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Effect of the combination of chemical-physical pretreatments for bonding PEEK to resin composites

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SUMMARY

Objective. Surface pretreatment of polyetheretherketone (PEEK) is necessary to increase the bond strength between PEEK and veneering materials. The objective of this in vitro study was to evaluate the effect of a combination of chemical and physical pretreatments on the surface of PEEK for the adhesion of resin composite.

Materials and methods. Five pretreatments were evaluated: grit-blasting (Al_2O_3), grit-blasting (Al_2O_3)+primer adhesive, 98% sulfuric acid, 98% sulfuric acid+primer adhesive, and an untreated control. The PEEK surface was evaluated with a scanning electron microscope, and the tensile bond strength (TBS) of the PEEK with the veneering resin composite interface was measured with a universal test machine at a crosshead speed of 5 mm/min. The failure was categorized as adhesive, cohesive or mixed.

Results. Pretreatment with grit-blasting+primer adhesive obtained the highest mean ± standard deviation TBS value (19.10±1.70 MPa) followed by 98% sulfuric acid+primer adhesive (17.90±1.20 MPa), while the pretreatments with grit-blasting (13.97±2.33 MPa) and 98% sulfuric acid (12.18±0.70 MPa) showed the lowest values. All pretreatments showed statistically significant differences with respect to the untreated PEEK control and the combined pretreatments with respect to the pretreatment with grit-blasting and 98% sulfuric acid ($p \leq 0.05$). A higher number of adhesive failures were observed except in the grit-blasting+primer adhesive group, which had 80% mixed failures. No cohesive failures were seen.

Conclusion. The combination of chemical-physical pretreatments induced an increased in the TBS values between PEEK and resin composite because of the topographical roughness and the micro-mechanical anchoring in the PEEK surface.

Keywords: PEEK, surface, bond strength, physical-chemical pretreatments, resin composite.

INTRODUCTION

Polyetheretherketone (PEEK) is an aromatic linear semicrystalline thermoplastic polymer with biological and mechanical properties and is extensively used for different biomedical applications. It was patented in 1981 as a material for implantation in the human body and accepted in 1990 by the United States Food and Drug Administration. Advantages of PEEK include maintaining its mechanical properties at high temperatures, a modulus of elasticity similar to that of bone, and a low coefficient of friction, and has been reported to maintain its physical and chemical integrity after being implanted (1-4).

In dentistry, PEEK has been used in the fabrication of dental implants, fixed dental prostheses, abutments, and implant-supported frameworks and has been used in patients with metal allergies or who decline metal prostheses (5, 6). As PEEK has a greyish color and low translucency, it is necessary to veneer its surface for acceptable esthetics. Resin composites have been typically used as the veneering material to replicate the appearance of natural tooth structure (7-9). However, the hydrophobic surface, the resistance to surface modification, and the low surface energy of PEEK make bonding to resin composites challenging (10).

Surface modifications are necessary to enhance the bond strength between resin composites, adhesives,

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and cements with PEEK. Dental laboratories have used plasma, ultraviolet and ozone, grit-blasting, acidic etching, and radiation as pretreatments. These pretreatments have been reported to increase surface functional groups, to promote morphological and chemical changes on the PEEK surface, and to improve adhesion and increase the bond strength values (7, 8).

According to the requirements of ISO 10477, the accepted minimum value at the interface of resin composite and substrate is 5 MPa (11). Values greater than 10 MPa between resin composite and PEEK have been reported to be clinically acceptable, and values without any pretreatment are reported to be from 0 to 12 MPa (12-16). However, these values may vary according to the chemical composition of the PEEK and the type of resin composite applied (17-19).

In clinical dental practice, the bonding of PEEK to resin composites continues to be a problem. A generalized established protocol for pretreatments for surface modification to improve the bond strength of PEEK to resin composite is lacking, and the mechanism of action of the bonding is still unclear. Nevertheless, the combination of micro-retention and chemical bonding is a pre-treatment that has been reported to provide a strong and durable bond in dental restorations (9, 15, 19, 20). However, evidence on the combination of chemical and physical pretreatments on the surface of PEEK is sparse, and information on the interaction of PEEK to resin composites that can improve the bond strength values between PEEK and veneering materials is lacking. Therefore, the objective of this *in vitro* study was to evaluate the micromorphology of the surface of PEEK and the bond strength of PEEK to resin composite after different combination surface pretreatments. The null hypothesis was that no difference would be found in the micromorphology and bond strength of PEEK to resin composite after different combined chemical and physical surface pretreatments.

MATERIALS AND METHODS

Specimen preparation and surface treatment

The compositions and details of the materials used in this study are shown in Table 1. A total of 125 circular-shaped PEEK specimens were fabricated (Ø 4.0 mm and 2.0 mm in thickness, BioHPP, Bredent; Senden, Germany) and were randomly divided into five test groups (n=25) according to the surface pretreatment (Table 2). The PEEK specimens were embedded in an autopolymerizing acrylic resin to facilitate manipulation and were polished with 400-, 800-, 1200-, and 2000-grit rotating silicon carbide paper for 1 min under running water, cleaned in an ultrasonic bath with 80% ethanol followed by distilled water for 10 min, and air-dried before surface pretreatments at room temperature (RT).

Scanning electron microscopy

Randomly selected specimens (n=5) from each group were examined with a scanning electron microscope (SEM) (JEOL, JSM-6510, Tokio, Japan) to evaluate the micromorphology of PEEK surfaces after surface pretreatments. The specimens were first gold sputter-coated (15-20 nm) and observed at 170× and 2000× magnification and a working distance of 10.0 mm and 15.0 kV.

Bonding procedure

To evaluate the tensile bond strength (TBS) (n=20) a resin matrix-composite block measuring 4 mm in thickness (Filtek Z350 XT: 3M ESPE, St. Paul, MN, USA) was built onto the PEEK surfaces of the specimens in 2-mm increments. The resin composite was light polymerized for 20 s at 800 mW/cm^2 (Kulzer Heraflash; Indiana, USA). The PEEK discs with the resin block were embedded in acrylic resin inside a metal base to be mounted on the universal testing machine clamp (Shimadzu AG-X; Maryland, USA), and the load was applied at a crosshead speed of 5 mm/min. The specimens were positioned parallel to the loading direction in the jig of the testing universal machine with the PEEK surface, and the TBS was calculated with the following formule: fracture load/bonding area (MPa=N/cm^2). For fracture type analyses, the debonded area was examined by one calibrated and blinded examiner using a stereomicroscope. The type of failure was classified into adhesive failure mode between PEEK and resin matrix-composite (1), cohesive failure mode within PEEK (2), and mixed failure mode with both cohesive and adhesive failures (3, 18). Photographs were

Table 1. List of materials used and their characteristics

Material	Manufacturer	Composition
Filtek Z350 XT	3M ESPE, St. Paul, MN, USA.	Bis-EMA, UDMA and TEGDMA with nanoparticles of non-agglomerated silica and zirconia/silica nanoclusters (59.5% vol%).
Williams Blasting Compounds	Ivoclar Vivadent, USA.	Aluminum trioxide poder 50 micron.
BioHPP® PEEK	Bredent, Senden, Germany.	Ceramic filled (20%) PEEK.
SR Connect	Ivoclar Vivadent, USA.	Methyl methacrylate (60–70%), poly(methyl methacrylate) (<10%), dimethacrylate (20–30%) and catalysts (3–5%).
Sulfuric acid	RCI Labscan, Samutsakorn, Thailand.	98% Sulfuric acid.

made to observe the PEEK surface and determine the type of failure mode.

Statistical analysis

The data were analyzed by using a statistical software program (Sigma Plot). Two-way repeated measures ANOVA and the t test statistical analyses were used to determine statistically significant differences in the means of the study variable among the study groups (α=0.05).

RESULTS

Surface morphology analysis

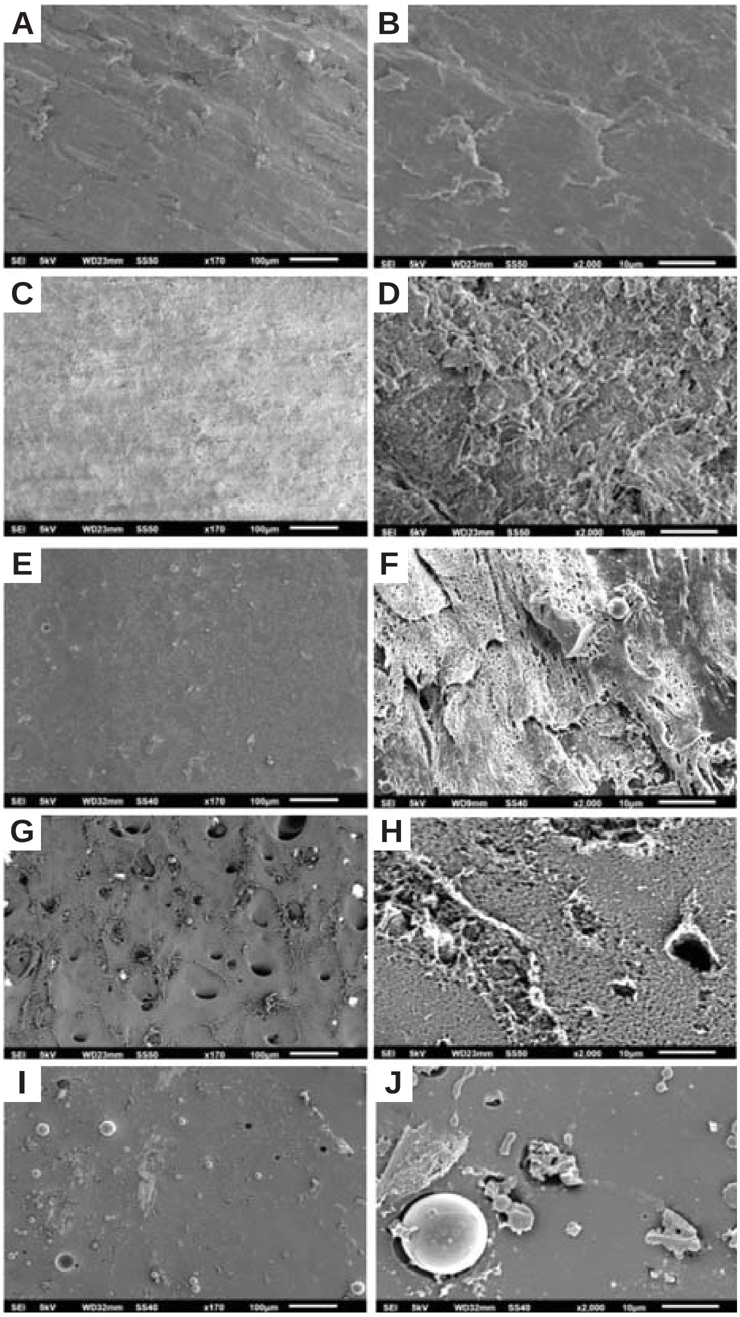

Fig. 1. SEM images of the PEEK surface topography after surface pretreatment at low magnification 170× and high magnification 2000×. Untreated surface (A, B), grit-blasting (C, D), grit-blasting+primer adhesive (E, F), 98% sulfuric acid (G, H) and 98% sulfuric acid+primer adhesive (I, J)

The characterization of the PEEK surfaces after the different surface pretreatments are shown in the SEM images at 170× and 2,000× magnification in Figure 1. A homogeneous and smooth surface without depressions and with minimal irregularities was observed on the untreated PEEK surface (Figure 1 A, B). The Al_2O_3 grit-blasting PEEK surface showed microroughness and porosity of different dimensions on most of the surface (Figure 1 C, D), while the combination of Al_2O_3 grit-blasting+primer adhesive showed an increased roughness with a porous, irregularly structured surface with deep and narrow pits (Figure 1 E, F). The pretreatment with 98% sulfuric acid resulted in a PEEK surface with localized pores of different dimensions and depth (Figure 1 G). At higher magnification, a porosity pattern was observed around the larger pores formed by the corrosion of the acid on the surface and large pits and round cavities were seen (Figure 1 H). Pretreatment with 98% sulfuric acid+primer adhesive caused irregularities, with localized depressions on the PEEK surface (Figure 1 I, J).

Tensile bond strength

The mean and standard deviations of bonding to PEEK with different surface pretreatments are shown in Table 3. Figure 2A shows the PEEK specimen with the resin block bonded to it and mounted

Table 2. Surface pretreatment protocols evaluated

Treatment group	Description
Untreated surface	No surface pretreatment was applied.
Grit-blasting	The surfaces of the PEEK were abraded with 50-µm Al_2O_3 particles for 10 s at a pressure of 2 bar at a working distance of 10 mm perpendicular to the treated surface for 10 s.
Grit-blasting-particle abrasion+primer adhesive	The surfaces of the PEEK were abraded with 50-µm Al_2O_3 particles for 10 s at a pressure of 2 bar at a working distance of 10 mm for 10 s + primer adhesive, the adhesive was used according to manufacturer's instructions and light polymerized at 800 mW/cm^2 for 90 s.
98% sulfuric acid	Etched with 98% sulfuric acid 1 min and then rinsed with deionized water for 1 min.
98% sulfuric acid+primer adhesive	Etched with 98% sulfuric acid 1 min and then rinsed with deionized water for 1 min, ultrasonically cleaned for 10 min + primer adhesive, the adhesive was used according to manufacturer's instructions and was and light polymerized at 800 mW/cm^2 for 90 s.

in the universal testing equipment; once the force was applied, the resin block detached (Figure 2B). The mean values of the surface pretreatments were from highest to lowest as follows: grit-blasting+primer adhesive, 98% sulfuric acid+primer adhesive, grit-blasting, 98% sulfuric acid, and untreated surface. Table 3 shows the values and the significant difference between the surface pretreatments. All surface pretreatments evaluated showed significant difference versus the untreated control group ($p<0.05$), and the combined pretreatments with grit-blasting+primer adhesive and 98% sulfuric acid+primer adhesive showed statistically significant difference versus the individual treatments of grit-blasting and 98 % sulfuric acid.

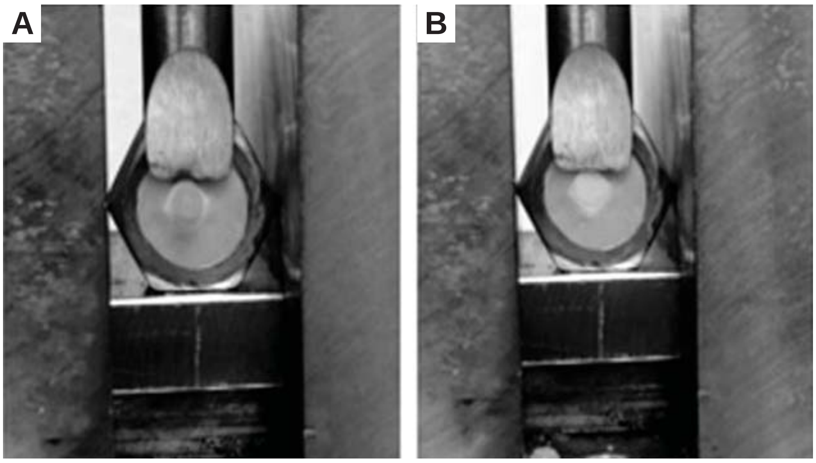

Fig. 2. Test mechanism before the test (A) and after the test without the resin composite block (B)

The failure mode analysis showed no cohesive failures, only adhesive and mixed failures in the 4 groups of surface pretreatments (Table 3). In the untreated surface (Figure 3A) and 98% sulfuric acid groups (Figure 3B), 100% of failures were adhesive. The pretreatments with grit-blasting (Figure 3C) and 98% sulfuric acid+primer adhesive showed 60% adhesive failures and 40% mixed failures (Figure 3D), and the PEEK specimens with grit-blasting+adhesives surface pretreatment, which had the highest TBS values, had 20% adhesive failures and 80% mixed failures.

DISCUSSION

Surface pretreatments on PEEK have been used to improve adhesion with restorative materials; however, each pretreatment has a different action on the surface according to its chemical and physical mechanism (21-26). This *in vitro* study evaluated the effects of combined chemical and physical pretreatments on the topography and the TBS of PEEK bonded to resin composite. The results obtained showed that the combination of 98% sulfuric acid+primer adhesive and grit-blasting+primer adhesive surface pretreatments had the highest TBS values between the surface of PEEK and resin composite, possibly because of the increase of roughness and the interaction with the adhesive. Therefore, this study suggests using chemical and physical pretreatments on the PEEK surface to increase microretentive areas to improve the TBS between PEEK and resin composite. Therefore, the null hypothesis that no difference would be found in the surface micromorphology and TBS of PEEK to resin composite after different combined chemical and physical surface pretreatments was rejected.

Our results were consistent with those of other studies reporting that increased PEEK surface roughness increased micromechanical anchorage, resulting in increased bond strength (27,28). In the present study, PEEK surfaces treated with grit-blasting (Al_2O_3)+primer adhesive showed the highest surface roughness, followed by pretreatment with 98% sulfuric acid+primer adhesive. Therefore, the combination of chemical and physical pretreatments increased the TBS between PEEK and resin composite, while pretreatments with only grit-blasting and 98% sulfuric acid showed lower TBS values.

Different parameters have been reported to be key factors in each of the surface pretreatments on PEEK, for grit-blasting, the particle size, application time, working distance, and pressure influence, so these working parameters should be controlled. The application of primer adhesive on the PEEK surface has been reported to increase surface wettability, improving the bonding between the inert PEEK surface and resin composite. Wettability is related to the content and solvents of the adhesive system, since the primer adhesive creates a chemical interaction between the surface of PEEK and resin composite (10). In the present study, combined surface pretreatments were tested with an adhesive to evaluate whether the synergy increased the bond strength. The adhesive (SR Connect; Ivoclar Vivadent) contains methyl methacrylate

Table 3. Mean and standard deviation (SD) for values of test groups (MPa). Failure mode in different pretreatment methods. Different lowercase letters represent statistical differences between the control group and the different pretreatment methods (column). P<.05 was accepted as significance level

Treatment group	Mean values (MPa) ±SD	Adhesive %	Mixed (adhesive-cohesive) %
Untreated surface	7.46±0.91	100%	0
98% sulfuric acid	12.18±0.70	100%	0
Grit-blasting	13.97±2.33	60%	40%
98% sulfuric acid+primer adhesive	17.90±1.20	60%	40%
Grit-blasting+primer adhesive	19.10±1.70	20%	80%

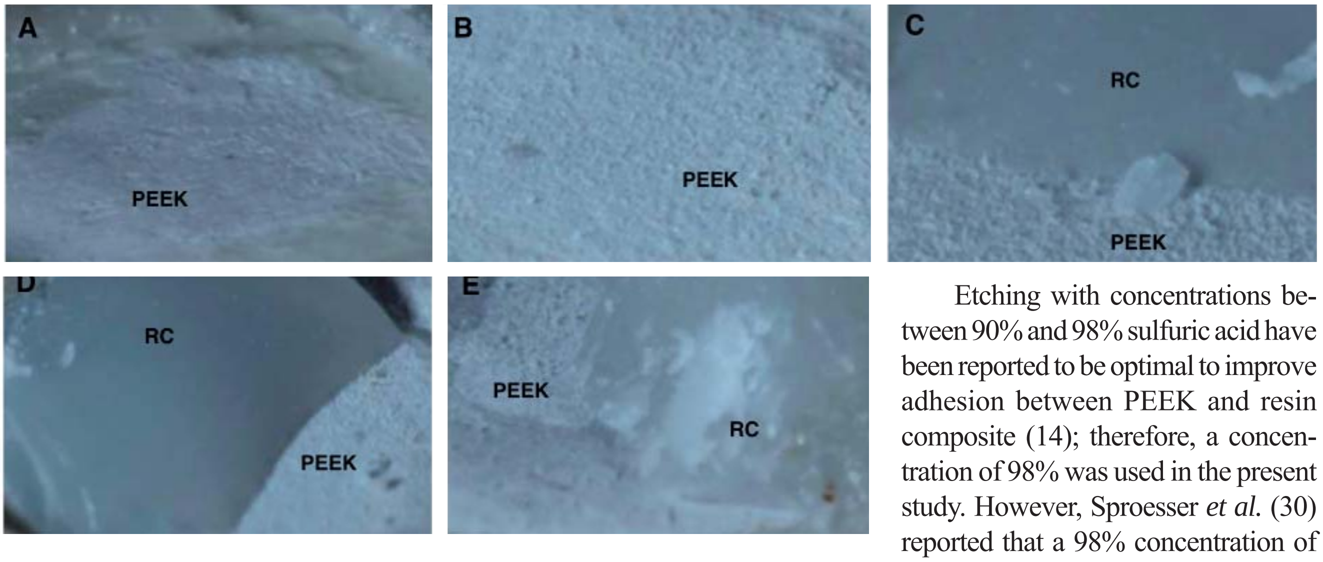

Fig. 3. Images of the adhesive failure type mode on PEEK bonding surface. In the untreated surface (A) and in the surface pretreatment with 98% sulfuric acid (B), the failures were adhesive, and in the PEEK surface with the pretreatments with grit-blasting 98% (C), sulfuric acid+primer adhesive (D), and grit-blasting+primer adhesive (E) were 40%, 40%, and 80% respectively of mixed failures (adhesive-cohesive). RC: Resin composite.

(MMA)-based liquid and is applied extraorally. As MMA monomers have been reported to increase the bond strength between PEEK and resin composites, the chemical components of the primer adhesive may influence the adhesion of the PEEK and the resin composite and may play a central role in creating a chemical bond between the different polymers (5, 9, 10, 17).

Our results were consistent with those of Caglar *et al.* (10) who reported that applying an adhesive system with MMA after the grit-blasting improved the resin bonding to PEEK. Çulhaoğlu *et al.* (15) reported that grit-blasting, laser irradiation, and silicoating procedures led to similar mean shear bond strength values and that the highest mean values were observed for acid-etched PEEK surfaces. Also, Schmidlin *et al.* (29) reported that the use of a hydrophobic dental bonding agent created an adequate adhesion to PEEK. The TBS values in the present study were higher for grit-blasting, 98% sulfuric acid, and the SR Connect adhesive, and the lowest TBS values were obtained when the pretreatments were applied individually.

A second pretreatment to obtain high TBS values in the present study was the combination of 98% sulfuric acid+primer adhesive. Pretreating PEEK with 98% sulfuric acid has been shown to increase porosity and surface permeability, as sulfuric acid attacks the functional ether and carboxyl groups between the benzene rings (21-23). The combination of 98% sulfuric acid+primer adhesive created sulfonate groups in the polymer chains of PEEK which chemically cross-linked to MMA dental adhesives (27, 28). However, the use of 98% sulfuric acid is contraindicated clinically because of its extremely corrosive nature, requiring appropriate use and manipulation.

Etching with concentrations between 90% and 98% sulfuric acid have been reported to be optimal to improve adhesion between PEEK and resin composite (14); therefore, a concentration of 98% was used in the present study. However, Sproesser *et al.* (30) reported that a 98% concentration of sulfuric acid may negatively affect the penetration of adhesives and resulted in weak points of bond interfaces, proposing lower sulfuric acid concentrations. Pisaisit *et al.* (14) evaluated the microtopographical changes of PEEK surfaces after sulfuric acid etching, demonstrating that the use of sulfuric acid enhanced the penetration of the resin adhesive. The present results showed that the surface of PEEK after pretreatment with 98% sulfuric acid+primer adhesive had increased roughness because of the dissolution of the PEEK matrix by a sulfonation reaction. A possible explanation for the increased TBS values following PEEK etching is because the larger surface area has more sulfonic groups which can be penetrated by the MMA-containing adhesive, improving bond strength (27, 28).

Future studies testing new surface pretreatments are necessary to improve the interaction between PEEK and veneering materials and under thermo-mechanical loading conditions. The lack of standardization of pretreatment parameters has prevented a generalized protocol for increasing the adhesion values of the PEEK surface. Limitations of the present study included the in vitro design that did not fully simulate clinical conditions. In vivo studies are necessary for clinical assessment and for investigating the long-term outcome of veneering resins bonded to PEEK.

CONCLUSION

The combination of physical and chemical pretreatments on the PEEK surface with grit-blasting+primer adhesive system and 98% sulfuric acid+primer adhesive system increased the surface microroughness of PEEK and the TBS values between the PEEK and resin composite.

STATEMENT OF CONFLICTS OF INTEREST

The authors state no conflict of interest.

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