

Impact of Resin Cement and Adhesive Systems on the Bond Strength of Polyetheretherketone Dental Restorations

Rezin Simanların ve Adeziv Sistemlerin Polietereterketon Dental Restorasyonlarının Bağlanma Dayanımı Üzerindeki Etkisi

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Keywords

Adhesive, PEEK, shear bond strength, resin cement

Anahtar Kelimeler

Adeziv, PEEK, makaslama bağlanma dayanımı, rezin siman

Received/Geliş Tarihi : 05.07.2021

Accepted/Kabul Tarihi : 31.07.2021

doi:10.4274/meandros.galenos.2021.55823

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Abstract

Objective: This study investigated the effect of adhesive systems and resin cements on the shear bond strength (SBS) of poly (ether ether ketone).

Materials and Methods: 60 disc-shaped PEEK samples were divided into 2 according to the adhesive system to be applied (n=30): (1) Visio.link; (2) Scotchbond Universal and further divided according to resin cement (n=10): (1) Panavia V5; (2) Panavia SA Universal; (3) Variolink Esthetic cement. Resin cements with a diameter of 3 mm and a height of 2 mm were bonded to the adhesive-applied specimens. After thermal-cycling (5,000x), SBS was measured, and failure types (cohesive, adhesive and mixed) were evaluated. Data were analyzed by two-way ANOVA and Tukey HSD test for pairwise comparisons (p<0.05).

Results: No significant differences were found among the adhesives. Variolink Esthetic showed the lowest SBS, followed by Panavia SA Universal and Panavia V5 cement, respectively. Only 21.4% of SBS could be explained by the cement, the interaction of adhesive and cement, or the adhesive. The partial eta-squared values revealed that cement had the highest effect on SBS values (0.244). All the groups showed predominant adhesive failure.

Conclusion: Chairside adhesives showed similar bond strength to standard PEEK adhesives, and all the cement were above the minimum acceptable bonding strength according to ISO 10477, but only dual-cure resin cement was clinically acceptable. Therefore, cementation with dual-cure resin cement following the universal adhesive application is a safe bonding procedure that can be applied in the clinic for the cementation of PEEK restorations.

Öz

Amaç: Bu çalışmada, adeziv sistemlerin ve rezin simanların polietereterketonun (PEEK) makaslama bağlanma dayanımı (SBS) üzerine etkisinin araştırılması amaçlanmıştır.

Gereç ve Yöntemler: Altmış adet disk şeklinde PEEK örnek üretildi ve uygulanacak adeziv sistemine göre (1) Visio.link ve (2) Scotchbond Universal olarak 2 gruba ayrıldı (n=30). Daha sonra her grup uygulanacak rezin siman tipine göre (1) Panavia V5, (2) Panavia SA Universal, (3) Variolink Esthetic siman olarak tekrar 3 gruba ayrıldı (n=10). Adeziv uygulanan örnekler 3 mm çapında 2 mm yüksekliğinde rezin simanlar bağlandı. Örnekler termal siklus uygulandı (5.000x) ve ardından SBS testi yapıldı. Başarısızlık tipleri (adeziv, koheziv ve miks) değerlendirildi. Veriler, iki yönlü ANOVA ve post-hoc Tukey HSD testi kullanılarak analiz edildi (p<0,05).

Bulgular: Simanlar arasında istatistiksel olarak anlamlı bir farklılık bulunmamıştır. Variolink Esthetic, en düşük SBS değerini gösterdi, bunu sırasıyla Panavia SA Universal ve Panavia V5 simanları izledi. SBS'nin sadece %21,4'ü siman, adeziv veya siman ve adezivin etkileşimi ile açıklanabildi. Kısmi eta-kare değerleri, SBS değerleri üzerindeki en yüksek etkiye (0,244) simanın sahip olduğunu gösterdi. Tüm gruplar ağırlıklı olarak adeziv başarısızlık gösterdi.

Sonuç: Klinikte kullanılan adeziv sistemleri, standart PEEK adezivlerine benzer bir bağlanma dayanımı göstermiştir. Tüm simanlar, ISO 10477'ye göre kabul edilebilir minimum bağlanma kuvvetinin üzerindedir, ancak yalnızca dual-cure siman klinik olarak kabul edilebilir bulunmuştur. Bu nedenle üniversal adeziv uygulamasını takiben dual-cure rezin siman ile yapılan simantasyon, PEEK restorasyonlarının simantasyonu için klinikte uygulanabilecek güvenli bir bağlanma prosedürü olabilir.

Introduction

Due to the increasing demand for the fabrication of metal-free restorations and advances in digital materials and technology, computer-aided design (CAD)/computer-aided manufacturing (CAM) high-impact polymers have been introduced to the market. These materials can be used as alternative materials to zirconia and titanium as dental framework materials or abutments to support single crowns to full-arch reconstructions (1-4). Polyetheretherketone (PEEK) is a semi-crystalline and thermoplastic high-performance polymer that has superior properties, such as heat resistance, chemical and mechanical strength, (5) and low solubility and water absorption values (6). On the other hand, due to its hydrophobic surface and low surface energy PEEK shows low adhesion to resin-based luting materials (5,7,8). Therefore, standard protocols for adhering resin-based composites to prosthetic substrates have not been effective for PEEK polymers, and the long-term adhesion of PEEK-based structures is still an issue (9). To overcome this problem, previous studies have evaluated the bonding between PEEK material and resin by applying different surface treatments, such as sandblasting, silica coating, surface piranha etching, and sulfuric acid (7,8,10-12). The minimum acceptable bonding strength of resin-based materials is 5 MPa, according to ISO 10477 (13). However, previous studies have reported that shear bond strength (SBS) values above 10-12 MPa are clinically acceptable for bonds between PEEK and resin-matrix composites (7,9,14,15). Studies have found that sandblasting on the PEEK surface improves the SBS with resin and have suggested this as one of the best initial pretreatment alternatives for PEEK surfaces. Surface treatments increase the micromechanical bonding of the PEEK surface, but still, additional adhesive applications are required to achieve a strong PEEK-resin bond (13).

In recent years, more user-friendly, less technically sensitive, and more simplified adhesive systems have been developed in line with clinicians' demands. For this purpose, universal adhesive systems have been introduced to the market (16).

In the course of the cementation of different superstructure materials, clinicians may prefer different cements due to their ease of use, lack of additional cementation step, or color stability. However, there is no established protocol regarding the bond strength of PEEK material and resin cements. Therefore, this study aimed to investigate the effect of different adhesives and resin cements with different polymerization properties on the SBS of sandblasted PEEK material. The null hypothesis was that different adhesive resin cements and adhesive material types would not affect the SBS of the PEEK material.

Materials and Methods

This *in vitro* study was approved by the Ethical Committee of Pamukkale University (decision no: 04, date: 16.02.2021). Table 1 shows the compositions and details of the materials used in this study. The dimensions of the PEEK specimens were designated and entered into the software program (CEREC 4.4; Dentsply Sirona, York, PA, USA) and produced by using the CAD/CAM method (10x2 mm) (n=60). All samples were embedded in chemically polymerized acrylic resin (Meliodent, Heraeus Kulzer, South Bend, IN, USA) with one side up and polished with a polishing machine (Presi Mecapol P230, Presi, Grenoble, France) under running water using 400, 800, 1000, and 1500 grain silicon carbide papers at 150 rotations/min for 40 s. Before initiating the bonding procedure, all specimen surfaces were sandblasted with 110 µm Al₂O₃ for 15 seconds at 0.2 MPa at an angle of 45°. Subsequently, the specimens were cleaned with 10% alcohol for 180 s in an ultrasonic cleaner and air dried. The 60 PEEK specimens were divided randomly into two test

Table 1. Manufacturers and compositions of the products used in this research

Coprapeek		Pure 100% PEEK	White Peaks Dental Systems GmbH & Co. KG., Essen, Germany
Visio.link		PETIA [†] , MMA [*] , Photoinitiators	Bredent GmbH & Co KG, Senden, Germany
Scotchbond Universal Adhesive		Bis-GMA [‡] , HEMA [¶] , MDP [§] , polyalkenoic acid copolymer, filler, ethanol, water, silane, initiators	3M ESPE, Seefeld, Germany
Panavia SA Universal	A	Monomer (Bis-GMA, 10-MDP, HEMA, TEGDMA ^{**} , other methacrylate monomer), filler (silanated colloidal silica, silanated barium glass filler), pigment, initiator	Kuraray Noritake, Tokyo, Japan
	B	Methacrylate monomer, filler (silanated sodium fluoride, silanated barium glass filler, aluminium oxide), silane coupling agent, pigment, accelerator, others	
Panavia V5	A	TEGDMA, Bis-GMA, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, accelerators, colloidal silica, silanated, fluoroaluminosilicate silanated barium glass filler, glass filler, initiators	Kuraray Noritake, Tokyo, Japan
	B	Bis-GMA, dl-camphorquinone, silanated aluminium oxide filler, silanated barium glass filler, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, pigments, accelerators	
Variolink Esthetic LC		Bis-GMA, TEGDMA, UDMA ^{††} , ytterbium trifluoride, boroaluminofluorosilicate glass, spheroidal mixed oxide, stabilizers, benzoylperoxide, pigments, Ivocerin [™]	Ivoclar Vivadent, Schaan, Liechtenstein

PEEK: Polyetheretherketone, ^{*}Methyl methacrylate, [†]Pentaerythritol triacrylate, [‡]Bisphenol a dimethacrylate, [§]Methacryloyloxydecyl dihydrogen phosphate, [¶]Triethylene glycol dimethacrylate, ^{**}Urethane dimethacrylate, ^{††}2-hydroxyethyl methacrylate

groups according to adhesive types (n=30): 1- Visio.link (Bredent GmbH & Co KG, Senden, Germany); 2- Scotchbond Universal (SU); 3- M, Seefeld, Germany). In group 1, Visio.link was applied on the PEEK surface with a brush and light cured for 90 s in a dual mode light curing unit (Labolight Duo, GC Europe, Leuven, Belgium), and in group 2, SU was applied and rubbed in for 20 s. Subsequently, a gentle stream of air was directed over the liquid for about 5 s, and the samples were light cured for 10 s with a light-emitting diode lamp (VALO[™] Cordless, Ultradent, South Jordan, UT, USA). After the adhesive application was completed, each specimen group was further divided into three groups (n=10) according to resin cement type: self-adhesive (Panavia SA Cement Universal, Kuraray Noritake Dental, Tokyo, Japan), dual cure (Panavia V5, Kuraray Noritake Dental, Tokyo, Japan), and light cure (Variolink Esthetic LC, Ivoclar Vivadent, Schaan, Liechtenstein). For the SBS measurements, a disc-shaped silicon mold with an inner diameter of 3 mm and a height of 2 mm was manually filled with one of the resin cements and subsequently polymerized according to the manufacturer instructions for the PEEK substrate. Then, all specimens were stored in

distilled water at 37 °C for 24 h and aged by thermal cycling for 5,000 cycles between 5° to 55 °C. After the aging procedure, the bonded specimens were placed in a universal testing machine and loaded with a crosshead speed of 1 mm/min. The maximum shear load was recorded immediately before debonding. The following formula was used to calculate the SBS data: fracture load/bonding surface area=N/mm²=MPa. Failure types were examined under a reflected-light microscope at 20x magnification. Failure modes were classified as adhesive failure, cohesive failure in adhesive resin cement, cohesive failure in PEEK, or mixed failure.

Statistical Analysis

Statistical analysis was performed by using SPSS v23 (IBM Armonk, NY, USA). The Kolmogorov-Smirnov test verified the existence of normal distribution, and the results were analyzed using Two-Way ANOVA (p≤0.05). The Tukey HSD test was used to compare the impact of different resin cements and types of adhesive (p≤0.05). General linear model with partial eta-squared statistics calculated the effect of the “cement” and “adhesive” parameters and their interaction on the SBS. The partial eta-squared value

reports the practical significance of each variable explained by the effect. Higher partial eta-squared values indicate a larger amount of variation calculated by the model effect.

Results

The mean and standard deviation values of SBS from resin cements in the test adhesives are illustrated in Figure 1. The Two-Way ANOVA results showed that the SBS was significantly affected by the cement type ($p < 0.001$), but the adhesive type was not significant ($p = 0.062$). The interaction between cement and adhesive was also found not to be statistically significant ($p < 0.987$) (Table 2). In terms of adhesives, Visio.link showed higher SBS values (12.32 MPa) than SU (10.41 MPa). The Tukey HSD test showed that the mean SBS value for the Panavia V5 resin cement group (14.26 MPa) was significantly higher ($p < 0.05$) than those of the Panavia SA cement (10.4 MPa) and Variolink Esthetic LC cement (9.43 MPa) groups. There was no statistical difference between the Panavia SA and Variolink Esthetic LC cements. Cement had the highest effect on SBS values according to the partial eta-squared values (0.244). Table 3 shows the distribution of the failure modes. No cohesive failure was observed among the test groups and adhesive

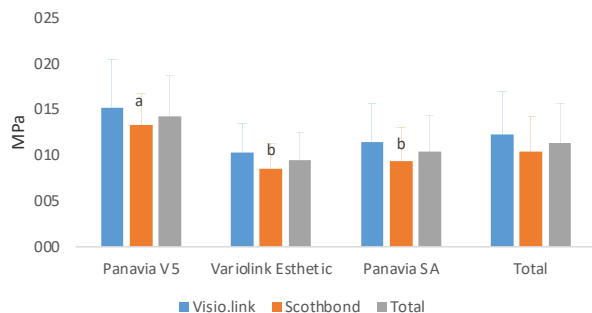


Figure 1. Boxplots of SBS for all tested groups (MPa)

^{a,b}There is no difference between the groups with the same letter, SBS: Shear bond strength

failure was the predominant failure type among the all-test groups.

Discussion

The reliable cementation of fixed dental prosthesis is a key factor in the long-term survival rates of dental restorations. Therefore, this study investigated the effect of different adhesive systems on long-term cementation in combination with varying cements. The null hypothesis of this study was partially rejected, as the dual-cure cement showed higher SBS than other cements when bonded to the PEEK material, and there were no significant differences between the adhesives.

Studies have shown that different surface treatments, including silane surface conditioning and helium plasma surface modification, do not significantly increase the SBS of PEEK with different veneering resins (14,17). On the other hand, it has been stated that 90-98% sulfuric acid etching increases the SBS of PEEK (18-20). However, these studies state that sulfuric acid is hazardous for clinical practice and chairside use and requires appropriate safety precautions that are not routinely found in dental laboratories or dental practices. Thus, sulfuric acid treatment was not used in the present study due to its low clinical applicability. Airborne-particle abrasion is frequently used in dental laboratories and clinics, especially in the surface preparation processes of oxide ceramics. Surface treatment with 110 μ m aluminum oxide was also reported for increasing the SBS of PEEK to different veneering resins (8,12,14,15,17). It has also been reported that airborne abrasion with 110 μ m Al₂O₃ for 15 seconds at 0.2 MPa is effective for bonding milled PEEK to veneering composite (21).

Some previous studies comparing different adhesive systems have reported that adhesives that contain methyl methacrylate, like Visio.link, improve the bonding characteristics of PEEK (12,22,23).

Table 2. Two-way ANOVA results of SBS with respect to cement, adhesive and interaction between cement and adhesive

	Sum of squares	df	Mean squares	F	p value	Partial eta-squared
Cement	261.638	2	130.819	8.695	0.001	0.244
Adhesive	54.489	1	54.489	3.622	0.062	0.063
Cement* adhesive	0.382	2	0.191	0.013	0.987	0.000

R²: 0.280, Corrected R²: 0.214 *Interaction. SBS: Shear bond strength

Table 3. Fracture types in research groups

Group	Mode of failure (percentage)		
	Adhesive	Cohesive	Mix
Visio.link + Panavia V5	6	0	4
Visio.link + Variolink Esthetic LC	8	0	2
Visio.link + Panavia SA Universal	10	0	0
Scotchbond + Panavia V5	9	0	1
Scotchbond + Variolink Esthetic LC	9	0	1
Scotchbond + Panavia SA Universal	10	0	0

However, Visio.link is a labside bonding material and needs a polymerization unit, and furthermore, there is no standard polymerization procedure because of the variety of polymerization units used in dentistry. Therefore, it is important to find an adhesive with clinical applicability for crowns cemented on substrates. In the present study, SU (one-bottle system) showed similar results to Visio.link. Two previous studies (8,24) reported similar outcomes for SBS with the use of Visio.link versus SU. The authors reported that this may be attributed to the content of SU, which includes 10-methacryloyloxydecyl-dihydrogenphosphat (MDP) monomers, dimethacrylate resins, and 2-hydroxyethylmethacrylate (HEMA), and since it is a single-bottle adhesive, it is immediately in contact with the PEEK surface.

In the present study, all of the cements were above the minimum acceptable bonding strength according to ISO 10477, and Panavia V5 was also above the clinically acceptable limit. All three resin cements used in this study contain bis-GMA and TEGDMA, stabilizers, fillers, and initiators. Panavia V5 also contains dimethacrylate polymers; Variolink Esthetic LC additionally contains urethane dimethacrylate; and Clearfil SA Cement Universal has HEMA, which is known from the literature to not provide long-term bonding, (24) and an MDP monomer, which means that, unlike for the other two cements, no prior bonding to the tooth substrate is required. Similar to the findings of Schmidlin et al. (7), this study found that conventional resin cements containing dimethylmethacrylate monomers showed higher SBSs than self-etching composite resin cements containing

adhesive phosphate monomers (MDPs). Although self-adhesive resin cements have the advantage of being economical and time saving, (10) they do not seem suitable for the bonding of permanent PEEK restorations. In addition, Sproesser et al. (20) investigated the effect of different sulfuric acid etching times on work of adhesion by using SBS and different surface parameters, such as contact angle measurement and surface free energy. As a result, they found that work of adhesion values could not clearly distinguish the bond between two materials. This study revealed a procedure that provides sufficient bonding for FPDs applied on PEEK infrastructures and can be applied in the dental clinic without the need for separate equipment. Nevertheless, the findings of the present study are insufficient for explaining the bonding between PEEK and resin cement when coefficient of determination values are considered. The first limitation of this study was using only one surface pretreatment. In addition, considering that cement type was the main effect on SBS in this study, future studies should evaluate cements with different ingredients and different surface pretreatments that can be applied in the dental clinic.

Conclusion

Chairside adhesives showed similar SBS values to standard PEEK adhesives, and all tested resin cements showed acceptable bonding according to the ISO 10477 standard, but only dual-cure resin cement was clinically acceptable. Therefore, cementation with a dual-cure resin cement following universal adhesive application is a safe bonding procedure that can be applied in clinical practice for the cementation of PEEK restorations.

Ethics

Ethics Committee Approval: This *in vitro* study was approved by the Ethical Committee of Pamukkale University (decision no: 04, date: 16.02.2021).

Informed Consent: *In vitro* study.

Peer-review: Externally peer-reviewed.

Authorship Contributions

Surgical and Medical Practices: I.K., S.C.Ş., H.L.E.S., Concept: I.K., S.C.Ş., H.L.E.S., Design: I.K., S.C.Ş., H.L.E.S., Data Collection or Processing: I.K.,

S.C.Ş., Analysis or Interpretation: I.K., S.C.Ş., H.L.E.S., Literature Search: I.K., Writing: I.K., S.C.Ş., H.L.E.S.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study received no financial support.

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