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# Evaluation of bond strength of polyether ether ketone (PEEK) to composite resin on hypobaric and hyperbaric conditions

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## ABSTRACT

The aim of this study was to investigate the shear bond strength of composite resin to PEEK under hypobaric and hyperbaric pressure changes in different PEEK surface treatments. Ninety-six PEEK specimens were divided into three groups ( $n = 32$ ). PEEK surfaces were sandblasted; acid-etched, and non-treated. Every group was then divided into three sub-groups ( $n = 10$ ) for simulating hyperbaric pressure (2.8 atm-18 m under sea level), hypobaric pressure (0.34 atm-8,200 m high altitude), and the control group (atmospheric pressure). The specimens underwent pressure cycles for 20 days. Results were compared by using ANOVA followed by the *post-hoc* Tukey test. Variations in bond strength within each group were also evaluated by the Weibull modulus. Regardless of the environmental pressure changes, it was determined that the bonding strength and the Weibull modulus were the highest in the acid-etched PEEK. Bonding strengths were lower in all groups after exposure to environmental pressure changes, but this difference was not statistically different. Bond strengths between the PEEK material and composite resin can be affected by environmental pressure, while surface modification of the PEEK material plays an important role in bond strength. Dentists should take importance in choosing the appropriate material on patients who are exposed to different barometric changes, as fractures on restorations may be life-threatening.

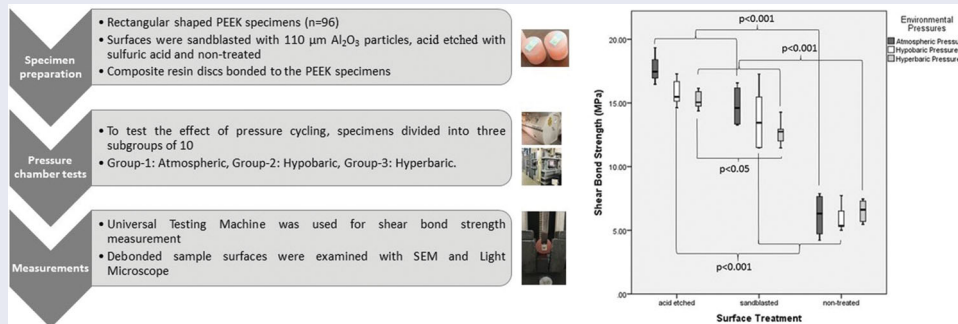
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Polyetheretherketone; composite resin; shear bond strength; aviation medicine; hyperbaric medicine

## GRAPHICAL ABSTRACT



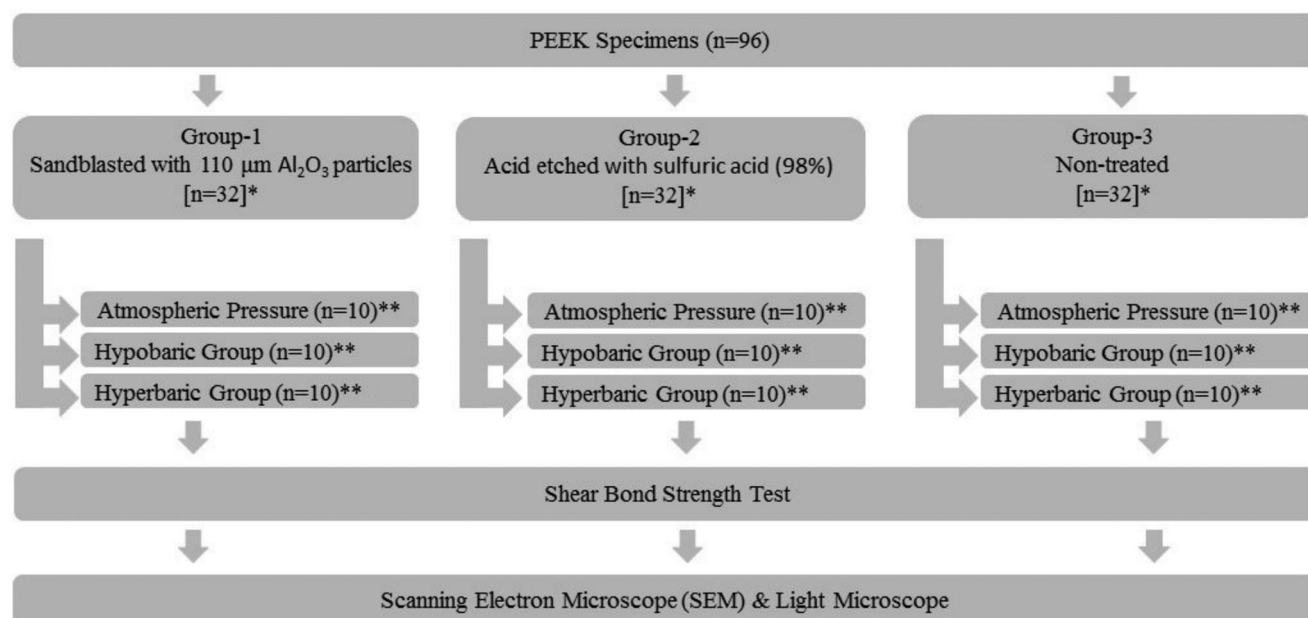
## 1. Introduction

Polyether ether ketone (PEEK) is a polycyclic, aromatic, thermoplastic polymer consisting of three aromatic ring repeating units linked together by two ether groups and a carbonyl group<sup>[1]</sup>. PEEK is used in various medical applications due to its excellent chemical, mechanical and thermal properties<sup>[2]</sup>.

In dentistry, PEEK is used in fixed prostheses, abutments, implant-supported bars, dental implants, partial prostheses<sup>[2]</sup>. However, PEEK has a disadvantage that limits its use because of its low translucency and grayish color. Therefore, composite resins are used for veneering. However, due to its low surface energy, PEEK needs surface treatment<sup>[3,4]</sup>. Application of different surface treatments, such as sandblasting, acid etching,

plasma treatment has been investigated to increase the bonding strength of PEEK material<sup>[5,6]</sup>. Previous studies have shown that the use of sandblasting for surface treatment and adhesive systems increases the adhesion strength of PEEK<sup>[5-8]</sup>. Additionally, sulfuric acid-treated PEEK surfaces have been reported to enhance the bonding strength of composite resins<sup>[9,10]</sup>.

Unlike thermal and mechanical conditions that affect dental materials and the oral cavity intermittently, barometric pressure changes have been regarded as another physical condition that may influence oral tissue and dental restorations<sup>[11]</sup>. In normal life, although the ambient pressure is almost constant, situations, such as high-altitude flight, diving, and working in hyperbaric conditions are subject to



**Figure 1.** Experimental design of the study. \*Two specimens from each group were selected for Scanning Electron Microscope (SEM) and Atomic Force Microscopy (AFM). \*\*Atmospheric pressure group was stored at ambient pressure for 20 days. The hyperbaric group pressure cycle regimen consisted of 20 pressure cycles ranging from 0 to 2.8 atm (18 m) at a rate of 0.5 atm/min. After 30 min at 2.8 atm, the decompression phase began at a descending rate over a period of ~5 min. The hypobaric group was decompressed to 1/3 atm (8,200 m altitude) in 5 min. After 30 min at 8,200 m, it recompressed to the normal atmospheric pressure over a period of 5 min. This process was repeated for 20 days.

pressure changes in the body and oral cavity. These adverse effects of barometric changes are mainly based on Boyle's Law, which states that at a constant temperature, the volume of an ideal gas is inversely proportional to its pressure<sup>[11–13]</sup>. Microleakage, secondary caries, reduced retention of dental restorations, and crowns are assumed to be the most important predisposing factors of dental barotrauma<sup>[14–16]</sup>. Few reports have been published on dental restorations and tooth fractures due to rapid changes of environmental pressure, both in hypobaric (in-flight) and hyperbaric (diving) conditions<sup>[11,12,14,15]</sup>.

The literature review has revealed very few studies on the effect of pressure changes on dental restorations. The studies examining environmental pressure changes in dentistry are mostly about cementation type, material, and techniques. Recently, the use of PEEK material in dentistry has increased. As crown dislodgements, restoration fractures can cause life-threatening risks in diving or flying, it is important to select the appropriate dental restoration. To the best of our knowledge, this is the first research that has assessed the effect of environmental pressure changes on the bonding strength of PEEK material with different surface treatments to composite resin. Therefore, this study aimed to test the shear bond strength of composite resin to different surface treated PEEK in barometric pressure changes. The null hypothesis was that neither pressure alteration cycles nor surface treatments would influence the shear bond strength between PEEK and composite resin.

## 2. Materials and methods

The experimental design of the study is in Figure 1.

### 2.1. Specimen preparation

Ninety-six rectangular shaped (5 × 10 × 2 mm) samples were prepared from PEEK blocks (Whitepeaks GmbH, Lange Heide, Essen, Germany; Lot no: E10002) and set in self-curing acrylic resin [Birlesik Group Dental (BGD), Turkey; Lot no: 170517]. For uniform surface treatment, PEEK samples were ground abraded with 600–800–1,000–1,200 grit silicon carbide sandpaper. After they were cleaned ultrasonically, and air-dried, the specimens were randomly divided into three groups ( $n = 32$  for each group) for surface modification procedures.

PEEK samples were treated with three surface modifications;

Group 1: PEEK surfaces were acid-etched with sulfuric acid (98%, BRTR, İzmir, Turkey, CAS 7664-93-9) for 1 min and then rinsed with deionized water for 1 min.

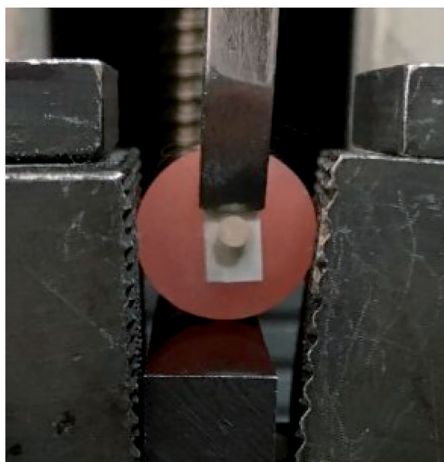
Group 2: PEEK surfaces were sandblasted with 110 μm Al<sub>2</sub>O<sub>3</sub> particles with 2 bar pressure at a distance of 1 cm for 1 min.

Group 3: A non-treated PEEK surface group.

After surface treatment, adhesive (Visio-Link, Bredent Group, Senden, Germany; Lot no: 171936) was applied on PEEK surfaces with a micro-brush and light-cured for 90 s (Labolight LV III, GC, Japan). An empty hollow plastic cylinder (3 mm diameter and 2 mm height) was placed on the surface of PEEK and 2 mm thickness composite resin (Estelite Posterior Packable Composite, Tokuyama Dental, Japan; Lot no: W110) was applied to the samples and immediately light-cured for 180 s according to the manufacturer's instructions. Materials used and their characteristics are listed in Table 1.

**Table 1.** List of materials used and their characteristics.

Material name	Manufacturer	Composition	Lot. number
PEEK-Block (CopraPeek light)	White peaks GmbH, Lange Heide, Essen, Germany		E10002
Autopolymerizing acrylic resin (Integra)	Birleşik Grup Dental (BGD), Turkey	%95 Methyl methacrylate (MMA), %5 Ethilenglicol dimethylacrylate (EGDMA)	170517
Visio-link	Bredent GmbH&Co KG, Senden, Germany	Methyl methacrylate (MMA) and pentaerythritol triacrylate (PETIA), photo initiators	171936
Sulfuric acid	BRTR Chemistry, Izmir, Turkey	98% Sulfuric acid	7664-93-9
Composite resin	Estelite Posterior Packable composite, Tokuyama Dental, Japan	Bisphenol A-glycidyl methacrylate (Bis-GMA), Bisphenol A polyethoxy dimethacrylate, CQ, dibutyl hydroxy toluene, MEQUINOL, triethylene glycol dimethacrylate (TEGDMA)	W110



**Figure 2.** Composite resin bonded to the PEEK surface was mounted in the jig of the testing machine with the PEEK surface parallel to the loading direction as seen in the figure.

## 2.2. Hyperbaric and hypobaric pressure exposures

The hyperbaric chamber used in this study was a custom-made device (Hipertech; Hipertech Electronic and Machine Industry Company, Istanbul, Turkey) that enabled electronic control of pressure changes. The pressure cycle regimen consisted of 20 pressure cycles ranging increasing from 0 to 2.8 atm at a rate of 0.5 atm/min, reaching the maximum pressure of 2.8 atm in ~6 min. After 60 min at 18 m (2.8 atm) deep, the decompression phase began at an ascent rate over a period of ~6 min. This process was repeated for 20 days.

The hypobaric chamber was a custom-made device (ETC; Southampton, PA, USA) that enabled electronic control of pressure changes. The hypobaric chamber was decompressed to 8,200 m (1/3 atm) in 5 min. After 60 min at 8,200 m altitude, the chamber was recompressed to the normal atmospheric pressure over a period of 5 min. This process was repeated for 20 days.

The control subgroups were stored at ambient pressure for 20 days.

## 2.3. Surface roughness measurement

For surface roughness measurement, one surface-treated PEEK specimen from each group was prepared with 96%

ethanol and air-dried, mounted on metallic stubs, sputter-coated with gold, and examined under a scanning electron microscope (SEM; QUANTA 400 F Field Emission SEM) at 1,000 $\times$  magnification. Atomic force microscopy (AFM, Veeco MultiMode V) was also used to analyze the topography of PEEK samples (one from each surface-treated group).

## 2.4. Shear bond strength measurement

The shear bond strength was measured with the Universal Testing Machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) at a crosshead speed of 0.5 mm/min. Specimens were put in the jig of the testing machine with the PEEK surface parallel to the loading direction at a 500 N load cell in the testing machine (Figure 2). The bond strength values were calculated by dividing the force at which bond failure occurred by the bonding area.

## 2.5. Failure mode analysis

The debonded surface of the specimens was observed under a light microscope (Olympus Corp., Tokyo, Japan) at 30 $\times$  magnification. Also, sample surfaces were examined with SEM (Leica MZ 12; Leica Microsystems, Bensheim, Germany) at 1,000 $\times$  magnification to assess the mode of failure. Type of failure identified as follows:

Type 1: Adhesive failure (<20% composite resin observed at the PEEK surface)

Type 2: Cohesive failure (more than 80% composite resin observed at the PEEK surface).

Type 3: Mixed failure (20–80% composite resin observed at the PEEK surface).

## 2.6. Statistical analysis

IBM SPSS Statistics 24.0 was used for statistical analysis. Shapiro Wilk's test was performed to evaluate the normality of data distribution. Differences between groups were tested using ANOVA followed by the *post-hoc* Tukey test. Values were expressed as mean  $\pm$  SD. The *p*-values for statistical significance were accepted <0.05.

Strength variations within each group were evaluated by calculating the Weibull modulus (*m*). A spreadsheet was used to rank the shear strength data in ascending order and appoint

a rank over the range 1–10; a line graph was then fitted through the points using the median rank regression method. Weibull modulus was then calculated by slope analysis.

### 3. Results

#### 3.1. Shear bond strength

The mean shear bond strength values, in decreasing order, were observed for acid-etched PEEK surfaces; in atmospheric pressure ( $17.68 \pm 1.20$ ), in the hypobaric group ( $15.84 \pm 1.10$ ), and the hyperbaric group ( $15.21 \pm 0.68$ ). This was followed by the sandblasted group: in atmospheric pressure ( $14.77 \pm 1.66$ ), in the hypobaric group ( $13.76 \pm 2.27$ ), and the hyperbaric group ( $12.70 \pm 0.95$ ). Non-treated PEEK surfaces provided the lowest mean shear bond strengths in

all environmental pressure changes. Significant differences were observed with acid-etched groups and sandblasted groups when comparing non-treated groups ( $p < 0.001$ ). However, no statistically significant differences were observed between hypobaric, and atmospheric pressure groups either with acid-etched or sandblasted ( $p > 0.05$ ). The mean shear bond strength values and standard deviations of each group are listed in Table 2 and statistical differences are shown in Figure 3.

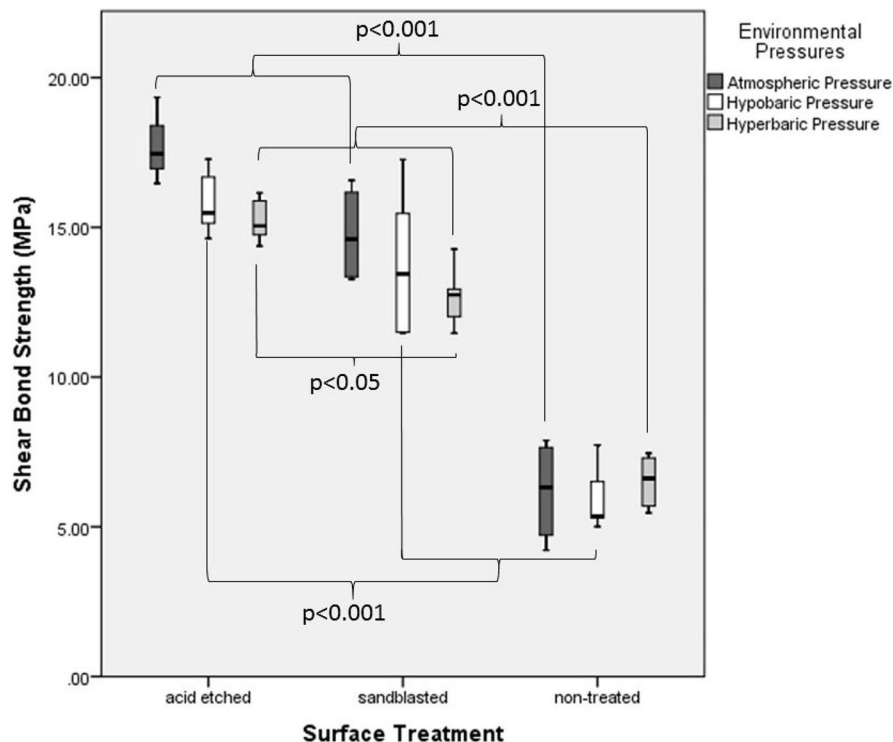
#### 3.2. Weibull modulus

The shear bond strength data of PEEK bonded to composite resin tested with different environmental pressures were further analyzed using the Weibull distribution function to predict the failure probability of the bond. The Weibull analysis

**Table 2.** Mean and standard deviation (SD) for shear bond strength values, min–max values of test groups, and the results of Weibull analysis and number of failure types.

Groups	Bond strength (MPa) (Mean $\pm$ SD)	Min	Max	Weibull modulus (m)	Weibull characteristic strength (MPa)	Failure analysis		
						Ad	Co	Mixed
1a	14.77 $\pm$ 1.66	13.27	16.58	8.23	15.57	10	–	–
2a	17.68 $\pm$ 1.20	16.48	19.34	13.87	18.27	9	–	1
3a	6.18 $\pm$ 1.75	4.22	7.88	3.31	6.92	10	–	–
1b	13.76 $\pm$ 2.27	11.48	17.26	6.16	14.77	10	–	–
2b	15.84 $\pm$ 1.10	14.63	17.28	14.37	16.36	10	–	–
3b	5.87 $\pm$ 1.05	5.01	7.73	5.40	6.37	10	–	–
1c	12.70 $\pm$ 0.95	11.47	14.27	13.62	13.14	10	–	–
2c	15.21 $\pm$ 0.68	14.37	16.15	22.78	15.53	10	–	–
3c	6.50 $\pm$ 0.91	5.46	7.46	7.04	6.92	10	–	–

Group 1a: sandblasted-atmospheric pressure; Group 1b: sandblasted-hypobaric condition; Group 1c: sandblasted-hyperbaric condition; Group 2a: acid etched-atmospheric pressure; Group 2b: acid etched-hypobaric condition; Group 2c: acid etched-hyperbaric condition; Group 3a: non-treated-atmospheric pressure; Group 3b: non-treated-hypobaric condition; Group 3c: non-treated-hyperbaric condition.



**Figure 3.** Boxplot graph of shear bond stress values. Significant differences were observed with acid-etched groups and sandblasted groups when comparing non-treated groups under all environmental pressure ( $p < 0.001$ ). There was a significant difference between the acid-etched group and sandblasted group under hyperbaric conditions ( $p = 0.003$ ).



for composite resin bonded to PEEK under different environmental pressures is shown in Table 2. Any kind of barometric pressure changes notwithstanding, the Weibull modulus was the highest for the acid-etched group. Weibull characteristic strength for the non-treated surface groups was lower than the other surface-treated groups. The Weibull analysis for composite resin bonded to PEEK under different environmental pressures is listed in Table 2.

### 3.3. Failure analysis

The failure analysis of each group is listed in Table 2. Almost all specimens exhibited adhesive failure. A representative microscopic image of failure type at a magnification of 30× in the light microscope is shown in Figure 4.

### 3.4. SEM and AFM

SEM and AFM demonstrated a tendency of increased surface roughness and irregularities of PEEK. SEM images at 1,000× magnification and AFM images are shown in Figures 5, 6. Surface roughness values (Ra) of the samples were as follows; non-treated: 65.1 nm; sandblasted: 87.3 nm; and acid-etched 83.4 nm. In the control group, there are irregular lines on the PEEK surface due to the polishing

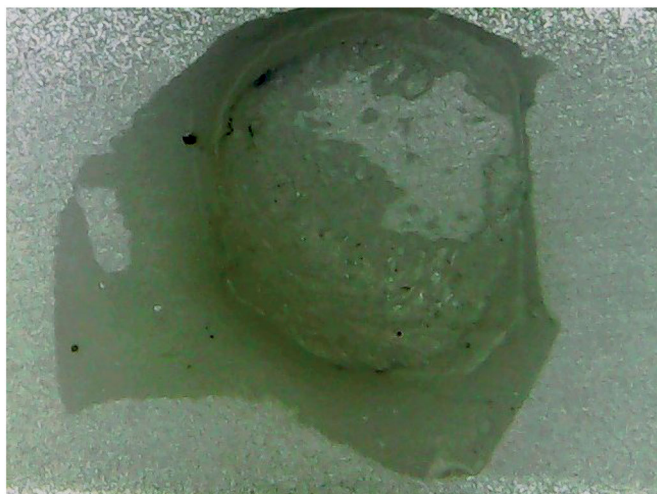


Figure 4. Representative light microscopy image (30×).

procedure. Irregular filler particles and small pits were observed on sample surfaces in the sandblasted and acid-etched groups. Pits and pores were larger in those etched with 98% sulfuric acid.

## 4. Discussion

To legitimize and validate the use of PEEK in dentistry, the bond strength between the composite resin and the PEEK is required to have strong adhesion. Our results showed that the environmental pressure changes and the surface roughness treatments of PEEK can affect the shear bond strength between PEEK and the composite resin. Therefore, the null hypothesis of the study was rejected.

In this study, higher bonding values were obtained in both acid-etched and sandblasted groups compared to the non-treated surface group. It is known that the surface roughness of a material is an important contributory factor for increasing the mechanical properties of the adhesive owing to increase surface contact<sup>[17]</sup>. Sandblasting and sulfuric acid etching treatment on PEEK surfaces create an active surface by removing the organic contaminant from the PEEK surface and provide micromechanical interlocking. It has been reported that sulfuric acid etching increases the concentration of the functional carbon-oxygen groups on the surface, allowing more functional elements to be available for the adhesive system<sup>[18,19]</sup>. Most of the previous studies concluded that sulfuric acid-etching improves the bond strength value of the PEEK material<sup>[9,10,19]</sup>. The results of this study are commensurate with previous studies that acid etching of PEEK surfaces provides stronger bond strength to resins. However, in this study, the surface roughened samples were more affected by the environmental pressure changes than the untreated group. The specific mechanism by which ambient pressure changes affect the bonded surfaces is unknown. The probable explanation for this result could be the possible air voids occurring inside the pits and pores of the PEEK surface that might affect the adhesion of resin. Untreated groups were less affected by pressure changes probably because of their flat regular surface.

In accordance with Boyle's law, the existing air space, expands or contracts due to the decrease or increase in

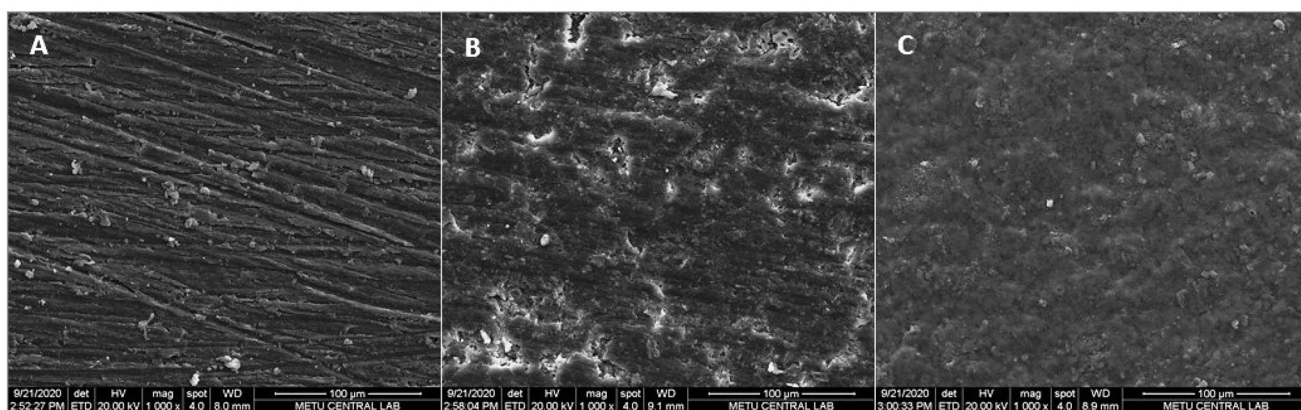
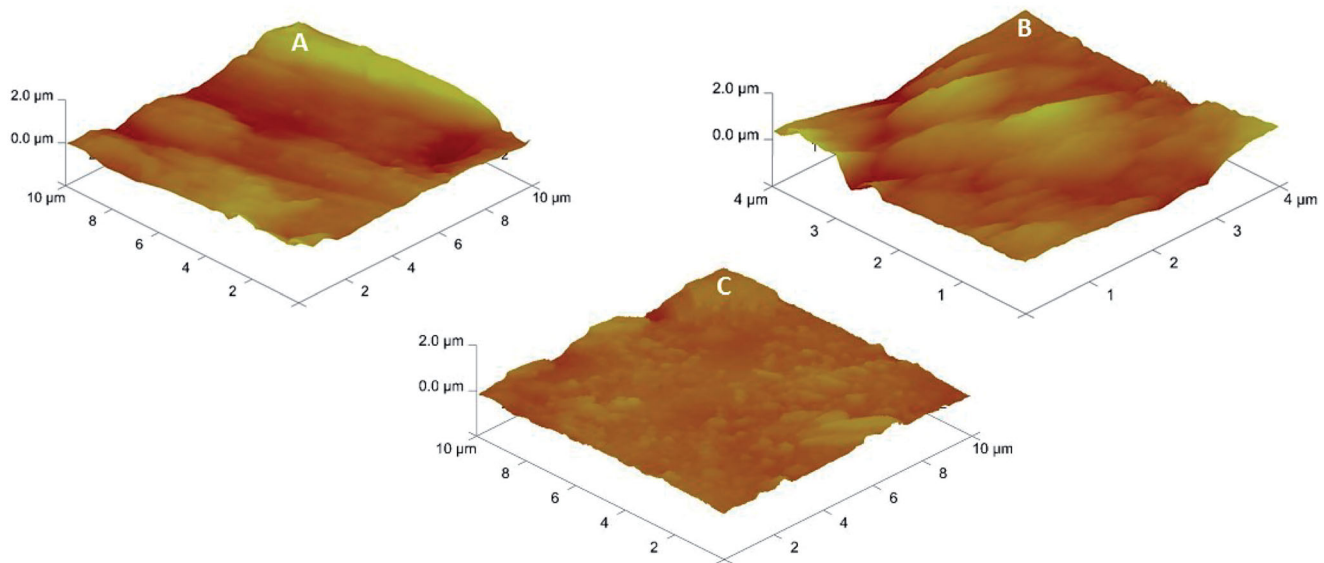


Figure 5. SEM micrographs of different surface treatment modalities: (A) non-treated; (B) sandblasted; (C) acid etched.



**Figure 6.** AFM images of the PEEK samples; (A) non-treated; (B) sandblasted; (C) acid etched.

pressure, weakens the structure of the dental restorations in divers and aircrew<sup>[20]</sup>. In the case of diving or flying, stress may occur in air-containing spaces, such as the pores in the resin layers, in bonding areas, or inside the dentinal or root canals. When returning to the sea level after diving or exposure to high altitude, the enclosed gas experiences expansion or contraction. The cumulative stress of compression expansion can produce fractures within the resin layer and/or along the interface surface<sup>[14]</sup>. This may explain the lower mean shear bond strength of the hypobaric and hyperbaric groups of this study.

The chemical structure and type of the adhesive systems and composite resins also affect the bond strength of PEEK materials. The non-pretreated PEEK surfaces are unable to adhere to the veneering composite resins<sup>[21,22]</sup>. Stawarczyk et al. reported Visiolink as an ideal adhesive bonding to improve the bond strength to PEEK surfaces<sup>[21]</sup>. It has been reported that Methyl methacrylate (MMA) and pentaerythritol triacrylate (PETIA) in Visiolink provide a reliable bonding dependently on the surface treatment<sup>[23]</sup>. In this study, Visiolink was used as a bonding agent.

In the present study, one type of composite resin was used for standardization. Composite resins consist of different components, such as photoinitiators, polymerization inhibitors, and organic monomers. The composition of resins can affect the bond strength. Bisphenol A-glycidyl methacrylate (Bis-GMA) is an organic monomer widely used in composite resins. It increases the viscosity of the material due to hydrogen bonding interactions between hydroxyl groups. A more fluid comonomer triethylene glycol dimethacrylate (TEGDMA) is added to the resin to solve the viscosity problem<sup>[24]</sup>. However, it was reported that TEGDMA reduces the mechanical properties of resin composites<sup>[24,25]</sup>. The composite resin used in this study has a high viscosity feature, which may have limited the filler loading capacity. The high viscosity property of the composite resin may prevent the filling of micro-pores and pits with resin. Possible air voids that occurred on the peek-resin bonded interface

may have caused microfractures during pressure changes. It was reported that flowable composite resin, vibration methods when applying composite or preheating composite could help limit the presence of air bubbles<sup>[26]</sup>. Further studies are needed to select the appropriate composite resin for veneering in individuals exposed to environmental pressure changes.

The bond strength decreased in both hypobaric and hyperbaric groups compared to atmospheric pressure groups in the current study. However, this reduction was not statistically significant. A study by Geramipناه et al compared the effect of different environmental pressures on the bond strength of fiber post to root canals<sup>[11]</sup>. They found a statistically significant decrease in the diver group. They concluded that rapid pressure changes in diving adversely affect the bond strength of dental restorations<sup>[11]</sup>. Shafigh et al. evaluated the fracture resistance and microleakage of amalgam and composite restorations under environmental pressure changes for 30 days<sup>[12]</sup>. They stated that the dive group had significantly lower fracture resistance values compared to the control group, whereas there was no significant difference between the control group and the flight group. However, in our study, there was no significant difference between hyperbaric and hypobaric groups. One of the important reasons for these contradictory results may be the bonding quality and performance of teeth. In both studies, the resins were bonded to dentin. Unfavorable histological factors, such as dentinal tubule densities, difficulty in moisture control may have affected their results. Additionally, the duration of exposure to pressure and the difference in materials, the variations in test conditions, cavity design, and dimensions may have all affected the results of our study.

The shear bond strength test was used to assess the bond strengths between PEEK and composite resin. Because this testing method has a simple application procedure<sup>[27]</sup>. It has been reported that micro-shear bond strength tests give more effective results in determining the bond strength of

smaller bonding areas<sup>[28]</sup>. However, Valandro et al. reported that results obtained for high-strength ceramics with the shear bond strength tests are similar to micro-shear bond strength test methods<sup>[29]</sup>. Therefore, the shear bond strength test was used in this study.

In our study, diving and flight conditions were simulated with hypobaric and hyperbaric chambers for 20 days, 1 h a day. The long-term effects of barometric pressure changes on dental health were examined in a 10-year study. Over this 10-year period, it was observed that personnel working in hyperbaric environments encountered more dental problems as compared to personnel working at ground level<sup>[30]</sup>. These findings pointed toward the fact that prolonged exposure to environmental pressure changes can cause a negative prognosis in oral dental health. As more harmful effects can be expected during longer periods of cyclic pressure changes<sup>[30]</sup>, this may be the limitation of our study as longer duration periods could change the effects of pressure changes and their consequences.

Weibull analysis was included in this study as it provides information about the variability of results. A high value of Weibull modulus indicates a close grouping of fracture stress values implying that material is more dependable. Regardless of the barometric changes, the Weibull modulus was the highest for the acid-etched group. According to the results of Weibull characteristics, it was observed that the characteristics values were low in non-treated groups. The low value of Weibull characteristics reflects the low reliability of the material. The low values of the Weibull modulus indicate that the material may weaken in the future and may experience unexpected failures. This failure most often manifests itself in barodontalgia. This puts flight or diving safety at risk. These results are consistent with shear bond stress results. Weibull distribution has shown to be an alternative method for the evaluation of the fracture probability of materials.

Analysis of the failure mode can help to explain bond strength results. All subgroup specimens revealed bond failures occurring at the PEEK interface. Moreover, there were almost no remnants of composite resins were observed on the surfaces of PEEK, suggesting a probable lack of missing adherence to PEEK surfaces, even though they were surface modified.

In conclusion, although the use of PEEK in dentistry is increasing day by day, its use in individuals exposed to hypobaric and hyperbaric pressure changes should be further investigated. Although, micromechanical locking from penetration of resins along pits and pores seemed to be an important factor in promoting adhesion between PEEK and resin materials, the effect of the environmental pressure changes on the adhesion of PEEK to resins are not known.


This is a pioneer study and further studies with different environmental pressure cycles, longer durations, different composite resins, and surface treatments and mechanical tests are needed to fully understand the effect of ambient pressure changes on PEEK material used in dentistry. Care should be taken in the selection of materials in aircrew and

divers, where fractures and cracks in dental restorations may cause life-threatening problems.

## Disclosure statement

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

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