Original Article

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Dentistry Section

Shear Bond Strength of Veneering Composite Versus Different Polyetheretherketone Materials after Various Surface Treatments: An In-vitro Study

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ABSTRACT

Introduction: Polyetheretherketone, also known as PEEK, is a semicrystalline thermoplastic material with many advantages. Since its grey material, it has to veneering with composite resin to enhance its aesthetic appeal. Bonding PEEK and composite veneers poses challenges due to its inert surface.

Aim: To assess and compare the Shear Bond Strength (SBS) between various polyetheretherketone materials and veneering composites following various surface treatments.

Materials and Methods: The in-vitro study was conducted at KSR Dental College and Hospital in Tiruchengode, Tamil Nadu, India, over a period of three months from December 2022 to February 2023. A total of 108 PEEK discs were prepared for the study, divided into three groups: unfilled PEEK, 30% carbon reinforced PEEK, and 10% carbon+10% graphite+10% Polytetrafluoroethylene (PTFE) reinforced PEEK. The specimens underwent different surface treatments, including no treatment, sandblasting with 110 um alumina particles, and acid etching with 98% sulphuric acid. Additionally, 108 composite discs were prepared and bonded to the PEEK specimens using adhesive and resin cement. The bonded specimens were immersed in distilled water for 24 hours, and the Shear Bond Strength (SBS) was determined using a universal testing machine. Statistical analysis was performed using one-way ANOVA, and mean values were compared using posthoc tests.

Results: The results indicated significant variation in SBS among the three groups without treatment (p=0.011) and after sandblasting with 110 um alumina (p=<0.001). The 30% carbon reinforced PEEK exhibited the highest SBS regardless of the surface treatments.

Conclusion: Among the tested materials, 30% carbon reinforced PEEK demonstrated the highest SBS, regardless of the surface treatments. Acid etching yielded the highest SBS among the various surface treatments, irrespective of the type of PEEK material used.

Keywords: Acid etching, Air abrasion, Alumina, Sandblasting

INTRODUCTION

Polyetheretherketone, also known as PEEK, is a semicrystalline thermoplastic material with several advantages, like excellent mechanical properties and a high melting point of approximately 335°C. It exhibits chemical stability with both organic and inorganic compounds, ease of processing, high stiffness, dimensional stability at high temperatures, and compatibility with common sterilisations methods. Due to its colour, radiolucency, stiffness, and lighter weight compared to natural teeth, PEEK is a preferred material for dental restorations [1,2].

The PEEK is a grey substance that does not contain any metals. While it offers better aesthetics than metal alloys, it falls short when compared to zirconia. Therefore, PEEK requires veneering with composite resin. However, bonding PEEK and composite veneers is challenging due to its inert surface [3]. Surface roughness, bacterial retention, colour stability, and wear qualities are important factors related to the long-term performance of dental prostheses [4]. Adhesion, influenced by material properties such as surface roughness, contact angle, wettability, and friction coefficient, is crucial for successful bonding and is greatly affected by surface modification [5,6]. A strong bond is formed when the adhesive securely attaches to the substrate and releases most of the applied energy. When both the substrate and adhesive contain reactive groups, a strong bond can be achieved. Surface roughness enhances the mechanical anchoring of the adhesive by increasing the surface contact area [7].

Numerous efforts have been made to increase the surface energy of PEEK using various surface treatment techniques. Studies have shown that surface treatment significantly increases the polar component of PEEK's surface free energy [8]. While individual research has been conducted on surface treatments of PEEK specimens [3,4], a direct comparison between different surface treatments within the same study is limited. Therefore, the present study aimed to investigate the Shear Bond Strength (SBS) of veneering composite to various polyetheretherketone materials and evaluate the effects of various surface treatments.

MATERIALS AND METHODS

The in-vitro study was conducted at KSR Dental College and Hospital in Tiruchengode, Tamil Nadu, India, over a period of three months from December 2022 to February 2023. The study received approval from the Institutional Review Board (IRB) and Institutional Ethical Clearance (IEC) committees of the institution (IEC-PG/FEB/2021/001). A total of 108 PEEK specimens were selected for the study, with each group included 36 specimens.

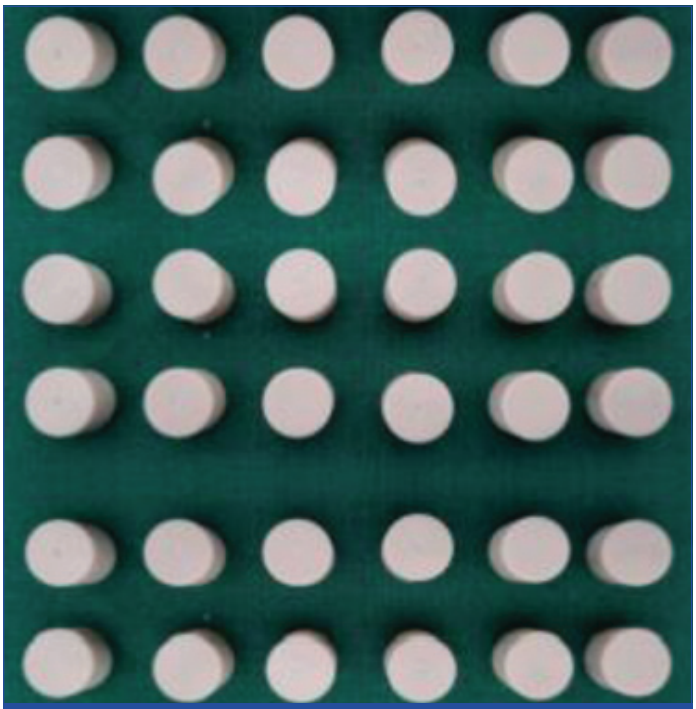
Sample size calculation: The sample size was calculated using G Power software, with an 80% effect size, 5% margin of error, and 80% power.

Study Procedure

The specimens were grouped into three groups based on the reinforcement material added to the unfilled PEEK: Group A (unfilled

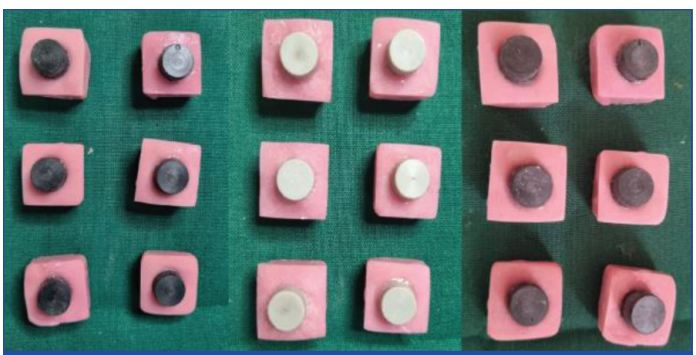
PEEK), Group B (30% carbon reinforced PEEK), and Group C (10% carbon+10% graphite+10% PTFE reinforced PEEK). Each group included 36 specimens.

Unfilled PEEK, 30% carbon reinforced PEEK, and 10% carbon+10% graphite+10% PTFE reinforced PEEK granules were obtained from the same manufacturer (Shree Krishna Polymers, Chennai). PEEK granules were used to prepare disc-shaped specimens (n=108) with a diameter of 10 mm and a height of 10 mm using the injection moulding process. Each group included 36 discs prepared from the respective PEEK materials [Table/Fig-1]. Total of 108 experimental specimens were polished using 800 grit sandpaper and cleaned with distilled water for 10 minutes.



[Table/Fig-1]: PEEK discs.

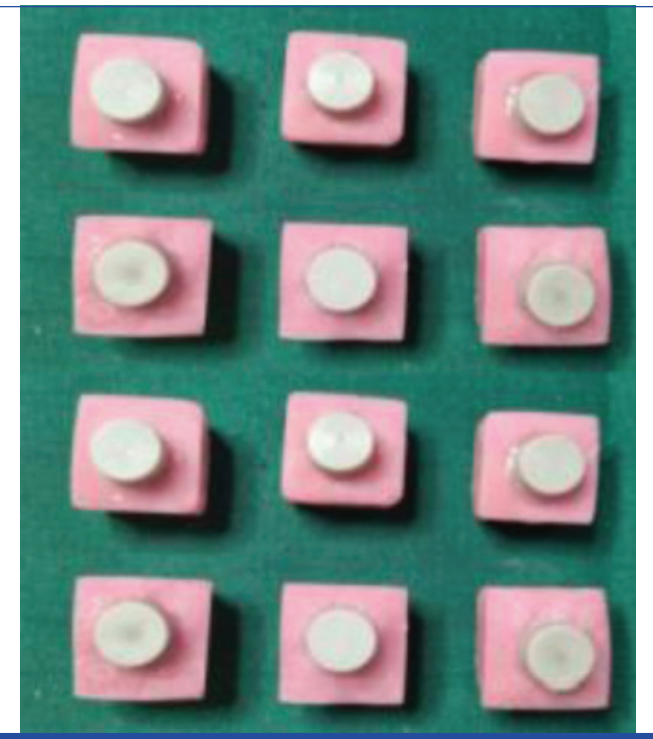
Self-cure acrylic resin was used to embed the PEEK specimens in an acrylic block. The specimens were embedded on an acrylic jig [Table/Fig-2]. The samples were then subgrouped based on different surface treatments. The first subgroup was left without treatment (Test-1). The second subgroup of each group was sandblasted with 110 um alumina particles for 15 seconds at 3 atm of pressure and a distance of 5 mm in the sandblaster at an approximate angle of 60 to 90 degrees (Test-2). The sandblasted specimens were air-dried with compressed air for 20 seconds [9]. The third subgroup was treated with acid etching using 98% sulphuric acid for 60 seconds. After cleaning with deionised water for one minute, the specimens were air-dried for 20 seconds [10].



[Table/Fig-2]: Mounted peek specimens.

Indirect composite resin (GC Gradia indirect composite) was condensed in a Teflon mold with a diameter of 8 mm and a height of 6 mm in increments. Each incremental surface was light-cured for 40 seconds using an Light Emitting Diode (LED) light curing unit. The excess material was removed, and a smooth surface was obtained [Table/Fig-3]. A thin layer of adhesive (3M Single Bond Universal Adhesive) was applied to the PEEK specimens, followed by light curing. Adhesive resin cement (G-CEM one-self-adhesive resin) was dispensed and mixed on a mixing pad. The composite disc was coated with the mixed cement and immediately seated on the PEEK specimen. Pressure was applied, and light curing was performed. Excess cement was removed, and light curing was done on all margins and surfaces [Table/Fig-4].

[Table/Fig-3]: Prepared composite discs.



[Table/Fig-4]: Composite bonded with peek.

The samples were soaked in distilled water for 24 hours before testing. Shear bond strength was tested using a universal testing machine (Model 3382, Instron) according to International Organisation for Standardisation (ISO) technical specification #11405 [Table/Fig-5]. A shear force was applied occlusally using a chisel rod parallel to the bonded surface of the specimen. Bond strength was calculated using

[Table/Fig-5]: Universal testing machine.

the formula: Bond strength (MPa)=Debonding Force (Newton)/Surface area of the composite $(mm)^2$.

The mean and standard deviation values for all groups were obtained and subjected to statistical analysis.

STATISTICAL ANALYSIS

Descriptive and inferential statistics were analysed using International Business Machines (IBM) Statistical Package for Social Sciences (SPSS) version 21.0 (IBM Corp, Version 21.0. Armonk, NY: IBM Corp). A test of normality was conducted to assess the data distribution, and one-way ANOVA was performed to analyse the differences in SBS between Group A (unfilled PEEK), Group B (30% carbon reinforced PEEK), and Group C (10% carbon+10% PTFE+10% graphite reinforced PEEK). Bonferroni posthoc tests were conducted to determine which pairs of groups differed significantly from each other. A p-value of less than 0.05 was considered statistically significant.

RESULTS

The influence of surface treatments (Test-1: without treatment, Test-2: sandblasting with 110 um alumina, and Test-3: acid etching with 98% sulphuric acid) on the Shear Bond Strength (SBS) of the veneering composite on unfilled PEEK resulted in values of 12.20 MPa, 13.82 MPa, and 14.48 MPa, respectively [Table/Fig-6]. There was a statistically significant difference (p=0.010) between Test-1 and Test-3 for the unfilled group.

Tests	Unfilled peek	30% Carbon reinforced peek	10%carbon+10% graphite+10% reinforced peek	F-value	p-value
Without treatment	12.20±1.59	14.10±1.32	12.62±1.59	5.310	0.011
Sandblasting with 110 um alumina	13.82±2.2	15.63±1.40	13.89±1.51	4.059	<0.001
Acid etching with 98% sulphuric acid	14.48±1.41	17.47±1.42	14.01±0.98	25.504	0.05

[Table/Fig-6]: Comparative evaluation of shear bond strength of different reinforced peek after various surface treatments.

The influence of surface treatments on the SBS of the veneering composite on 30% carbon reinforced PEEK resulted in values of 14.10 MPa, 15.63 MPa, and 17.47 MPa, respectively. There was a statistically significant difference between Test-1 and Test-2 (p=0.032), Test-1 and Test-3 (p=<0.001), and Test-2 and Test-3 (p=0.008) [Table/Fig-7].

Tests	Groups*	p-value
Unfilled PEEK	Test-1 vs Test-2	0.097
	Test-2 vs Test-3	1.000
	Test-1 vs Test-3	0.010
30% carbon reinforced PEEK	Test-1 vs Test-2	0.032
	Test-2 vs Test-3	0.008
	Test-1 vs Test-3	<0.001
10% carbon+10% PTFE+10% graphite reinforced PEEK	Test-1 vs Test-2	0.105
	Test-2 vs Test-3	1.000
	Test-1 vs Test-3	0.066

[Table/Fig-7]: Multiple comparisons of shear bond strength of various surface treatments for different PEEK materials.
*Test-1-untreated
Test-2-sandblasting with 110 um alumina
Test-3-acid etching with 98% sulphuric acid

The influence of surface treatments on the SBS of the veneering composite on 10% carbon+10% PTFE+10% graphite reinforced PEEK resulted in values of 12.62 MPa, 13.89 MPa, and 14.01 MPa, respectively. There was no statistically significant difference observed among the groups.

When comparing the SBS among the different groups, statistically significant differences were found between Group A and Group B (p=<0.001), Group B and Group C (p=<0.001), and Group A and Group C (p=<0.001) for the surface treatment of acid etching with 98% sulphuric acid [Table/Fig-8].

Tests	Groups*	p-value
Without treatment	Group-A vs Group-B	0.011
	Group-A vs Group-C	1.000
	Group-B vs Group-C	0.069
Sandblasting with 110 um alumina	Group-A vs Group-B	0.051
	Group-A vs Group	1.000
	Group-B vs Group-C	0.064
Acid etching with 98% sulphuric acid	Group-A vs Group-B	<0.001
	Group-A vs Group-C	<0.001
	Group-B vs Group-C	<0.001

[Table/Fig-8]: Multiple comparisons of shear bond strength of different PEEK materials for various surface treatments.

DISCUSSION

The PEEK is considered an advanced biomaterial and is used in dentistry for creating temporary crowns for implants, using plastic temporary abutments. PEEK has an extremely low elastic modulus, but when reinforced with materials such as carbon fibres or glass fibres, its elastic modulus can increase significantly, making it advantageous for endosseous implants. Carbon Fibre-reinforced PEEK (CFR-PEEK) has attracted interest from the medical implant community due to its favourable properties [11].

In present study, two surface treatments were performed on PEEK specimens: sandblasting with 110 um alumina (mechanical method) and acid etching with 98% sulphuric acid (chemical method). Previous research has shown that sandblasting with 110 um alumina particles improves bond strength to resin cements with PEEK [12]. Sandblasting creates a microporous surface with increased wettability, enhancing micro retention [13]. Similarly, sulfuric acid treatment increases the number of functional groups on the PEEK surface, creating larger micro porosities that promote bonding with composite materials.

Adhesive resin cements, specifically G-CEM ONE Paste Pak, were used in present study as luting agents for bonding the veneering

composite to the PEEK specimens [14]. Indirect composite restorations have advantages over direct composites when veneered with PEEK, including reduced incidence of problems and less polymerisation shrinkage stress.

When comparing the SBS between the veneering composite and PEEK specimens after sandblasting with 110 um alumina particles (Test-2), higher SBS values were observed in all three groups compared to specimens without treatment (Test-1). This is attributed to the roughness created by sandblasting, which provides micromechanical interlocking and improves wetting properties.

When comparing the SBS between the veneering composite and PEEK specimens after acid etching with 98% sulphuric acid (Test-3), the highest SBS values were observed compared to Test-1 and Test-2, regardless of the type of PEEK material. This is likely due to the chemical bonding between the sulfonate groups produced by sulfuric acid and the adhesives, as well as the micromechanical bonding created by resin tags penetrating the surface pits and pores of PEEK [13].

When comparing the SBS between the veneering composite and PEEK specimens of different groups, regardless of surface treatments, the group with 30% carbon reinforced PEEK showed the highest SBS. This is attributed to the influence of carbon fibres, which create a rougher surface and improve wettability, thus enhancing bond strength.

A study by Li W et al., showed the alteration in surface topography of PEEK specimens due to influence of carbon fibres. A 30% carbon reinforcement created rougher surface [15]. A study was done by EL-Wassefy NA to compare acid etching with 98% sulphuric acid is an effective surface treatment for improving the bond strength between the veneering composite and PEEK, regardless of the type of PEEK material [3]. Carbon fibre reinforcement also contributes to higher bond strength. It is important to consider these factors when selecting surface treatments for PEEK restorations in dental applications.

Limitation(s)

One limitation of present study is that it was conducted in-vitro, so it did not precisely replicate the conditions in the oral cavity. Another methodological flaw is the absence of thermocycling or long-term water storage to simulate artificial aging, which could have provided insights into the long-term endurance of the veneering process.

CONCLUSION(S)

Within the limitations of present study, it can be concluded that the highest SBS was observed in 30% carbon-reinforced PEEK when comparing it to the veneering composite and different PEEK materials after various surface treatments. Additionally, when comparing the effect of different surface treatments on SBS with the veneering composite and different PEEK materials, the highest value was observed in the acid-etched group, regardless of the type of PEEK specimen.

To better understand the chemical implications of the surface treatments on SBS and to evaluate the long-term endurance of the veneering process, further research is needed. This could include analysing the chemical structures on the PEEK surfaces and conducting studies with longer exposure periods in distilled water.

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