

# Effect of Particle Size and Application Pressure in Air-Borne Particle Abrasion on the Adhesion of Polyetheretherketone

## Keywords

Air-Borne Particle Abrasion  
Air-Abrasion  
Indirect Veneering Composite Resin  
PEEK  
Shear Bond Strength  
Surface Roughness

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## ABSTRACT

*Introduction:* This study aims to investigate the effect of different particle sizes and pressures on the shear bond strength (SBS) and the roughness (Ra) of polyetheretherketone. *Materials and methods:* A total of 117 polyetheretherketone specimens were fabricated and divided into a control (CN: no air-particle abrasion) and eight pre-treatment groups (air-abraded with 50- or 110- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles at 1(A), 1.5(B), 2(C), and 2.5(D) bar). The adhesive, Visio.link was used. Thermal aging was performed. Surface properties, SBS and failure mode were assessed. Data was analyzed by linear regression, Pearson correlation and Dunnett's T3 test for pairwise comparisons ( $p < 0.05$ ). *Results:* The highest and lowest Ra were found in the 110 $\mu$  and CN groups respectively, and the highest SBS results were obtained from the 50 $\mu$  group. Pressure and particle size showed significant difference on the investigated properties ( $p < 0.001$ ). Only adhesive failure was observed in the CN, and resin cohesive failure was observed in addition to adhesive failure in the overall study groups. *Conclusion:* The air-borne particle abrasion procedures and the adhesive material combination are critical to the strength of the polyetheretherketone bond. The combination of visio.link with the pre-treatment option of 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles applied at 1.5-bar pressure delivered the most favorable results.

## INTRODUCTION

All modifications of the polyaryletherketone (PAEK) family are semi-crystalline polymers, and the most well-known member of this family is polyetheretherketone (PEEK).<sup>1-4</sup> Because of its superior milling properties that are suitable for use with computer-aided design/computer-aided manufacturing (CAD/CAM) technology and excellent mechanical properties, PEEK can be used in many different dental applications.<sup>4-6</sup>

The long-term success of PEEK is possible if the material can provide an ideal bond strength and bonding continuity to other dental materials (e.g., acrylic resins, direct or indirect composite resins, titanium, and metallic frameworks or attachments) and dental tissues.<sup>7,8</sup> However, inert nature, low surface energy, and poor wettability of PEEK adversely affect the bonding capacity of the veneering materials, which are recommended to overcome the aesthetic deficiencies of PEEK, especially those related to color.<sup>4,5</sup> PEEK materials have a grayish-brown or pearly white opaque color that might not meet the aesthetic expectations. A possible solution to

Received: 09.08.2023

Accepted: 31.01.2024

doi: 10.1922/EJPRD\_2610Sahin08

this issue is to use resin-based restorative materials to veneer PEEK surfaces by employing either direct or indirect methods. However, it is important to note that the most significant risk associated with these applications is the difficulty in achieving strong adhesion to the PEEK surface. This is widely considered the primary clinical disadvantage of PEEK materials.<sup>4,7</sup> For this reason, pre-treatment applications and surface modification processes constitute a very important step in establishing successful long-term material adhesion.<sup>5,8,9</sup>

Many methods, such as air-borne particle abrasion, tribochemical silica coating, etching with sulfuric acid or piranha solution, laser applications, different types of plasma applications, and adhesive material applications, have been used to activate the inert surface of PEEK.<sup>2,4,5,10-16</sup> From studies examining the methods used for PEEK surface activation, it has been determined that the air-borne particle abrasion method increased the adhesion strength of PEEK and positively affected the adhesive properties.<sup>4,5,8,10-12,17</sup> Therefore, the most common approach recommended for PEEK surface modification currently is to abraded the surface of the material with Al<sub>2</sub>O<sub>3</sub> and apply an adhesive material containing methyl methacrylate (MMA) to the surface.<sup>8,10-12,15</sup>

The effect of different sizes of Al<sub>2</sub>O<sub>3</sub> particle and air pressures on the bond strength between PEEK and the veneering material has been investigated in previous studies,<sup>2,4,10,13,14,18</sup> but the results of the studies were insufficient.<sup>4,5,8,10</sup> In order to reach a general conclusion on this subject, it is necessary to simultaneously evaluate the effect of particle size and pressure differences used in the air-borne particle abrasion method on the bonding properties and surface roughness of the PEEK.<sup>5</sup> This study investigates the effect of different Al<sub>2</sub>O<sub>3</sub> particle sizes and pressures on the shear bond strength (SBS) of PEEK bonded to indirect veneering composite resin and the surface roughness of the PEEK.

The null hypotheses of the study are different particle sizes and application pressures will not affect the SBS of the PEEK bonded to indirect veneering composite resin, and the surface roughness of the PEEK.

## MATERIALS AND METHODS

### SPECIMEN PREPARATION

A total of 117 PEEK (CoproPeek Light, Whitepeaks Dental Solutions; polyetheretherketone (≈ 80%), titanium dioxide (< 20%), and other additives (< 0.1%)) specimens (10 × 10 × 2 mm) were fabricated by milling CAD/CAM production process according to ISO 10477. All the specimens were embedded in autopolymerizing acrylic resin (Meliodent, Heraeus Kulzer) and polished with 200, 500, 800, and 1000 grit silicon carbide abrasive papers (Struers) under continuous water cooling in an automatic polishing machine (Mecapol P 230, Press) at 180 rpm for 1 minute at each grade. The polished specimens were randomly divided into a negative control group and eight

study groups (n=12 for SBS analysis, n=1 for scanning electron microscopy analysis for each group) that air-borne particle abrasion with Al<sub>2</sub>O<sub>3</sub> particles with different properties (Renfert Basic Classic, Renfert) as a pre-treatment of the PEEK surface:

CN group:	no air-borne particle abrasion
Group 50 <sub>A</sub> :	50-µm particle size with 1-bar pressure
Group 50 <sub>B</sub> :	50-µm particle size with 1.5-bar pressure
Group 50 <sub>C</sub> :	50-µm particle size with 2-bar pressure
Group 50 <sub>D</sub> :	50-µm particle size with 2.5-bar pressure
Group 110 <sub>A</sub> :	110-µm particle size with 1-bar pressure
Group 110 <sub>B</sub> :	110-µm particle size with 1.5-bar pressure
Group 110 <sub>C</sub> :	110-µm particle size with 2-bar pressure
Group 110 <sub>D</sub> :	110-µm particle size with 2.5-bar pressure

All air-borne particle abrasion procedures were performed at a 45° angle and 10 mm from the specimen surface for 15 seconds. After the air-borne particle abrasion process was completed, all specimens were cleaned in 70% isopropanol in an ultrasonic cleaner (Eurosonic Energy, Euronda) for 15 minutes, washed with distilled water for 10 minutes, and dried under light air pressure.

### SURFACE ROUGHNESS AND TOPOGRAPHY ANALYSIS

Surface roughness measurements were used to analyze the surface properties of all groups after the air-borne particle abrasion protocol. Measurements were made with a diamond-tipped contact profilometer (Mahr Perthometer M2, Mahr) applying a measuring force of 0.7 mN with a trace length of 6 mm. Measurements were made at three different areas on each specimen by moving the diamond tip of the device along the specimen surface in parallel, and the average roughness data (R<sub>a</sub>) of the specimens were calculated. After each measurement, the profilometer was calibrated with a special calibration block.

One random specimen selected for analysis by SEM, and the surfaces of the specimens were coated with 80% gold and 20% palladium using a sputtering device (Q150R ES, Quorum Technologies). The specimens were evaluated using 1000× magnification at 20 kV.

### INDIRECT COMPOSITE VENEERING PROCESS

The surfaces of the specimens were treated with visio.link (Bredent) adhesive material according to the manufacturer's instructions for the indirect veneering process. Then, indirect veneering composite resin (Gradia Plus, GC Dental Products) was applied to the surface of the specimens using a disk-shaped silicone mold with a 2-mm diameter and a 3mm height. The silicone mold was removed after each composite resin was polymerized according to the manufacturer's instructions. Information about the composition of the adhesive

and indirect veneering composite resins and the application protocol details are summarized in Table 1. After the veneering process was completed, the specimens were kept in distilled water for 1 day at room temperature in a dark environment. Next, all the specimens were aged by thermal cycling in distilled water for 5000 cycles between 5 °C and 55 °C with a dwell time of 20 seconds in an automated thermocycling machine (Gökceler Machines, İstanbul, Türkiye).

## SHEAR BOND STRENGTH TEST AND FAILURE MODE OBSERVATION

A universal test machine (Autograph AGS X, Shimadzu) was used for the SBS test at a crosshead speed of 1 mm/min. The SBS values were calculated in megapascals (MPa) by dividing failure load (N) by the area of the composite resin ( $a=P/A$ ). The failure types of all the specimens were analyzed using an optical microscope (MP 320; Carl Zeiss) at 50× magnification. The failure types were categorized as adhesive failure at the interface between the PEEK and the indirect veneering composite resin, cohesive failure in the PEEK, cohesive failure in the indirect veneering composite resin, and mixed failure (adhesive and cohesive failure of at least 25% of the surface).

## STATISTICAL ANALYSIS

The sample size for the study was calculated using the G-POWER software (version 3.1.9.2; Heinrich Heine Universität, Düsseldorf, Germany). It stipulated nine independent groups according to an effect size of 0.4, 80% power, and 0.05 sampling error. The SPSS program for Windows, version 21.0 (SPSS Inc., Chicago, IL), was used for statistical analysis. The normal distribution of the data was evaluated by the Shapiro-Wilk test. In the comparison of the  $R_a$  and SBS results of the groups, MANOVA with Dunnett's T3 *post hoc* test was used for pairwise analysis. Pearson correlation analysis was used to determine the correlation between the  $R_a$  and SBS values. The main effect of the particle and pressure differences was determined by linear regression analysis. Significance was evaluated as  $p<0.05$  and  $p<0.001$ .

## RESULTS

### SURFACE ROUGHNESS

The highest  $R_a$  was found in the 110<sub>D</sub> group, and the lowest  $R_a$  was observed in the CN group. Statistically significant differences were found between the CN group and all study groups ( $p<0.001$  for all comparisons). The  $R_a$  values of all groups and statistical differences between groups were summarized in Table 2.

### SHEAR BOND STRENGTH

The highest SBS results were obtained in the 50<sub>B</sub> group, followed by the 50<sub>D</sub>, 110<sub>D</sub>, 110<sub>B</sub>, 110<sub>C</sub>, 50<sub>C</sub>, 50A, 110<sub>A</sub>, and CN groups. Significant differences were found between the SBS results of all test groups ( $p<0.001$  for all comparisons) and the CN group (Table 2). When the particle sizes and pressure values were evaluated together in the study groups, the highest SBS value was determined in the 50- $\mu$ m particle size with 1.5-bar pressure (50<sub>B</sub>) group and the lowest value was determined in the 50- $\mu$ m particle size with 1-bar pressure group (50<sub>A</sub>,  $p<0.001$ ). In the 110- $\mu$ m particle size groups, the highest SBS values were obtained in the group with a 2.5-bar pressure (110<sub>D</sub>), and the lowest values were observed in the 1-bar pressure (110<sub>A</sub>) group ( $p<0.001$ , Table 2).

In addition, when the 50- $\mu$ m and 110- $\mu$ m particle groups were evaluated within themselves, it was observed that the SBS values decreased in the 2-bar pressure groups compared to the 1.5-bar and 2.5-bar pressure groups. Despite the decrease in the SBS values, no significant difference was found between the 110<sub>C</sub> group and the 1.5-bar or 2.5-bar (50<sub>B</sub>, 50<sub>D</sub>, 110<sub>B</sub>, and 110<sub>D</sub>) groups ( $p>0.05$ ), while the 50<sub>C</sub> group showed a significant statistical difference with the 50B and 50<sub>D</sub> groups ( $p=0.001$  and  $p=0.029$ , respectively) (Table 2). There was positive correlation between  $R_a$  and SBS results ( $r=0.441$ ,  $p<0.001$ ).

**Table 1. The composition and the application of the materials used in the study.**

Product	Composition*	Application recommendation*
visio.link	MMA, 2-prepenoic acid reaction products with pentaerythritol, diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide	Apply the product to the material surface with a brush thinly and only once. Then immediately apply 90 seconds of light with a dental laboratory polymerization device (GC Labolight DUO, GC Dental Products) at 370-400 nm wavelength range.
Gradia Plus® indirect composite (nano-hybrid)	1%–5% Bis-GMA, 5-10%TEGDMA,1%–5% UDMA, ceramic filler	3 minutes of light application (at a wavelength of 380-510 nm with approximate peaks at 395 and 475 nm) in full mode with a dental laboratory polymerization unit (GC Labolight DUO, GC Dental Products).

\*Material contents are presented according to the manufacturer's information. BisGMA: Bisphenol-A glycidyl dimethacrylate, MMA: Methyl methacrylate, TEGDMA: triethyleneglycol-dimethacrylate, UDMA: urethane dimethacrylate.

**Table 2.** SBS and surface roughness data ( $R_a$ ) of all groups.

GROUPS (n=13)	$R_a$			SBS (MPa)		
	Mean $\pm$ SD	Min-Max (Median)	p*	Mean $\pm$ SD	Min-Max (Median)	p**
CN Group	0.29 $\pm$ 0.04 <sup>a</sup>	0.23 – 0.38 (0.3)		11.24 $\pm$ 3.6 <sup>a</sup>	2.9 – 14.4 (11.71)	
Group 50 <sub>A</sub>	0.45 $\pm$ 0.04 <sup>b</sup>	0.4 – 0.56 (0.44)		74.58 $\pm$ 37.43 <sup>b</sup>	18.32 – 142.9 (82.32)	
Group 50 <sub>B</sub>	0.55 $\pm$ 0.04 <sup>b,c,d</sup>	0.46 – 0.59 (0.57)		156.82 $\pm$ 19.0 <sup>c</sup>	98.3 – 175.41 (160.7)	
Group 50 <sub>C</sub>	0.63 $\pm$ 0.05 <sup>c,e</sup>	0.49 – 0.73 (0.63)		86.25 $\pm$ 33.54 <sup>b,f</sup>	32.4 – 174.47 (87.72)	
Group 50 <sub>D</sub>	0.54 $\pm$ 0.08 <sup>b,c</sup>	0.44 – 0.7 (0.54)	<0.001	139.05 $\pm$ 29.42 <sup>c,d,e</sup>	97.71 – 178.93 (141.58)	<0.001
Group 110 <sub>A</sub>	0.61 $\pm$ 0.05 <sup>c</sup>	0.51 – 0.69 (0.61)		57.1 $\pm$ 13.51 <sup>b</sup>	34.5 – 77.39 (52.63)	
Group 110 <sub>B</sub>	0.72 $\pm$ 0.07 <sup>e,f</sup>	0.63 – 0.89 (0.72)		101.95 $\pm$ 29.59 <sup>b,e</sup>	50.94 – 146.88 (113.21)	
Group 110 <sub>C</sub>	0.81 $\pm$ 0.09 <sup>f</sup>	0.68 – 0.96 (0.81)		106.05 $\pm$ 32.31 <sup>b,c</sup>	60.36 – 176.72 (96.82)	
Group 110 <sub>D</sub>	0.88 $\pm$ 0.08 <sup>f</sup>	0.67 – 1 (0.88)		132.47 $\pm$ 30.45 <sup>c,d,e,f</sup>	87.28 – 192.74 (133.88)	

\* MANOVA with Dunnett's T3 post hoc multiple comparisons, \*\*Kruskal–Wallis test and Mann–Whitney U test with pairwise analysis:  $p < 0.05$  and  $p < 0.001$ . No statistically significant difference existed between groups represented by the same letters, but a statistically significant difference was found between the groups with different letters. SD: standard deviation, Min: minimum, and Max: maximum.

A regression model was established for different pressures and different particles, and control groups were excluded in the regression analysis. The linear regression model established to examine the effect of pressure and particles differences on SBS was statistically significant ( $F=34.637$ ;  $p < 0.001$ ). When the 50  $\mu\text{m}$  air-borne particle abrasion process was taken as a reference, the SBS value obtained in the 110  $\mu\text{m}$  air-borne particle abrasion process was 5.259 lower, but this value was not statistically significant ( $p=0.416$ ). When 1-bar pressure value was taken as reference for the pressure variable, the SBS value obtained at 1.5-bar pressure was 82.592, the SBS value obtained at 2-bar pressure was 49.36, and the SBS value obtained at 2.5-bar pressure was 88.967, and these values statistically significant ( $p < 0.001$ , Table 3). Significance was also obtained in the linear regression model established to examine the effect of pressure and particles on  $R_a$  ( $F=120.233$ ;  $p < 0.001$ ). When the 50  $\mu\text{m}$  air-borne particle abrasion procedure was taken as reference, the  $R_a$  was obtained 0.238 units more when the particle size was applied as 110  $\mu\text{m}$  ( $p < 0.001$ ). When the 1-bar pressure application was taken as a reference, the  $R_a$  value was 0.152 higher for those with 1.5-bar pressure, 0.23 higher for those with a pressure value of 2-bar and 0.221 higher for those with a pressure value of 2.5-bar, and these values were statistically significant. ( $p < 0.001$ , Table 3).

When 1-bar pressure value was taken as reference for the pressure variable, the SBS values for 1.5 bar, 2-bar and 2.5 bar were 82.592 MPa, 49.36 MPa and 88.967 MPa, respectively and statistically significant ( $p < 0.001$ , Table 3). The effects of pressure and particles on  $R_a$  were also statistically significant ( $F=120.233$ ;  $p < 0.001$ ). When the 50  $\mu\text{m}$  air-borne particle abrasion procedure was taken as reference, the  $R_a$  was 0.238

unit higher than 110  $\mu\text{m}$  ( $p < 0.001$ ). When the 1-bar pressure application was taken as a reference, the  $R_a$  value was 0.152 unit higher than 1.5-bar pressure, 0.23 unit higher than 2-bar and 0.221 unit higher than 2.5-bar, and these values were statistically significant. ( $p < 0.001$ , Table 3).

## FAILURE MODE EVALUATION

Only adhesive failure was detected in in the CN and 110<sub>A</sub> groups. In addition to adhesive failure, cohesion failure was observed in the indirect veneering resin material in the 50<sub>A</sub>, 50<sub>B</sub>, 50<sub>D</sub>, 110<sub>B</sub>, 110<sub>C</sub>, and 110<sub>D</sub> groups. In the 50<sub>C</sub> group, cohesive failure was detected in both the indirect veneering resin material and the PEEK structure in addition to adhesive failure. Mixed failure was not found in any of the study groups (Table 4).

## SURFACE TOPOGRAPHY

SEM images of the surface topography of specimens after applying air abrasion with different  $\text{Al}_2\text{O}_3$  particle sizes and pressures was shown in Figure 1. The CN group (no air-particle abrasion procedure) exhibited less roughness than the others (Figure 1A). A homogeneous and dense microporosity was observed in the 50<sub>B</sub> group (Figure 1B), and alumina particles embedded on the surface of the specimen were observed in the 50<sub>D</sub> group (Figure 1E). In the 110<sub>A</sub> group, the embedding of particles on the surface was intense, and there were particle ruptures on the surface of the PEEK (Figure 1F). A cleaner specimen surface was observed in the 110<sub>B</sub> and 110<sub>C</sub> groups, with fewer alumina particles embedded than in the 110<sub>A</sub> group (Figure 1). In the 110<sub>D</sub> group, deep craters were formed on the surface, and the embedded alumina particles or particle ruptures were almost absent (Figure 1I).

**Table 3.** Investigation of the effect of pressure and particle differences on SBS and Ra by linear regression

	SBS a						Ra b						
	$\beta_0$ (%95 CI)	SD	$\beta_1$	t	p	VIF	$\beta_0$ (%95 CI)	SD	$\beta_1$	t	p	VIF	
<b>Dependent</b>	49.425 (37.773 – 61.077)	5.881		8.404	<0.001		0.378 (0.35 – 0.406)	0.014		26.971	<0.001		
<b>Particle</b>	<b>50 <math>\mu\text{m}^*</math></b>												
	<b>110 <math>\mu\text{m}</math></b>	-5.259 (-18.023 – 7.506)	6.442	-0.052	-0.816	0.416	1.026	0.238 (0.207 – 0.268)	0.015	0.644	15.478	<0.001	1.026
<b>Pressure</b>	<b>1-bar*</b>												
	<b>1.5-bar</b>	82.592 (65.309 – 99.875)	8.723	0.686	9.469	<0.001	1.316	0.152 (0.111 – 0.193)	0.021	0.345	7.330	<0.001	1.316
	<b>2-bar</b>	49.36 (32.077 – 66.642)	8.723	0.410	5.659	<0.001	1.316	0.23 (0.189 – 0.271)	0.021	0.522	11.071	<0.001	1.316
	<b>2.5-bar</b>	88.967 (71.684 – 106.25)	8.723	0.739	10.200	<0.001	1.316	0.221 (0.18 – 0.262)	0.021	0.501	10.643	<0.001	1.316

\*Reference parameter. <sup>a</sup>F=34.637, p<0.001; R<sup>2</sup>=0.553, Adjusted R<sup>2</sup>=0.537; <sup>b</sup>F=120.233, p<0.001; R<sup>2</sup>=0.811, Adjusted R<sup>2</sup>=0.804,  $\beta_0$ : Standardized beta coefficient,  $\beta_1$ : Nonstandardized beta coefficient, SD: Standard deviation.

**Table 4.** Failure mode evaluation results of the specimens.

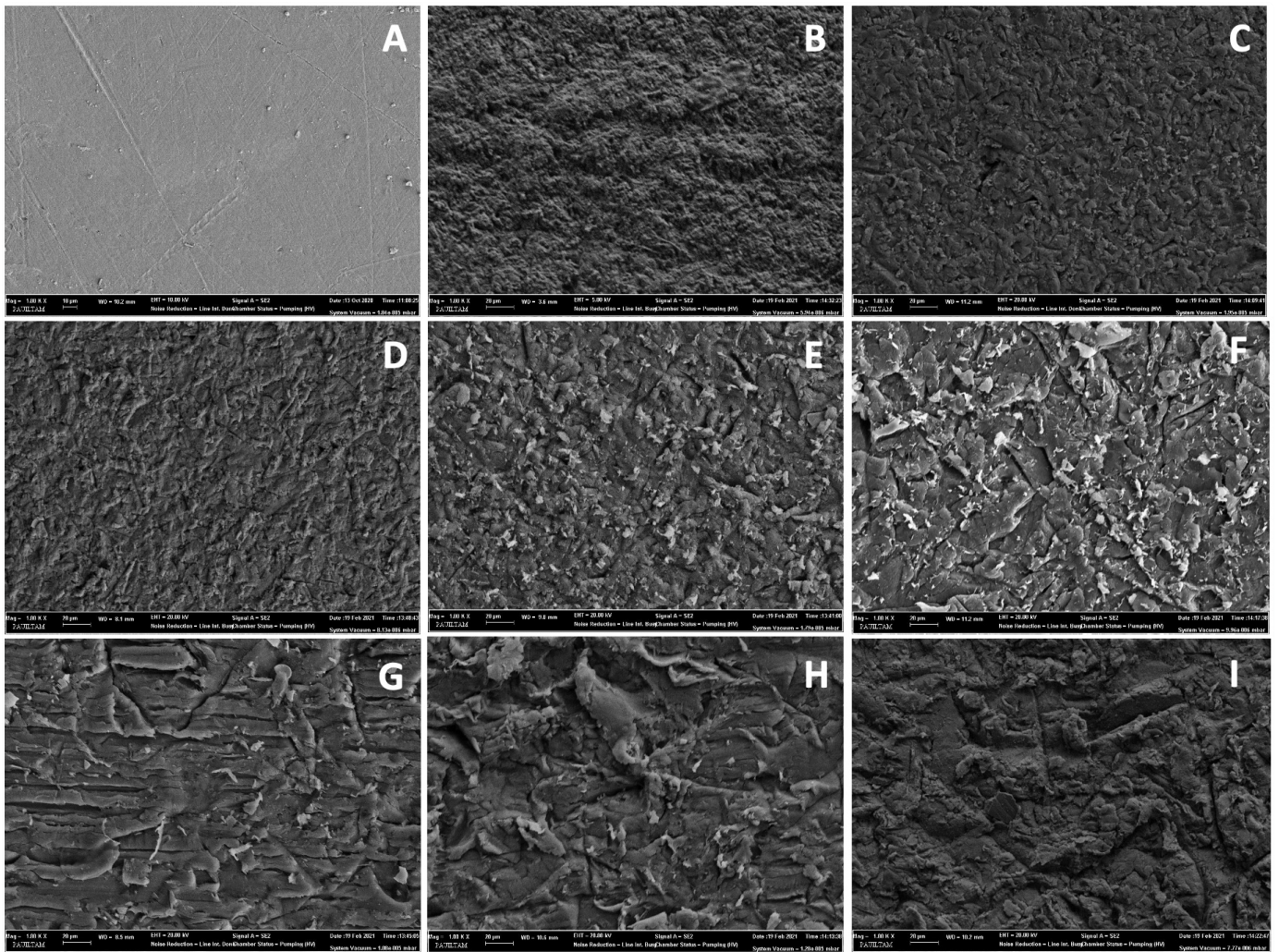
Groups	Adhesive	Cohesive (Veneering resin)	Cohesive (PEEK)	Mix
CN	12	-	-	
50 <sub>A</sub>	10	2	-	
50 <sub>B</sub>	1	11	-	
50 <sub>C</sub>	9	2	1	
50 <sub>D</sub>	6	6	-	None
110 <sub>A</sub>	12	-	-	
110 <sub>B</sub>	10	2	-	
110 <sub>C</sub>	9	3	-	
110 <sub>D</sub>	2	10	-	

## DISCUSSION

The first and second null hypotheses, that different particle sizes and application pressures would not affect the SBS of the PEEK bonded to the indirect veneer composite resin or the surface roughness data ( $R_a$ ), were rejected in line with the results of the study.

Like previous studies that tested different alumina particle sizes and application pressures,<sup>14,18</sup> increased pressure increased the SBS values in the current study. Regardless of particle size, the specimens abraded at 1-bar pressure exhibited lower numerical SBS values than other groups in agreement with previous studies. However, this numerical difference did not create a statistical difference. Also, the application of 50- $\mu\text{m}$  particles at a pressure of 1.5 bar showed higher numerical SBS results than 110- $\mu\text{m}$  particles at 1.5-, 2-, and 2.5-bar application pressures, and 50- $\mu\text{m}$  particles at 2- and 2.5-bar application pressures. While these numerical differences caused a statistical difference between some groups, they did not create a statistical difference between other groups. Although the  $R_a$  values were numerically higher in the specimens abraded with 110- $\mu\text{m}$  particles at 1.5-, 2, and 2.5-bar pressures and with 50- $\mu\text{m}$  particles at 2-bar pressure, the SBS results were numerically lower than those of the specimen prepared with 50- $\mu\text{m}$  particles at 1.5-bar pressure. Similar to the SBS results, the numerical differences observed in terms of  $R_a$  did not cause a statistical difference for each group. On the other hand, the changes in SBS values positively correlated with the  $R_a$  according to the results of the correlation analysis performed in the current study ( $r=0.441$ ,  $p<0.001$ ).

Many factors, such as the amount of substrate, viscosity and molecular weight of the adhesive agent and veneering composite, and the penetration capacity of the adhesive materials, affect the bonding between materials.<sup>5</sup> Micromechanical locking, which is provided by the penetration of the bonding agent into



**Figure 1:** SEM images at 1000× magnification of PEEK after air-borne particle abrasion with different  $\text{Al}_2\text{O}_3$  particle sizes and different application pressures. A) CN group. B) 50<sub>A</sub> group. C) 50<sub>B</sub> group. D) 50<sub>C</sub> group. E) 50<sub>D</sub> group. F) 110<sub>A</sub> group. G) 110<sub>B</sub> group. H) 110<sub>C</sub> group. I) 110<sub>D</sub> group.

the microporosity, is an important factor in strengthening the adhesion between PEEK and resin materials.<sup>14,18,19</sup> For this reason, the different SBS values obtained in this study may be due to the differences in particle size and application pressure and the penetration capacity of the visio.link adhesive material into the microporous areas. In addition, although surface roughness values increased in some of the study groups, the changes in SBS values did not correlate with the  $R_a$  since the visio.link adhesive material could not reach the same penetration depth.

In contrast, after applying high pressure, the roughness of the specimen surfaces will increase, and the bond strength will increase accordingly. However, the increase in pressure may negatively affect the SBS by causing the alumina particles to be more easily embedded in the treated surface structure.<sup>5,14,18</sup> After the pre-treatment, in addition to the non-homogeneous roughness on the surface of the PEEK specimens in the SEM images of the current study, embedded alumina particles were also observed on some surfaces. In particular, the number of alumina particles embedded in the surface increased in the 2.5-bar pressure group (50<sub>D</sub>) compared to the other groups abraded with 50- $\mu\text{m}$  particles. This increase

led to a decrease in SBS values in the 2.5-bar pressure group (50<sub>D</sub>) compared to the 1.5-bar pressure group (50<sub>B</sub>), which presented the highest values among the specimens abraded with 50- $\mu\text{m}$  particles. These results were similar to the results of Silthampitag *et al.*,<sup>14</sup> in which similar particle sizes and pressures (50  $\mu\text{m}$  and 2 bar) were tested. Similar to the present study, irregular cracked grooves and crack patterns were observed in the SEM images, and it was concluded that air abrasion generally caused  $\text{Al}_2\text{O}_3$  to be embedded in the PEEK surface. The increased pressure may further increase this effect and embedding  $\text{Al}_2\text{O}_3$  particles in the PEEK surface may negatively affect the bonding capacity.<sup>14</sup>

Bond strength is also associated with surface oxygen concentration, surface roughness, and wettability.<sup>7</sup> In this study, it was assumed that increasing SBS values following air-borne particle abrasion with increasing application pressures led to a higher wettability by increasing the surface roughness, which in turn increased the bond strength. This finding was also supported by SEM images showing increased surface roughness and crater formation (especially in the 110- $\mu\text{m}$  groups) when compared with the surface properties of the

low-pressure groups in terms of increased particle size or applied pressure. Although more studies are needed to confirm the present results, the combinations of 50- $\mu\text{m}$  particles with 1.5-bar pressure, 50- $\mu\text{m}$  particles with 2.5-bar pressure, 110- $\mu\text{m}$  particles with 1.5-bar pressure, and 110- $\mu\text{m}$  particles with 2.5-bar pressure are suitable for increasing the bonding properties of the PEEK to the indirect veneering resin.

In previous studies investigating the bond strength, lower SBS values were observed in the specimens where adhesive material was applied without air-borne particle abrasion (similar to the CN group).<sup>7,11,12,17</sup> However, even in the CN group, the SBS was  $11.24 \pm 3.6$  MPa. Considering the results of previous studies on adhesive materials, this result may be due to 2-propenoic acid reaction products with pentaerythritol-triacrylate (PETIA) in the visio.link adhesive material, which has been reported to significantly affect PEEK surface activation while bonding to the dimethacrylate polymer.<sup>4,9-11,15</sup> This finding led to the expectation that the dominant failure type in the CN group would be adhesive rupture of the interface, and the study results supported this expectation.

In addition, as in previous studies,<sup>16</sup> the most common failure type observed in this study was adhesive failure between the PEEK and the indirect resin material bond surface (62.5%). It is not unexpected that groups featuring lower SBS values will solely experience adhesive failure, owing to a weak interface connection. It has been determined that with an increase in SBS values, cohesive fractures are observed, specifically within the resin structure, in conjunction with adhesive failure. As the strength of the bond increases, it is common to observe cohesive fracture in the resin material. This is due to the fact that the resin material has a lower fracture strength when compared to the PEEK material. In a recent study, cohesive failure in the resin material was found to be the most common type of failure in groups 50<sub>B</sub> and 110<sub>D</sub>, which exhibited the highest SBS data. The results of the study indicate that when PEEK is combined with additional restorative materials that possess high bond strength, there is a risk of failure in the material that has lower fracture strength. This finding could serve as a valuable resource for clinicians, enabling them to anticipate complications that may occur during the clinical use of the material, and subsequently plan for the appropriate solution to such complications.

Determination of  $R_a$  following the air-borne particle abrasion procedures allowed accurate identification of the specimen surfaces in terms of the effectiveness of the surface activation by the adhesive materials.<sup>7</sup> In this study, the analysis of the  $R_a$  and the examination of the surfaces with SEM imaging helped interpret the SBS results. Thus, a better analysis of the parameters (particle size, pressure type, or adhesive material) that can be used to increase the bond strength on the sample surfaces is provided.<sup>7</sup> Correct analysis of these evaluated parameters will also be beneficial in shaping future study designs.

In this study, a thermal aging protocol was applied, which aimed to imitate the changes in the oral environment during eating, drinking, and breathing with 5000 cycles between 5 °C and 55 °C.<sup>20,21</sup> However, there have been studies reporting that the current procedure may cause microcracks in the material and bond surfaces, volumetric changes resulting in weakening of the chemical bonding, and stress, in addition to not affecting or increasing the bond strength.<sup>11,22,23</sup> In short, conflicting results have been reported for the effect of thermal cycling on bond strength for PEEK. The first limitation of the current study was that there is no additional control group that was not aged, so no result isolating the effect of the aging protocol has been obtained. There is a definite need for further and comprehensive studies investigating the effect of artificial aging on chemical binding to PEEK substrates in this controversial area.

In increasing the bond strength between the PEEK and the veneering resin composite materials, air-borne particle abrasion can be an effective pre-treatment in terms of cost, time, ease of access, and applicability. The application of suitable adhesive materials, in addition to the pretreated surfaces, is critically important. Since the current study tested the combination of the visio.link adhesive material most commonly used in laboratory applications with PEEK, as well as the specific particle size and application pressure, additional studies are needed to improve on the current results. In addition to the study groups, only the negative control group, designated as the no pre-treatment group, was tested in this study. It may be recommended to include more clinical application scenarios in the methodology to determine the method to be used as a positive control group in future studies. For example, testing another innovative dental material in terms of SBS values may help to better understand the bond strength limits of PEEK material. Therefore, including such materials as a positive control group in future studies may increase the importance of the study results.

Although positive correlation was detected between SBS and  $R_a$  in the current study, the fact that the numerical differences detected in SBS and  $R_a$  values did not create a statistical difference for each group is a finding that should be taken into consideration. Increasing the power of the study and the number of samples may cause the existing numerical data to change and this change to be reflected as a statistical difference. Therefore, methodological changes in sample size and statistical power of the study may be planned in future studies. The *in vitro* design of this study cannot fully simulate the intraoral clinical situation. With the current study design, preliminary information can be given to the clinician regarding the possibilities required to achieve ideal bonding to the PEEK. To reach more general conclusions, all scenarios tested *in vitro* should be evaluated with long-term clinical studies.

## CONCLUSION

Deposition parameters of the air-borne particle abrasion procedure and the selection of the adhesive material for surface conditioning are critical in obtaining improved bond strength. In order to increase the adhesion between the PEEK and the indirect resin composite and obtain stronger bonds, the combination of visio.link with the pre-treatment option of air-borne particle abrasion with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles at 1.5-bar pressure was shown to be a successful option. The SBS values are affected by alumina particle size and applied pressure. High application pressures can cause alumina particles ( $\text{Al}_2\text{O}_3$ ) to be embedded in the PEEK surface, reducing the bond strength.

## ACKNOWLEDGEMENTS

The study was approved by the Medical Ethics Committee of Pamukkale University (Approval number: 60116787-020/54320 and 60116787-020/328882).

## DECLARATION OF CONFLICTING INTERESTS

The authors declared that there is not any conflict of interest and financial interest in the companies are included in this article.

## FUNDING

The authors declared that there is not any financial interest in the companies included in this article.

## AUTHOR CONTRIBUTIONS

Conceptualization: Sahin SC; Methodology: Sahin SC, Mutlu-Sagesen L; Formal analysis: Sahin SC, Mutlu-Sagesen L; Investigation: Sahin SC, Mutlu-Sagesen L, Karaokutan I; Özcan M, Writing-original draft preparation: Sahin SC; Writing- review, editing and discussion: Mutlu-Sagesen L, Karaokutan I., Özcan M.

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