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Original Article

Comparison of two adhesive systems of various polyetheretherketone (PEEK) composites on the shear bond strength

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Abstract

Aim: In this in vitro study, the effect of two adhesive systems applied to surfaces of different polyether-ether-ketone (PEEK) composites on the shear bond strength (SBS) of a composite resin was compared.
Methodology: Eighty PEEK specimens were divided into four groups (n=20): Unfilled PEEK (UF), carbon-fiber-reinforced PEEK (CFR), glass-fiber-reinforced PEEK (GFR), and ceramic-reinforced PEEK (CR). Each group was further divided into two subgroups (n=10): Visio.link (VL) and Single Bond Universal (SB). The specimens with 8-mm diameter and 5-mm thickness were prepared. SBS was examined using a universal testing machine. Results were statistically analyzed by multivariate analysis of variance and Tukey's post-hoc test. Failure modes were analyzed using a stereomicroscope at 20× magnification. Surface properties were examined by scanning electron microscopy. The surface properties of the specimens were examined by scanning electron microscopy (SEM).
Results: Effect of different PEEK and adhesive systems on SBS was found to be statistically significant ($p<0.05$). SBS values for CFR-VL and UF-VL groups were statistically more significant than those for CFR-SB and UF-SB groups. SBS values for the GFR-VL group were statistically more significant than that for the UF-VL group. SBS values for the CF-SB group were statistically more significant than those for CFR-SB and CFR-SB groups. Failure modes were examined using a stereomicroscope at 20× magnification, and adhesive and mixed failure modes were observed.
Conclusion: PEEK composites with different contents and properties can be used in fixed prosthetic restorations. However, additional experimental and clinical studies are required to investigate different PEEK frameworks and composite veneers.

Keywords: adhesion, PEEK, PEEK composites, Polyetheretherketone, shear bond strength, veneering

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Introduction

Polyether-ether-ketone (PEEK) is a semi-crystalline thermoplastic composite polymer comprising repeating monomers of two ethers and one ketone. Owing to its interesting properties such as biocompatibility, low plaque affinity, lightweight nature, high strength, and wear and fatigue resistance, PEEK has been attracting increasing attention in dentistry. PEEK materials exhibit physical, chemical, and mechanical properties that can be easily modified by using fillers, such as carbon fiber, glass fiber, and ceramics (1, 2). The elastic modulus of unfilled PEEK (UF-PEEK) is ~4 GPa. Carbon-fiber-reinforced PEEK (CFR-PEEK) and glass-fiber-reinforced PEEK (GFR-PEEK) composites can be modified by the addition of various fiber fillers, thereby affording an elastic modulus similar to those of bone and dental hard tissues. Therefore, stress-related problems can be reduced as the elastic modulus of the material is similar to that of oral biological tissues (1, 3, 4). In addition, when PEEK composites are used with composite resin materials, which exhibit more similar mechanical properties (e.g., toughness modulus and energy dissipation capacity), the composites can compensate for the absence of the natural periodontal damping effect in implant-supported restorations (5). Although the fracture strength of GFR-PEEK is less than that of CFR-PEEK, it is suitable for use as a fixed dental restoration. GFR-PEEK is a mechanically and clinically acceptable material, but it has not been used thus far for prosthetic restorations (6). Ceramic-reinforced PEEK (CR-PEEK) is used in fixed and removable prosthodontics for several functions, such as abutment, framework, primary crown, temporary denture, and inlay (2, 7). In addition, PEEK, which can be produced by injection or computer-aided design and computer-aided manufacturing (CAD-CAM) methods, can be used for different clinical scenarios (2).

Owing to the poor optical properties of PEEK, such as low translucency and color, the use of PEEK as full-coverage monolithic restorations is limited. Hence, PEEK requires conventional or CAD-CAM milled composite veneers with additional aesthetic materials, such as composite resins, to meet aesthetic expectations (7). Although veneering can be applied to PEEK frameworks with different ceramics, such as lithium disilicate, composite veneers provide facile application and repair (8, 9). The wettability of a solid surface such as PEEK is directly related to the surface chemistry and morphology. Owing to the chemical inertness of PEEK and its low surface energy, problems associated with bonding to the composite resin may arise. This disadvantage has been improved by different chemical or mechanical surface modification methods, such as airborne-particle abrasion, laser and plasma applications, and etching the PEEK surface with sulfuric acid (H_2SO_4) or a piranha solution. Several studies have reported the chemical or mechanical surface modification of PEEK (10-22). In chemical processes, besides etching with H_2SO_4 and a piranha solution, adhesive systems of different manufacturers also are applied (10-12). Airborne-particle abrasion strongly affects the hydrophobic behaviour of PEEK and its composites via the change in its surface morphology (13). Several studies have reported successful results for the combination of mechanical (such as airborne-particle abrasion) and chemical (such as adhesives) surface treatments to improve the wettability of PEEK and its bonding strength to composite resins (10, 12, 14-20, 22). In several studies, the bonding of UF-PEEK, CR-PEEK, or TiO_2-containing PEEK composites to the composite resin was evaluated in terms of shear bond strength (SBS) (13, 15-18, 20, 22). However, a limited number of studies have compared the bonding strengths of the UF-PEEK, GFR-PEEK, CFR-PEEK, and CR-PEEK composites to the composite resin in terms of SBS. Also, long-term studies related to the successful surface modification of the composite resin in terms of the bonding properties of PEEK composites are required.

In this in vitro study, the objective was to compare the effects of two adhesive systems applied to surfaces of different PEEK composites on the SBS of a veneering composite resin. The null hypothesis was that the types of PEEK composites and adhesive system would not increase the bonding strength between PEEK and the veneering composite resin.

Materials and Methods

Eighty PEEK specimens were divided into four groups (n=20): Unfilled PEEK (UF, Tecapeek MT Natural, Ensinger Gmbh, Nufringen, Germany), carbon-fiber-reinforced PEEK (CFR, Tecapeek MT CF30 Black, Ensinger Gmbh, Nufringen, Germany), glass-fiber-reinforced PEEK (GFR, Tecapeek GF30 Natural, Ensinger Gmbh, Nufringen, Germany), and ceramic-reinforced PEEK (CR, BioHPP, Bredent, Senden, Germany). Each group was further divided into two subgroups (n=10): VL (Visio.link, Bredent GmbH & Co KG, Senden, Germany) and SB (Single Bond Universal, 3M ESPE, Deutschland GmbH, Neuss, Germany). Table 1 lists the details about the materials and their manufacturers. The UF, CFR, and GFR specimens were cut into dimensions of 8 mm in diameter × 5 mm in thickness from cylinder rods with a diameter of 8 mm at 400 rpm under water cooling by using a precision cutting device (Minitom, Struers A/S, Ballerup, Denmark). They were prepared by considering the thickness of the cutting disc. The CR specimens were prepared using a CAD/CAM device (Redon Hybrid, Istanbul, Turkey) from 98.5×24 mm prefabricated discs (Brecam BioHPP, Bredent, Senden, Germany) with a diameter of 8 mm and a thickness of 5 mm. Next, the specimens were embedded in a self-cure acrylic resin (Procryla; President Dental, Munich, Germany) and polished with 600-, 800- and 1200-grit silicon carbide (SiC) paper under running water. To modify the PEEK surface, the specimens were subjected to airborne-particle abrasion with 50-µm Al_2O_3 at a pressure of 2 bar and at a distance of 10 mm for 20 s. After surface conditioning, the specimens were cleaned using an ultrasonic cleaner (Digital Dental Ultrasonic CD-4820, Shenzhen Codyson, Guangdong, China) with deionized water for 10 min and dried in air. Both adhesive systems were applied and polymerized according to the manufacturer's instructions. The veneering composite (Gradia, GC, Tokyo, Japan) resin was manually applied in plexiglass tubes (inner diameter of 5 mm and a thickness of 4 mm) and polymerized. After polymerization, the specimens were stored at 37°C in deionized water for 24 h and aged by thermocycling with 5000 cycles at 5°C and 55°C in high-purity water for 20 s each with 10 s between baths for thermal stabilization (Thermocycler THE 1100; SD Mechatronik Feldkirchen-Westerham, Germany). Compressive load was measured by using a universal testing machine (MTS Criterion Model 42, MTS, MN, USA) at a crosshead speed of 0.5 mm/min. A compressive load was applied to the PEEK-resin interface by using a mono-bevelled chisel-shaped steel tip (Fig. 1).

Table 1. Product names, manufacturers, composition properties of test materials, and procedures used in the present study

Code	Materials	Product names and Manufacturers	Compositions	Applications
UF	Unfilled PEEK	Tecapeek MT Natural, Ensinger Gmbh	Unfilled Polyetheretherketone	50 µm Al_2O_3, 2 bar
CFR	Carbon fiber-reinforced PEEK	Tecapeek MT CF30 Black, Ensinger Gmbh	30% Carbon fiber-reinforced Polyetheretherketone	50 µm Al_2O_3, 2 bar
GFR	Glass fiber-reinforced PEEK	Tecapeek GF30 Natural, Ensinger Gmbh	30% Glass fiber-reinforced Polyetheretherketone	50 µm Al_2O_3, 2 bar
CR	Ceramic-reinforced PEEK	BioHPP, Bredent	20% Ceramic-reinforced Polyetheretherketone	50 µm Al_2O_3, 2 bar
VL	Visio.link	Bredent GmbH & Co KG	MMA, PETIA, dimethacrylates, photoinitiators	1) Apply a thin layer 2) Light cure 90 s. (bre.Lux PowerUnit, intensity 220 mW/cm^2, bredent, Senden, Germany)
SB	Single Bond Universal	3M ESPE, Deutschland GmbH	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane	1) Apply a thin layer by rubbing for 20 s 2) Gentle air stream for 5 s 3) Light cure 10 s. (Elipar Freelight 2, 1200 mW/cm^2,3M ESPE, Seefeld, Germany)
	Veneering composite resin	Gradia, GC	UDMA, inorganic-organic composite filler, silica nano powder, glass powder	1) Apply with the layering technique 2) Light cure 360 s. in a laboratory curing division (bre.Lux PowerUnit, intensity 220 mW/cm^2, bredent, Senden, Germany)

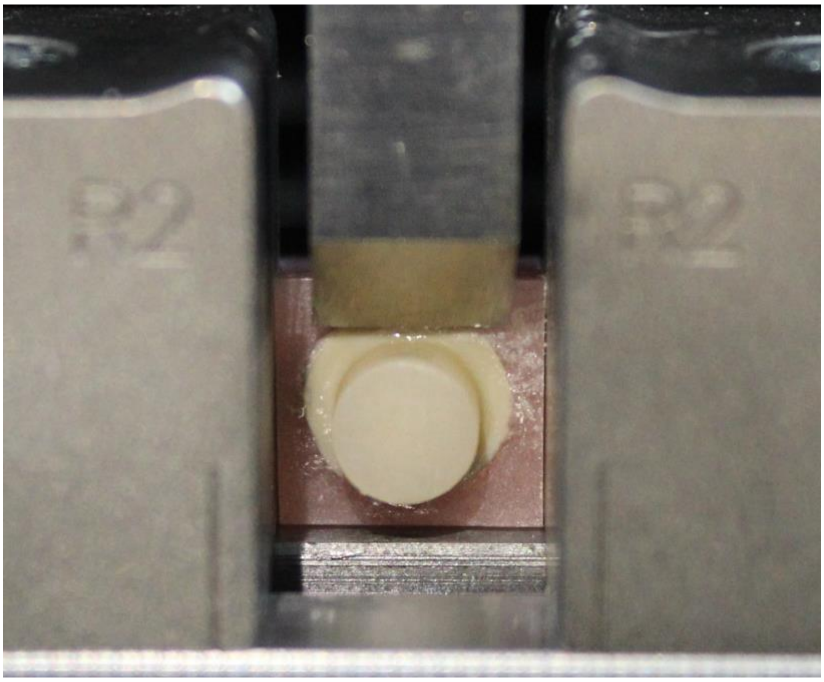

Figure 1. Specimen prepared for SBS test placed on the testing machine.

SBS values were calculated according to the following equation: s = F/A (s: shear bond strength [MPa], F: peak load at failure divided by the specimen surface area [N], A: bonded area [mm^2]). Failure modes were analyzed by using a stereomicroscope at 20× magnification (ZeissStemi 508, Carl Zeiss Microscope, Göttingen, Germany) and were classified into adhesive (between the PEEK framework and veneering composite resin), cohesive (in the veneering composite resin), and mixed (adhesive and cohesive failure modes occurring simultaneously) (15,16).

The surface properties of the specimens were examined by SEM at 10–1000×magnification at 20 keV (Hitachi Regulus 8200, Tokyo, Japan). Before analysis, non-conductive specimens were coated with gold using a device for insulating material coatings (Quorum SC7620, QuorumTech, East Sussex, UK) to make it suitable for imaging.

Statistical analysis

Data were evaluated using a statistical software program (IBM SPSS Statistics, v20.0; Armonk, NY, USA). The MANOVA test was performed to evaluate effects of the material and adhesive on the SBS for each specimen group. Before the MANOVA test, the normality analysis of the data was performed using the Kolmogorov-Smirnov distribution test, and the data were found to exhibit a normal distribution. The independent variable t-test was performed to compare the mean SBS values of different adhesives in the same material. Tukey's post-hoc test was conducted to compare each specimen batch and analyze the interaction between groups. The level of significance was set at 0.05.

Results

As a result of the MANOVA test performed according to the obtained data, statistically significant results were observed for the effect of different PEEK composite and adhesive systems on the SBS ($p<0.05$). Table 2 lists the mean SBS values, standard deviations, and p values of all groups. The SBS values for the CFR-VL (20.20±3.81 MPa) and UF-VL (17.65±2.04 MPa) groups were statistically higher than the SB values (15.62±3.09 and 13.79±4.01 MPa) for both composite groups, respectively. The SBS values for the GFR-VL (22.52±5.84 MPa) group were statistically higher than those for the UF-VL (17.65±2.04 MPa) group. The SBS values for the CR-SB (20.31±3.72 MPa) group were statistically higher than those for the CFR-SB (15.62±3.09 MPa) and UF-SB (13.79±4.01 MPa) groups. The evaluation of the mean SBS and standard deviation values in all groups revealed that the SBS values for the VL (19.88±4.21 MPa) group are higher than those for the SB (16.95±4.35 MPa) group.

After the fracture test, the failure modes of the specimens were examined by using a stereomicroscope at 20× magnification. Table 3 lists the failure modes of the specimens. The specimens exhibited adhesive and mixed failure modes, but a completely cohesive failure mode was not observed. After the examination of the failure modes in 80 specimens, 44 specimens exhibited the adhesive failure mode, and 36 specimens exhibited the mixed failure mode. Figures 2 and 3 show the SEM images of the specimens in the adhesive and mixed failure modes at 90× magnification, respectively.

Table 2. MANOVA test results and Means±SD for SBS of the specimens of PEEK composites with different adhesive systems

Groups	Means±SD (MPa)		Mean method	
	SB	VL		
GFR	18.07±3.94	22.52±5.84	20.29±5.36	
CFR	15.62±3.09	20.20±3.81^{A}	17.91±4.11	
UF	13.79±4.01	17.65±2.04^{Aa}	15.72±3.67^{a}	$p<0.05$
CR	20.31±3.72^{bc}	19.16±3.19	19.73±3.43^{c}	
TOTAL	16.95±4.35	19.88±4.21^{A}		

SD: Standart deviation. Within the same column or row, the same superscripted letters indicate significant differences ($p<0.05$). a: GFR, b: CFR, and c: UF. Statistically significant differences between the specimens of PEEK composites (within the same adhesive material). A: SB. Statistically significant differences between different adhesive systems (within the same of the specimens of PEEK composites).

Table 3. Failure modes of the specimens of PEEK composites with different adhesive systems

Groups	Adhesive		Mixed	
	VL	SB	VL	SB
GFR	5	5	5	5
CFR	8	7	2	3
UF	3	6	7	4
CR	4	6	6	4

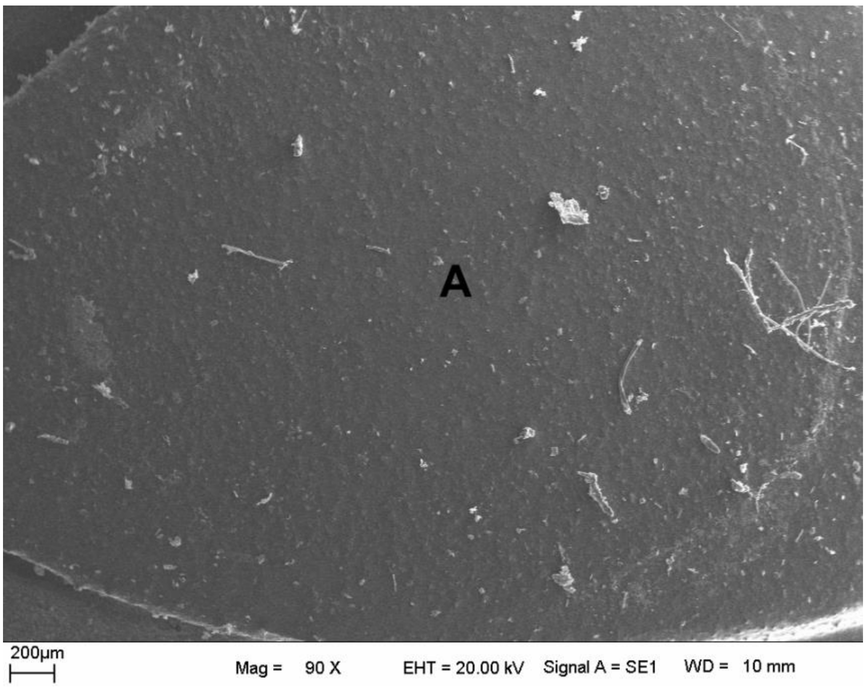

Figure 2. Type of adhesive failure mode: A, adhesive failure from PEEK surface. SEM: 90x magnification.

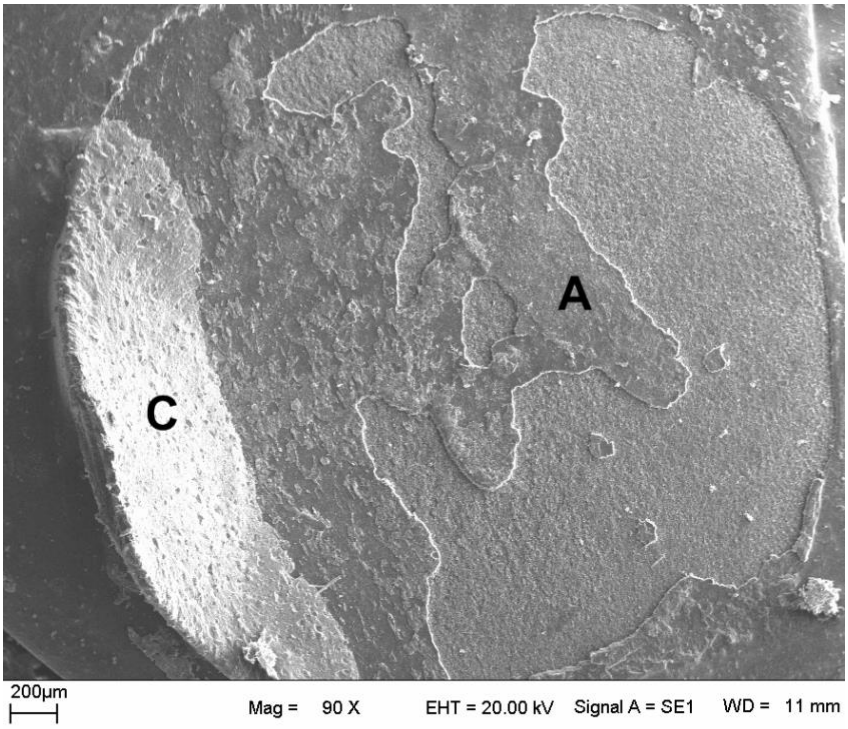

Figure 3. Type of mixed failure mode: A, adhesive failure from PEEK surface; C, cohesive failure in composite resin. SEM: 90x magnification.

Discussion

In this in vitro study, the effect of two adhesive systems applied to the surfaces of UF-PEEK, GFR-PEEK, CFR-PEEK, and CR-PEEK composites on the bond strength of a veneering composite resin were compared in terms of the SBS test. The type of the PEEK composite and adhesive system can affect SBS ($p<0.05$). With the data obtained in this study, the null hypothesis of this study was rejected.

In this study, similar surface treatments were applied to the GFR (20.29±5.36 MPa), CFR (17.91±4.11 MPa), UF (15.72±3.67 MPa), and CR (19.73±3.43 MPa) groups, and average SBS values were obtained. In several studies, SBS values of greater than 10 MPa are argued to be acceptable although there is no clear consensus (17, 23, 24). In this study, acceptable SBS values of greater than 10 MPa were observed in all groups.

Micro-shear and micro-tensile tests are more current and consistent to identify bonding properties. However, when both micro methods are used, a higher bonding strength versus a lower bonding area is obtained. Compared to macro-test methods, these methods also exhibit considerable technical sensitivity and detail. Despite this, macro test methods are more widely employed (25, 26). Hence, in this study, the SBS test, which is one of the macro test methods, is employed to obtain direct and rapid results and to reduce the possibility of error due to its facile operation and technical sensitivity.

In this study, the diameter of the composite resin specimens was 5 mm. Airborne-particle abrasion with 50-µm Al_2O_3 was conducted on the PEEK surface, and the SBS value for the CR group was 19.16±3.19 MPa. Jin et al (18). evaluated the bonding strength of the composite resin by airborne-particle abrasion with 110-µm Al_2O_3 and by the application of an adhesive material (e.g., Visio.link) to the CR-PEEK (BioHPP) and titanium surfaces, and the SBS value for CR-PEEK was reported as 31.1±3.5 MPa. In this study, by using composite resin specimens with a diameter of 14 mm and performing airborne-particle abrasion with 110-µm Al_2O_3 to PEEK specimens possibly led to higher SBS values compared with those obtained herein. Ourahmoune et al. (13) reported that particle size utilized for airborne-particle abrasion affects the PEEK surface morphology. On the contrary, Stawarczyk et al. (15) reported that the different sizes of Al_2O_3 particles do not significantly affect PEEK bonding properties. Hence, there are different opinions regarding the airborne-particle abrasion process for PEEK surface modification.

In dentistry, fibers are used for the structural reinforcement of high-performance polymers (11). The addition of short fibers to PEEK, which is a high-performance thermoplastic polymer, renders considerable improvement to its properties, such as stiffness, strength, and hardness. The presence of carbon or glass fibers in PEEK composites renders a significant impact on the results of the airborne-particle abrasion process. In fact, the roughness of PEEK composites is higher than that of UF-PEEK composites after airborne-particle abrasion under the same processing conditions (13). In this study, the SBS values for GFR (20.29±5.36 MPa) and CFR (17.91±4.11 MPa) groups were greater than that observed for the UF (15.72±3.67 MPa) group with similar surface treatments. In addition, the crystallinity ratio in semi-crystalline thermoplastic materials such as PEEK considerably affects the surface morphology created by airborne-particle abrasion due to its rheological properties (13)

Henriques et al. evaluated the composite resin cementing effect of H_2SO_4 etching and laser structuring of UF-PEEK, GFR-PEEK, and CFR-PEEK surfaces by the SBS test and reported the effect of surface modification on the PEEK composites (1). No statistically significant difference was observed in SBS values for the CFR-PEEK and GFR-PEEK specimens, while the SBS values for the UF-PEEK specimens decreased. Contrary to this study, in this study, surface modification by airborne-particle abrasion with Al_2O_3 and adhesive systems in the UF, CFR, and GFR groups is successful. Hence, similar techniques (such as airborne-particle abrasion and adhesive systems) can be applied before the application of the composite resin cement (27). Nevertheless, this topic must be investigated further.

Universal adhesives, which can be used as etch & rinse and self-etch, are theoretically one of the new-generation adhesive systems developed for all restoration materials. In addition, Single Bond Universal (SBU) contains Vitrebond (3M ESPE), which is a polyalkenoic acid copolymer. The high bonding strength of SBU is thought to be related to the presence of the polyalkenoic acid copolymer (28). The manufacturer has declared that Scotchbond Universal (3M ESPE) and SBU are the same adhesive with different product names sold in different regions of the world. As another adhesive system, Visio.link (VL) contains methylmethacrylate (MMA) and pentaerythritol triacrylate (PETIA) (Bredent). Previous studies have reported that an increase in surface area achieved by airborne-particle abrasion and the use of MMA-containing adhesive systems improve the bonding properties of PEEK (10, 15, 20, 29). In addition, the PETIA component in VL has been reported to exhibit a high capacity to modify the PEEK surface (15) In this study, both adhesive systems used for the surface modification of PEEK composites exhibited acceptable SBS values. In addition, the examination of the SEM images of adhesive and mixed failure modes (Figure 2 and 3) as well as the similar number of both failure modes revealed that both adhesive systems are successful herein. In addition, other substances that are not named by the SBU and VL manufacturers also may be present, which are not known yet to promote attachment to PEEK surfaces.

Stawarczyk et al. evaluated the effect of adhesive systems and airborne-particle abrasion parameters on the PEEK and composite resin bonding and reported that the airborne-particle abrasion pressure (0.5 and 3.5 bar) significantly affects the bonding properties as opposed to the effect of different particle sizes (50 and 110 µm) on Al_2O_3 particles (15). VL and SBU exhibited similar bonding properties, especially at high pressures, by using 50-µm Al_2O_3 at a pressure of 3.5 bar. In addition, SBU systems exhibited excellent adhesion with the increase in the pressure of airborne-particle abrasion. In this study, an airborne-particle abrasion pressure of 2 bar was selected as the average of the two airborne-particle abrasion pressures (0.5 and 3.5 bar, respectively) to accurately compare the two adhesive systems. In parallel, similar SBS values were observed for the VL and SB adhesive groups.

Bunz et al. investigated the effect of aging on SBS values by the application of a universal adhesive to PEEK specimens for surface treatment by airborne-particle abrasion using 50-µm Al_2O_3 (21). Depending on the aging time in PEEK specimens, SBS values of 8.75–14 MPa were obtained. The SBS values herein are less than those obtained in a study reported by Bunz et al. (21); the authors did not indicate the waiting time between the pre-treatment, conditioning, and veneering stages for application to the PEEK surface. Composite resin and adhesive material must be applied immediately as the PEEK surface properties can change within a few minutes after air-abrasion pre-treatment. The long residence time of PEEK after airborne-particle abrasion affects its good bonding properties. In addition, as methacrylates in adhesive systems are hydrophobic, bond strength can be strongly affected (15).

One of the limitations of this study is that specimens were produced and tested under ideal conditions that may not reflect actual clinical conditions. In addition, although the situation in the mouth was simulated by a thermal aging process, heat, moisture, and chewing forces could not be exactly simulated. Another limitation is that a control group was not formed to evaluate the two adhesive systems and the four PEEK composites. However, considering previous studies, airborne-particle abrasion with Al_2O_3 particles and adhesive systems provide sufficient adhesion. However, the possible effect of different adhesive systems on the bonding properties of PEEK composites should be investigated in future studies.

Conclusions

Within the limitations of this in vitro study, the following conclusions were made: Adhesive systems can affect the shear bond strength of PEEK composites. Both adhesive systems are applicable for the surface modification of different PEEK composites. Additional laboratory and clinical studies to investigate the effect of different PEEK composites on surface modification are required to validate the results of this study in the future.

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