

Assessment of different surface treatments and shear bond characteristics of poly-ether-ether-ketone: An *in vitro* SEM analysis

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Abstract

Aim: The aim of this study is to assess the surface roughness and shear bond characteristics of poly-ether-ether-ketone after different surface treatments.

Setting and Design: An *in vitro*, prospective.

Materials and Methods: One hundred and twenty disc-shaped samples of 10 mm diameter and 2 mm thickness were milled and subjected to following surface treatments: 110 μm alumina particles, 98% concentrated sulfuric acid, and 10–20 μm synthetic diamond particles. Surface characteristics of treated sample were studied under SEM with $\times 500$ and $\times 1000$ magnification. Shear bond strength (SBS) with composite resin discs embedded in acrylic blocks after luting with self-etch resin cement and resin-modified glass ionomer cement (RMGIC) was evaluated using the universal testing machine (Instron®, Massachusetts U. S. A).

Statistical Analysis Used: The data collected were evaluated using the Analysis of variance and Tukey's honest significant difference *post hoc* test.

Results: Highest SBS and SR were noted with self-etch resin cement in the given order: 98% sulfuric acid ($2.106 \pm 0.186 \mu\text{m}$), followed by alumina particles ($1.706 \pm 0.160 \mu\text{m}$) and synthetic diamond particles ($1.101 \pm 0.167 \mu\text{m}$).

Conclusion: The SBS of self-etch resin cement was higher compared to RMGIC for all three surface treatments done on test samples. Hundred percent samples treated by all three surface treatment methods showed mixed type of failure.

Keywords: Adhesion, polyetheretherketone, SEM, shear bond strength, surface roughness

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INTRODUCTION

Polyetheretherketone (PEEK) is a linear thermoplastic, aromatic, and semicrystalline polymer. It is an attractive material for the application in dentistry because of its excellent thermomechanical characteristics, chemical stability, biological inertness, stability with almost all organic and inorganic chemicals, good strength, stiffness, toughness, and fatigue properties. The uniqueness of PEEK is that it can be processed without the need of any additives, making it highly advantageous for medical and dental application.^[1,2] PEEK is currently being used for wide range of dental applications such as dental clasp, healing abutments, and transitional abutments and also as an alternative rigid material in removable partial dentures and fixed dental prostheses.^[3-7] As a restorative material, it has good strength to withstand masticatory forces in the posterior region and has also shown a lot of potential for metal-free and ceramic-free crown and bridge.^[8] Another advantage it possesses is that, it does not cause wear of the opposing natural dentition when used as a crown or a bridge.^[9] Without any complexities, PEEK can be easily reshaped and can be modified or trimmed with regular burs.^[8]

Despite its numerous advantages, PEEK has failed to grab attention in the field of restorative and prosthetic dentistry, due to difficulties in establishing a strong and durable adhesion to composite resin because of PEEK's chemical inertness, low surface energy, and resistance to surface modification by chemical treatments.^[10-14] Adhesion is an indispensable property that depends on numerous material characteristics such as surface roughness (SR), wettability, reflectivity, and coefficient of friction which are significantly affected by surface treatment and processing.^[13-15]

Air-borne particle abrasion is the most popular procedure which simultaneously roughens and cleans the surface ultimately increasing the available surface area.^[7,16-19] Alumina particles are most widely used for air abrasion as it creates more wettable surface. Diamond particles of 30–50 μm have been used in dentistry for the surface treatment of metals and ceramics. Diamond particle being harder than alumina particle creates more SR but, in some studies, bigger particles of diamond have led to the formation of surface defects.^[20,21] Current literature has shown that etching with concentrated sulfuric acid creates a rough and chemically altered surface which enables it to bond more effectively with hydrophobic resin composites.^[7,14,22]

Another factor which is important in achieving a strong bond is the luting cement which acts as a connecting link between a restoration and substrate.^[23] In recent years,

resin cements have rapidly gained popularity as luting agents. This category of cements has high tensile and compressive strengths and the lowest solubility of the available cements.^[24] Flexural properties, including modulus and strength are important to prevent de-bonding during function, and resin cements have both a high modulus and strength. Resin-modified glass ionomer (RMGI) cement combines some of the desirable properties of glass-ionomer cement (fluoride release and chemical adhesion) with high strength and low solubility of resins.^[25]

In this study, air abrasion with alumina particles (110 μm), synthetic diamond particles (10–20 μm), and chemical treatment with concentrated sulfuric acid (98%) was used to find out if the air abrasion with diamond particles proves superior to both the other two already existing methods of surface treatment. Luting agents used in this study were Multilink N, Ivoclar Vivadent, Germany (Dual cure resin cement) and RelyX Luting 2, 3M ESPE, USA (RMGI cement). Air abrasion with synthetic diamond particles was chosen as an alternative in this study with the reasoning that as the diamond particles are harder than the alumina particles, they would create deeper pits resulting in a rougher surface and thereby might enhancing the bond strength.

The null hypothesis of this study was that there is no difference in shear bond strength (SBS) between the test specimens exposed to three types of surface treatments luted with two types of cements used.

MATERIALS AND METHODS

The study was approved by the Institutional Review Board for ethics committee is ECR/511/inst/MH/2014/RR-20.

Preparation of specimens and surface treatment:

One hundred and twenty disc-shaped samples of 10 mm diameter and 2 mm thickness were milled with the help of a computer-aided design/computer-aided manufacturing device (Yenadent d43, France), from commercially available PEEK blanks (Jinan Carved Technology Co. Ltd., China). The samples were polished with 800 grit sandpapers and ultrasonically cleaned in distilled water for 10 min.

A customized metal mold of 10 mm diameter and 2 mm thickness was used to fabricate composite resin discs (Restofill, Anabond Stedman Pharma Research (P) Ltd, India). The samples were light cured (Woodpecker, China; Model No: LED D) for 40 s. Following this, the composite specimens were retrieved from the mold and additional external curing of 40 s was done on the other

side to ensure complete polymerization. One hundred and twenty composite specimens were fabricated in this manner. Auto-polymerizing acrylic resin (DPI RR Cold Cure, Dental Products of India, Wallace Street, Mumbai) was poured in the rectangular metallic mold in early dough stage to prepare a resin block mount for SBS analysis. Each composite specimen was partly embedded in the center of the partially set rectangular resin block.

One hundred and twenty PEEK samples ($n = 120$) were randomly divided into four groups with 30 samples in each group. The samples in each group were subjected to the respective surface treatment.

- Group 1 ($n = 30$): No treatment was done (control)
- Group 2 ($n = 30$): Air-abrasion with alumina particle ($110 \mu\text{m}$) at air pressure of 0.1 MPa (1 bar) at a distance of 10 mm for 10 s
- Group 3 ($n = 30$): Concentrated 98% sulfuric acid was applied over the surface of samples for 60 s
- Group 4 ($n = 30$): Air-abrasion with synthetic diamond particles ($10\text{--}20 \mu\text{m}$) at air pressure of 0.1 MPa (1 bar) at a distance of 10 mm for 10 s.

Following surface treatments, all samples were ultrasonically cleaned in a water bath (C-80-N; Confident Dental Equipment's Ltd, Bangalore, India) and air dried.

Determination of surface roughness and surface analysis
SR of five specimens from each group was measured using surface profilometer (Mitutoyo, Japan). The direction of measurement was at the right angle to the direction of abrasion. After surface treatment, specimens of each group were studied using scanning electron microscope and four readings were noted at different spots on same sample (SEM) (Zeiss, Sigma, Germany) at $\times 500$ and $\times 1000$ [Figures 1 and 2].

Bonding procedure

Each group having 30 samples was subdivided into two subgroups having 15 samples each according to the cement used.

- Subgroup A– Self etch resin cement-Multilink N Resin Cement (Ivoclar Vivadent, Schaan Liechtenstein, Germany).

Following the manufacturer's instructions, silane coupling agent (Monobond-N; Ivoclar-Vivadent, Liechtenstein, Germany) was applied with an applicator tip on the PEEK sample and after 60 seconds, dried with a strong stream of air to achieve a thin, uniform layer. Resin cement was then applied on the center of PEEK sample and luted with composite disc under a constant load of 5N. The cement was

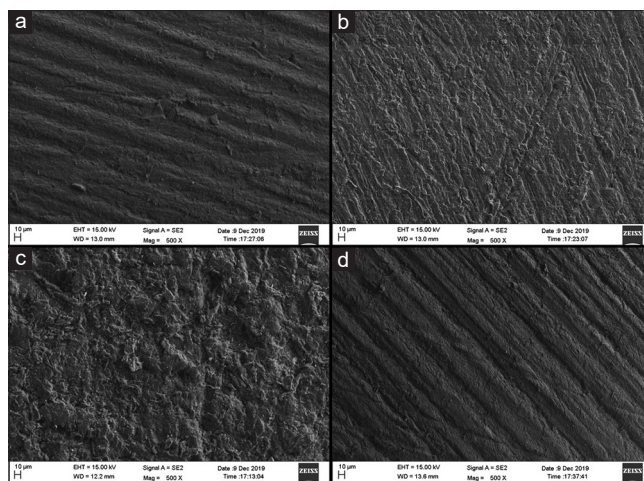


Figure 1: SEM images at 500 \times magnification, (a) PEEK surface with no surface treatment, (b) PEEK surface after air abrasion with aluminum oxide ($110 \mu\text{m}$), (c) PEEK surface after air abrasion with concentrated 98% sulphuric acid, (d) PEEK surface after air abrasion with synthetic diamond particles ($10\text{--}20 \mu\text{m}$)

then tac cured for 2 s using LED dental light curing device. (Woodpecker, China; Model No: LED D) with light intensity of $850\text{--}1000 \text{ MW}/\text{cm}^2$. Excess cement was removed with the help of an applicator tip. The cement was then light cured for 20 s and allowed to set completely for 24 h.

- Subgroup B-RMGI cement-RelyX Luting 2 (3M USPE (USA)).

Following the manufacturer's instructions, equal amount of base and catalyst paste of the cement was dispensed on a mixing pad with the help of a clicker. The two pastes were mixed for 20 s to attain a mix of uniform consistency and color. This was done within the working time of 2 min. The cement was then applied on the center of PEEK specimen and luted with composite disc under a constant load of 5N. The cement was tac cured for 5 s. The excess cement was removed with a brush. The cement was allowed to set completely for 24 h.

Thermocycling of test specimen

After 24 h, all specimens were subjected to thermocycling regime of 5000 cycles, immersed cyclically with water baths of 5°C and 55°C in an Automatic Thermocycling Dipping Machine that was designed for the simulation of oral temperature changes with a dwell time of 15 s and transfer time between the baths was 15 s. The machine was controlled by programmable logic controller system.

Determination of shear bond strength and type of fracture

The auto-polymerizing resin block was stabilized with the help of a customized metal holder in the clamps of the universal testing machine (Instron®, Massachusetts

U. S. A). A SBS test was performed to evaluate the bond strength between PEEK and the resin cement used in the study ($n = 120$). The samples were secured in a horizontal position with the assistance of a metal fixture. A metal blade with an edge thickness of 0.5 mm was moved vertically at 90°, at a crosshead speed of 0.5 mm/min until the sample fractured. The load was applied at the PEEK-cement interface. All tests were performed under uniform atmospheric conditions of 23.0°C ± 1°C and 50% ± 1% relative humidity.

The values for SBS were derived with the use of following formula:

$$\text{SBS (MPa)} = \text{Force (N)} / \text{Area (mm}^2\text{)}.$$

The de-bonded samples of all four groups were examined under a stereomicroscope (CSM2) under

a magnitude of ×40 [Figure 3]. This examination was done to determine whether the type of fracture was (a) adhesive failure mode between materials and resin-based luting materials, (b) cohesive failure mode within resin-based luting materials or within material, and (c) mixed failure mode with both cohesive and adhesive failures.

Statistical analysis

Statistical analyses were performed to compare SBS for different materials and resin-based luting materials by the one-way analysis of variance (ANOVA) for all four groups and two subgroups. Intergroup analysis of subgroup was done by Tukey's honest significant difference (HSD) *post hoc* test. A value of $P < 0.05$ was considered to indicate statistical significance. The Statistical Package for the Social Sciences software (SPSS) version 22.0 (IBM Analytics, New York, U. S. A) was used to carry out the statistical analysis.

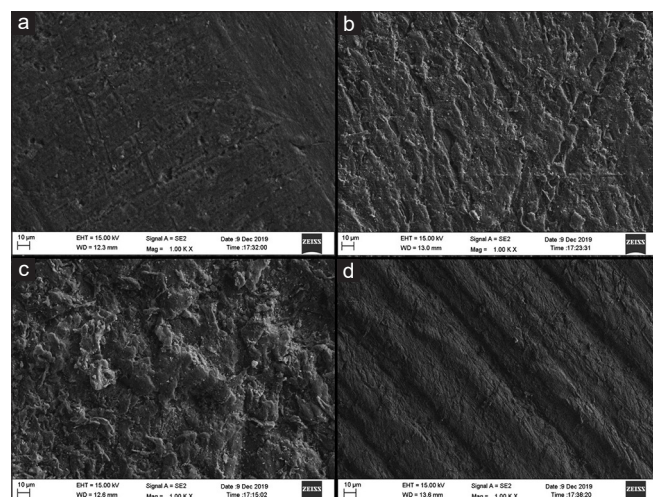


Figure 2: SEM images at 1000× magnification, (a) PEEK surface with no surface treatment, (b) PEEK surface after air abrasion with aluminum oxide (110 μm), (c) PEEK surface after air abrasion with concentrated 98% sulphuric acid, (d) PEEK surface after air abrasion with synthetic diamond particles (10-20 μm)

Table 1: Values of surface roughness in microns

Group 1	Group 2	Group 3	Group 4
0.147±0.0238	1.706±0.160	2.106±0.186	1.101±0.167

Table 2: Inter-group analysis of Subgroup A and Subgroup B

	P-value of Subgroup A	P-value of Subgroup B
Group 1 versus 2	0	0.005
Group 1 versus 3	0.0049	0.005
Group 1 versus 4	0	0
Group 2 versus 3	0	0
Group 2 versus 4	0	0
Group 3 versus 4	0.0002	0.005

Table 3: Comparison of shear bond strength values in Megapascals between all four groups and two subgroups with one-way analysis of variance test

	Group 1	Group 2	Group 3	Group 4
Subgroup A	-	3.91±0.59	7.52±1.20	2.27±0.39
Subgroup B	-	2.66±0.30	3.85±0.36	1.03±0.21

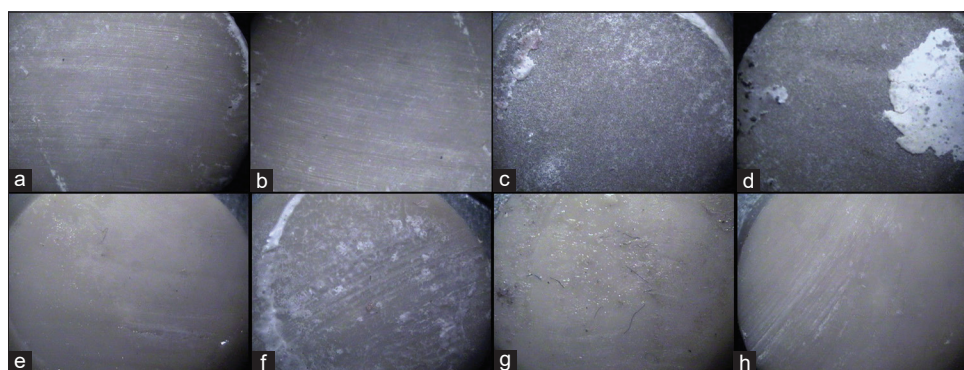


Figure 3: Failure analysis under a Stereomicroscope (CSM2) under a magnitude of 40×, (a) No Treatment + Self Etch Resin cement, (b) No Treatment + RMGIC, (c) Air abrasion with Al₂O₃ + Self Etch Resin cement, (d) Air abrasion with Al₂O₃ + RMGIC, (e) Concentrated sulphuric acid + Self Etch Resin cement, (f) Concentrated sulphuric acid + RMGIC, (g) Air abrasion with synthetic diamond particles + Self Etch Resin cement, (h) Air abrasion with synthetic diamond particles + RMGIC

RESULTS

The values of SR of five samples from each Groups 1, 2, 3, and 4 were measured by a surface profilometer, as shown in Table 1. Highest SR values were seen in Group 3 (treated with 98% sulphuric acid) with values of $2.106 \pm 0.186 \mu\text{m}$.

Among the subgroups; subgroup A (Self-etch resin cement-Multilink N, Ivoclar Vivadent, Germany) showed higher SBS values for all four groups, which was statistically significant as compared to subgroup B (RMGIC– RelyX Luting 2, 3M ESPE, USA) [Table 2]. Highest SBS was seen in self-etch resin cement-Multilink N of samples treated with 98% sulfuric acid with value of $7.52 \pm 1.20\text{MPa}$ and least was seen in RMGIC– RelyX Luting 2 of samples treated with 10 – 20 μm diamond particles with values of $1.03 \pm 0.21\text{MPa}$ [Table 3]. The least SBS values were shown by RMGIC– RelyX Luting 2 for all four test groups.

The results revealed that for each cement there was a statistically significant increase of SBS of Group 3 and Group 2 compared to Group 4 compared to Group 1. ($P = 0.00$ i.e., $P < 0.05$). Table 4 shows the distribution of the failure modes. All samples from Groups 2, 3, and 4 showed mixed failure, i.e. adhesive and cohesive type of failure. In Group 1, all 30 samples showed adhesive type of failure.

DISCUSSION

PEEK is a promising material and is used in many industries including electronics, automotive, aerospace, and medical equipments.^[2,26,27] Owing to its good biocompatibility and attractive mechanical properties such as heat resistance, solvent resistance, excellent electrical insulation, good wear resistance, and high fatigue resistance, PEEK is ideal for restoring the lost tooth structure.^[15] The study done in the recent past showed a visible deformation of three-unit fixed dental prostheses (FDPs) at 1200N, but this value was higher than the masticatory forces in posterior region which are up to 600N. PEEK has been demonstrated to be worthy in load-bearing areas.^[7]

Whitish appearance of PEEK makes it a good esthetic tooth-colored restorative material. However, its retention has been a drawback.^[9] Retention of crown plays an

imperative role in its clinical success. This is influenced by multiple factors, of which the bond formed between the intaglio surface of the crown and cement used is most crucial because clinical failures are commonly observed at this interface. This critical bond between PEEK and cement is dependent on various parameters, of which SR and treatment are the key factors. Literature on adhesion of surface-treated PEEK to teeth using different luting agent and its durability is very limited. Effective bonding of PEEK to tooth structure is a prerequisite for using it as a prosthetic restorative material.^[9] Sufficient SR of PEEK is required to obtain sufficient mechanical retention for a successful bond. However, for PEEK, limited number of surface roughening methods have been successfully employed owing to its hardness and strength.^[15] The commonly used methods of surface treatment of PEEK include air-abrasion with alumina particles and with corrosive solutions such as concentrated sulfuric acid and piranha solution.^[28,29]

In recent times, diamond particles of 30–50 μm have been used in dentistry for surface treatment of metals and ceramics.^[20,21] As the diamond particles are harder than alumina, it was hypothesized that air abrasion with diamond particles would produce more irregular and rougher surface and hence improve SBS compared to air abrasion with alumina particles.^[20] However, there is no literature present on the use of diamond particles for the surface treatment of PEEK.

In this study, a new surface treatment approach of PEEK, i.e., synthetic diamond particles (10-20 μm) were compared to conventionally used technique of air-abrasion of aluminum oxide particle (110 μm) and surface treatment by concentrated 98% sulfuric acid.

The result of SR indicated that the SR value of Group 3 (2.106 ± 0.186) was highest among the four groups followed by that of Group 2 (1.706 ± 0.160) which was more than Group 4 (1.101 ± 0.167). The least SR value was recorded with Group 1 (0.147 ± 0.0238). SEM analysis was used for the microanalysis of the specimens by generating high-resolution images at high magnification. Scanning electron microscopy images of surface of each group are shown in Figures 1a-d and 2a-d.

Various studies have successfully reported the use of concentrated sulfuric acid for the surface treatment of PEEK. This corrosive acid results in microporosities by dissolution of PEEK matrix by a process known as sulfonation reaction resulting in micromechanical bonding with the luting agent.^[28,30] A study by Chaijareenont *et al.* evaluated the effect of concentrated sulfuric acid etching

Table 4: Failure type analysis

Samples	Adhesive failures	Cohesive failures	Mixed failures
Group 1	30	-	-
Group 2	5	2	23
Group 3	2	1	27
Group 4	11	-	19

of PEEK at different concentrations, i.e., 70%, 80%, 85%, 90%, and 98% for 60 s on bond strength with resin cement and concluded that sulfuric acid concentration of 90% and 98% was the optimal concentrations to improve adhesion between PEEK and resin-based materials.^[31]

In aluminum oxide sandblasting, the alumina particles move at great speed, and their collision with the surface leads to the transformation of most of their kinetic energy into heat. This results in microporosity on the surface which enhances the micro-retention by creating a larger and more “active” surface and by increasing its wettability.^[32,33] In literature, alumina sand blasting for PEEK has been done with particle sizes of 50 μm and 110 μm . Rosentritt *et al.* compared the efficacy of the surface treatment of PEEK with 50 μm and 110 μm alumina particles on SBS with various resin cements and concluded that alumina particles of 110 μm showed higher SR value and better bond strength to resin cements.^[34]

After a thorough literature search, the following cements were selected: Self-etch resin cement-Subgroup A (Multilink^R N-Ivoclar Vivadent, Liechtenstein, Germany) and RMGIC-Subgroup B (RelyX Luting 2, 3M ESPE, USA).

The SBS values of each cement in all four groups were analyzed by a one-way ANOVA, and mean values were compared using Tukey’s HSD test, illustrated in Tables 3 and 4 which summarizes the performance of both cements used in this study for each group. For each cement, the difference in values of SBS shown by each group was statistically significant.

- Group 1: SBS test could not be established with the PEEK samples in this group, as they de-bonded during the oral simulation test, before being subjected to SBS test
- Group 2: The samples treated with alumina particles showed SBS values that were greater than that of Group 1 and Group 4 with the two cements, which can be correlated with greater SR of Group 2 (1.706 ± 0.160) as compared to that of Group 1 (0.147 ± 0.0238) and Group 4 (1.101 ± 0.167). In Group 2, the higher SBS was seen with subgroup A (self-etch resin cement – 3.91 ± 0.59 MPa) as compared to that of subgroup B (RMGI- 2.27 ± 0.39 MPa)
- Group 3: The samples treated with concentrated sulfuric acid showed highest SBS values amongst all groups with both cements. The higher SBS values were recorded with subgroup A (self-etch resin cement – 7.52 ± 1.20 MPa) compared to subgroup B (RMGI- 3.85 ± 0.36 MPa) which can be attributed to the use of a concentrated sulfuric acid in

conjunction with a resin cement. Concentrated sulfuric acid created a rougher surface as compared to other treatment methods in this study by dissolving the PEEK surface which can be demonstrated in the SEM findings [Figures 1c and 2c]. This was in accordance with the studies done by Zhou *et al.*^[15] and Silthampitag *et al.*,^[35] the use of an adhesive facilitated the deeper penetration of resin cement in contrast to RMGIC

- Group 4: The samples treated with synthetic diamond particles showed SBS values that were lower as compared to Groups 2 and 3 with both cements. This can be correlated with SR value for Group 4 (1.101 ± 0.167) which was significantly lower than the SR value of Group 3 (2.106 ± 0.186) and Group 2 (1.706 ± 0.160). The reason that could possibly be attributed to the lower SR value was the size of the synthetic diamond particles which did not penetrate deep into the PEEK surface which is demonstrated in the SEM findings. The higher SBS value in this group was recorded with subgroup A (self-etch resin cement – 2.27 ± 0.39 MPa) compared to that of subgroup B (RMGI – 1.03 ± 0.21 MPa) which can be attributed to the ability of self-etch resin cement in the presence of an adhesive system to possess a stronger bond in contrast to RMGIC which did not contain any adhesive.

Better results were seen with resin cement which could be attributed to the adhesive system which facilitated bonding with the functional groups in PEEK, thereby improving bonding of PEEK with the cement. Furthermore, Albert and El-Mowafy^[36] explained that the insoluble resin cement absorbs water which may help the relaxation of the internal stress caused by polymerization shrinkage, consequently reducing the potential of internal failure of the resin cement during thermo-cycling.^[37] This also contributes to superior SBS of this cement. Moreover, since there is a general agreement that resin cements possess lower polymerization shrinkage stresses, thereby provide good marginal integrity and low micro-leakage.^[36]

Types of failure: The results revealed that 100% of the samples from Group 1 showed adhesive type of failure [Figure 3a and b]. The samples from Groups 2, 3, and 4 showed 100% mixed failure, i.e., adhesive and cohesive type of failures, [Figure 3c-h] and also higher SBS values indicative of a strong bond between the PEEK and cement.

This study had limitations. First is the *in vitro* nature of the study. Second, the SBS test is not entirely representative of the mode of load application in a clinical situation.

Moreover, finally, it is not clinically feasible to use such highly corrosive solutions, as it needs careful handling. It may also imply the destruction of the etched superficial PEEK surface.^[22] This might have a weakening effect on PEEK structure and cause bond degradation at the interfaces.

CONCLUSION

- The SR SBS of samples treated with concentrated 98% sulfuric acid for 60 s was maximum, followed by the samples treated by sandblasting with 110 μm aluminum oxide particles, samples treated by 10–20 μm synthetic diamond particles was lowest with both the test cements
- The SBS of self-etch resin cement was higher compared to RMGI cement for all three surface treatments done on test samples
- 100% samples treated by concentrated 98% sulfuric acid, 110 μm aluminum oxide particles, and synthetic diamond particles showed mixed type of failure, i.e., mixture of adhesive and cohesive failure.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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