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Shear bond strength of different luting agents to polyether ether ketone

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ABSTRACT

Background: Polyether ether ketone (PEEK) was recently introduced to dentistry. However, difficulty in provision of a strong durable bond is its main drawback. Thus, precise surface treatment and use of a suitable luting agent are imperative for bonding of PEEK restorations. This study aimed to assess the effect of type of luting agent on shear bond strength (SBS) of PEEK.

Materials and Methods: In this *in vitro* study, 60 square-shaped PEEK samples were fabricated and sandblasted with 110 μm Al_2O_3 particles. The samples were then divided into four groups based on the type of cement used (n = 15): zinc phosphate cement, Panavia F2, Panavia V5, and resin-modified glass-ionomer (RMGI) cement. After bonding, the samples were thermocycled for 5000 cycles. The SBS was measured by a universal testing machine. The surface of samples was inspected under a video measuring machine to determine the mode of failure. Data were analyzed using the Kruskal–Wallis test via SPSS version 24 (α = 0.05).

Results: RMGI did not bond to PEEK. The SBS values were 4.02 ± 2.87 megapascals (MPa) for Panavia V5, 10.84 ± 6.05 MPa for Panavia F2, and 10.50 ± 2.88 MPa for zinc phosphate. The SBS in the Panavia V5 group was significantly lower than that in the Panavia F2 (P = 0.001) and zinc phosphate (P < 0.001) groups. No significant difference existed between the Panavia F2 and zinc phosphate groups in this respect (P > 0.05).

Conclusion: Panavia F2 resin cement and zinc phosphate conventional cement provided the highest bond strength to PEEK, while RMGI did not bond to PEEK.

Key Words: Dental cement, glass-ionomer cement, resin cement, shear strength

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INTRODUCTION

Polyether ether ketone (PEEK) was recently introduced to dentistry.[1] PEEK is a thermoplastic, semi-crystalline polymer from the polyaryl ether ketone family with high performance, which has a linear aromatic structure.[2] It has a number of advantages including high mechanical properties such as high melting temperature and high fatigue resistance, high thermal and chemical resistance, optimal biocompatibility, tooth-like appearance, and easy shaping with bur.[3,4] Among the available thermoplastic polymers, PEEK has lower water sorption than polymethyl methacrylate.[5] In contrast to composite resin and polymethyl methacrylate, PEEK does not undergo polymerization shrinkage.[6]

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It has optimal dimensional stability[4] and possesses a modulus of elasticity in-between that of cortical and cancellous bones.[7] Moreover, PEEK is radiolucent and is therefore compatible with the imaging modalities such as computed tomography, magnetic resonance imaging, and radiography,[8] allowing examination, diagnosis, and treatment of disease conditions without the need for retrieval or replacement of PEEK restorations.[9] In dentistry, PEEK is mainly used for dental implants, temporary abutments, healing abutments, implant-supported bars, and dental clasps.[10] Moreover, due to nonmetallic color, low weight, and high strength, it can be used as a rigid material in removable and fixed partial dentures (FPDs).[11]

The clinical success of FPDs highly depends on their cementation process.[12] Schwartz *et al*. demonstrated that loss of crown retention was the second most common cause of failure of crowns and the conventional FPDs.[13] Although creation of resistance and retention forms is among the primary principles of tooth preparation, cement are still required for a strong and durable bond between the restoration and the underlying tooth structure.[14] They also increase the fracture resistance of the restored teeth and the restorations.[15] Cement also serve as a barrier against microbial leakage and seal the interface between heterogeneous materials.[14] The main clinical drawback of PEEK is that it cannot form a strong and durable bond to dental materials due to its low surface energy and high resistance to chemical surface treatments[6] because the chemical and aromatic structure of ketone and other constituents of PEEK provide an inert surface with suboptimal bonding capability.[16,17] Thus, precise surface treatment and use of an appropriate cement are imperative for bonding of PEEK restorations.[18] Although resin cement have been used for cementation of PEEK restorations, the manufacturer claims that different cement types such as zinc phosphate, glass-ionomer, and self-adhesive cement can be used for cementation of PEEK restorations. It is important since resin cement cannot be used when ideal isolation cannot be achieved.[19] Furthermore, the use of resin cement may not be suitable for implant restorations, because one criterion that needs to be addressed in cement selection for implant restorations is the easy removal of excess cement.[20] The possible risk of damage to titanium implants should also be considered.[21] According to a study by Agar *et al*., zinc phosphate cement residues can be easily removed while resin cement are the most difficult to remove.[22] Thus, the use of traditional cement may be the solution for such cases as studies on the use of these cement for zirconia restorations have reported positive results.[23-25] Moreover, it has been shown that adhesive properties that are important for stability of a restoration are influenced by the type of resin cement.[9] Thus, selection of the type of resin cement is important as well.

Many studies have assessed the efficacy of different surface treatments for providing a stronger bond between PEEK and resin cement.[6,9,26-29] Moreover, several studies have evaluated the use of different resin cement for this purpose;[9,11,27,30,31] however, further studies are still required on this topic. Nonetheless, no previous study has assessed the use of traditional cement for cementation of PEEK restorations in comparison with resin cement. Thus, this study aimed to compare the shear bond strength (SBS) of different luting agents to PEEK. The null hypothesis of the study was that no significant difference would be found in SBS of PEEK to different cement.

MATERIALS AND METHODS

In this *in vitro* study, sample size estimation was performed by PASS version 11 by using one-way ANOVA sample size calculation formula and considering the mean values of 2.97, 1.88, 2.44, 1.03, and 0.43 and standard deviation of 1.80 according to a previous study,[32] assuming the statistical power of 0.84, and error rate of 0.05. The minimum sample size was calculated to be 11. However, 15 samples were included to increase the study power.

Sixty square-shaped samples measuring 7 mm in length and width and 2 mm in thickness were fabricated from PEEK discs (breCAM Bio-HPP: Bredent, Senden, Germany) using a cutting machine under water coolant. The bonding surface of the samples was polished with 400–1000-grit silicon carbide papers for 10 s with finger pressure. Each sample was separately mounted in auto-polymerizing acrylic resin (Technovits 4000; Heraeus Kulzer GmbH and Co., Wehrheim, Germany) such that only the bonding surface measuring 7 mm × 7 mm remained exposed. Next, the samples were randomly divided into four groups (n = 15) based on the type of cement to be used by the block randomization method with block length of four: zinc phosphate cement (Richter and

Hoffmann: Berlin, Germany), Panavia V5 dual-cure resin cement (Kuraray, Tokyo, Japan), Panavia F2 dual-cure resin cement (Panavia F2: Kuraray, Osaka, Japan), and resin-modified glass-ionomer (RMGI; GC Fuji II: GC America, Illinois, USA) cement. Table 1 presents the details regarding the cement used in this study.

Prior to the initiation of bonding process, all samples were cleaned in an ultrasonic bath containing deionized water for 10 s and air-dried. Next, the bonding surface of the samples was sandblasted with 110 μm aluminum oxide particles (2.5 bar pressure, 3 cm distance, 10 s time, and 45° angle). All cement were used at room temperature (23°C ± 1°C) and relative humidity (50% ± 5%) according to the manufacturer's instructions. A total of 60 plastic cylinders (Tygon tubes: Saint-Gobain, Courbevoie, France) with an internal diameter of 3.5 mm and height of 5 mm were obtained and filled with composite resin by 2 mm thickness (Filtek Z250: 3M ESPE, St. Paul, MN, USA). Composite resin was applied in two layers, and each layer was cured for 20 s using a light-curing unit (Signum: Heraeus Kulzer, Hanau, Germany). The remaining part of the cylinders was filled with the respective cement according to the manufacturers' instructions. The cylinders were then placed on PEEK discs upside down [Figure 1]. All procedures were performed by an experienced clinician. For the application of zinc phosphate and RMGI cement on the bonding surface, no adhesive layer was applied on the bonding surface according to the manufacturers' instructions. However, for the application of Panavia F2 and Panavia V5 resin cement, a thin layer of adhesive (Visio.link: Bredent, Senden, Germany) was applied on the PEEK surface with one stroke of a microbrush according to the manufacturers' instructions and immediately light cured for 90 s using a light-curing unit (Labolight: LV-III, GC, Tokyo, Japan). Excess cement was removed from the margins at the bonding surface using a disposable microbrush.

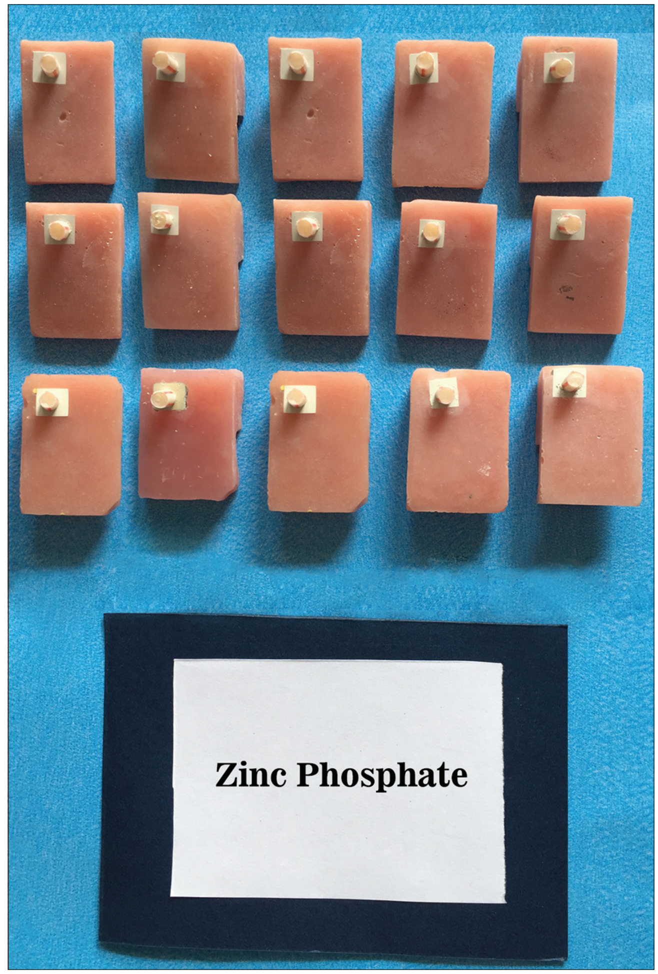

Figure 1: Samples prepared in zinc phosphate group prior to thermocycling.

Table 1: Luting agents used in this study

Type	Cement	Manufacturer	Composition
Zinc phosphate	Hoffmann's zinc phosphate	Hoffmann Dental Manufacturing	Powder: Zinc oxide, magnesium oxide Liquid: O-phosphoric acid
Dual polymerizing adhesive resin cement	Panavia F2.0	Kuraray Noritake Dental	MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators, silanated barium glass filler, surface treated sodium fluoride, accelerators, pigments
Dual polymerizing adhesive resin cement	Panavia V5	Kuraray Noritake Dental	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica, silanated aluminum oxide filler, dl-camphorquinone, pigments
RMGI	Fuji II LC	GC America	Liquid: 20%-22% polyacrylic acid, 30%-40% HEMA, 5%-7% 2,2,4, trimethyl hexamethylene dicarbonate, 4%-6% TEGDMA, 5%-15% proprietary ingredient Powder: Aluminosilicate glass

RMGI: Resin modified glass ionomer, HEMA: Hydroxyethylmethacrylate, TEGDMA: Triethyleneglycol-dimethacrylate, Bis-GMA: Bisphenol A diglycidylmethacrylate, LC: Light-cured, MDP: Methacryloyloxydecyl dihydrogen phosphate

Next, the samples cemented with resin cement were light cured for 40 s using a light-curing unit. The light intensity of the device was controlled with Optilux radiometer to be 430 mW/cm^2.

After the bonding process, all samples were incubated in an aqueous medium at 37°C ± 1°C for 24 h. The samples were then thermocycled for 5000 cycles between 5°C and 55°C with a dwell time of 20 s in each bath [Figure 2]. The samples that were debonded during thermocycling were categorized as pretest failure with 0 megapascal (MPa) bond strength.

The SBS was measured using a universal testing machine (STM-20: Santam, Tehran, Iran). The load was applied by the crosshead tip at a speed of 1 mm/min [Figure 3]. The maximum load at failure in Newtons was divided by the surface area in square-millimeters (mm^2) to report the bond strength in MPa.

In order to determine the failure mode, the debonded surface was inspected using a video measuring machine (Easson, Guangdong, China) at ×88.4 magnification. Accordingly, the mode of failure was categorized as adhesive (no cement remnant on the PEEK surface), cohesive (fracture in PEEK or cement), and mixed (a combination of adhesive and cohesive failures).

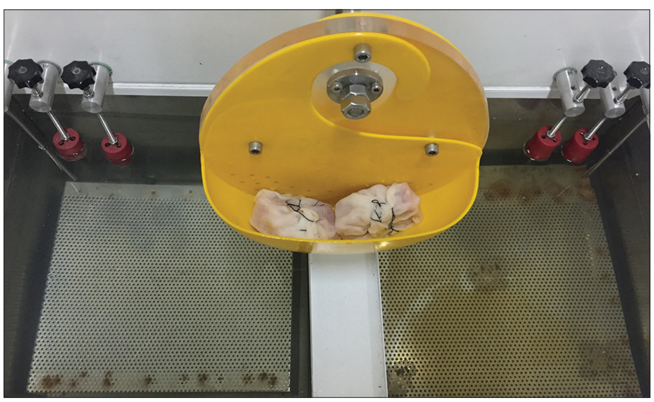

Figure 2: Thermocycling of cemented samples.

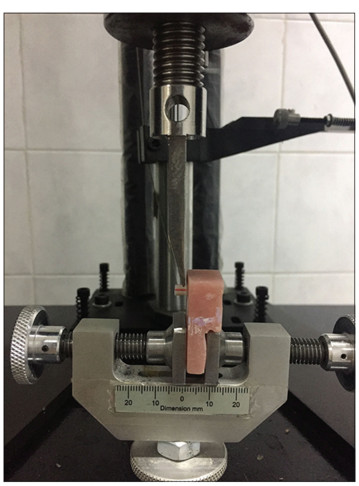

Figure 3: Shear bond strength test.

Data were analyzed using SPSS version 24 (SPSS Inc., Chicago, IL, USA). Normal distribution of data was evaluated using the KolmogorovSmirnov test, which revealed that data were not normally distributed. Thus, the mean SBS of the groups was compared using nonparametric Kruskal–Wallis test. Pairwise comparisons were carried out using the Mann–Whitney test with Bonferroni adjustment. Level of significance was set at 0.05.

RESULTS

All samples cemented with RMGI failed during thermocycling (pretest failure). Thus, they were not included in statistical analysis. The mean and standard deviation of SBS in each group are presented in Table 2. According to the Kruskal–Wallis test, a significant difference existed in the mean SBS of the groups ($P < 0.001$). The maximum SBS was noted in the Panavia F2 group (10.84 ± 6.05 MPa) while the minimum SBS was noted in the Panavia V5 group (4.02 ± 2.87 MPa). Pairwise comparisons of the groups showed that the SBS of the Panavia V5 group was significantly lower than that of the Panavia F2 ($P = 0.001$) and zinc phosphate ($P < 0.001$) groups. No significant difference was noted in SBS of the Panavia F2 and zinc phosphate groups ($P > 0.05$). Evaluation of the samples by the video measuring machine revealed that the mode of failure was adhesive in all samples (between the PEEK surface and cement).

DISCUSSION

Bond failure at the cement-restoration interface is the most common cause of restoration failure, which can lead to development of secondary caries.[31] Thus,

Table 2: Mean, standard deviation, and median of shear bond strength (megapascal) of samples cemented with different luting agents (*n*=5)

Group	*n*	Mean±SD (MPa)	Median	Minimum–Maximum
Zinc phosphate	15	10.50±2.88	9.78	3.00–16.29
Panavia F2.0	15	10.84±6.05	9.18	1.92–26.11
Panavia V5	15	4.02±2.87	3.08	1.02–11.46

SD: Standard deviation, MPa: Megapascal

a durable and predictable bond between restoration and tooth structure can guarantee the function and long-term clinical service of restorations.[27] Although an effective bond to PEEK is a prerequisite for its application as a dental material in prosthesis,[17] limited information is available on the bond to PEEK and its durability using different cement types. Thus, this study aimed to measure the SBS of PEEK to different cement. The results showed that Panavia F2 resin cement yielded the highest, and Panavia V5 resin cement yielded the lowest SBS. However, samples cemented with RMGI did not bond to PEEK and were all debonded during thermocycling. Thus, the null hypothesis of the study regarding no significant effect of cement type on SBS to PEEK was rejected.

All bond strength tests have advantages and disadvantages, and no consensus has been reached on any test. However, the SBS test is the most common among all bond strength tests.[33] Tensile tests are also commonly used; however, they can lead to unequal stress distribution.[34] Moreover, sample preparation for tensile test is complex, and if not correctly controlled, torque stresses are generated which can decrease the bond strength.[34] On the other hand, very small samples should be used for microtensile test in order to obtain more uniform stress distribution.[35] Nonetheless, conduction of microtensile test is difficult and it is easily affected by different parameters.[34] Although conduction of microshear test is easier than the microtensile test, its superiority to conventional shear tests has not been confirmed.[36] Shear test is easily performed and is suitable for prediction of the function of dental materials.[34] On the other hand, it is believed that shear stresses comprise a major part of stresses involved in bond failure of restorative materials.[37]

The effect of thermocycling on bond strength of PEEK crowns with different surface treatments can predict the long-term clinical service of PEEK restorations.[6] Evidence shows that thermocycling is an appropriate method for simulation of thermal alterations that occur in the oral environment as the result of eating, drinking, and respiration.[38] Limited studies on the bond to PEEK have performed aging.[6,26,38] In this study, all samples were subjected to repeatable standardized stress. Aging was performed by thermocycling for 5000 cycles corresponding to 4–5 years of clinical service.[39] Many studies have assessed the surface treatments of PEEK, and they have all stated that surface treatment is imperative to enhance wettability and achieve an optimal bond to PEEK.[6,9,26,27,40] Although etching with 98% sulfuric acid yields the best results in achieving a durable bond,[27,30,40] it is highly corrosive and dangerous for chairside use in the clinical setting and cannot be the first choice for surface treatment prior to cementation.[27] A more common and safer method is to use a combination of sandblasting with 50–110 μm Al2O3 particles and application of adhesive systems containing methyl methacrylate,[26] which was employed in this study. Sandblasting increases the surface roughness and subsequently enhances the micromechanical interlocking of the cement.[9] Moreover, it completely removes the organic impurities from the material surface and cleans and activates the surface.[9] Visiolink adhesive was used for resin cement in this study, which contains pentaerythritol triacrylate, methyl methacrylate monomers, and dimethacrylates.[18] It is assumed that pentaerythritol triacrylate dissolves the surface of PEEK, and subsequently, methyl methacrylate monomers cause swelling of the dissolved surface, and eventually, dimethacrylate monomers result in bonding of composite resin to the two methyl groups.[41]

The mode of failure in this study was entirely adhesive, which is the most common mode of failure observed in the literature.[6,9,11,27,30] Tsuka *et al*. used Panavia V5 resin cement in their study for bonding to PEEK. They sandblasted the PEEK surface with AL_2O_3 particles. All failures were adhesive.[11] Song *et al*. assessed the bond strength of posts fabricated from PEEK. The samples that were treated with sandblasting and application of Visiolink and cemented with Panavia F2 mainly showed adhesive failure.[42] No similar study is available on the cement used in this study. On the other hand, comparison of bond strength values reported in studies would be difficult and inaccurate due to the variability in study designs, methodologies, surface treatments, cement types, and methods of assessment of bond strength.

In this study, the bond strength of samples cemented with Panavia F2 was significantly higher than that of Panavia V5. The only difference between these two cement is the presence of methacrylate monomers and phosphate groups in the composition of Panavia F2, which are not present in Panavia V5. The ceramic specific primer of Panavia V5 that contains MDP was not used in this study since its application has not been recommended in the cement manufacturers' instructions for PEEK, and only Visiolink was applied. MDP has a phosphoric acid group that forms optimal bond to ceramic oxides.[43] This may be the possible reason for higher bond strength of Panavia F2 to PEEK although MDP alone is not a key factor for optimal bonding to PEEK and some studies have questioned its efficacy.[9,29] Factors such as the interaction effect of several adhesive components and their amount, viscosity, molecular weight, and penetration depth into PEEK are among other possible factors that affect the bond strength to PEEK.[26] An interesting finding of this study was related to zinc phosphate cement, which yielded the highest bond strength to PEEK after Panavia F2. This finding was in contrast to the results of other studies on other restorative materials[44,45] and calls for further investigations regarding this cement and its interactions with PEEK. All samples bonded with RMGI cement were debonded during thermocycling, which indicates that RMGI is not a suitable cement for bonding to PEEK. This finding may be due to the presence of high amounts of hydroxyethylmethacrylate (HEMA) in its structure. HEMA is a mono-methacrylate, which according to the existing evidence, does not provide an optimal durable bond.[26] Evidence shows that restorations bonded with adhesives containing HEMA are more susceptible to water sorption and subsequent hydraulic degradation.[46]

Relatively small sample size and assessment of limited number of resin cement were among the limitations of this study. Moreover, this study had an *in vitro* design, which cannot perfectly simulate the clinical setting. Thus, generalization of results to the clinical setting is difficult. Long-term clinical studies are required on this topic.

CONCLUSION

Within the limitations of this study, it may be concluded that Panavia F2, zinc phosphate, and Panavia V5 cement yielded the highest bond strength, in descending order. The SBS values of Panavia F2 resin cement and conventional zinc phosphate cement were not significantly different while the SBS of Panavia V5 resin cement was significantly lower than that of the other two cement. Therefore, in case of appropriate preparation of PEEK restorations according to the manufacturer's instructions, the conventional zinc phosphate cement can provide an acceptable bond comparable to that of Panavia F2 resin cement. The specimens cemented with RMGI were debonded during thermocycling; thus, RMGI cannot be suitable for bonding of PEEK restorations. Furthermore, the mode of failure was adhesive in all cement groups.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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