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Effect of Surface Treatments on the Shear Bond Strength of High-Performance Polymers to Resin Cements: An Experimental Study

Yüksek Performanslı Polimerlerin Rezin Siman ile Olan Makaslama Bağlanma Dayanımı Üzerine Yüzey İşlemlerinin Etkisi: Deneysel Çalışma

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ABSTRACT Objective: This study aimed to evaluate the shear bond strength (SBS) between high-performance polymers and resin cement. **Material and Methods:** A total of 104 disc-shaped specimens-52 from polyetheretherketone (PEEK) and 52 from polyetherketoneketone (PEKK) (8×2 mm)-were obtained from computer-aided design/computer-aided manufacturing blocks under water cooling. The specimens were divided into 4 groups based on surface treatment (n=13): Group 1-silica coating; Group 2-acid etching; Group 3-laser irradiation with 100 µm groove depth; Group 4-laser irradiation with 150 µm groove depth. One specimen from each group was randomly selected for scanning electron microscope analysis. SBS was measured using a universal testing machine. The effects of material type, surface treatment, and their interaction on SBS were analyzed using a generalized linear model. "Post hoc" comparisons were performed using Bonferroni correction. The significance level was set at $p<0.05$. **Results:** Material type, surface treatment, and their interaction significantly affected SBS ($p<0.05$). PEEK exhibited a higher average SBS (16.47 MPa) than PEKK (11.06 MPa). Among surface treatments, the highest SBS was observed in the silica coating group (25.56 MPa), followed by acid etching (13.45 MPa), laser irradiation at 150 µm (8.17 MPa), and 100 µm (7.88 MPa). The silica-coated group showed significantly higher SBS values for both materials ($p<0.05$), with the highest recorded SBS in silica-coated PEEK. **Conclusion:** Silica coating or acid etching can be recommended as effective surface treatments. Laser treatment with these parameters was not suitable for improving the bond strength of high-performance polymers.

Keywords: Polyetheretherketone; polyetherketoneketone; polymers; resin cements; shear strength

ÖZET Amaç: Bu çalışmanın amacı, yüksek performanslı polimerler ile rezin siman arasındaki makaslama bağlanma dayanımını [shear bond strength (SBS)] değerlendirmektir. **Gereç ve Yöntemler:** Su soğutması altında bilgisayar destekli tasarım/bilgisayar destekli üretim bloklarından elde edilen, 52'si polietereterketon (PEEK) ve 52'si polieterketonketon (PEKK) olmak üzere toplam 104 adet disk şeklinde örnek (8×2 mm) hazırlandı. Örnekler, yüzey işlemine göre 4 gruba ayrıldı (n=13): Grup 1-silika kaplama, Grup 2-asit pürüzlendirme, Grup 3-100 µm oluk derinliğinde lazer uygulaması, Grup 4-150 µm oluk derinliğinde lazer uygulaması. Her gruptan rastgele seçilen bir örnek çevresel taramalı elektron mikroskobu ile analiz edildi. Örneklerin SBS üniversal test cihazı ile ölçüldü. Materyal tipi, yüzey işlemi ve bu 2 değişkenin etkileşiminin SBS üzerindeki etkileri, genelleştirilmiş lineer model ile analiz edildi. "Post hoc" karşılaştırmalar Bonferroni düzeltmesi ile yapıldı. Anlamlılık düzeyi $p<0,05$ olarak belirlendi. **Bulgular:** Materyal tipi, yüzey işlemi ve bu ikisinin etkileşimi SBS üzerinde anlamlı etki gösterdi ($p<0,05$). PEEK'in ortalama SBS değeri 16,47 MPa iken, PEKK'in ortalaması 11,06 MPa olarak belirlendi. Yüzey işlemleri arasında en yüksek SBS değeri silika kaplama grubunda (25,56 MPa) gözlendi; bunu sırasıyla asit pürüzlendirme (13,45 MPa), 150 µm lazer (8,17 MPa) ve 100 µm lazer (7,88 MPa) izledi. Her iki materyalde de silika kaplama grubu anlamlı şekilde daha yüksek SBS değerleri gösterdi ($p<0,05$) ve en yüksek değer silika kaplı PEEK'te kaydedildi. **Sonuç:** Silika kaplama veya asit pürüzlendirme, etkili yüzey işlemleri olarak önerilebilir. Bu çalışmada, kullanılan parametrelerle uygulanan lazer işlemi, yüksek performanslı polimerlerin bağlanma dayanımını artırmak için uygun bir yöntem değildir.

Anahtar Kelimeler: Polietereterketon; polieterketonketon; polimerler; rezin simanları; kayma mukavemeti

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Polyetheretherketone (PEEK) belongs to the polyaryletherketones (PAEK) group, has an aromatic molecular structure, and is a combination of ketone and ether functional groups between aryl rings.[1] The PEEK polymer was first produced from PAEK by Bonner in 1962. It is non-toxic and mutagenic and is also biocompatible *in vivo* and *in vitro*. No clinically significant cases of an allergy to PEEK have been reported.[2]

Currently, polymers are cost-effective materials that offer high strength relative to their weight and possess a lower density than metals. Unlike ceramics, composite indirect restorations are softer, more adaptable, and easier to finish and polish, and cause less wear on opposing teeth. Moreover, polymers allow for easier additional modifications.[3] Polyetherketoneketone (PEKK), a significant component of the PAEK family, consists of 80% PEKK and 20% titanium dioxide. The inclusion of an additional ketone group in comparison to PEEK, improves its mechanical and physical attributes. Its notable benefits include superior compressive strength, minimal plaque adhesion, a bone-like elastic modulus, and simple polishing characteristics.[4]

In addition to such positive properties, bond strength in dental materials poses an important clinical problem. Adequate bond strength is critical for the long-term clinical success of restorations, as it ensures durable adhesion between the restorative material and the tooth structure.[5] Bond strength is a key determinant in the long-term clinical success of restorations involving high-performance polymers, particularly in implant-supported frameworks and definitive prostheses. Insufficient adhesion may lead to restoration failure, marginal leakage, or debonding under functional loads, making surface optimization strategies essential for reliable clinical performance.[6]

Providing adequate bond strength between high-performance polymers and dental materials is difficult due to their low surface energy and resistance to surface treatments.[7] Due to its aromatic chemical structure and low surface energy, PEEK exhibits significant resistance to chemical surface treatments, necessitating mechanical or energetic surface modification techniques to improve its bonding properties.[8]

Studies have focused on methods for improving the bond strength between high-performance polymers and resin cement. One of them is the application of various surface treatments. Surface modification enhances the mechanical and biological characteristics of a material by altering its surface properties without impacting its overall attributes. This process ensures that the material's beneficial properties remain intact.[9]

Surface treatments frequently applied to increase bond strength include sandblasting, acid etching, tribochemical silica coating, laser etching, and plasma spray applications.[10] Increasing surface roughness through various surface treatments, including laser irradiation, has been shown to enhance adhesion by improving wettability, enlarging surface area, and enabling micromechanical retention.[11] A recent study highlighted that different laser types, such as Er:YAG, Nd:YAG, diode, and femtosecond lasers, have different effects on PEEK and PEKK surfaces, emphasizing the need for material-specific laser protocols to optimize bonding performance.[12]

A study reported that a sulfuric acid application is more effective than other surface treatment applications in terms of providing bond strength.[13] Although high-concentration sulfuric acid treatment can significantly improve bond strength, its clinical application remains limited due to the potential for mucosal damage upon accidental exposure.[8]

Tribochemical silica coating is a technique that can be applied chairside, utilizing a specially adapted aluminum sandblasting method to coat particle surfaces with silica.[14] Rosentritt et al. reported that the tribochemical silica coating process can be preferred as an alternative surface treatment to achieve successful bonding.[15]

Conversely, Çulhaoğlu et al. indicated that silica-coated PEEK surfaces provide lower bonding despite higher wettability compared to sandblasted, laser, and acid-treated surfaces.[16] Lasers, which can be used in many areas of dentistry including the removal of caries, the prevention of sensitivities, and bleaching treatment are also used to increase the usability of low-energy polymers by making their surfaces functional.[17]

Different laser parameters such as energy output and frequency can significantly influence the effectiveness of surface conditioning and the resulting bond strength, requiring careful parameter selection for each substrate.[18] Although there are many types of lasers, the CO_2 laser is often used as a soft tissue laser while Nd: YAG and Er: YAG lasers are preferred for both hard and soft tissues.[19] A recent study demonstrated that laser surface treatments can significantly enhance the surface roughness and bonding performance of dental materials, especially in low-energy surfaces like ceramics and polymers.[20]

The Er: YAG laser, widely used for surface treatments of dental materials, emits light at a wavelength of 2,940 nm, which is highly absorbed by water and hydroxyapatite, making it effective for modifying polymer surfaces without inducing thermal damage.[21,22] However, excessive laser energy may cause melting of the glassy phase, negatively impacting bonding performance, highlighting the need for moderate energy settings.[18]

Although increasing laser energy and irradiation time can enhance surface roughness, excessive intensities may cause structural damage, adversely affecting the bonding performance.[11] In the present study, moderate Er: YAG laser parameters were selected based on previous evidence suggesting that higher energy levels may compromise surface integrity, while lower energies fail to provide sufficient roughness for micromechanical retention.[11] A study by Tsuka et al. demonstrated that Neodymium doped yttrium vanadate laser groove treatments with depths of 100 μm, 150 μm, and 200 μm significantly increased both surface roughness and shear bond strength (SBS) of PEEK to resin-based luting agents.[23] The 150-200 μm treatments showed the highest bond strengths, although the difference between them was not statistically significant, suggesting a saturation point in surface effectiveness. Despite numerous investigations on laser-based surface treatments, no standardized protocol or consensus has yet been established for optimizing the SBS of high-performance polymers to resin-based materials.[24] Hence, this study aims to compare the effects of different surface treatment methods-sulfuric acid application, tribochemical silica coating, and 2 distinct Er:YAG laser parameters-on the bond strength between high-performance polymers and resin cement. The null hypothesis of the study is that these treatments do not significantly affect the bond strength of polymers to resin cement.

MATERIAL AND METHODS

SPECIMEN PREPARATIONS AND SURFACE TREATMENTS

Ethical Statement

This study did not involve human participants or animals. Therefore, ethical approval and informed consent were not required.

Employing the G*Power software (v3.0.10), a power analysis (power=75%, α=0.05, f=0.40) determined the sample size as 104, with 13 specimens per group across 8 subgroups. A total of 104 specimens, each measuring 8×2 mm, were prepared: 52 from PEEK (CopraPeek, Whitepeaks Dental Solutions, Germany) and 52 from PEKK (Pekkton Ivory, Cendres+Métaux, Sweden). Sample dimensions were verified using a digital caliper (Guanglu, Taizhou, China). The specimens were embedded in auto-polymerizing acrylic resin (Imicryl SC, Imicryl Dental Materials, Inc., Konya, Türkiye). Surface irregularities were removed, and a smooth surface was achieved by polishing the specimens with 600- and 800-grit water-cooled silicon carbide paper for 15 seconds with finger pressure. After polishing, specimens were cleaned in an ultrasonic cleaner (CD-4800, Jeken, Dongguan, China) for 10 minutes and air-dried prior to surface treatments. All specimens were allocated to 4 groups at random (n=13) using a simple randomization method, with each group receiving a different surface treatment as outlined in Table 1.

Group 1: Silica Coating

The material surfaces were treated using 30-μm silica-coated Al2O3 particles with the Cojet system (3M ESPE, Seefeld, Germany) for 15 seconds, maintaining a 10 mm distance and 2.8 bar pressure.

Group 2: Acid Etching

PEEK surfaces were treated with 98% sulfuric acid (CAS 7664-93-9) through acid etching for 60 seconds, followed by a 1-minute rinse with water.

TABLE 1: Surface treatment applied to specimens	
Groups	**Surface treatments**
Group 1	Silica coating
Group 2	Acid etching with H_2SO_4
Group 3	Laser irradiation (with 100 µm laser groove dept)
Group 4	Laser irradiation (with 150 µm laser groove dept)

Group 3 and Group 4: Laser Irradiation

The Er: YAG laser (AT Fidelis, Fotona, Ljubljana, Slovenia)-wavelength 2,940 nm, frequency 10 Hz, irradiation time 15 seconds, pulse duration 300 µs-was used. The laser beam was aligned at a 90 °C angle to the specimen surface, maintaining a 10 mm distance, with water irrigation applied throughout the procedure. A dental handpiece (R14-C) was used with a cylindrical sapphire tip (1.3×12 mm). The depth of the laser groove was formed under 2 conditions, 100 µm (3) and 150 µm (4).

ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY ANALYSIS

The specimens were examined using an environmental scanning electron microscope (ESEM) (Quanta FEG 250, FEI Inc., USA) at 5000× magnification to evaluate the impact of surface treatments on the material surfaces.

Adhesive Application

Following the surface treatment, an adhesive (Visio-Link, Bredent, Senden, Germany) was applied as a thin layer to the specimen surfaces using a disposable brush in circular motions, as per the manufacturer's instructions. The adhesive was air-dried and then light-cured for 90 seconds with a dental laboratory polymerizer (Valo GRAND, Ultradent, South Jordan, UT, USA).

Resin Cement Application

A dual-cured self-adhesive resin cement (Calibra, Dentsply Sirona, Konstanz, Germany) was placed into a custom-designed mold having a 5 mm inner diameter and a 3 mm height. The specimens were then polymerized following the manufacturer's instructions. A summary of all materials used is provided in Table 2.

SBS Test

Following the application of resin cement, all specimens were immersed in distilled water and stored at 37°C for 24 hours prior to testing in order to simulate intraoral humidity and temperature conditions. The tip used for the SBS test was positioned at a 90°C angle to the specimens. The SBS was evaluated using a universal testing machine (Shimadzu Corporation, Kyoto, Japan) operating at a crosshead speed of 1 mm/min. According to International Organization for

TABLE 2: Summary of the materials and equipment used in this study	
Materials and equipments	**Manufacturer**
PEEK	CopraPeek Whitepeaks Dental Solutions GmbH&Co.KG, Wesel, Germany
PEKK	Pekkton Ivory Cendres+Metaux, SA Sweden
Er: YAG laser	AT Fidelis, Fotona, Ljubljana, Slovenia
Digital caliper	Guanglu, Taizhou, China
Ultrasonic machine	CD-4800, Jeken, Dongguan, China
Autopolymerizing acrylic resin	Imicryl SC, Imicryl Dental Materials, Inc., Konya, Turkey
Cojet	3M ESPE, Seefeld, Germany
Sulfuric acid	RCI Labscan, Samutsakorn, Thailand
ESEM	Quanta FEG 250, FEI Inc., USA
Adhesive	Visio-Link, Bredent, Senden, Germany
Dental laboratory polymerizer	Valo GRAND, Ultradent, South Jordan, UT, USA
Resin cement	Calibra, Dentsply Sirona, Kanstanz, Germany
Universal test machine	Shimadzu Corporation, Kyoto, Japan

PEEK: Polyetheretherketone; PEKK: Polyetherketoneketone; ESEM: Environmental scanning electron microscope

Standardization (ISO)/TR 11,405 standards, the recommended speed for the cutting tip during SBS testing ranges from 0.45 mm/min to 1.05 mm/min.

In this study, the cutting tip speed was set at 1 mm/min following previous studies.[25, 26] A previous study reported that loading speeds between 0.5 and 1 mm/min do not significantly affect the results, whereas higher speeds may lead to unreliable or erroneous outcomes.[27] Therefore, 1 mm/min was chosen as a reliable and standardized value within this range. Force was applied to each sample until fracture occurred, and the maximum load values were recorded. SBS forces were recorded in newtons (N) and calculated in megapascals (MPa) by dividing the peak load at failure by the bonded surface area.

STATISTICAL ANALYSIS

Statistical analysis was performed using IBM SPSS Statistics (version 25, IBM Corp., Armonk, NY, USA). The Shapiro-Wilk test was performed to evaluate the normality of the SBS data, while the Levene test was used to assess the homogeneity of variances. The effects of the group and surface treatment, as well as their interactions on bond strength, were analyzed using the generalized linear model method. "Post hoc" pairwise comparisons were performed using Bonferroni correction. A significance level of $p<0.05$ was applied.

RESULTS

ESEM ANALYSIS

In the ESEM analysis of Group 1 (Figure 1a, Figure 2a), the silica coating did not create pores on the materials and exposed some fibers, creating a rough surface. However, in PEKK, deep micro-voids and rougher surface morphology were observed. In Group 2 (Figure 1b, Figure 2b), PEEK has light scratches that are close to smooth, while PEKK had a rough surface with more frequent and deep scratches. For Group 3 (Figure 1c, Figure 2c), ESEM analysis showed regular light scratches in the PEEK while laser ablation maintained the integrity of some fibers in the center and scratches on the periphery in the

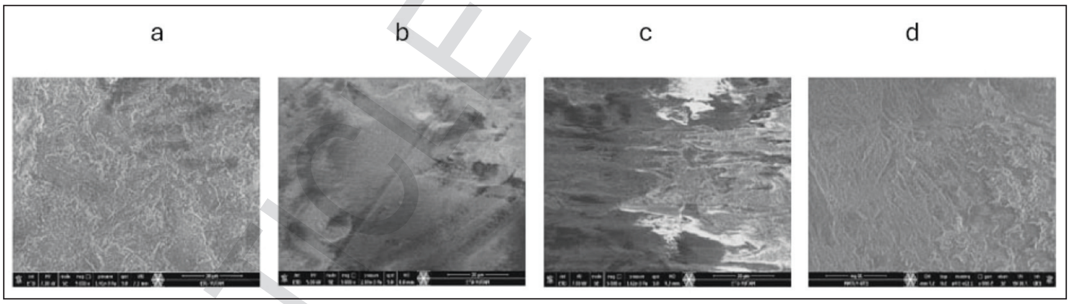

FIGURE 1: a: ESEM image (×5000 magnification) of PEEK after silica coating; **b:** ESEM image (×5000 magnification) of PEEK after acid etching; **c:** ESEM image (×5000 magnification) of PEEK after laser irradiation (100 µm groove depth); **d:** ESEM image (×5000 magnification) of PEEK after laser irradiation (150 µm groove depth)

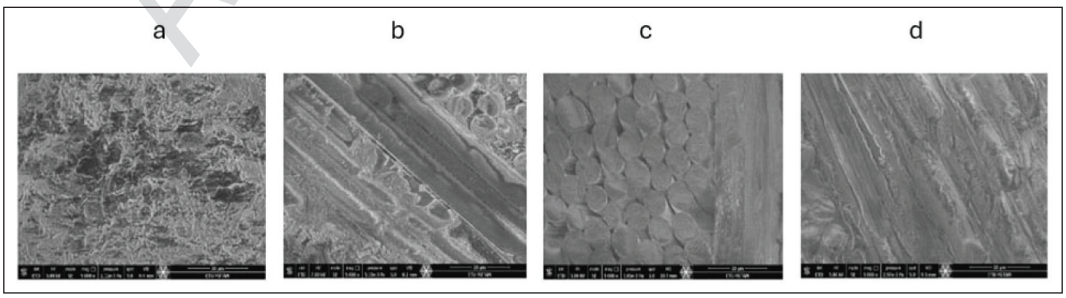

FIGURE 2: a: ESEM image (×5000 magnification) of PEKK after silica coating; **b:** ESEM image (×5000 magnification) of PEKK after acid etching; **c:** ESEM image (×5000 magnification) of PEKK after laser irradiation (100 µm groove depth); **d:** ESEM image (×5000 magnification) of PEKK after laser irradiation (150 µm groove depth)

PEKK. In the ESEM analysis of Group 4 (Figure 1d, Figure 2d). It was observed that the PEEK surface was homogeneously modified, resulting in a rough texture with minor pits. On the other hand, uniformly distributed scratches were observed in PEKK, and there were micro hills on some parts of the surface.

Figure 1a-d and Figure 2a-d illustrate the representative surface morphologies of each group, where characteristic features described in the text-such as scratches, pits, and fiber exposure-are clearly visible at 5000× magnification.

SBS Evaluation

The main effect of the material was found to be significant on SBS ($p<0.05$). While the average value of SBS in PEEK was 16.47 MPa, the average value of SBS in PEKK was found to be 11.06 MPa. The main effect of surface treatments was also determined to be statistically significant ($p<0.05$). While the SBS value was 25.56 MPa in the silica coating surface treatment group and 13.45 MPa in the acid etching group, it was obtained as 7.88 MPa in the 100 µm depth laser applied group and 8.17 MPa in the 150 µm depth laser applied group. Material and surface treatment interactions were also found to be statistically significant on SBS ($p<0.05$). The highest SBS value was found in the silica coating group of PEEK. The difference between this group and the groups where other surface treatments were applied to PEEK was determined to be statistically significant ($p<0.05$). The SBS value in the acid etching group of PEEK material was obtained as statistically significantly higher than the 2 groups in which laser treatment was applied at 100-150 µm depth on the same material. While the lowest bond strength value in PEEK was obtained in the group where the laser was applied at a depth of 100 µm, no significant difference was obtained between the groups where the laser was applied at a depth of 100-150 µm.

In the PEKK, the SBS was highest in the silica coating group, as in the PEEK, and this value was obtained as statistically significantly higher than all other surface treatment groups of the PEKK ($p<0.05$). The lowest SBS value in PEKK was obtained in the group where the laser was applied at a depth of 150 µm. No significant difference was obtained in terms of SBS between the acid etching and laser-applied groups at 2 different depths on the PEKK. Surface treatment was found to have the greatest effect on SBS. 69.8% of the bond strength is explained by material and surface treatment (Table 3). Other multiple comparison results are presented in Table 4. The mean and standard deviation graph of SBS according to material and surface treatment is given in Figure 3. Since all SBS values were greater than 5 MPa, they complied with the ISO 10,477 standards.[28]

DISCUSSION

In this study, the bond strength of high-performance polymers to resin cement was investigated according to the different surface treatments. The results showed that the silica coating increased the SBS significantly. On the other hand, for both PEEK and PEKK, the bond strength in the laser-treated groups was found to be significantly lower than in the other surface-treated groups. Therefore, the null hypothesis that surface treatments will not make a difference in the bond strength of polymers with resin cement was rejected.

The systems frequently used in the tribochemical silica coating process are the Rocatec and Cojet (3M ESPE) systems. The Cojet system is a system

TABLE 3: Examination of the effects of materials and surface treatments on SBS

	Sum of square	df	$\bar{X}$ Square	F	p value	Partial Eta square
Material	468.109	1.000	468.109	17.712	<0.001	0.240
Surface treatment	3,280.550	3.000	1,093.517	41.375	<0.001	0.689
Material*surface treatment	290.211	3.000	96.737	3.660	<0.001	0.164

df: degree of freedom

TABLE 4: Descriptive statistics of shear bond strength for groups

	Material		
	PEEK (n=52)	**PEKK (n=52)**	**Total**
Surface treatments			
Group 1 (n=13)	29.97±13.18^{c}	21.14±1.19^{b}	25.56±10.13^{A}
Group 2 (n=13)	18.44±3.66^{b}	8.45±1.89^{a}	13.45±5.88^{B}
Group 3 (n=13)	7.61±2.11^{a}	6.50±1.02^{a}	7.88±1.68^{C}
Group 4 (n=13)	9.85±3.51^{a}	8.16±1.20^{a}	8.17±3.04^{C}
Total	16.47±11.21^{A}	11.06±6.10^{B}	13.76±9.36

A-CThere is no difference between surface treatments with the same letter; A-BThere is no difference between materials with the same letter; a-cThere is no difference between material and surface treatment interactions with the same letter.
PEEK: Polyetheretherketone; PEKK: Polyetherketoneketone

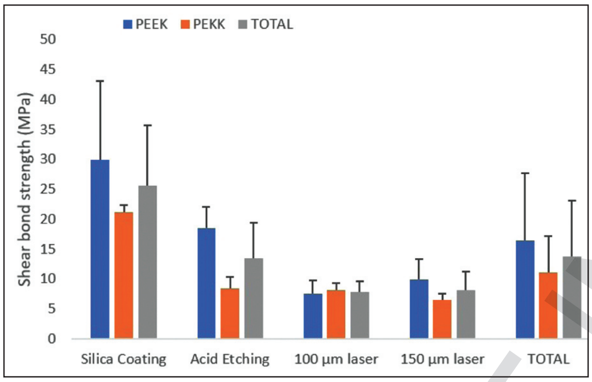

FIGURE 3: Bond strength mean and standard deviation graph according to material and surface treatment
PEEK: Polyetheretherketone; PEKK: Polyetherketoneketone

that can be easily applied in the clinic where 30 µm aluminum oxide sand modified with silane is applied to the material surface. In cases where restorations need to be repaired intraorally, it is a preferred method for providing mechanical roughness. In this study, the Cojet system was used in the tribochemical silica coating process. Schmidlin et al. applied various surface treatments to the PEEK and concluded that silica coating treatment significantly enhanced bond strength (11.5±3.2 MPa) compared to the control group.[29] Similarly, in the current study, silica coating was found to significantly improve the bond strength of both high-performance polymers. The superior bond strength observed with tribochemical silica coating may be attributed to a combination of micromechanical interlocking and chemical interaction facilitated by the silica layer, which increases the surface reactivity and enhances adhesion with resin cement.[5] This is consistent with previous findings, where the tribochemical coating system not only increased surface roughness but also enhanced chemical bonding via silica incorporation, providing an effective interface for silane coupling and subsequent resin adhesion.[30]

Studies have reported that bond strength values increase when Visio. link adhesive is applied to PEEK samples with different surface treatments.[31,32] Based on this result, Stawarczyk et al. recorded the Visio link applied group as the positive control group in their study.[33] Thus, Visio link adhesive was applied to all samples after surface treatments in the current study. The acid etching process changes the chemical structure of the PEEK and bonds with the carbonyl and ether groups of the polymer. This increases the reactivity of the material to the resin cement by revealing functional groups on the surface of the PEEK.[34] In the current study, it was reported that the acid etching treatment increased the bond strength of PEEK, which supports this. However, due to its corrosive nature, sulfuric acid can cause serious damage when it comes into direct contact with the skin, so it is not suitable for clinical use. Additionally, it has been noted that the sulfur functional groups abundantly generated through sulfuric acid application can negatively impact human cells. These groups may lead to DNA damage by producing low-value sulfur compounds, free oxygen radicals, and harmful cellular effects, and sulfur dioxide.[9,35]

In this respect, the silica coating process can be chosen as an alternative to sulfuric acid since the bond strength of PEEK in the current study was found to be high in the silica coating group, as in sulfuric acid. On the other hand, in PEKK, the highest bond strength was found in the silica coating group, followed by the sulfuric acid group. It is thought that this difference between the materials may be due to the 2nd ketone group present in the PEKK. Recent studies evaluating PEKK materials have also demonstrated lower bond strength values compared to other high-performance polymers like PEEK when subjected to certain surface treatments, particularly laser-based protocols. For instance, Asik and Ozyilmaz observed that laser irradiation failed to significantly improve PEKK bond strength, possibly due to the material's higher crystallinity and the presence of titanium dioxide (TiO_2) particles, which may scatter laser energy and limit effective surface modification.[12] It has been reported that the 20% TiO_2 content in PEKK contributes to increased hardness and wear resistance, and due to the lower energy output of dental lasers compared to the melting point of titanium, these particles may not be adequately affected by irradiation and tend to remain unmodified on the surface. This could lead to suboptimal micromechanical interlocking and reduced adhesive interaction with resin cement, as also discussed in prior literature.[36,37] These intrinsic material differences may explain the lower performance of PEKK observed in our study, especially in laser-treated groups.

The association between surface morphology and bond strength in this study aligns with previous structural equation modeling -based reports. Schmidlin et al. showed that sulfuric acid and silica coating produced porous or microrough surfaces on PEEK, enhancing micromechanical retention. Similarly, our ESEM images (Figure 1a, Figure 2a) revealed granular, irregular morphologies in silica-coated groups, supporting their superior SBS values.[29] Park and Lee also confirmed that silica application via the Rocatec system improved surface roughness and bonding.[30] In contrast, laser-treated groups-especially PEKK-exhibited smoother or shallow-grooved surfaces (Figure 1c-d and Figure 2c-d), which likely limited resin infiltration. This is consistent with Asik and Ozyilmaz who reported that laser irradiation left PEKK surfaces irregular and poorly reactive due to unmodified TiO_2 particles.[12] These findings highlight the key role of surface topography in bonding performance, shaped by both material composition and treatment methods. These differences in surface reactivity suggest that clinicians should carefully consider the specific polymer type when selecting surface treatment protocols, as PEKK may require more aggressive or alternative conditioning approaches to achieve reliable bonding in clinical applications.

Various aging methods are employed to replicate intraoral conditions. In laboratory experiments, artificial aging methods like thermal cycling or water immersion are frequently employed to assess bond strength. Likewise, in this study, to simulate clinical conditions, the samples were maintained in distilled water at 37 °C for 24 hours following the application of adhesive resin cement, aligning with prior research.[8]

Various methods are available for measuring bond strength *in vitro*, with shear, tensile, and microtensile bonding tests being the most commonly used techniques.[38] Although it has been stated that stress distribution is more homogeneous in tensile tests in theory, this may not be suitable in clinical practice because the slightest deviation in the direction of the force and the position of the sample under examination will affect the results. Therefore, in practical applications, it can be said that the load distribution in shear tests is more uniform.[39] In addition, shear tests were observed to be more successful in simulating the forces in the oral environment compared to tensile tests.[40] In this respect, the shear test was preferred for evaluating the bond strength in the current study.

As a limitation of the study, different luting cement materials and surface modification methods could be examined to evaluate the bond strength of high-performance polymers to resin cement. As this study was conducted under *in vitro* conditions, it may not entirely represent the clinical scenario. The results of the *in vitro* study may differ under various static-dynamic aging conditions. Moreover, fracture mode analysis was not performed following debonding, which limits the ability to characterize the nature of failures (adhesive, cohesive, or mixed). Future studies should include such analyses to better understand the failure mechanisms. Furthermore, long-term durability of the bond remains a concern, as previous studies have reported inconsistent outcomes under thermocycling and water storage conditions.[41,42] This highlights the need for future investigations utilizing dynamic aging protocols to more accurately simulate intraoral conditions.

From a clinical standpoint, the choice of surface treatment should be tailored to the specific application. Tribochemical silica coating may be preferred for definitive prostheses or intraoral repairs due to its reliable bonding performance. Although sulfuric acid is effective, it is limited in clinical use due to safety concerns. Laser treatments offer the advantage of non-contact application but their success depends on the polymer type and laser parameters. Moreover, the long-term stability of bonding remains uncertain, highlighting the need for further studies incorporating thermomechanical aging to validate these protocols clinically.

Bond strength in dental materials poses an important clinical problem. Research has concentrated on techniques for achieving sufficient bond strength between prosthetic materials and luting cements. When the literature is reviewed, there are not enough studies investigating the bond strength of PAEKs with resin cement, especially by applying laser surface treatment. In the current study, PEEK and PEKK were included, changing the laser parameters, and comparing the mechanical and chemical surface treatments together.

CONCLUSION

Within the limitations of this in vitro study, the following conclusions can be drawn:

i. Silica coating and sulfuric acid etching significantly improved the bond strength of PEEK to resin cement; however, due to the corrosive nature of sulfuric acid, the tribochemical Cojet system is a more clinically feasible alternative.

ii. Among all treatments, silica coating yielded the highest bond strength values for PEKK as well.

iii. Laser surface treatments applied with the tested parameters were ineffective for both polymers, resulting in significantly lower bond strength values compared to other methods.

iv. Despite their structural similarities, PEEK and PEKK exhibited different responses to the same surface treatments, highlighting the importance of material-specific protocols.

These findings underscore the need for careful selection of surface conditioning methods based on the material type and clinical applicability.

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Conflict of Interest

No conflicts of interest between the authors and / or family members of the scientific and medical committee members or members of the potential conflicts of interest, counseling, expertise, working conditions, share holding and similar situations in any firm.

Authorship Contributions

Idea/Concept: *Büşra Tosun;* ***Design:*** *Büşra Tosun, Nuran Yanıkoğlu, Tuğrul Kırtıloğlu;* ***Control/Supervision:*** *Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan;* ***Data Collection and/or Processing:*** *Büşra Tosun, Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan;* ***Analysis and/or Interpretation:*** *Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan;* ***Literature Review:*** *Büşra Tosun, Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan;* ***Writing the Article:*** *Büşra Tosun, Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan;* ***Critical Review:*** *Büşra Tosun, Nuran Yanıkoğlu, Tuğrul Kırtıloğlu, Gözlem Ceylan.*

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