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ORIGINAL ARTICLE

Effect of Different Surface Treatments and Application Times on Shear Bond Strength Between Polyetheretherketone and Composite Resin

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ABSTRACT

Objective: To investigate the effect of various surface treatments and different application times on shear bond strength between polyetheretherketone and composite resin. **Methods:** A total of 110 disc-shaped polyetheretherketone specimens were randomly divided into 9 groups and 4 different surface treatments (control, sandblasting, tribochemical silica coating, sulfuric acid) were applied. The sandblasting, tribochemical silica coating, and sulfuric acid processes were performed 3 different times (10s, 15s, 20s). Then, the composite resins were applied to the treated surfaces of the polyetheretherketone specimens. The shear bond strength test and scanning electron microscopy analysis were performed. The data were statistically analyzed using analysis of variance and Duncan honest significant difference test. **Results:** One-way analysis of variance revealed differences in shear bond strength among the groups ($p < 0.001$). While the control group showed the lowest bond strength values (4.24 ± 1.53 MPa), 20 s of sulfuric acid process showed the highest bond strength values (27.91 ± 4.44 MPa). **Conclusion:** Depending on the application time to the material, additional surface treatments increase the polyetheretherketone composite bonding. Surface treatments with sulfuric acid applications provide higher surface bonding values than other treatments.

Key words: CoJet, composite, PEEK, sandblasting, sulfuric acid

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INTRODUCTION

Polyetheretherketone (PEEK) is a member of the polyaryletherketone family with impressive physical and chemical properties.[1] It consists of aromatic benzene molecules that use functional ether or ketone groups in bonding.[2] Due to its advanced mechanical properties and biocompatibility as a high-performance polymer, its usage areas in dental applications are becoming increasingly common due to its resistance to almost all organic and inorganic chemicals.[2,3] Furthermore, it is assumed that its elastic modulus of 3-4 GPa, which is much closer to the bone than metal alloys or ceramics, provides benefits to the maxillofacial system with unique mechanical dynamics.[4,5] In addition to its very low density of 1.265 g/cm^3, PEEK is also impressive with its excellent tensile strength, bending strength, and abrasion resistance values, which indicate high ease of use and durability.[6] Many areas such as infrastructure material in fixed dentures, framework of removable dentures, production of the clasp and other components, temporary abutments, healing caps, and implant material can be listed as the usage areas of PEEK in dentistry.[2,6–8] High compatibility can be achieved using computer aided design-computer aided manufacturing (CAD-CAM) milling systems in manufacturing.[9] Based on these positive properties, PEEK has become a reliable and aesthetically pleasing alternative to metal-based materials for dental restorations in a short time.[10] Furthermore, its positive biological properties, radiolucency property, high fracture resistance, and acceptable dimensional stability support this situation.[6] Despite all its advantageous structural properties, the natural optical properties, low translucency, and grayish pigmentation of the material are the most important limitations for fixed partial dentures,[11] which eliminates the option of using PEEK as a stand-alone coating material. Its production in full contour is not possible due to its disadvantageous properties mentioned above. It should definitely be veneered with an aesthetic

material.[11] The chemically inert behavior of the PEEK material indicates a potential bonding problem at the interface of the PEEK core and the veneering resin and at the interface of the PEEK core and resin adhesive cement. In addition to its low surface energy, due to its resistance to surface modification provided by different mechanical chemical processes, providing sufficient bond strength between resin composites and PEEK surfaces is an additional difficulty,[12] which remains a problem in the clinical use of PEEK.

Previous on the bonding of PEEK to composite resins have shown that the bond strength is insufficient when surface treatment is not applied.[6] The first step to achieve a good adhesion with PEEK materials is to increase the surface roughness by surface treatments and increase the bond strength by allowing the resin material to flow into the microretentive areas formed.[13] Increased surface roughness decreases the surface tension, increases hydrophilicity and surface area, and provides micromechanical retention.[3] Nevertheless, durable resin bonding can be achieved by applying methacrylates containing primer on the material surface.[14,15] As indicated previously, it was recommended to apply various surface treatments, such as hydrofluoric acid etching, laser treatments, tribochemical silica coating, plasma treatment, piranha solution (peroxymonosulfuric acid, 10:3 hydrogen peroxide), sandblasting (Al_2O_3), or the application of concentrated sulfuric acid, to the PEEK surface to achieve higher bond strengths.[14,16,17] Acid etching of the PEEK surface leads to the occurrence of carbon-oxygen compounds and thus provides more functional groups to which adhesive systems can be bonded.[18] The application of primers and adhesives on the surfaces after surface roughening treatments is among the factors that increase the bond strength. Etching the PEEK surface before conditioning it with methylmethacrylate (MMA) based primers and coating may also increase the free surface energy and roughness and the tensile bond strength.[19]

The aim of this study was to examine the effect of mechanical and chemical surface roughness treatments applied at different times on the PEEK-composite resin bonding. The null hypothesis of the study was that the 20 s sulfuric acid applied to the PEEK surface would be more successful in shear bond strength values compared to other surface treatments and other application times.

METHODS

The power analysis was carried out using the G*Power software program (v3.0.10) to obtain the highest power level with the smallest sample size. The analysis showed that at least 11 specimens were required for the highest power level (power = 80, $\alpha = 0.05$), and a total of 110 specimens were used in the study.

Table 1. Manufacturer's informations of the materials.

Material	Manufacturer
PEEK Blocks	CopraPeek; Whitepeaks Dental Solutions GmbH&Co, Essen, Germany
50 µm Al_2O_3 Sand	Akrodent; Koca Chem&Dent, Ankara, Turkey
30 µm silanized Al_2O_3 Sand	CoJet Sand; 3M ESPE, Neuss, Germany
98% sulfuric acid	Honeywell Fluka, Germany
Primer+Bond	Prime&Bond Universal; Dentsply DeTrey GmbH, Konstanz, Germany
Composite Resin	G-aenial; GC Dental Products Corp., Tokyo, Japan

Table 2. Surface treatments applied to specimens.

Group Code	Description
C	No surface treatment
SB10	Sanblasting with 50 µm Al_2O_3 for 10 s
SB15	Sanblasting with 50 µm Al_2O_3 for 15 s
SB20	Sanblasting with 50 µm Al_2O_3 for 20 s
CJ10	Coating with 30 µm silanized Al_2O_3 for 10 s
CJ15	Coating with 30 µm silanized Al_2O_3 for 15 s
CJ20	Coating with 30 µm silanized Al_2O_3 for 20 s
SU10	Etching with 98% sulfuric acid for 10 s
SU15	Etching with 98% sulfuric acid for 15 s
SU20	Etching with 98% sulfuric acid for 20 s

Specimen preparation

The specimens were produced from PEEK blocks by milling with the CAD-CAM unit with a disc-shaped (diameter of 10 mm and a height of 2 mm). The polishing procedure was applied to the surfaces of the obtained specimens as specified: The surfaces of the specimens were ground with P600 and P800 grit silicon carbide paper (English Abrasives & Chemicals Ltd, London, UK) for 60 s and polished with a fine pumice stone (Ernst Hinrichs Dental, Goslar, Germany) and goat hair brushes (Jiffy; Ultradent Products, South Jordan, UT, USA) for 60 s in an automatic polishing device (Reco Dental, Wiesbaden, Germany) with a vertical force of 25 N to produce a standard surface. After the polishing treatment was completed, the specimens were cleaned in an ultrasonic cleaner for 10 minutes and kept in distilled water at 4 °C until the surface treatments were applied. Information about the materials used in the study is presented in Table 1.

The specimens were randomly divided into 10 groups according to the surface roughening procedures (n =11),

and the surface treatments were applied as indicated in Table 2.

All surface treatments were applied by a single researcher. Sandblasting processes were performed with a sandblasting device (Airsonic; Hager &Werken, Duisburg, Germany) at a distance of 10 mm under 4 bar pressure with 50 µm Al_2O_3, and the specimens were washed with distilled water and dried for 60 seconds after sandblasting. Tribochemical silica coating treatments were applied with an intraoral pen sandblasting device (CoJet Prep; 3M ESPE, Seefeld, Germany) at a distance of 10 mm under 2.8 bar pressure. No cleaning process was applied to the surfaces of the specimens after the treatment in order not to damage the salinization. After the application of %98 sulfuric acid, the surfaces of the specimens were washed with distilled water and dried for 60 seconds. All applied surface treatments were applied in 3 different working times as 10, 15, and 20 seconds.

Shear bond strength test

After the surface treatments were completed, the bonding agent was applied to the surfaces of the specimens with the help of a cotton pellet for 10 seconds and polymerized for 10 seconds with a light device (Valo Grand; Ultradent Products, South Jordan, USA). The molds with a diameter of 3 mm and a height of 3 mm were prepared from transparent additive type silicone (Elite Glass; Zhermack, Badia Polesine, Italy) to standardize the composite resins to be bonded to the surfaces of the specimens. The prepared molds were placed so that they would coincide with the center of the PEEK specimens. The composite resins were placed in these mold cavities, teflon tape was placed on them by removing the excess amount, and they were polymerized with a light device under constant load (400 g) for 20 seconds. The silicone molds on the specimens, the polymerization of which was completed, were removed. The specimens were tested in shear bond strength (SBS) test setup with a head speed of 1 mm/min in the shear mode of a universal test device (Model 2519-106; Instron Corp., Norwood, MA, USA). The bond strength values, and fracture types of each specimen were recorded. The shear bond strength values obtained in Newton were converted to the MPa unit. The SEM image of a randomly selected specimen from each group was taken at × 2000 magnification and recorded. Figures 1-4 show the SEM images of the specimens at ×2000 magnification.

Statistical analysis

Statistical analysis was performed with the Kolmogorov-Smirnov test for normality test and analysis of variance (ANOVA) using IBM SPSS Statistics v. 20 (SPSSv20.0; SPSS Inc., Chicago, IL, USA). Duncan's multiple comparison test was used for intergroup comparisons ($\alpha = 0.05$).

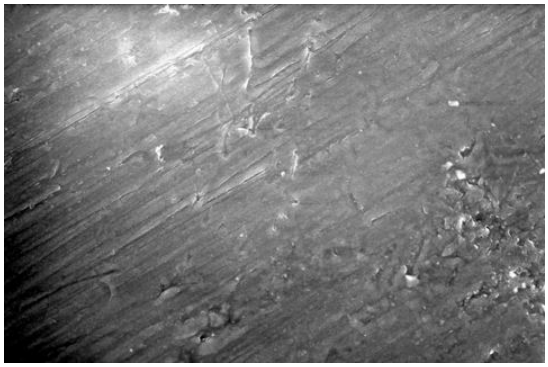

Figure 1. Scanning electron microscope images of specimen from C group. Original magnification × 2000.

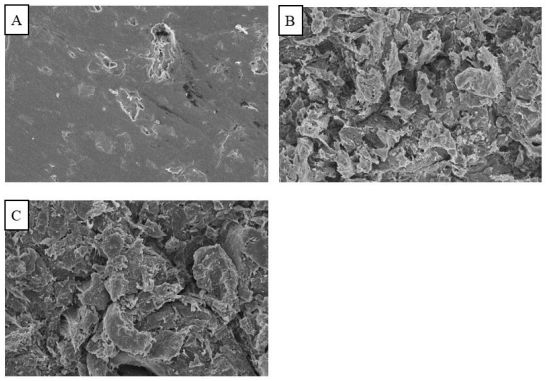

Figure 2. Scanning electron microscope images of specimens from SB group. Original magnification × 2000. A, 10s. B, 15s C, 20s.

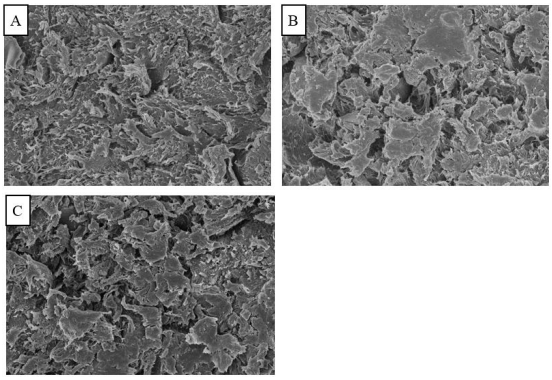

Figure 3. Scanning electron microscope images of specimens from CJ group. Original magnification × 2000. A, 10s. B, 15s C, 20s.

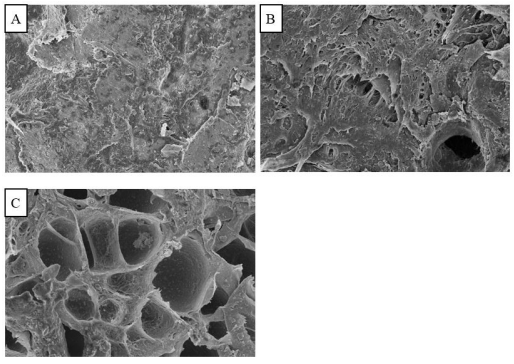

Figure 4. Scanning electron microscope images of specimens from SU group. Original magnification × 2000. A, 10s. B, 15s C, 20s.

Table 3. Least square means and standard deviation (SD) for SBS test.

Groups	Mean ± SD	N	p
C	4.24 ± 1.53[a]	11	0.000
SB10	7.20 ± 4.02[a]	11	
SB15	15.01 ± 6.63[b,c]	11	
SB20	14.38 ± 5.42[b,c]	11	
CJ10	13.75 ± 5.09[b]	11	
CJ15	15.81 ± 5.15[b,c]	11	
CJ20	18.81 ± 4.75[c]	11	
SU10	27.08 ± 4.54[d]	11	
SU15	27.38 ± 5.42[d]	11	
SU20	27.91 ± 4.44[d]	11	
Total	17.16 ± 9.14	110	

Different lowercase letters mean significantly different.

Table 4. Distribution of the observed failure mode.

Failure mode	C	SB10	SB15	SB20	CJ10	CJ15	CJ20	SU10	SU15	SU20
Adhesive	11	11	11	11	9	10	9	7	9	9
Cohesive	-	-	-	-	-	-	-	-	-	-
Mixed	-	-	-	-	2	1	2	4	2	2
Total	11	11	11	11	11	11	11	11	11	11

RESULTS

The ANOVA detected significant differences among the surface treatments of the PEEK specimens ($p < 0.001$). Duncan's multiple comparison test found that the specimens treated with sulfuric acid at different times were significantly different from the control, sandblasting, and CoJet applied specimens ($p < 0.05$). It was found that there was no difference ($p > 0.05$) between the C and SB10 groups. However, these two groups differed ($p < 0.05$) compared to all other groups, and there was a statistical difference ($p < 0.05$) only between CJ10 and CJ20 among the sandblasted and CoJet applied specimens. The highest SBS values were observed in SU20 (27.91 ± 4.44 MPa), while the lowest SBS values were observed in C (4.24± 1.53 MPa) specimens. Table 3 shows the results of ANOVA, and the mean and standard deviation values. Table 4 shows the results of the failure modes.

When the SEM images of the specimens (× 2000) were examined;
It was observed that C and SB10 specimens had appearances close to each other and had very small cracked areas on their surfaces, that the specimens in the SB15, SB20, CJ10, CJ15, and CJ20 groups had appearances close to each other and their surface areas consisted of dense lattice structures, and that large porosities were formed on the surfaces of the specimens in the sulfuric acid group due to the increase in time together with dense lattice structures and irregular areas (Fig. 1-4). The bonding material penetrates these irregular areas. When the SEM images of the SU10, SU15 and SU20 groups are examined, it is seen that the surface morphology of the PEEK samples contains in accordance with the penetration of the resin material. it is seen that these do not weaken the structure of the surface but provide more indented surface than sandblasting and tribochemical coating groups.

DISCUSSION

Although the effect of surface roughening treatments on the bond strength between PEEK and composite resin has been examined in previous studies, the effect of different application times on this bond is unknown. In this study, it was aimed to examine the connection of different roughening methods applied to the surface of the PEEK material at different times with the composite resin. According to the study results, the null hypothesis of the study was accepted since the application of sulfuric acid increased the bond strength values compared to other surface treatments.

A large number of mechanical tests, such as shear bond strength, pull-out, tensile, and microtensile tests, are used to measure the bond strength between materials.[20] The shear test is the most commonly used test type for this purpose since it is easy to apply and can stimulate loads in the oral environment.[21] Therefore, in the current study, the shear bond strength test was preferred to evaluate the bond strength of the materials.

It is recommended that the bond strength be increased by applying the methods of chemical etching or mechanical abrasion to the PEEK surface.[19] While airborne particle abrasion leads to an increase in the roughness of the material surface,[10] the etching treatment leads to an increase in functional carbonoxygen groups on the superficial layer of PEEK.[22] Within the scope of this study, sandblasting, tribochemical silica coating, and sulfuric acid application were preferred as mechanical and chemical surface roughening treatments.

The material surface has great importance for the placement of resins in the pits and cavities in terms of bonding. It is recommended to apply low-viscosity adhesive systems before applying the veneer material to the PEEK surface.[2,23] The applied primer and bond systems contain alcohol, acetone, methyl methacrylate monomer, or silane.[19] In the present study, the adhesive system was a primer&bond etch&rinse system and chemically contains dimethacrylate resin and PENTA (phosphoric acid modified acrylate resin).

In the literature, it is reported that the minimum value for acceptable bonding should be 5 MPa for the PEEK-composite resin bonding,[19] and that there should be values of 10 MPa and higher for an ideal bonding.[19] According to the results of the present study, it was observed that only group C had values below the acceptable bonding limits. However, group SB10 had mean values slightly above the acceptable bonding limits, and all other groups had higher bond strength values than the accepted ideal bonding values, which suggests that surface roughening treatments should be certainly applied to the PEEK surface for an ideal PEEK-composite resin bond. Culhaoglu et al.[24] reported in their study that 15 s sandblasting, 15 s CoJet, and 60 s sulfuric acid treatments significantly increased the bond strength of PEEK materials to the composite resin compared to the control group ($p < 0.05$), and they indicated that there was no statistically significant difference between the surface treatments applied ($p > 0.05$). While Ates et al.[21] could not find a difference between 15 s sandblasting and 15 s CoJet treatments, they reported that both surface treatments increased the bond strength values compared to the control group. According to the present study results, it was observed that 15 s sandblasting and 15 s CoJet treatments statistically significantly increased the bond strength values ($p < 0.05$), which was similar to the data in the literature. In another study, 10 s sandblasting, 12 s CoJet, and 60 s sulfuric acid were applied to the PEEK surface, and its bond with the composite resin was examined. While no difference was observed between the groups, it was reported that all surface treatments increased the bond strength values compared to the control group.[17] According to the results of current study, it was observed that 10 s sandblasting treatment did not differ significantly compared to the control group ($p > 0.05$), and 10 s CoJet treatment significantly increased the bonding values compared to the control group ($p < 0.05$). We think that this difference with the literature may be due to the fact that 10 s sandblasting treatment did not produce sufficient roughness on the PEEK surface, and that the increase in the 10 s CoJet treatment may be due to the silanization treatment. In a study on the concentration of sulfuric acid, while 70 and 80% sulfuric acid treatments were not significantly different from the control group, 85%, 90%, and 98% concentrations had better bonding compared to the others.[25] In the present study, sulfuric acid at a concentration of 98% was preferred due to its successful effect on bonding. A study found that the application of 60 s 98% sulfuric acid to the PEEK surface was more successful compared to the control, silica coating, and sandblasting groups.[24] Schimidlin et al.[17] and Zhou et al.[3] also shared similar results in their studies. Sproesser et al.[8] reported that the application of 98% sulfuric acid for 90 s was successful in the PEEK-composite bond and that the application for 15 s did not differ compared to the control group. According to the results of the present study, it was revealed that 98% sulfuric acid significantly increased the PEEK-composite resin bonding values compared to the control, sandblasting, and tribochemical silica coating groups in all application times; however, there was no difference between the application times. We considered that it was due to the ability of high concentrations of sulfuric acid to significantly roughen the surface of PEEK, a polymeric material, even in a short application time. We thought that the SEM images of the specimens and surface examinations of the specimens also supported the test results, that the porosity areas formed especially in the sulfuric acid group increased the bonding values, and that the sand applied in the sandblasting and CoJet treatments contributed to bonding by forming a lattice layer on the material surfaces.

This study has some limitations. The first one is that in this study, while testing the bond strength of the specimens, the roughness values or the roughness results of the applied surface treatments were not expressed numerically, although they were displayed by SEM (no correlation was established between roughness and bond strengths). Another limitation is that the thermal cycle treatment was not applied to the specimens within the scope of this study.

CONCLUSION

Within the limits of the present study, the following conclusions were achieved:

1. Different surface roughening procedures could increase the bond strength of the PEEK-composite resin, and 98% sulfuric acid groups had the highest bonding values.
2. 98% Sulfuric acid application applied in 10,

15 and 20 second applications and applied at is more costly than sandblasting and tribochemical coating.
3. There was no difference in terms of bonding values between different application times of sandblasting and tribochemical silica coating applications, except for the 10 s sandblasting treatment.

CONFLICT OF INTEREST

Authors declare that they have no conflict of interest.

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