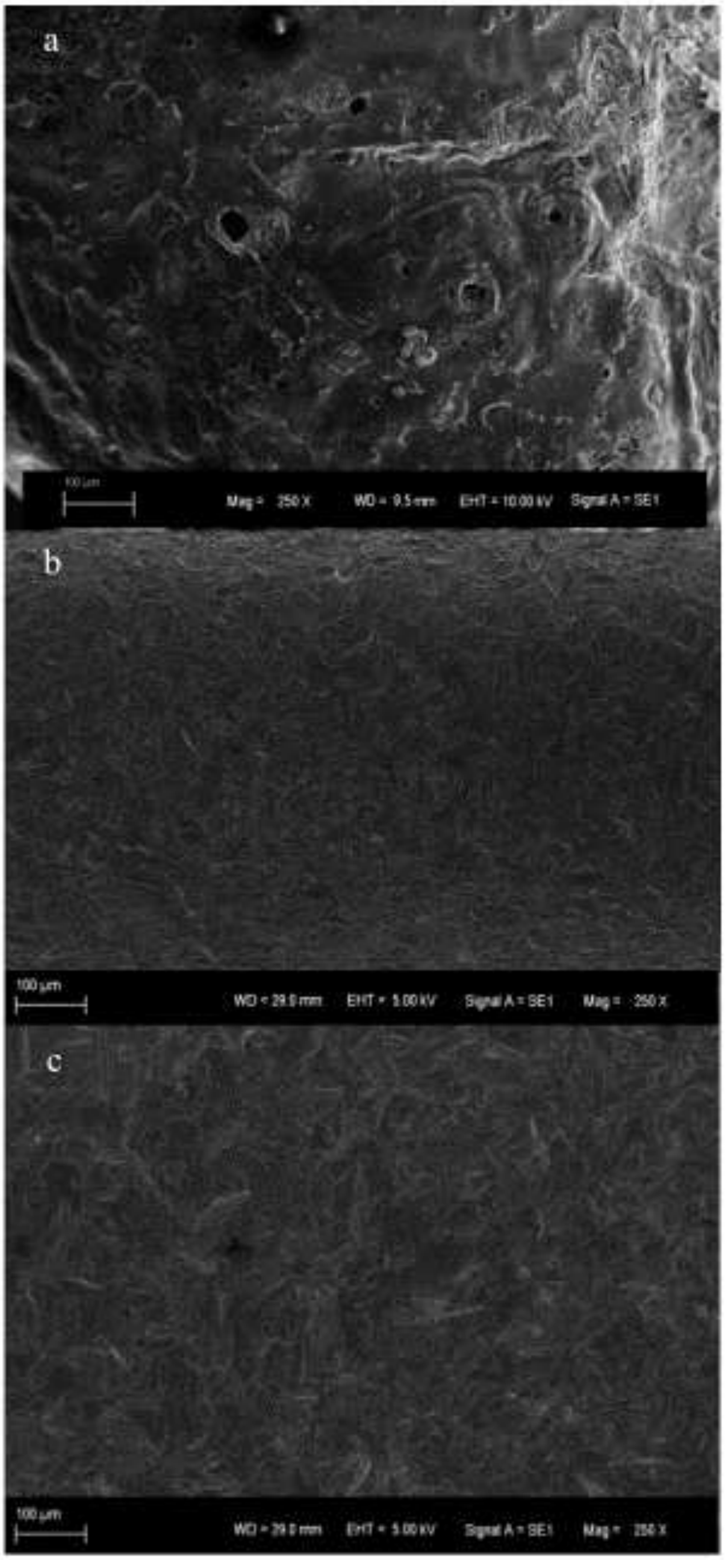

Figure 2(a-c). SEM images of each study group after surface treatment.**(a)** PEEK(Polyetheretherketoue); **(b)** Metal post; **(c)** Glass-fiber post.

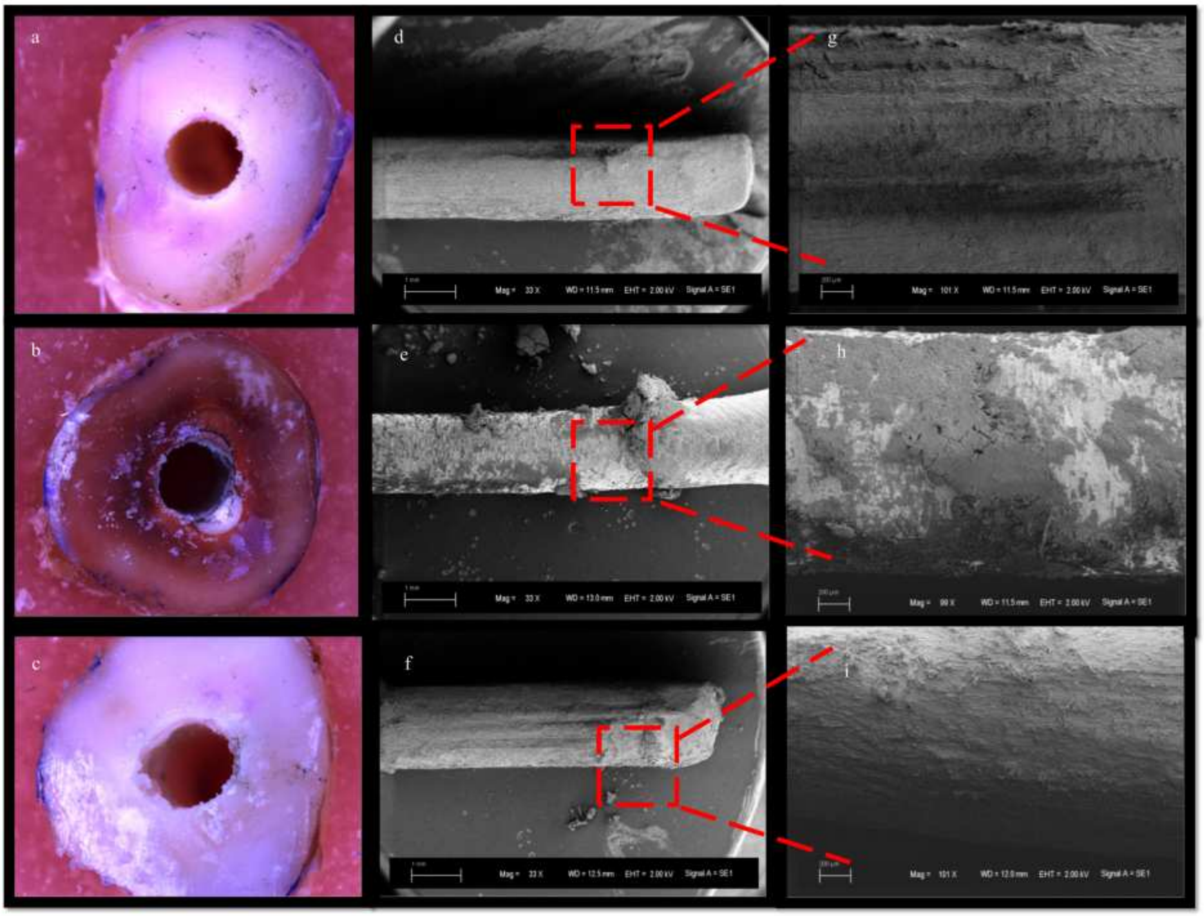

Figure 3(a-i). Optical microscope images **(a,b,c)** of the roots and SEM images **(d, e, f, g, h, i)** of the post materials following tensile bond test. **(a,d,g)** PEEK(Polyetheretherketone);**(b,e,h)** Metal post; **(c,f,i)** Glass-fiber post.