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An in vitro comparative evaluation of bond strength between polyether ether ketone and resin cement using different adhesive systems after various surface pretreatments

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Abstract

Aims: To evaluate and compare shear bond strength between PEEK and resin cement using different adhesive systems after various surface pre-treatments.

Settings and Design: In vitro comparative study

Methods and Material: Seventy two specimens of PEEK were fabricated, polished and divided into 3 pretreatment Groups (n= 24/group): no surface pretreatment (control group), Sand blasting for 15 seconds with 110 µm alumina and sulfuric acid (98%) etching for one minute. Specimens from each group were into divided into subgroups according to three adhesive systems (n= 8 subgroup): no adhesive, MMA based adhesive and Bis-GMA based adhesive system. All specimens were bonded with luting resin cement and incubated in distilled water for 24hrs at 37°C. SBS was measured in Universal Testing Machine (UTM) and bond failure was assessed with stereomicroscope.

Statistical analysis used: Multivariate CRD ANOVA was used to analyze the data. Level of significance was set at $p \leq 0.05$.

Results: PEEK specimens with no surface pretreatment without adhesive system exhibited least SBS and specimens etched with sulfuric acid as surface pretreatment and Bis-GMA based adhesive system exhibited highest SBS. Among all groups, the most frequent failure mode was adhesive failure between the material and the resin luting cement.

Conclusions: Adhesion between PEEK and luting resin cement appears to be micromechanical locking from bonding agent after surface pretreatment.

Keywords: Polyetheretherketone, Shear Bond Strength, Stereomicroscope, Adhesive System, Sulfuric acid, Bond Failure

Introduction

The focus of every prosthetic treatment is set on the rehabilitation of the masticatory, phonetic, and esthetic functions of the patient. Different materials must be considered carefully according to every clinical situation. Due to their new properties and advantages, innovative techniques and materials must be regarded as potential alternatives to traditional materials. There is a great interest and ongoing research with regard to dental polymer which shows better properties than conventional materials Polyaryletherketones (PAEKs) are belongs to family of high-performance thermoplastic resins, which consists of an aromatic backbone molecular chain interconnected by ketone and ether groups. PAEKs have strong strength, fatigue and flexural strength, good stability stability at high temperatures and high wear resistance1. PEEK has very favourable chemical and mechanical properties, which make it attractive for dental applications. These properties created a wide range of indications for PEEK in the field of dental restorations, which has a growing trend in using metal-free materials. It allows magnetic resonance imaging (MRI). Radiation heat does not cause disintegration. Laboratory stages are simple. Biologically when PEEK material and components are examined, no evidence has been shown of cytotoxicity, mutagenicity, carcinogenicity or immunogenicity in the toxic form. It is a biologically inert material2,3. Because of its non-metallic color, low weight and high strength, PEEK can be use as an alternative rigid material in partial dentures and fixed dental prosthesis. Because PEEK's mechanical properties are similar to those of dentin and its enamel, PEEK may have a higher chance of alloy and ceramic refinement. When used in crowns and fixed dental prosthesis for the posterior teeth, PEEK offers advantages in resisting the forces and attrition due to the opposing teeth4. Dental bonding agents provide a link between scheduled dentures and dental support configurations. It also reduces the microleakage of organisms around the dental restorations because it has been implicated in adverse pulpal response and reduce the durability. Adhesive luting cements improves retention; lack of retention is a common cause of fixed prosthesis failure. An adhesive luting cement provides gap-free restoration margins, minimizing microleakage and reduces the risk of secondary caries. It can strengthen the restoration and the remaining tooth structure4. Adhesion between resin-based luting materials used in crowns and fixed dental prosthesis is an indispensable property of dental restorative material. The chemical composition and low energy of PEEK can lead to difficulty connecting the cement elements A durable bond between the Luting material and the PEEK framework is a prerequisite for long-term stable and functional outcomes. A durable bond between the Luting material and the PEEK framework is a prerequisite for long-term stable and functional outcomes. PEEK has low adhesion to resin based luting material. The bond strength of PEEK to resin is low because PEEK possess its chemical inertness, low surface energy and resistance to surface modification. Two methods to obtain a strong bonding performance between the resin and PEEK include the alteration of the PEEK surface and conditioning with an adhesive system to enable the chemical interactions. There are no studies evaluating the use of PEEK as a surrogate of other substructure materials for fixed dental prosthesis. All PEEK studies have reported that bonding to PEEK must be improved to achieve adequate, long-term adhesion clinically; however, information concerning the potential and limitations of PEEK in bonding to dental material is still insufficient. The aim of this in-vitro study was to evaluate and compare bond strength between PEEK and resin cement using different adhesive systems after various surface pre-treatments. Further it determined the effect of interaction between surface pretreatments and adhesive systems on bond strength between PEEK and resin cement and evaluate the type of bond failure between PEEK and resin cement[5].

Subjects and Methods

A total of 72 PEEK (figure-1) standard specimens were prepared by CAD-CAM milling (Arum CAD/CAM milling Machine) of PEEK blocks(PEEK Natural,Blomden). The dimensions of specimens were 7x7x3 mm. Specimens were cleaned in an ultrasonic machine and divided into 3 pretreatment Groups (n= 24/group): no surface pretreatment (control group)(Group I), Sand blasting (mechanical pretreatment)(Group II)(SSamit, Dento KEM) and Acid etched with sulfuric acid (98%) (chemical pretreatment)(Group III)(Qualigem, Thermo Fisher Scientific Indian pvt Ltd). Specimens from each group were divided into subgroups according to three adhesive systems (n=8 subgroup): no adhesive (Subgroup A), MMA based adhesive(Subgroup B)(Mono Bond, Ivoclar Vivadent) and Bis-GMA based adhesive system(Subgroup C)(Teric N Bond Universal, Ivoclar Vivadent).

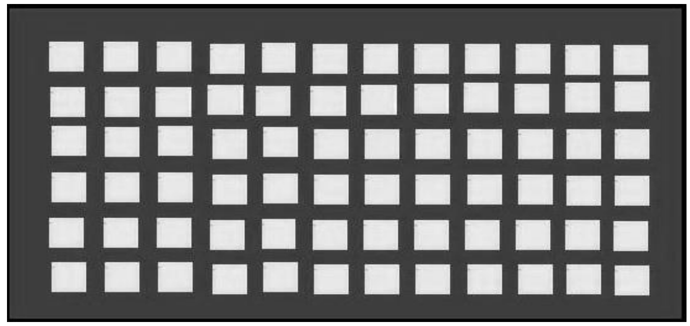

Figure 1: Milled test specimen

Specimens of Group I were underwent no surface pretreatment. Specimens of Group II were subjected to airborne particle abrasion with 110 µm sand particles with pressure 0.35 MPa at a working distance of 10 mm for 15 sec. Specimens of group III were etched with 98% Sulfuric acid using 100 µl of acid for 60 seconds, followed by careful rinsing with deionized water (Merck Life Science pvt ltd) for 30 seconds. Special care was taken to rinse in a constant motion in a single direction to avoid any additional directional changes of the delicate new surface topography. Thereafter, specimens were air dried for 10 seconds. After surface pretreatment all specimens were examined under Stereomicroscopic (Lawrence & Mayo, Germany) at 20x magnification. All surface pretreated specimens were placed in acrylic jig which were fabricated according to inner diameter of custom made metal mould for UTM (Enteck Instruments India pvt ltd). After placement no adhesive was applied to specimens of Subgroup IA, IIA and IIIA. Specimens of Subgroup IB, IIB and IIIC were subjected to application of Methyl methacrylate based adhesive system and Specimens of Subgroup IC, IIC and IIIC were subjected to application of Bisphenol glycidyl methacrylate based adhesive system. Bonding agent was applied on the surface pretreated specimens using micro brush. Single coat of adhesive system was applied in single direction followed by drying with gentle oil free air for 30 seconds. Dried specimens were then light cured with light curing unit (SKD BLUE RAY) at wavelength of 460 Nm output of 900 mw/cm^2 for 20 seconds, as per manufacturer's instructions.

After applying different adhesive systems all specimens were bonded with resin cement**.** Dual sided adhesive tapes were used as mold for bonding Resin cement. 7x7 mm sized dual side adhesive tape with 3mm inner diameter and 3mm height were placed over PEEK specimens and filled with resin cement (Teric N Intro, Ivoclar Vivadent). Dual cure resin cement's base and catalyst paste were dispensed equal quantity on plastic coated mixing pad. Dispensed dual cure cement was mixed with help of agate spatula for 30 seconds, as per manufacturer's instructions. Mixed cement was pressed onto the bonding area with help of composite filling instrument, and light-cured under a LED curing light wave length of 460 nm and output of 900 mw/cm^2 for 40 second, as per manufacturer's instructions. The tube axis should be perpendicular to the bonding surface**.** After curing of the luting resin cement dual side adhesive tape were removed from the specimens. Excess adhesive and cement were removed carefully. Specimens of all groups were incubated in deionized water for 24 hours at 37^{0}C for aging.

The shear bond strength was examined by a Universal Testing Machine (figure-2). During the testing procedure, all specimens were placed in custom made mounting jig. A shear apparatus applied force at a crosshead speed of 5 mm/min with 0 load. Visually confirming the position, load was gradually applied at speed of 5mm/min by auto operating software, till the specimen fractured. The load of fracture and the displacement of the bonded area were noted. Using the fracture load shear bond strength was calculated. The shear bond strength was calculated according to the following equation: **D = F/S** Where, D is the shear bond strength, F is the fracture load (N) at failure, S is the bonded area (mm^2).

All fracture specimens were investigated under a stereo-microscope at a 20× magnification for examine Failure (figure-3). Failures were classified as cohesive failure in PEEK, cohesive failure in resin cement, adhesive failure, or mixed failure. The data collected was tabulated and subjected to statistical analysis for interpretation of results. Multivariate CRD ANOVA was used to analyze the data. Level of significance was set at p≤0.05.

Figure 2: Evaluation of Shear Bond strength in UTM

(a) Specimen placed in UTM

(b) Fractured specimen after load application

(c) Reading of fracture load

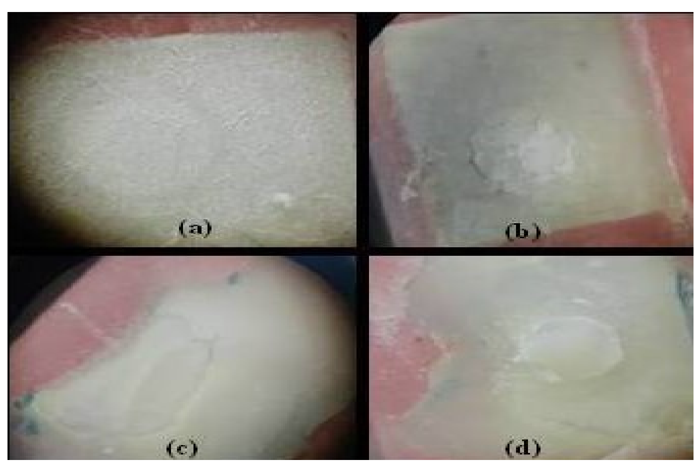

Figure 3: Stereomicroscopic view of different bond failures

(a) Adhesive bond failure

(b) Mixed bond failure

(c) Cohesive bond failure within PEEK

(d) Cohesive bond failure within luting resin cement

Results

Result shows descriptive statistics and summary of two way interaction between Groups (different surface pretreatment) and different adhesive systems used. Here if the difference of mean values between different groups is 0.56 MPa then there is statistically significant difference. In the present study Group IA exhibited least shear bond strength and Group IIIC exhibited highest shear bond strength. The statistical significance among the groups was as – IA ≃ IIA< IIB ≃ IB< IIIA ≃ IC< IIC< IIIB< IIIC.

Table -1 shows summary of mean Shear Bond strength of all groups after application of different adhesive system

Table -2 shows relative frequency of bond failure with respect to Groups (surface pretreatment) irrespective of adhesive system used. It was found that adhesive failure accounted for most of the fractures in the specimens as follows: Group I 83.3%, Group II 87.5% and Group III 58.3%. Specimens which exhibited cohesive failure were as follows: Group I 16.7%, Group II 8.3% and Group III 16.7%. Specimens which exhibited mixed failure were as follows: no mixed type bond failure in Group I, for Group II 4.2% and Group III 25% respectively.

Table 1: Summary of mean Shear Bond strength of all groups after application of different adhesive system

Group	Mean Shear Bond strength (MPa)			
	Subgroup A	Subgroup B	Subgroup C	Total mean for Group
I	1.62 ± 0.14^{a}	3.05 ± 0.22^{b}	4.08 ± 0.21^{c}	2.93^{I}
II	2.15 ± 0.33^{a}	2.85 ± 0.27^{b}	5.06 ± 0.52^{d}	3.35^{II}
III	3.95 ± 0.39^{c}	5.66 ± 0.76^{e}	12.65 ± 1.26^{f}	7.42^{III}
Total mean for Subgroup	2.58^{A}	3.85^{B}	7.27^{C}	

*The different numeric superscripted indicate that Shear Bond strength value were significantly different at p ≤ 0.05 among Groups.

*The different uppercase superscripted letters indicate that Shear Bond strength value were significantly different at p ≤ 0.05 among Subgroups.

*The different lowercase superscripted letters indicate that Shear Bond strength value were significantly different at p ≤ 0.05 among two way interaction.

Table 2: Relative frequency of type of bond failure in specimens of all groups

Groups	Bond failure type					
	Adhesive		Cohesive		Mixed	
	No.	percentage	No.	percentage	No.	percentage
IA	8	100%	0	0%	0	0%
IB	7	87.5%	1	12.5%	0	0%
IC	5	62.5%	3	37.5%	0	0%

IIA	8	100%	0	0%	0	0%
IIB	7	87.5%	1	12.5%	0	0%
IIC	6	75%	1	12.5%	1	12.5%
IIIA	7	87.5%	1	12.5%	0	0%
IIIB	6	75%	2	25%	0	0%
IIIC	1	12.5%	1	12.5%	6	75%
Total	55	76.38%	10	13.88%	7	9.71%

Discussion

The present study evaluated the bonding properties of resin and materials using shear bond strength test which is aimed to measure the stress required to break the bond between two materials. In the physiological activities of the oral cavity, the most external force received by the prosthesis is shear stress. Finite element analysis by Della Bona et al. demonstrated that shear strength could best represent the chewing state. The shear bond strength was tested to characterize the bond strength between the resin cement and PEEK6. PEEK specimens which were treated with sulfuric acid (98%) pretreatment exhibited strongest bond with the resin cement. This can be attributed to the fact that sulfuric acid attacks carbonyl and ether groups of PEEK which may lead to more functional groups improve hydrophilicity of the surface by introducing sulfonic acid group (-SO3) into the polymer chain of PEEK and result in better crosslinking that provide with polymer and the surface roughness values were significantly increased in this group. In a study conducted by Kosuke Kurahashi et al7, Oliver Sproesser et al8, Patcharawan Silthampitag et al9 and Pisaisit Chaijareenont et al10 shows similar result. The use of 98% sulphuric acid is not clinically viable because of its extremely corrosive nature. Also it is a hazardous material, and direct contact may cause serious damage. The bond strength between PEEK and the resin cement after surface treatment with sand blasting (110µm) exhibited better bond strength than specimens with no surface treatment. This can be attributed that after sand blasting there increases the surface roughness, creates afresh active surface layer by removing organic contaminant from the material surface and advances micromechanical interlocking of polymer. Similar results have been demonstrated by Kosuke Kurahashi et al7 and Christine Keul et al11 in their study. On the other hand Oliver Sproesser et al8 stated that untreated PEEK surface exhibited weak bond to the resin cement due to a complete lack of mechanical retention. Further, it was evident that Bis-GMA based adhesive system application on treated PEEK exhibited strongest bond with the resin cement. This can because Bis-GMA based adhesive system creates a bond between two surfaces that swelled the dissolved surface and dimethacrylate monomer provided the connection to the methyl group as binder side. Similar result was demonstrated by Oliver Sproesser et al, Ipek Caglar et al, Bogan Stawarczyk et al and Chrisstine Keul et al. The bond strength between PEEK and the resin cement after application of MMA based adhesive system better than no adhesive. In MMA based adhesive system one functional group of the bifunctional MMA monomer is occupied by phosphate group that once react with PEEK and the resin cement and further no reaction occurred. Similar result was demonstrated by Christine Keul et al11 and Bogna Stawarczyk et al12. This result can be attributed to the fact that chemical composition of the adhesive system plays an important role in creating bond between PEEK and luting adhesive cement. Similar result was demonstrated by Christine Keul et al11, Patcharawan

Silthampitag et al10 and Ipek Caglar et al12. In general, the fracture mode is influenced by the composition of the binder and the actual bonding method. Fracture can occur in the interior of adhesive and PEEK or the interface of bonding. In present study adhesive failure is most common than cohesive and mixed type bond failure. One possible reason may be that a bonding strength of adhesive is less than the cohesive strength of adhesive. In addition, cohesive failure and mixed failure mode contributes a less proportion which manifest that the bond strength and cohesion strength of the adhesive are similar to PEEK. Similar result was observed in Yue Wang et al14, Bogna Stawarczyk et al12 and Ahmet Kursat Culhaoglu et al13. Various surface pretreatments increase the bond between PEEK and resin cements. Among the procedures tested application of sulphuric acid surface pretreatment resulted in highest bond strength between PEEK and resin cement. Additional adherence programs of Bis-GMA have resulted in significantly higher bonds15. In vitro learning is an important aspect of the development of new materials and techniques, as it can provide valuable information for further clinical trials in clinical trials. Additional in vitro studies are used to test new possibilities or techniques for further testing in vivo, that is, in animals and humans, an important aspect of teeth. One advantage of in vitro research is that it enables researchers to perform single-variable experiments under controlled conditions16. Based on the results of the present study it can be inferred that various surface pretreatments of PEEK prosthesis leads to an improvement in bond with luting cements. Though surface pretreatment with 98% sulphuric acid leads to highest bond strength but keeping in mind the risk and hazards of the technique, it should be used with caution. Sand blasting is an acceptable and feasible procedure for surface pretreatment of PEEK prosthesis. Further the present study also emphasized the role of bonding agents on improving the bond between PEEK prosthesis and luting cements. This can serve helpful to clinicians for increasing the success and serviceability of the prosthesis in patient's mouth. Clinical implications of the study: Bond strength between FDP and luting cement is prime requisite for serviceability of prosthesis. Alteration in surface properties can affect the bond strength between prosthesis and luting cement. Mechanical surface treatment may lead to surface roughness that helps in improving mechanical bond between prosthesis and luting cement.Chemical surface treatment may lead to changing in polymer chain and creates surface roughness that improves bond strength. Use of adhesive systems improves the bond strength between prosthesis and luting cement. Adhesive bond failure are most commonly observed between prosthesis and luting cement.

Conclusion

Within the limitation of this in-vitro study it can be concluded that:

- The ability of Shear bond strength of PEEK with resin cement has improved after various surface pretreatments.
- 98% Sulfuric acid pretreatment exhibited highest shear bond strength. But as handling of sulfuric acid is dangerous, this should be used with caution.
- Surface pretreatment with sandblasting also improved shear bond strength, and also serves as a safer and feasible option.
- Shear bond strength of PEEK with resin cement has improved after application of different adhesive systems.
- Bis-GMA based adhesive system exhibited higher bond strength than MMA based adhesive system.
- The major factor in promoting adhesion between PEEK and luting resin cement appears to be

micromechanical locking from bonding agent after surface pretreatment.

- Further comparative studies are needed to be more conclusive about the shear bond strength between PEEK and resin cement using different surface pretreatments and bonding agents in clinical environment.

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