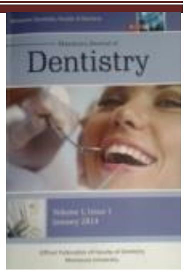
Effect of surface pretreatments on the surface roughness and shear bond strength of a modified polyetheretherketone (PEEK) material



Ahmed M . Shehab-Eldin[a], El-Sayed M. Gorab[b], Manal F. Osman[c].

[a] *Researcher of Dental Biomaterials, Faculty of Dentistry, Mansoura University.*
[b] *Assistant Professor of Dental Biomaterials, Faculty of Dentistry, Mansoura University.*
[c] *Professor of Dental Biomaterials, Faculty of Dentistry, Mansoura University..*

Abstract:

Aim of study: This study is aimed to evaluate the effect of different surface pretreatments on the surface roughness and shear bond strength of a modified polyetheretherketone (PEEK) material bonded to dentin.

Materials and methods: Eighty disk shaped PEEK specimens with dimensions (10 mm X 2 mm) were machined and allocated into 4 pretreatment groups, Group 1; no treatment, Group 2; etching with 98% sulfuric acid for 1 min, Group 3; air abrasion with 50 µm Al_2O_3, and Group 4; nitrogen plasma treatment. Surface roughness (Ra) was determined by a profilometer. Shear bond strength (SBS) to human dentin was tested using a universal testing machine at a crosshead speed of 1mm/min perpendicular to the bond interface. Data were statistically analyzed using ANOVA and Tukey's HSD, $\alpha = 0.05$.

Results: Air abrasion with 50 µm alumina significantly increased the surface roughness ($P<0.001$). The highest shear bond strength values were observed for sulfuric acid etched group.

Conclusions: A modified PEEK material strengthened with 20% submicron ceramic fillers, could be treated by etching with 98% sulfuric acid for 1 min, to enhance bond strength to human dentin.

Keywords: Surface roughness, air abrasion, PEEK, shear bond strength.

Introduction

Polyetheretherketone (PEEK) is considered as one of the most common high-performance engineering plastics recently available. It has a high-temperature semi-crystalline thermoplastic polymer become from the group of polyaryletherketone (PAEK) that composed of an aromatic backbone molecular chain, linked together alternately by ether or ketone functional groups.[1] PEEK is extremely beneficial in many applications for industries, such as automotive, aerospace, electronic devices, and medical equipments, that is because of its perfect mechanical properties, like thermal resistance, solvent resistance, great wear resistance, high fatigue resistance and great electrical insulator.[2, 3]

The material moreover has great dimensional stability; its modulus of elasticity is partially located between the cancellous and cortical bone.[4] Also, the PEEK is actually radiolucent and convenient to imaging procedures, such as X-rays, magnetic resonance imaging (MRI), and computed tomography (CT). The PEEK permits to examination, diagnosis, and treatment of clinical situation without requiring substructure expulsion or substitution because of its radiolucency. Thus, PEEK can displace the conventional materials (like stainless steel and titanium) as metallic implants in orthopedics, spinal surgery, and traumas.[3, 5, 6] In dentistry, PEEK is basically applied for the production of the frameworks of fixed and removable dental prosthesis, implant frameworks, implant fixtures, and restorative implant parts.[7, 8]

Modification procedures, like a filling, blending, and fiber reinforcement, are essential for fabrications of new PEEK-based material with ideal features required in different applications field.[9-12] The dental prostheses can be fabricated by PEEK either by using CAD-CAM or injection molding procedures. The benefits of using this material are eliminating allergic reactions, excellent polishing features, and low plaque affinity. A modified PEEK material (BioHPP; Bredent GmbH) containing 20% ceramic fillers was introduced in dentistry with great mechanical properties and amazing biocompatibility.[13, 14]

In addition to all these positive aspects; however, establishing a strong and durable adhesion of a PEEK infrastructure to resin cement and dentin is difficult owing to its low surface energy, inert chemical performance, and resistance to surface modification.[15] Therefore, enhancing the surface features of PEEK has gotten to be a research hotspot. Adhesive properties, which are critical for the stabilization of dental prosthesis, are affected by the surface modifications. Therefore, studying the effect of different surface pretreatment on surface characteristics and bond strength of a modified polyetheretherketone (PEEK) material adhesively luted to human dentin might be of value.

MATERIALS AND METHODS:

The materials used in the present study are shown in Table 1.

I. Preparation and grouping of specimens

A total of eighty disk-shaped specimens with dimensions (10 mm X 2 mm) were cut out of PEEK blanks under copious cooling water using CAD/CAM technique. The specimens were polished and smoothed by 800 grit SiC papers (Struers Ballerup, Denmark) to accomplish a standardized surface.

Thereafter, the polished specimens were collected and washed by distilled water in an ultrasonic cleaner for 30 min (Codyson, Ultrasonic Cleaner, China) and then air-dried. The specimens were classified into four tested groups (n=20 for each group; 10 specimens for studying the surface roughness and 10 specimens for measurement of shear bond strength) according to surface treatment methods as follow;

- Group 1: The specimens was not be subjected to any treatment.

Group 2: Specimens were treated using sulfuric acid, the bonding surfaces of the PEEK materials were etched with 98% sulfuric acid (Sigma- Aldrich, Chemie GmbH, Steinheim, Germany) for 60 sec. Then, acid was washed off for 60 sec with distilled water and then dried with oil-free compressed air.

Group 3: Specimens were treated using air abrasion, the surfaces of PEEK materials were sandblasted (Basic Master, Renfert GmbH, Hilzingen, Germany) with 50 µm Al2O3 particles (Shera, Germany) for 15 sec at a distance of 10 mm perpendicular to the treated surface and a pressure of 0.4 MPa. After that, cleaned the specimens for 60 sec with distilled water and then dried with oil-free compressed air.

- Group 4: The specimens were treated using a low-pressure plasma system (Pico, Diener, Electronic GmbH, Germany). PEEK specimens were positioned on the stand of samples, and the system was set at 0.2 mbar pressure. The flow of nitrogen was adjusted at 22 sccm (standard cubic centimeters per minute), and the specimens were treated for 1 min with 400v power plasma.

Table 1: Materials used in this study.

Materials	Composition	Batch number	Manufacturer
breCAM.BioHPP (modified PEEK CAD/CAM material)	Polyetheretherketone , 20wt% titanium dioxide ceramic filler	56654456	Bredent GmbH &co., senden, Germany
G-CEM LinkAce ™ (self-adhesive dual-cure resin cement)	**Past A:** Fluoro-alumino silicate glass, UDMA, dimethacrylate, Sio_2 , initator , inhibbitor **Paste B:** Sio_2 , UDMA, dimethacrylate, intiator, inhibitor	1810101	GC Corp., Tokyo, Japan

II. Surface roughness measurement

Ten specimens were selected randomly from each group to evaluate surface roughness using a profilometer (SURFTEST SJ-201, Mitutoyo Corp., Japan). Measurements were accomplished in three various directions after placement the probe in the center of the surface of each specimen with a cross length of 0.8 mm and a fixed measuring speed of 0.5 mm/sec. The resolution of the recorded data was 0.01 µm. The average surface roughness (Ra) of each specimen was calculated.

Ⅲ. Evaluation of shear bond strength (SBS)

Teeth selection and dentin preparation

Forty freshly extracted human molar teeth with caries-free were collected from the outpatient dental clinic, in oral surgery department of Mansoura University, (age; 40-65) and these teeth extracted due to periodontal disease, The teeth were cleaned from the surface debris, sterilized in 0.5% sodium hypochlorite directly after the extraction, and then stored in deionized water at 3-4° C until be used.

The occlusal dentin surface was exposed by cutting vertically to the long axis of the tooth by using a low-speed diamond disk (PICO-155 Precision Cutter, PACE Metallographic Technologies, USA) underwater irrigation. The prepared dentin surface was observed by using a stereomicroscope (Olympus SZ61TR Trinocular Zoom, Tokyo, Japan) at a magnification of 40X for detecting any residual enamel. The teeth were polished using #600-grit silicon carbide sandpapers by hand under running water for 60 seconds to expose a flat standardized dentin surface. The polished surfaces have been cleaned for 5 min in an ultrasonic cleaner. Each tooth was inserted in auto-polymerizing acrylic resin (Acrostone, Anglo-Egyptian Company. Hejaz, Cairo, Egypt) in plastic cylinders with the flat dentin surface exposed.

Bonding procedures

Disc-shaped PEEK specimens were cemented onto the dentin with dual-cured self-adhesive resin cement (G-CEM LinkAce, GC Corp., Tokyo, Japan). The cementation technique application was done according to the manufacturer’s guidance. The cement was mixed through the auto-mixing tips in a 1:1 base to catalyst ratio, a thin and even layer of the resin cement was applied to the PEEK pretreated surface. The PEEK specimens were placed above the dentin surface and kept in place under a load of 500gm for 10 min. Excess resin cement was removed with a hand instrument, and the dentin/PEEK set was light-cured by an LED light-curing unit (Monitex LiteQ LD-107, Taiwan) at the intensity of 1000 mW/cm2 for 20 sec in various direction (top of the PEEK and then at bonding margins). Specimens were stored in distilled water for 24 h at 37° C and then subjected to 5000 thermal-cycles from 5°C to 55°C, at 20 sec staying time between baths in a thermocycler machine (Theromocycler, Robota, Alexandria, Egypt).

Shear bond strength (SBS) testing

The shear bond strength has been experimented with using a universal testing machine (Instron Model 3345, Instron Corp., MA, USA) at a crosshead speed of 1 mm/min and used a chisel loading applicator positioned accurately perpendicular to the bonded margins and as close as possible to the interfacial zone. A shear load was applied until failure occurred. The bond strength (s) values, expressed in MPa, were calculated using the following formula:

$\tau = F/A$, where τ is the shear bond strength (MPa), F is the fracture load (N), and A is the bond area (mm^2).

Determination of mode of failure

After debonding, the bonding surfaces of each specimen were inspected using an optical stereomicroscope (Olympus SZ61TR Trinocular Zoom, Tokyo, Japan) with magnification 15x for the PEEK disc and 10x for the tooth structure to determine the mode of failure. Failure mode was categorized into three types:

(A) Adhesive failure between resin luting agent and dentin or PEEK surface;
(B) Cohesive failure within the resin luting agent; and
(C) Mixed mode of failure.

V. Statistical analysis

The data obtained were tabulated for statistical analysis which was conducted using Statistical Package of Social Science (SPSS) v25.0 software (SPSS Inc., Chicago, IL, USA). The normality of data was first tested with Shapiro test. Means and standard deviation of surface roughness and shear bond strength (SBS) to dentin were obtained for each group. One way ANOVA test was used to compare between the 4 groups, while post hoc Tukey's test was used for in-between groups’ comparison.

The threshold of significance is fixed at 5% level (p-value). The results were considered:

- Non-significant when the probability of error is more than 5% ($p > 0.05$).
- Significant when the probability of error is less than 5% ($p \leq 0.05$).

RESULTS:

I. Surface roughness

All groups' means, as well as standard deviations of surface roughness (µm) are shown in Table 2. The diagrammatic presentation of these values is shown in Figure 1. The highest surface roughness was noted with the Sand-blasting group (3.465 ± 0.203), followed by the Sulfuric acid group (0.764 ± 0.317), then the Plasma group (0.146 ± 0.035), and the lowest surface roughness was noted with the Non-treated group (0.133 ± 0.014). One-way analysis of variance (ANOVA) results of surface roughness are displayed in Table 3. There was a significant difference in surface roughness between the tested groups ($P < 0.001$). Tukey's statistical test displayed a significant difference between group 4 and both 2 and 3 and between group 3 and both groups 1 and 2 and between group 1 and group 2 ($P < 0.001^*$). On the other hand, there was no significant difference between group 1 and group 4 ($P = 0.999$).

Table 2: Tukey's test's Means, standard deviations and results for different groups' surface roughness (µm).

Groups	Mean ± SD
No treatment	0.133 ± 0.014^a
Sulfuric acid etched	0.764 ± 0.317^b
50 µm sandblasting	3.465 ± 0.203^c
Plasma	0.146 ± 0.035^a

Means with the same superscript letters are not significantly different (Tukey's test, $P > 0.05$).

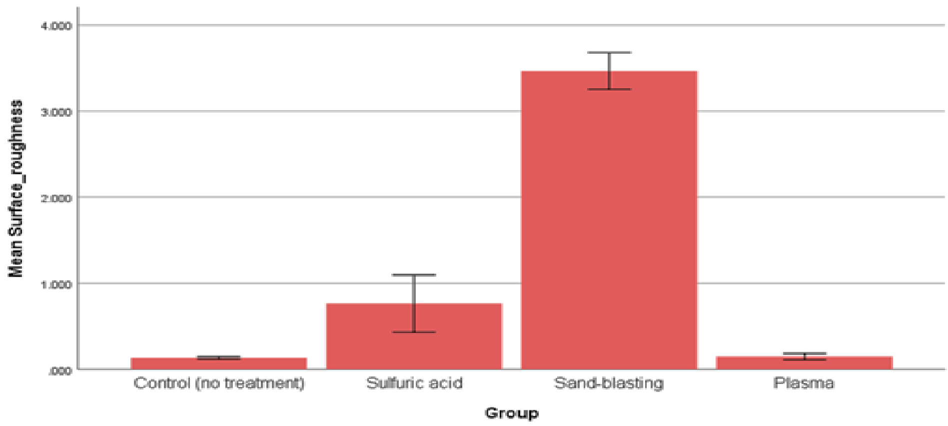


Fig.1. Means and standard deviations of the tested groups' surface roughness (µm).

Table 3: One-way ANOVA for surface roughness of different groups.

Source	Df	Sum of Squares	Mean Square	F	P
Groups	3	45.303	15.101	421.97	$< 0.001^*$
Error	20	0.716	0.036		
Total	23	46.018			

*Statistically significant difference at $P \leq 0.05$

Ⅱ. Shear bond strength

All groups' means and standard deviations of shear bond strength (MPa) are shown in Table 4 and graphically represented in Figure 2. The mean shear bond strength comparison between the tested groups to dentin showed that the sulfuric acid group exhibited the highest value (16.691 ± 5.233 MPa), while the non-treated group showed the lowest value (2.060 ± 0.559 MPa). One-way ANOVA results of shear bond strength are shown in Table 5. The studied groups were significantly different in shear bond strength ($P < 0.001$). Tukey's statistical test showed a significant difference between group 2 and other groups ($P < 0.001$*). Other way, no significant difference was shown between group 1 and both groups 3 ($P = 0.091$) and 4 ($P = 0.748$) and between group 3 and group 4 ($P = 0.476$).

Table 4: Means, standard deviations and results of Tukey's test for shear bond strength (MPa) of different groups.

Groups	Mean ± SD
No treatment	2.06 ± 0.559^{a}
Sulfuric acid etched	16.69 ± 5.233^{b}
50 µm sandblasting	$5.636 \pm 1.245^{a,c}$
Plasma	$3.517 \pm 0.442^{a,d}$

Means with the same superscript letters are not significantly different (Tukey's test, $P > 0.05$).

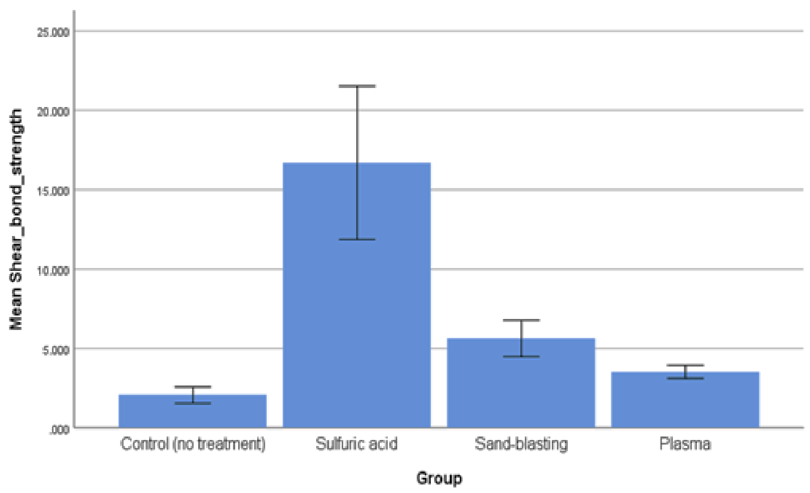


Fig.2. Means and standard deviations of the tested groups' shear bond strength (MPa).

Table 5: One-way ANOVA for shear bond strength of different groups.

Source	Df	Sum of Squares	Mean Square	F	P
Groups	3	926.217	308.739	41.945	< 0.001*
Error	24	176.654	7.361		
Total	27	1102.871			

*Statistically significant difference at $P \leq 0.05$

Mode of debonding

Examination by stereomicroscopic at the debonded area showed three modes of failure; adhesive failure when no resin luting agent residue is left on the dentin or PEEK surface, cohesive failure within resin luting agent, and mixed failure when resin luting agent remnants were partially left on dentin or PEEK surface. There was a variation in modes of failures between the tested groups as shown in Table 6, where more adhesive failure was showed in non-treated and plasma treatment groups, while cohesive and mixed ones were showed in sulfuric acid etching and 50 µm sandblasting groups.

Table 6: Modes of failure for the different groups after debonding under stereomicroscope.

Mode of failure / Surface treatment	Adhesive failure	Cohesive failure	Mixed failure
No treatment	7	2	1
Sulfuric acid etched	1	6	3
50 µm sandblasting	2	4	4
Plasma	5	3	2

DISCUSSION:

PEEK is a biocompatible thermoplastic engineering with special material properties which put it as an attractive material in the dental field, and can be considered as ideal dental restorative material causing of its great biocompatibility and superior mechanical properties. In this study, using a PEEK material reinforced with ceramic fillers modification increased the PEEK biomechanical properties, makes it more suitable for use as fixed dental prostheses, especially in height stress-bearing regions. The strong and successful bonding of PEEK material is required for its application as dental prosthetic materials.

Through the bonding procedure, the PEEK material surface should have sufficient roughness to get suitable mechanical retention. However, the surface roughening process of PEEK material was impeded because of its high strength and hardness. In the current study, the bond strength of the modified PEEK material to tooth structure was enhanced by using different pretreatment procedures. Furthermore, the use of the shear bond strength test could reproduce simply the clinical situation, and it is considered more convenient for estimating the bonding abilities of resin cement to PEEK.

The etching method is predominantly used as a surface treatment. The surface pores of the pretreatment PEEK can be penetrated by the adhesive to create adhesion collateral that can produce micro-mechanical retention. The results of the current study displayed that the etching of PEEK with 98% concentrated sulfuric acid demonstrated the highest shear bond strength, as compared to other groups. That could be demonstrated by the fact of sulfuric-acid to generate sulfonate groups (−SO3) with PEEK polymer chains that became chemically cross-linked to methylmethacrylate-based adhesives. Furthermore, the PEEK surface pores and pits have a dispersal of resin tags that lead to micromechanical bonding. This result shows that concentrated sulfuric acid could be etching the material efficiently. Therefore, using of sulfuric acid with 98% concentration is an effective procedure for improving the bonding properties of the material. It is necessary to know that sulfuric acid is a strong acid and needs special caution in handling, disposal, and storage. It could be used in dental office or prosthetic laboratories if the bond strength obtained a significant improvement.

The surface treatment by airborne particle abrasion now is considered as the easiest methods for surface treatment. In dentistry, sandblasting is consider to be one of the most widely application used for surface treatment of implant, cementation of orthodontic bracket, restoration of collapsed porcelain resin, and other parts. Sandblasting is increasing the surface roughness and activation of the surface by eliminating the organic contaminants thoroughly from the composite material surface to produce micro-mechanical interlocking with luting cement. In this study, the results exhibited that 50 µm alumina air-abrasions did not distinctly improve the bond strength to resin cement; however, it increased the (Ra) values when comparing to other tested groups. A possible explanation is that the high porosities and rough surfaces produced with alumina particles on the air-abraded Peek surface may have an adverse effect on the cement penetration and caused a few weak points of the bonding interface. This result is comparable to that achieved by Stawarczk B et al., who perceived observed that; however the highest values were achieved in surface roughness is air abraded group, the greatest SBS were observed for the acid-etched group.

The using of a plasma treatment procedure to modify PEEK surfaces is a reasonable approach idea in biomaterials to can be used in orthopedics, traumas, and a spinal implant. [3] It seems to enhance biocompatibility in terms of a respective cell to substrate interactions.[16, 17] Concerning the bonding to materials, different effects can be made by plasma application because the PEEK material mainly represents a thermoplastic organic polymer with highly cross-linked structures but a lack of sufficient functional groups being capable to react with methacrylate, which exist within the most dental composite resin materials. Thus, the chemical bonding among these substrates could be predicted to be limited. However, some polymers surfaces with plasma treatment can be improved in terms of hydrophilicity by creating oxygen-containing functional groups, such as C=O and –OH.[18] Therefore, the chemical structure of the polymer should preferably match the composition of the plasma gas to improving the adhesive properties.[19] Oxygen, Nitrogen, hydrogen, argon, and so on are the most used in low-temperature plasma surface treatment of polymer materials. In the current study, low-temperature plasma treatment was applied using nitrogen gas. The results of the present study displayed that plasma treatment could not provide a significant improvement concerning the shear bond strength. This can be attributed to lower surface roughness (Ra) values obtained with the plasma treatment group and therefore decreasing mechanical retention. This result goes to the finding obtained by Stawarczyk et al[20] who reported that plasma treatment with helium gas had no impact on the adhesion between self-adhesive resin cement and PEEK using methyl methacrylate (MMA)-based adhesives.

The clinical success of PEEK as a fixed restoration must be durable and functional. However, the oral cavity environment is complex. When we have a change in oral temperature, the PEEK, adhesive system, and the dental hard tissue changed differently in their thermal expansion coefficients, which produce stress at the bonded area. This series of changes generate an adverse effect on the long-term bonding stability of PEEK. In the present study, the aging method consisted of thermocycling for 5000 thermal cycles (5 ºC/55 ºC; staying time, 20 seconds). The effect of retention strength of the differently pretreated PEEK by thermocycling is playing an important role in durability expectation. Thermocycling is a satisfactory way for the laboratory simulation of intraoral thermal varieties caused by breathing, drinking, and eating.[21, 22] During this procedure, all specimens encountered standardized and reproducible stress.[23]

The fracture analysis revealed that modes of failures were adhesive, mixed, and cohesive for all adhesive tested groups. It was found that the retentive adhesive strength depends on the amount of the substrate fracture. Thereby, the detected mode of failure supports the achieved result values of the bond strength in this study. Furthermore, the appearance of cohesive and mixed failures might be attributed to the unequal stress distribution at the bonded area at the time of the loading procedure.[24]

CONCLUSION:

Based on the result and within limitation of the study, the following conclusion can be made:

1. Air abrasion with 50µm Al_2O_3 significantly increased the surface roughness ($P < 0.001$).
2. The highest shear bond strength value can be attained when etching with 98% sulfuric acid for 1 min.
3. Although 50µ air abrasion showed the highest roughness, it didn't clearly increase the SBS of PEEK to dentin.
4. Plasma treatment of PEEK couldn't achieve a significant improvement in SBS compered to controlled group.

RECOMMENDATIONS:

- Further studying we are recommended to evaluate other pretreatment method for PEEK such as other etching solution, silica coating and silanization techniques, and the application of other adhesive system.
- Supplementary in-vivo studies are needed to evaluate the long-term bonding stability.

periodontal ligament and alveolar bone with pocket formation, recession or both.(1)Intra-ossseous periodontal defects are at a higher risk of disease progression when left untreated.(2)

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