Official Dental Journal of Suez Canal University
DENTAL SCIENCE Updates
Print ISSN 2636-3836
Online ISSN 2636-3844
www.dsujournals.ekb.eg
DSU — Vol. 5, No. 2, September (2024) — PP. 275:284

EFFECT OF SURFACE MODIFICATIONS OF POLY ETHER ETHER KETONE ON SURFACE ROUGHNESS, WETTABILITY AND BONDING TO VENEERING RESIN COMPOSITE

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DOI: 10.21608/dsu.2024.224242.1193
Manuscript ID: DSU-2307-1193 (R2)

KEYWORDS

Air abrasion,
Shear bond strength,
Surface roughness, PEEK.

ABSTRACT

Introductio: PEEK was widely utilized for medical applications, showing obvious recent focus in dentistry. Using PEEK for complete-coverage monolithic replacements was constrained through various aesthetic issues due to its off-white shade and little transparency. Consequently, further veneering is required to achieve acceptable aesthetics. **Aim:** In this study, a modified polyetheretherketone (PEEK) material was bonded to a resin composite veneer with the aim of evaluating the impact of various surfacing preparation methods for roughness, wettability also shears bond strength. **Material and Methods:** 160 disk-shaped PEEK specimens with dimensions (5×5×2mm) were cut to 4 groups: Group 1(without any modification), Group 2(98% sulfuric acid etched for one minute), Group 3(sandblasted through 50 µm Al_2O_3), then Group 4(combination of airborne particle abrasion and sulfuric acid etching). Surface roughness (Ra) was determined using a profilometer, while wettability was determined according to the static sessile drop method using deionized water at 22°C for contact angle measurements. Shear bond strength (SBS) was assessed on universal test device. Statistics were applied to the data using ANOVA. **Results:** Surface roughness of the specimens was significantly raised ($p \leq 0.001$) by air abrasion with 50 µm alumina. The most significant shear bond strength measurements were found for the combination of airborne particle abrasion and acid etching. **Conclusion:** A reinforced PEEK that contains 20% submicron ceramic fillers, could be treated by sandblasting and etching, to development durable bond.

INTRODUCTION

PEEK was highly efficient a semi-crystalline polymeric bio materials component. It falls under the polyaryl-ether-ketones (PAEK) category[(1)]. An aromatic linear chain is linked to this methacrylate-free polymer by ether and ketone groups[(2)]. The PEEK polymer is resistant to mechanical stress, oxidative assaults, and high temperatures because to its aromatic rings. Polyether-ether-ketone is a desirable material for the medical and dentistry fields because to its noteworthy mechanical qualities, resisting both inorganic and organic substances, in addition biocompatibility[(3)].

PEEK is a substance with a long history of use in the medical field that is currently gaining popularity in the field of dentistry[(4)]. Its grayish-white colour and limited translucency cause certain aesthetic

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issues. Therefore, more dental composites are still required for veneering in order to achieve satisfactory aesthetics[5]. Furthermore, PEEK's hydrophobic, chemically inert character, together with its resistance to alterations by various chemical and mechanical treatments, have restricted its usage in prosthetic dentistry. To effectively apply PEEK in dentistry with long-lasting constancy, procedures aimed at obtaining a lasting connection between PEEK and resin composites essential be established[5,6,7].

Proper diffusion of the adhesive substance on the surface of the adherent or substrate is the primary prerequisite for long-lasting bonding, and this can only be accomplished if the adherent's surface free energy is larger than the adhesive's own. Adhesives often have a surface energy higher than untreated PEEK, which results in poor bonding capabilities. Many efforts were made to enhance the PEEK's surface energy through utilizing various modifications procedures, including laser, sandblasting, plasma and acid etching treatments, since it is difficult to change the surface energy of the adhesive[6,8,9]. Their final findings demonstrated that PEEK's surface free energy rose in the polar component after surface treatment.

Through acid etching and sandblasting, PEEK has gained a wide field of application in dentistry to overcome its adhesive problem to resin composite materials. The purpose of the current education be situated to evaluate how different mechanical also chemical superficial treatments affected the PEEK core material's and a veneering composite's shear bond strength.

MATERIAL AND METHODS

Table (1) *Constituents used, Compositions, Producer, and Lot number*

Constituents	Composition	Producer	Lot number
PEEK	Titanium dioxide ceramic filler (20 weight percent) with polyetheretherketone	Bredent GmbH and Co.KG, Germany	510046
Sulfuric Acid	98% sulfuric acid (H_2SO_4)	Merk KGaA, Darmstadt, Germany	K53316780
Single bond universal Adhesive	Methyl methacrylate, activators, stabilisers, and diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide	3M Deutschland GmbH, 41453Neuss, Germany	10806A
Filtek Z350 XT universal restorative® resin composite	UDMA, BDDMA, titanium dioxide, iron oxide pigments, stabilisers, activators, and pyrogenic silicic acid.	3M Deutschland GmbH, Neuss, Germany	NC86771
Filtek™ Z350XT Flowable restorative resin composite	Bis-GMA, TEGDMA, and Bis-EMA methacrylate resin monomers; dimethacrylate polymers; silica (75 nm) and zirconia (5-10 nm) nanofiller; around 65% weight filler load.	3M ESPE, St.Paul, MN, USA	NF26198

I. Sample preparation and grouping

Overall, 160 samples, 40 samples/group (5×5×2 mm^3) were sliced into sections using a low-speed diamond saw cutting machine from a PEEK disc. (Isomet® 4000 saw, Lake Bluff, Illinois, USA: Buehler) under copious water. Specimens were polished and smoothed with a silicon carbide paper (four hundred up to 800-grit) (Denmark's Struers Ballerup) to achieve an identical surface. Subsequently, the improved samples were gathered, cleaned for 30 minutes with distilled water in an ultrasonic cleaner (Codyson, China, Ultrasonic Cleaner), and later dried by air.

Samples grouping: Four test groups were created from the specimens (n=40 /group; three specimens for SEM and EDX, three specimens for FTIR analysis, seven specimens for studying the surface roughness, seven specimens for wettability testing, and 20 samples for shear bond strength assessment) based on surface treatment techniques applied as follows:

***Group* I:** Samples were not given any kind of treatment.

***Group* II:** Samples have been etched with 98% H_2SO_4 for one minute, followed by careful washing by purified water, formerly drying at standard temperature.

***Group* III:** Samples have been exposed to sandblasting with 50 μm (Al_2O_3) at a pressure of 2 MPa for 10 s, then a 10 mm separation between the specimen surface and the nozzle kept at a 90° angle.

***Group* IV:** Specimens were exposed to sandblasting with 50 μm Al_2O_3 at a pressure of 2 MPa for 10 s vertically at a 10 mm distance from the disc surface, followed by etching through 98% H2SO4 once a minute. Then, after properly cleaning the discs with distilled water, they were allowed to air dry at room temperature.

Subgrouping of the samples: All groups were further divided into 2 sub-groups (n=10), based on the method of veneer application, either with and without flowable composite for the shear bond strength test: **Subgroup A:** without flowable composite application, **Subgroup B:** with flowable composite application.

II. Fourier transforms infrared spectroscopy (FTIR) analysis

Three randomly selected specimens (n=3) of each group were analyzed using a Fourier Transform Infrared Spectroscope (FTIR) in the transmittance modes.

III. Surface roughness measurement

The Profilometer has been used to measure the roughness of the surface of seven samples chosen at random from each group. After placing a central probe with a fixed measurement speed of 0.5mm/sec and a cross length of 0.8 mm on the surface of each specimen, measurements were carried out in three distinct directions with a final resolution of the recorded data of 0.01 mm. Each specimen's average surface roughness (Ra) was calculated.

IV. Evaluation of Wettability and Water Contact Angle

With the help of an electronic contact angle goniometer with remote control, seven specimens from each group were chosen to assess their wettability. Three measurements of the contact angle(θ) were made under controlled circumstances (T = 22.1°C, RH = 40.5%) for each sample with a sessile droplet measure appliance and water medium. The reduced contact angle values were thought to be a sign of the specimen's improved wettability.

V. Evaluation of shear bond strength (SBS)

An unfilled resin material (Universal Single Bond Adhesive from 3M Deutschland GmbH in Neuss, Germany) has been applied to the residual samples (n=20 for each group) through micro brushing. Next, Oil-free air was used to blast the resin, creating a thin coating, followed by 15 seconds of light curing. To ensure reproducibility, a customized split mould cylinder (3.0 mm inner diameter, and 2.0 mm in height) was put on the surfaces that were glued. (Figure 1). For subgroup (A), the mould has been occupied with a nanofilled composite (Filtek Z350 XT universal restorative® A3 Body shade, 3M ESPE) that was cured for 40s, while for subgroup (B), a small layer of flowable composite restoration (0. 5 mm) was inserted into the mould by using a dental probe, which was light-cured for 40 s. Afterwards, the nano-filled resin composite was applied then cured for 40 seconds. All samples were kept in distilled water at 37°C for 24 hours after polymerization before the SBS test.

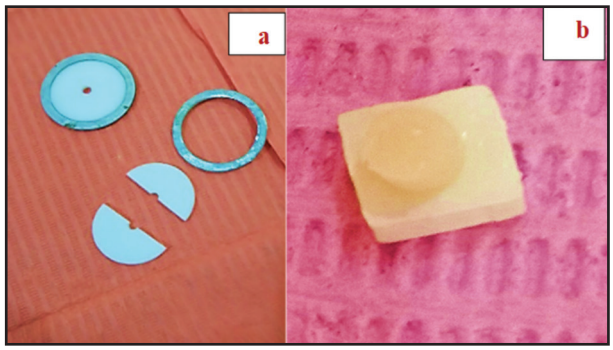

Fig. (1) **(a) Customized split mould. (b) PEEK disc veneered with a resin composite.**

A universal testing device (INSTRON, USA) was used to evaluate the shear bond strength, and the greatest load that could be applied before the veneer separated the surface of the PEEK was used as the failure load. Shear bond strength has then been determined via the following equation: SBS (MPa) = Load (N) / area (mm^2).

The effectiveness of the shear bonds formed by the various surface conditioning techniques was examined using one-way ANOVA. Subsequently, Tukey's test was then utilised for comparisons, adjusting the arithmetical significance at $p \leq 0.05$.

RESULTS

I. Scanning electron microscope (SEM) investigation:

SEM microphotos depicted a smooth layer that was obvious for the untreated group ***(Figure2a)***. Acid-etched samples displayed a rough sponge-like porous fiber network ***(Figure 2b)***, while an uneven, irregular surface was consistent with how the alumina particles were distributed for air-abraded samples ***(Figure2c)***. In the category of acid etching and air abrasion, deep cavities with sharp lines and irregularities, revealed roughened surfaces as well as agglomeration of alumina particles throughout the entire surface ***(Figure 2d)***.

II. Evaluation using Energy-Dispersive X-ray Spectroscopy (EDX):

Dispersion of Sulphur groups on surface of the PEEK polymer matrix was investigated using an EDX study.***Figure 3 (a and b)*** display the chemical elements presented on PEEK's surface. Sulphur groups were not identified in EDX at the limit of detection, most probably being related to the limitation method for EDX.

III. Fourier transforms infrared spectroscopy (FTIR) analysis:

PEEK sample (Group-I) exhibited relatively strong absorption bands. These bands are due to stretching vibrations of C=C-H, $-CH_2$, C=O, and skeletal in-plane vibrations of the aromatic ring. The chemical modification of PEEK with 98%

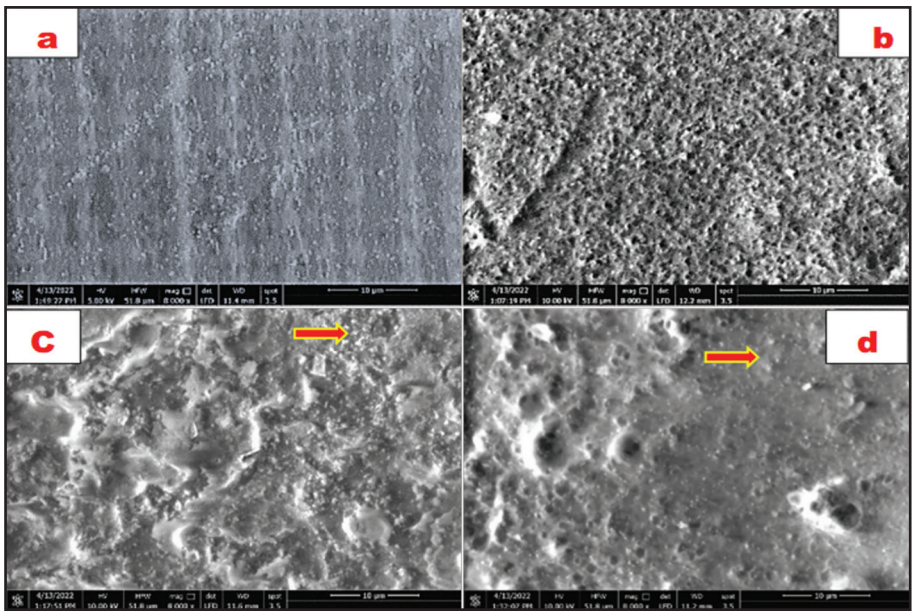

Fig. (2) SEM images of specimens' surfaces **(a)** Control group. **(b) Etched** group **(c)** Sandblasting group. **(d) The** combination group of etching and sandblasting. (The arrows refer to the presence of aluminum oxide particles.)

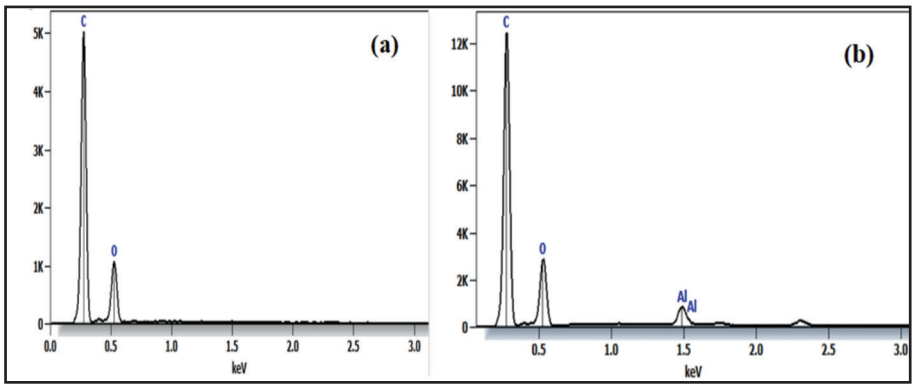

Fig. (3) EDX analysis of (a) Etched group, (b) Combination group.

sulfuric acid (H_2SO_4) (group-II) to get sulfonated poly (ether ether ketone) (SPEEK) has modified the polymer through the sulfonation procedure. With the sulfonation procedure, sulfonic collections (–SO_3H) were additional to the hydroquinone part, representative sulfonation grade [10]. On sandblasting PEEK sample, most of the relative intensities of the vibration bands were reduced and shifted to the lower frequencies. This may be attributed to the surface morphology modification. On the other hand, sandblasting with H_2SO_4 etching (group-IV) caused some shifts in vibration bands. Due to the ease of modifying the hydrophobic polymer chain, chemical change of PEEK through (H_2SO_4) to get sulfonated polyetheretherketone (SPEEK) and/or sandblasting or both has been an alternative for obtaining bioactive materials[11]. Each pretreatment group's FTIR pattern spectrum was displayed in ***Figure (4)***.

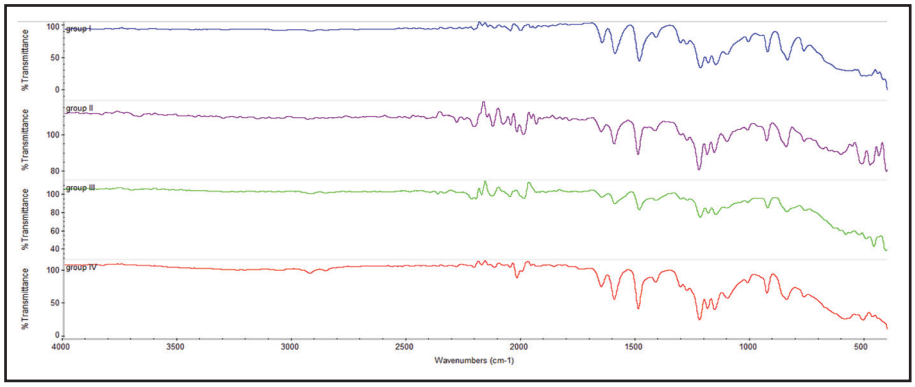

Fig. (4) Each pretreatment group's FTIR pattern spectrum

IV. Surface Roughness Measurement

Means and SD values for Roughness (Ra) of different tested groups are presented in ***table (2)*** and ***figure (5)***, being significantly different at $p \leq 0.001$. The least significant Ra values were recorded for GI, followed by GII, GIII, and GIV.

Table (2) *Means and SD for Roughness (Ra) for different tested groups.*

	Ra		Rank	p-value
	Mean	SD		
GI	0.07	0.02	C	<0.001*
GII	0.31	0.07	B	
GIII	0.67	0.07	A	
GIV	0.71	0.1	A	

*NS= Non-significant, *= significant*

Different lowercase letters within rank column indicates significant difference ≤ 0.001.*

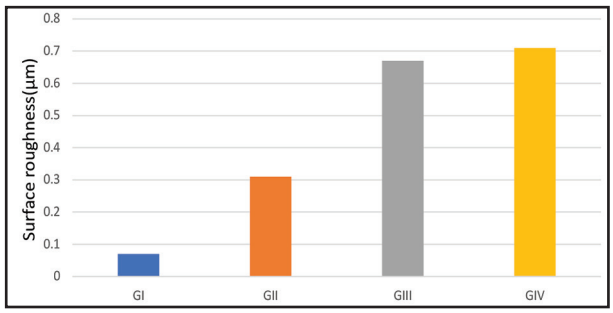

Fig. (5) Bar chart showing the mean Roughness (μm) values for different tested groups.

V. Wettability and water contact angle measurement

Means and SD for contact angle of tested groups are presented in ***table (3)*** and ***figure (6)***. Significant differences between tested group were detected at $p \leq 0.001$. GI showed the highest contact angle followed by GIII, followed by GII, with the lowest contact angle recorded for GIV.

Table (3) *Means and SD for contact angle for different tested groups*

	Contact angle		Rank	p-value
	Means	SD		
GI	87.03	6.7	A	<0.001*
GII	77	4.6	Bc	
GIII	83.62	5.19	Ab	
GIV	73.1	4.4	C	

*NS= Non-significant, *= Significant*

Different lowercase letters within rank column indicates significantly different ≤ 0.001.*

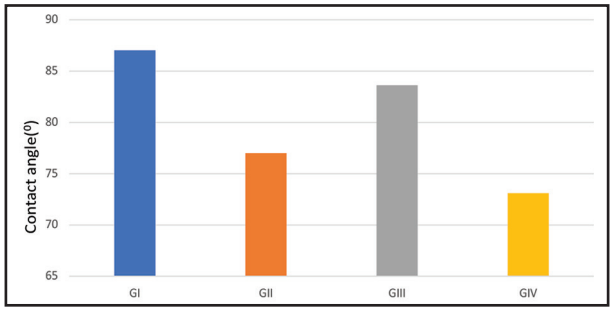

Fig. (6) Bar chart showing the mean contact angle for different tested groups.

VI. Shear bond strength (SBS)

The surface treatments had a substantial impact on the specimens' SBS values, per the two-way ANOVA ($p \leq 0.001$). Sub group A and Sub group B, revealed significant differences between tested groups at $p \leq 0.001$. GI and GIII showed the lowest significant shear bond strength values compared to GII and GIV which showed the highest bond strength values. While for all groups, insignificant differences between subgroup A and Subgroup B were recorded at $p \geq 0.05$.

Table (4) *Means and SD values for shear bond strength of different tested collections*

Groups	Shear bond strength at Maximum Load [MPa]			
	SG A		SG B	
	Mean	SD	Mean	SD
GI	0.32	0.25	1.12	0.49
GII	11.90	5.17	14.55	3.09
GIII	5.37	4.51	6.22	2.54
GIV	16.44	6.03	14.65	4.14
p-value	≤ 0.001*		≤ 0.001*	

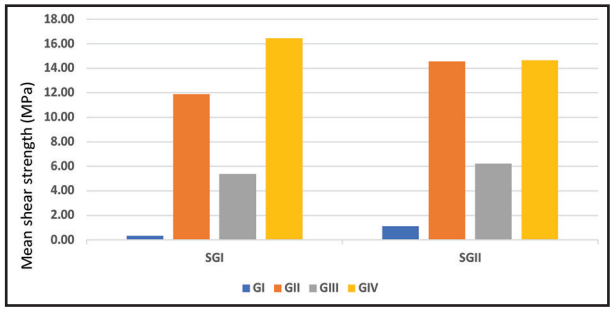

Fig. (7) The mean shear bond strength values for tested collections

DISCUSSION

The material polyetheretherketone (PEEK), which has long been utilized in medicinal applications, is now gaining popularity in the field of dentistry[4]. PEEK is a biocompatible thermoplastic restorative material which shows great biocompatibility and superior mechanical properties over acrylics and other polymeric restorative materials, making it more attractive, and even considering it an ideal restorative dental material [12]. PEEK materials that have been strengthened with ceramic fillers have improved biomechanical capabilities, making them even more appropriate for utilisation in permanent dental prosthesis.

Using PEEK for complete-coverage single restorations was constrained by various aesthetic issues due to its off-white shade and lack of transparency. Therefore, more composites designed for covering are motionless required to achieve acceptable esthetics[5]. The hydrophobic, chemically inert properties of PEEK as well as its surface resistance to alterations through several chemical in addition mechanical treatments have further restricted its usage in prosthetic dentistry. There must be a strong connection between PEEK and veneer composite resins to be able to effectively use PEEK for dental requests that exhibit enduring stability [5,6,7]. This study evaluated the impact of mechanical and chemical modifications on the veneering composite, besides PEEK shear bond strength.

The bonding strength of dental plastic materials can be significantly improved by surface roughening[13,9]. Due to PEEK's increased strength and hardness, only a select few surface roughening processes work well with it. Airborne particle abrasion was chosen as the mechanical surface treatment in the current investigation based on earlier research that identified it as one of the greatest first pretreatment choices designed

for PEEK surfaces[13,14]. As opposed to that, past research found that etching PEEK with H_2SO_4 may create a strong connection that would last for a long time. Other acids, such as hydrochloric acid and nitric acid, on the other hand, did not result in any surface alterations even at the greatest concentrations[13,15,16]. To improve the outer layer's roughness and bonded area, multiple treatment techniques were used in PEEK materials because it was discovered that surface roughness improved adhesive approaches[17].

The results of the current study showed a surface roughness of airborne particle abrasion on Al_2O_3 (0.67μm) that was higher than that of an etched peek surface (0.31μm), being significantly different. The higher surface roughness values for the sandblasted PEEK specimens were also demonstrated in the pictures of SEM, revealing an irregular and fissured rough surface for sandblasted specimens, with some surface-embedded alumina particles originating from sandblasting (***fig.2(c)***). This result is an agreement with previous studies that reported that 50μm air abrasion produced more roughness than acid etching ***Gorab et al.***[12] and also, ***Çulhaoğlu et al.***[18] who reported increased mean surface roughness values for airborne particle abrasion with Al_2O_3.

Also, the results of this study recorded a smaller contact angle of acid etched specimens (77˚) compared to sandblasted specimens (83.62˚). The production of polar groups, including hydroxyl, carboxyl, and peroxide groups, can be used to explain why acid etching-treated objects have greater wettability. This method proved to be more suitable for biological applications because the surface of treated samples is depleted of debris, thus increasing its hydrophilicity. Also, the wettability of sandblasted specimens showed a decrease rather than an increase as expected. This controversy may be linked to the wettability property's qualities, which is influenced by both the surface chemical and the contact angle. This finding is consistent with the findings of ***Culhaoglu et al.*** [18] demonstrated a lower contact angle of an acid-etched PEEK (76.0°) than that of a sandblasted PEEK (84.83°).

Several test methodologies can be used to evaluate bond strength. They include the shear/micro-shear (SBS/SBS) bond strength test, the tensile/micro-tensile (TBS/TBS) bond strength test, and additional tests[19]. The current study chose the shear bond strength test because it is relatively simple to conduct, may cause shear stress, which is a significant factor in the weakening and debonding of restorative materials, and because it can measure the shear bond strength of materials[20,21].

The findings of the current research showed a higher shear bond strength of chemically etched PEEK specimens (11.90 MPa) than that of sandblasted PEEK specimens (5.37 MPa), being significantly different. The higher bond strength values for the acid-etched PEEK specimens may be explained as acid etching alters the biochemical characteristics of the PEEK construction by eliminating organic leftovers, aggressive the aromatic constructions and dissolving the ether and carbonyl functional groups between the benzene ring[13,22,23]. Thus, with the increased surface energy, diffusion of the bonding agent into PEEK surface porosities allowed. This result is in accordance with previous education that reported the greatest values for shear bond strength for PEEK specimens etched with 98% sulfuric acid for 1 min[12,24,25]. According to our FTIR results, the chemical modification of PEEK with 98% sulfuric acid (H_2SO_4) to get sulfonated poly (ether ether ketone) (SPEEK) has modified the polymer over the sulfonation response. With the occurrence of the sulfonation procedure, sulfonic collections (–SO3H) are added to the hydroquinone section as

a representative sulfonation grade [10]. The bands were found at 3667 and 1484 due to the sulfonation of the PEEK sample (Group-II) [26]. The extensive band at 3667 cm-1 is credited to the OH-vibration of the OH-group of the sulphonic functional acid group (SO_3H) [27].

The air-abraded PEEK specimens' high porosities and rough surfaces might have a detrimental impact on bonding agent diffusion, besides resulting in a few frail spots at the bonding interface, are another possible explanation. This outcome is comparable to what ***Stawarczk B et al. (2014)*** were able to accomplish[12] who yet, the greatest values were not those that were obtained in superficial roughness for the sandblasted group, and the highest SBS was experiential for the acid-etching group. The agglomeration of alumina particles on the sandblasted PEEK specimens, which blocked the pores, could be the cause, according to the present SEM examination. As a result, there was a limited amount of bonding agent that could enter the pores, resulting in a general decrease in the shear bond strength.

Also, the consequences of this study showed the highest bond strength standards in case of combining air abrasion plus acid etching (16.44 MPa) likened to treatments with acid etching and air abrasion independently. This outcome was clarified by the detail that the airborne-particle abrasion changed the PEEK surface and etching treatment destroyed more chemical bonds, leaving an additional polar surface as an extra binding site, improving the adhesive's ability to penetrate surface pores. In the current study, combining airborne particle abrasion with acid etching compared to when both methods were used, produced greater surface roughness values and increased wettability for PEEK surfaces. All tested groups' SBS findings exceeded the optimal clinical service limits of 10 MPa (limits between 10.47 and 22.55 MPa) and the ISO 10477 minimum threshold level of 5 Mpa[24].

CONCLUSIONS

1. While it is true that 50 μm sandblasting produced the maximum irregularity, the SBS of composite resin veneer to PEEK was not significantly increased.
2. The etching promoted by 98% sulfuric acid increase the SBS of composite resin veneer to PEEK.
3. Compared to applying each of the two treatments separately, combining airborne-particle abrasion and acid etching treatments improved the PEEK surface's wettability and roughness surfaces, strengthening the connection between the veneering and PEEK.

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