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Effect of Adhesive System on Bond Strength of Polyetheretherketone (PEEK) and Polyetherketoneketone (PEKK)

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Abstract: It is uncertain whether the interchangeable use of two adhesive systems would yield comparable shear bond strength (SBS) for both Polyetheretherketone (PEEK) and Polyetherketoneketone (PEKK); investigating this was the main objective of this study. Milled PEEK (Bredent, Senden, Germany) and PEKK (Pekkton Ivory, AnaxDent, Stuttgart, Germany) blocks were prepared with standardized roughness (0.20 μm) and randomly assigned into two groups ($n = 72$): with and without aluminum oxide air abrasion (AquaCare Twin, Medivance Instruments, London, UK). Two adhesive systems (Visio.link, Bredent, Senden, Germany, or PEKKBond, AnaxDent, Stuttgart, Germany) were randomly applied ($n = 36$). Flowable gingival composite (AnaxGum Gingiva, AnaxDent, Stuttgart, Germany) was bonded, and the samples were stored in water (37 °C, 24 h). SBS was measured (MPa) and data were analyzed using three-way ANOVA, followed by Tukey's test ($\alpha = 0.05$). All main effects and interactions were significant ($p < 0.05$), except for polymer ($p = 0.163$) and the triple interaction ($p = 0.601$). In the PEEK group, Visiolink showed higher SBS ($p < 0.001$), regardless of prior air abrasion. For the PEKK group, PEKKBond significantly increased SBS values ($p < 0.001$) for both pre-treatment groups. Previous air abrasion only significantly increased the SBS of controls without adhesive. This study highlights the importance of material-specific adhesive selection, rather than interchangeable use, for optimal results. The bond strength of PEEK and PEKK is influenced by the adhesive system applied. Moreover, PEKK consistently demonstrated higher SBS values in comparison to PEEK, even without the need for pre-treatment or adhesive conditioning. This characteristic renders PEKK a preferred choice for the fabrication of adhesive restorations.

Keywords: adhesives; polyetheretherketone; polyetherketoneketone; shear bond strength



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1. Introduction

Polyetherketonaketone (PEKK) and polyetheretherketone (PEEK) are high-performance semicrystalline thermoplastic polymers belonging to the polyaryletherketone (PAEK) family, characterized by the presence of ketone and ether groups [1,2]. These polymers offer

biocompatibility, superior electrical insulation, dimensional stability under high temperatures, and resistance to heat, wear, fatigue, tension, and flexion [1,3]. Both PEEK and PEKK can be reinforced with materials such as glass, titanium, ceramic, and carbon fibers, expanding their applications in dentistry, including in dental implants, abutments, and frameworks of removable and fixed partial dentures [1,3–5]. Their unique properties also make PAEKs valuable in the medical, food, aeronautics, and automobile industries [6].

Despite the versatility and wide applications of these polymers, esthetic concerns remain, particularly in dental prostheses [5–7]. These materials typically range in color from gray to white, with low translucency and high opacity, which poses challenges for achieving natural-looking results, and often requires esthetic veneering. To improve appearance, different materials, such as methyl methacrylate (MMA)- or dimethacrylate (DMA)-based resins, or ceramics, as well as tooth-colored and/or gingiva composites, can be used [5–7]. However, for the veneering to be durable, it is imperative to achieve strong adhesion between the substrate polymer and the veneering materials [5–7]. To improve the bonding properties of these polymers, surface treatments are necessary, due to their inherent inertness [3].

The literature lacks consensus on the effects of the manufacturing processes and surface treatments on these polymers [5]. Manufacturing processes include milling and pressing, while surface treatments range from airborne particle abrasion and sulfuric acid application to plasma and laser modifications. Adhesives such as Visio.link (Bredent, Senden, Germany) and PEKKBond (Anaxblend, Stuttgart, Germany) are also commercially available and recommended for enhanced bonding [4,6,8–12]. Some studies [3,5,13] and a systematic review [14] have demonstrated that pre-treatment with air-borne aluminum oxide particle abrasion increases surface roughness, improving tensile and shear bonding forces between polymers and resins.

The manufacturers of adhesive materials indicate that PEKKBond is designed for PEKK, while Visio.link is designed for PEEK. However, to simplify processes and reduce costs, there is a growing need to minimize the number of products and streamline techniques. This prompts the question of whether a single adhesive can achieve comparable bond strength for both polymers, due to their comparable composition. The aim of this *in vitro* study was to evaluate the shear bond strength (SBS) of two adhesives on PEEK and PEKK, and to assess whether aluminum oxide air abrasion improves adhesion. The null hypothesis proposed that no significant differences in SBS would be observed between the two adhesive systems, suggesting they could be used interchangeably for both polymers.

2. Materials and Methods

The compositions and details of the materials used in this study are shown in Table 1. A total of 72 specimens, each measuring 1 cm³, were tested in this study. There were 36 blocks obtained from a PEKK milling blank (Pekkton Ivory, Cendres+Métaux, Biel, Switzerland) and 36 blocks from a PEEK milling blank (Bredent, Senden, Germany). This sample size was determined based on data from a pilot study, using G Power software (version 3.0, 2008, Dusseldorf, Germany). According to the t-test, having 30 specimens in each group would be sufficient to detect a 3.36 MPa difference in shear bond strength (SBS), assuming a standard deviation of 5.5 MPa, a power of 80%, and a significance level of 5%. However, to ensure robustness, the sample size was increased by 20%, resulting in a total of 36 specimens per experimental condition.

Table 1. Materials used in study.

Material	Composition	Manufacturer
PEKK milling blank (Pekkton Ivory)	PEKK, TiO ₂	Cendres+Métaux SA, Biel, Switzerland
PEEK milling blank	PEEK, 20% weight TiO ₂	Bredent GmbH, Senden, Germany
Visio.link	MMA, dimethacrylates PETIA, 2-propenoic acid, ligroin, activators, stabilizers	Bredent GmbH, Senden, Germany
PEKKBond	Urethandimethacrylate, MMA, stabilizers, activators	AnaxDent, Stuttgart, Germany
Fluid gingival-colored composite (AnaxGum light pink flow)	UDMA, BDDMA, BisGMA, glass powder, pyrogenic silicic acid, activators, stabilizers, pigments (74 wt%, 0.7 µm)	AnaxDent, Stuttgart, Germany

Legend: PEEK: polyetheretherketone; PEKK: polyetherketoneketone; TiO₂: titanium oxide; MMA: methyl-methacrylate; UDMA: urethane dimethacrylate; BDDMA: butanediol dimethacrylate; BisGMA: bisphenol glycidyl dimethacrylate.

To facilitate polishing, each block was placed inside polyvinylsiloxane cylinders (20 × 25 mm) filled with colorless acrylic resin (Auto TDV, TDV Dental Ltda., São Paulo, Brazil). The specimens were then polished using a metallographic polisher (Ecomet 250 Grinder Polisher, Buehler, IL, USA) with rotating silicon carbide abrasive paper (200, 400, 600, and 1200 µm), while being continuously rinsed with water, until the surface achieved an average roughness of 0.20 µm. A threshold of 0.2 µm is considered clinically acceptable for dental materials, while higher roughness can cause discoloration, biofilm accumulation, and early fractures [14].

Afterward, the specimens were cleaned in an ultrasonic bath (CD-4820, Kondentech, São Carlos, Brazil) for 10 min, and dried with absorbent paper. Surface roughness (Ra) was measured and calibrated using a digital profilometer (SurfTest SJ-400, Mitutoyo Sul Americana, Susano, Brazil).

After random allocation, the specimens were divided into two distinct groups for surface pre-treatment: the control group (no treatment) and the air abrasion group. Air abrasion was performed using an intraoral air abrasion unit (AquaCare Twin, Medivance Instruments, London, UK) with 100 µm aluminum oxide particles. The equipment's tip was positioned perpendicular to the samples, maintaining approximately 10 cm, and subjected to a 20 s application at a pressure of 5 bar.

Following the pre-treatment, the specimens were further randomly divided into four groups ($n = 18$) based on the adhesive material applied, either Visio.link (Bredent, Senden, Germany) or PEKKBond (AnaxDent, Stuttgart, Germany), and the different polymers. Adhesive application and polymerization followed the manufacturer's instructions (40 s at 800 mW/cm² with Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein). After completing the adhesive applications, a fluid gingival composite (AnaxGum light pink flow, AnaxDent, Stuttgart, Germany) was inserted into cylindrical tubes (2 × 1.5 mm), previously printed in a 3D printer machine (Asiga Max™, Asiga, Sidney, Australia), and light-cured for 40 s (800 mW/cm²). An LED light-curing device (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein) was used to light-cure the composite according to the manufacturer's instructions. The specimens were then stored in distilled water for 24 h at 37 °C.

All SBS tests were conducted in an Instron universal machine (EMIC DL 100, Emic, São José dos Pinhais, Brazil) at a speed of 0.5 mm/min, and the results were expressed in MPa. Descriptive statistics, including the mean and standard deviation, were obtained, and the data normality distribution was assessed with the Kolmogorov–Smirnov test. Statistical analysis was based on three-way ANOVA, followed by Tukey's post hoc test for multiple

comparisons ($\alpha = 0.05$). All statistical analyses were performed by using the SPSS software (version 20, IBM SPSS, Armonk, NY, USA) at the 95% level of confidence.

3. Results

All main effects were found to be significant ($p < 0.05$), except for the polymers ($p = 0.163$). Concerning interactions, only the triple interaction was not significant ($p = 0.601$) (Table 2).

Table 2. Three-way factorial ANOVA results as a function of polymer (PEEK and PEKK), surface treatment (with and without air abrasion), and adhesive system (Visiolink and PEKKBond) and their interactions.

Effect	Mean Square	df	F Value	p-Value
Polymer	690.5	1	1.96	0.163
Surface treatment	3507.1	1	9.94	0.002
Adhesive system	31,672.9	2	89.75	<0.0001
Polymer $\times$ surface treatment	9255.5	1	26.22	<0.0001
Polymer $\times$ adhesive system	47,057.9	2	133.35	<0.0001
Surface treatment $\times$ adhesive system	6483.8	2	18.37	<0.0001
Polymer $\times$ surface treatment $\times$ adhesive system	179.9	2	0.51	0.601

In the PEEK group, the application of Visiolink resulted in higher SBS values ($p < 0.05$), irrespective of prior air abrasion ($p > 0.05$). The PEKKBond was barely efficient, only when air abrasion was not applied. Air abrasion alone increased the SBS values only for the control group without adhesive application (Table 3 and Figure 1).

Table 3. Mean values ($\pm$ SD) of shear bond strength (SBS) (MPa) between flowable gingiva-colored composite and different polymers, pre-treatments, and adhesive systems applied.

Material	Pre-Treatment	Adhesive	SBS ($\pm$ SD)	Significance
PEEK	None	Without	23.05 ($\pm$ 12.15)	Aa
		PEKKBond	42.94 ($\pm$ 16.69)	Ba
		Visiolink	58.74 ($\pm$ 27.90)	Ca
	Al ₂ O ₃	Without	54.92 ($\pm$ 18.25)	Ab
		PEKKBond	50.60 ($\pm$ 15.21)	Aa
		Visiolink	64.19 ($\pm$ 21.28)	Ba
PEKK	None	Without	32.76 ($\pm$ 11.46)	Aa
		PEKKBond	90.10 ($\pm$ 26.42)	Ba
		Visiolink	37.33 ($\pm$ 17.00)	Aa
	Al ₂ O ₃	Without	42.76 ($\pm$ 14.27)	Ab
		PEKKBond	84.29 ($\pm$ 25.10)	Ba
		Visiolink	22.42 ($\pm$ 6.99)	Cb

Capital letters mean differences between adhesives ($p < 0.05$). Small letter means differences between surface treatments ($p < 0.05$). No differences were observed between polymers ($p > 0.05$).

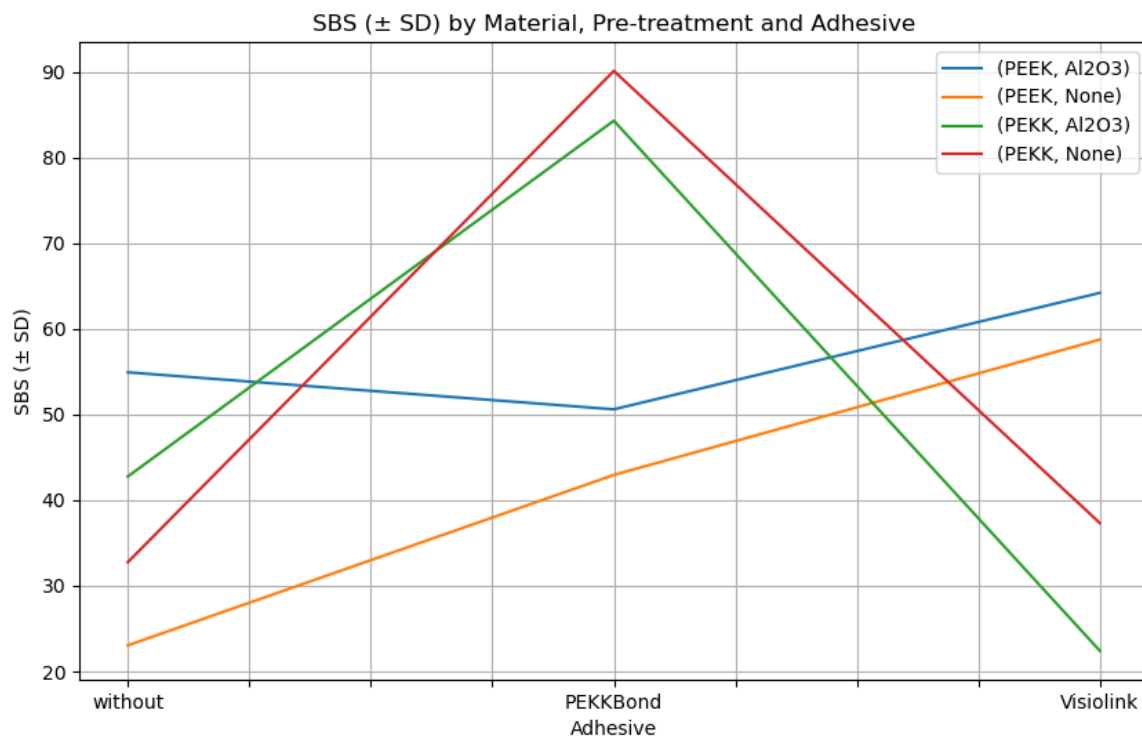


Figure 1. The graph shows the SBS values (with standard deviations) for different adhesives.

Similarly, in the PEKK group, the use of PEKKBond significantly increased the SBS values for both pre-treatment groups ($p < 0.05$), without differences between them ($p > 0.05$). In the air abrasion PEKK specimens, Visio.link significantly reduced the bond strength, and the values were lower than those observed in the controls ($p < 0.05$) (Table 3).

4. Discussion

The null hypothesis of this study, which proposed no differences in the SBS of two adhesive systems when applied to both polymers, was rejected. The results revealed that Visio.link exhibited superior performance on the PEKK surface, whereas PEKKBond significantly increased the SBS of PEKK samples. Furthermore, the impact of air abrasion was only evident in controls without adhesive. These findings are consistent with previous studies [5,7,9,12] and the recommendations of the manufacturers.

Successful bonding is a multifaceted process that relies on various interactions involving chemical, physical, and mechanical factors [15]. The observed results appear to be influenced by the chemical interactions between the adhesive components and the surface of each polymer [16]. For instance, Visio.link contains methyl methacrylate (MMA) and dimethacrylate monomers, including pentaerythritol triacrylate (PETIA) [7,17,18]. PETIA acts as a solvent on the PEEK surface, facilitating interaction with MMA monomers [7,16,17]. This, in turn, leads to surface swelling, while the dimethacrylate monomers provide PEEK with two methyl groups, creating potential bonding sites for the composite resin coating [17,18]. This chemical connection may be responsible for the higher SBS values observed on the PEEK surface when Visio.link was applied [18].

The compositions of PEKKBond and Visio.link share similarities, but the precise composition of PEKKBond has not been fully disclosed by the manufacturer. It is known that PEKKBond contains methylmethacrylate, which can dissolve the PEKK surface, thereby facilitating composite bonding [19]. However, the additional ketone group present in the PEKK molecular chain seems to create a chemical barrier that affects the efficacy of PETIA. This may explain why Visio.link did not perform efficiently on PEKK surfaces. A previous

study [7] also reported lower tensile bond strength (TBS) when PEEKBond was applied to PEEK surfaces. According to the authors, PEEKBond requires prior air abrasion at 0.4 MPa to achieve TBS results similar to those of Visio.link when applied to the PEEK surface [7]. In our study, prior air abrasion did not significantly impact the SBS values observed in PEKK samples. Unfortunately, it is difficult to understand the chemical interactions between the PEKKBond and PEKK surface closely. It has been speculated that MMA monomers in PEKKBond contribute to the swelling of the dissolved PEKK surface, while the dimethacrylate monomers provide a connection to the veneering composites by offering two methyl groups as binding sites [10]. This phenomenon may account for the remarkable performance of PEKKBond when applied to PEKK surfaces. Moreover, PEKK consistently demonstrated higher SBS values in comparison to PEEK, even without the need for pre-treatment or adhesive conditioning. This characteristic renders PEKK a preferred choice for the fabrication of crowns, fixed dental prostheses, and removable prostheses [15,20].

While the literature has highlighted aluminum oxide air abrasion as an effective method for enhancing surface roughness, improving the adhesion of polymers [2,7,15], our study presents a noteworthy observation. We found that air abrasion significantly increased the SBS of PEEK ($p < 0.0001$), but it did not yield the same level of effectiveness for PEKK ($p = 0.164$) (Table 2). These disparities in performance can be attributed, once again, to the additional ketone chain in PEKK, which appears to heighten surface resistance and diminish the efficacy of pre-treatment with air abrasion [6].

It is widely acknowledged that *in vitro* studies aimed at assessing the long-term durability of bonding should replicate the full range of oral environmental conditions by the use of artificial aging methods [21]. In the current study, our approach was limited to storing the specimens in distilled water at 37 °C for 24 h, without subjecting them to thermocycling. It is a well-established fact that thermocycling poses a more rigorous challenge, and can significantly reduce SBS values [21–23]. Therefore, future research that takes into account the oral cavity environment is essential for gaining a more comprehensive understanding of bonding behavior, particularly when considering the use of PEKKBond on PEKK surfaces.

In this study, the choice of 5 bar sandblasting pressure was based on the need to enhance the surface roughness of PEEK and PEKK to improve adhesion. However, previous authors have suggested that higher pressures and larger alumina particles may damage PEEK surfaces by increasing hydrophobicity and embedding alumina residues, potentially affecting bond durability [24]. According to these authors, the use of larger blasting particles at high pressure could mechanically degrade the PEEK surface [24]. While our study did not explicitly assess structural damage, potential surface alterations, including the exposure of TiO₂ fillers in PEEK, should be considered when using high-pressure abrasion. Future research should investigate whether lower pressures (e.g., 2–3 bar) could achieve comparable shear bond strength (SBS), while minimizing potential surface damage, when combined with the adhesive systems tested here.

Another study confirmed that sandblasting significantly improves the shear bond strength of PEEK compared to a polishing protocol alone, aligning with our methods [25]. However, while the authors used 2 bars with a 1 cm distance, we used 5 bars with 10 cm distance. Despite this difference, both studies are comparable. In addition, the previous study highlights the role of MMA-containing adhesives in enhancing SBS, suggesting that the functional monomers in our adhesive systems (MMA, UDMA, PETIA) likely contributed to adhesion [25]. Additionally, their results show that thermocycling negatively affected SBS, emphasizing the importance of evaluating long-term bond durability. Since our study did not include aging tests, we acknowledge this as a limitation, and recommend that future research incorporate thermocycling or water storage tests to assess bond stability.

over time. Furthermore, additional analysis of water contact angles and SEM imaging could help to determine the balance between mechanical interlocking and chemical adhesion.

The differences in bonding performance between Visiolink for PEEK and PEKKbond for PEKK can be attributed to the specific chemical compositions of these adhesives and how they interact with the surfaces of PEEK and PEKK. Visiolink, which contains MMA, dimethacrylates (PETIA), 2-propenoic acid, ligroin, activators, and stabilizers, performed better for PEEK, likely due to the strong interaction between MMA and the PEEK surface. MMA enhances wettability and promotes adhesion through polymerization into PMMA, forming a polymerized layer that increases mechanical interlocking [26]. Additionally, PETIA, a crosslinking agent, may have contributed to improved bond strength by forming a highly crosslinked polymer network, reinforcing adhesion [26]. PEKKbond, containing UDMA, MMA, stabilizers, and activators, performed better for PEKK, which may be linked to UDMA's superior mechanical properties. UDMA has lower polymerization shrinkage than MMA. We could have tested the hypothesis that it would last longer than MMA, but aging was not performed. The structural differences between PEEK and PEKK, particularly the higher ketone content in PEKK, could also affect their wettability and interaction with bonding agents. The higher ketone content in PEKK may enhance its compatibility with UDMA, leading to stronger and more durable bonding. This aligns with previous findings that primers containing both MMA and UDMA demonstrated higher SBS values and lower degradation after thermocycling, compared to MMA-only primers [26].

Although this study provides valuable insights into the comparison of two adhesive systems in terms of shear bond strength, certain limitations must be acknowledged. Using distilled water instead of artificial saliva with varying pH conditions may not fully replicate the intraoral environment, potentially affecting adhesive performance. Future studies should consider testing in artificial saliva to better simulate clinical conditions. Additionally, the absence of surface characterization techniques such as SEM, AFM, and FTIR limits our understanding of adhesive interactions, particularly in terms of roughness, morphological changes, and chemical bonding. The study's *in vitro* nature and lack of artificial aging conditions warrant further investigation. Future research should incorporate thermocycling, long-term water storage, and advanced surface characterization to better understand the durability and chemical interactions of these adhesive systems. Exploring alternative surface treatments, such as plasma or laser modifications, may also provide new insights into optimizing bonding performance. While these factors do not diminish our findings, they highlight opportunities for further research.

5. Conclusions

This study demonstrates that the choice of adhesive system plays a crucial role in achieving optimal bond strength for PEEK and PEKK substrates. Visiolink exhibited superior adhesion to PEEK, likely due to its MMA-PETIA interactions, while PEKKbond was significantly more effective for PEKK, potentially owing to its compatibility with the ketone-rich PEKK structure and lower polymerization shrinkage. These findings highlight that material-specific adhesives should be selected, rather than assuming interchangeability. Moreover, while air abrasion enhanced bond strength in PEEK, its impact on PEKK was minimal, suggesting that surface modifications must also be made with consideration of specific polymers. Adhesive selection and surface treatment strategies should be carefully chosen and combined to enhance the durability and clinical effectiveness of adhesive restorations using high-performance polymers.

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