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Yasmin ElFiky
Master Candidate, Department of Fixed Prosthodontics, Faculty of Dentistry, Cairo University, Egypt

Omnia Nabil
Lecturer, Department of Fixed Prosthodontics, Faculty of Dentistry, Cairo University, Egypt

Hesham Alansary
Professor, Department of Fixed Prosthodontics, Faculty of Dentistry, Cairo University, Egypt

Corresponding Author:
Yasmin ElFiky
Master Candidate, Department of Fixed Prosthodontics, Faculty of Dentistry, Cairo University, Egypt

The effect of different air blasting pressure of polyetheretherketone (PEEK) on surface roughness and bond strength to adhesive resin cement: An *in-vitro* study

Yasmin ElFiky, Omnia Nabil and Hesham Alansary

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Abstract
Statement of the Problem: Polyetheretherketone material shows good physical and mechanical properties but research is lacking optimum air blasting parameters for good bond strength to resin cement.
Aim: To evaluate the effect of different air blasting pressure of PEEK on surface roughness and shear bond strength to adhesive resin cement.
Materials and Methods: 24 discs of BioHPP PEEK (bredent, UK) were manufactured. Samples were air blasted with 110 µm Al_2O_3 particles for 10 seconds at distance of 10 mm and divided into 3 groups according to pressure.
Group A: 3 bar, Group B: 2.5 bar and Group C: 3.5 bar. Surface roughness was measured by SEM, Visio Link primer (Bredent, Germany) was applied and cured then G-CEM resin cement (GC, Japan) was applied and cured. Shear bond strength was measured using universal testing machine. EDX analysis was done, and failure mode was detected by stereo-microscope.
Results: Statistical analysis showed that roughness average (Ra) increased by increasing pressure, Group B was significantly the lowest ($p < 0.05$), while there was insignificant difference between other groups ($P > 0.05$). Regarding shear bond strength, Group A recorded (12.6864 ± 2.486 MPa), Group B (11.2842 ± 1.555 MPa) and Group C (10.1024 ± 2.317 MPa), there was significantly statistical difference between Groups A & C ($p < 0.05$). The failure mode was mainly adhesive in group C and mixed in groups A & B. EDX analysis showed an increase in Carbon and Silica and a decrease in Oxygen and Aluminum weight percentages after the bond strength test.
Conclusion: Increasing the air blasting pressure up to 3 bar increases the surface roughness and bond strength to resin cement, but more than 3 bar showed unfavorable surface roughness and decreased bond strength.

Keywords: Shear bond strength, PEEK, air blasting pressure, surface treatment

Introduction
Polyetheretherketone (PEEK) is a polyaromatic, semi crystalline, thermoplastic polymer and is a member of polyaryletherketones (PAEK) family. PEEK has numerous uses in dentistry today, it can be used in the fabrication of single crowns, endocrowns, bridges up to four units (two pontics), bonded bridges (Maryland bridges) and implant prosthesis [1, 2].
It possesses mechanical characteristics close to that of dentin and bone of human body, it has low modulus of elasticity (3–4 GPa), which is similar to bone's, resulting in less stress shielding compared to titanium's and zirconia's extremely high modulus of elasticity. With this flexibility, PEEK gives natural feeling in the mouth and remain resilient even after years of biting and chewing. Due to its hardness, it doesn't cause attrition to the opposing teeth which is a major advantage [3-5].
The stability, durability, and long-term clinical success of the restoration are significantly influenced by the quality and durability of adhesive bonding. According to Kurtz *et al*, surface treatment is regarded as a necessary step for establishing adhesion of restorations since it impacts the surface's wettability, roughness, and area and so affects the bonding with resin cement [6].

PEEK has low surface energy and is chemically inert, so its adhesion to resin cement may be affected. Several methods of surface treatment of PEEK have been considered such as mechanical, physical and chemical to enhance adhesive bonding [7].

Mechanical surface treatment causes surface irregularities (roughness) that increases the bond to resin cement by micromechanical interlocking. Air blasting is the most commonly used method, since it is a safe, easy, available, affordable method of surface treatment [8].

The most suitable parameters of air blasting (using Alumina particles) for surface treatment of PEEK should be fully understood for proper treatment. The current literature revealed that the most optimum particles size which is 110 µm at distance of 10 mm for 10 seconds duration, but the most optimum air pressure was not mentioned.

So, the purpose of our work was to determine how the surface roughness of PEEK and its bond strength to adhesive resin cements are affected by different air blasting pressure using alumina particles.

Materials and Methods

Ethical Approval

The study was approved by research ethics committee at Faculty of Dentistry Cairo University with an approval number of 28-7-20.

Sample size

A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that air blasting pressure has no influence on the shear bond strength between PEEK and the used resin cement and the surface roughness of PEEK. By adopting an alpha level of (0.05) a beta of (0.2) i.e., power=80% and an effect size (f) of (0.699) calculated based on the results of Stawarczyk, Bogna, *et al.*; the predicted sample size (n) was a total of (24) samples. Sample size calculation was performed using G*Power version 3.1.9.7

Samples preparation

A cylinder of BioHPP PEEK (bredent, UK) with 18 mm length and 10 mm diameter was designed using Windows 3D builder and Meshmixer software and dry milled by a 5-Axis Dental Milling Machine (Redon GTR, Turkey) to produce 4 cylinders. Each cylinder was sectioned into 8 discs, each of 2 mm thickness using a bench lathe sectioning machine (BV20L, Xi'an Industrial Machinery, China) with a stainless-steel cutting disk. A total of 32 discs were produced, any disc with any defect was discarded to end up with 24 discs. The measurements of width and thickness of each disc were confirmed with a Digital caliper (Digital Vernier Caliper IP54, USA). Each sample was polished on its bonding surface with a 600-grit silicon carbide (SiC) abrasive paper under water for 40s and then air dried with dry air for 15 seconds.

Surface treatment for PEEK

The PEEK samples were air blasted by a dental laboratory sandblaster (Bego, Germany) using 110 µm Al_2O_3 particles for 10 seconds measured by a stop watch at a distance of 10 mm and at a 90-degree angle with 3 bar pressure for group A, 2.5 bar pressure for group B and 3.5 bar pressure for group C. Then they were cleaned with oil free dry air for 10 seconds.

For standardization of air blasting conditions, a custom designed wooden holder was fabricated, with a fixed distance between its arms of 10 mm, Figure (1). The sample is fixed on one of the arms and on the other arm there is a special groove which fits on the airblaster machine nozzle to adjust the 90 degrees angle projection, Figure (2).

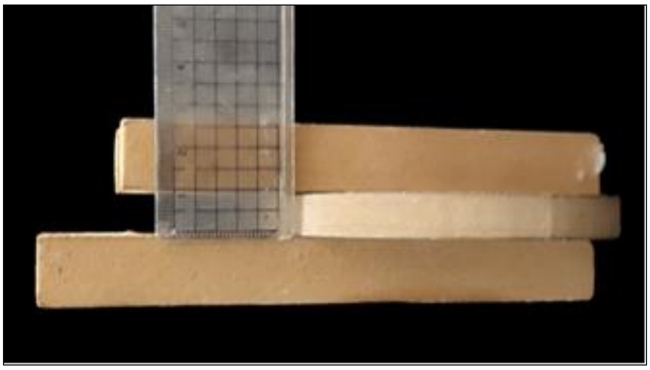

Fig 1: A custom designed wooden holder

Fig 2: One of the arms of the wooden holder fits on the airblasting machine nozzle to adjust the angle of projection

Topographic analysis

Surface images were recorded using Quanta FEG 250 scanning electron microscope (FEI Company, Hillsboro, Oregon-USA). The samples were mounted onto SEM stubs and examined and PEEK sample with no surface treatment were also examined for comparison. The SEM was operated at a working distance of 10.1 mm, with an in-lens detector with an excitation voltage of 20 kV. Each sample was analyzed at x1000, x5000 and x10000 magnification.

Representative images of different samples were selected to analyze by Image J software version 1.53 (USA) to detect the average roughness parameter (Ra) from SEM images.

Samples were selected for scanning electron microscope (SEM) model Prisma E (thermofisher company) attached with EDX unit for analyzing the elemental compositions of one of them before bonding and for the three of them after shear bond strength testing to compare alteration in the elemental composition. Samples were fixed on aluminium stubs with standard diameter using a carbon double sticky tape. SEM examination of each sample was operated at an accelerating voltage 20 kV. The examination of all groups was done at 500X and 1000 X magnifications.

Resin bonding

Adhesive agent application

Bonding was done immediately after air blasting of the surface to avoid any moisture contamination (9). A customized tape with a central hole of 3 mm diameter was put on the PEEK samples to define and standardize the bonding area [10]. Then a clean brush was used to apply visio. link primer (Bredent, Germany) in a thin coat over the exposed surface of the air-blasted surfaces of PEEK samples, Figure (3). Then it was light cured for ninety seconds by a bench top light polymerization device (bre. Lux Power Unit 2, Bredent, Germany) with LED light that provides careful and gradual increase of luminous power with a wavelength range of 370-400 nm and a semi matte finish of PEEK samples was obtained

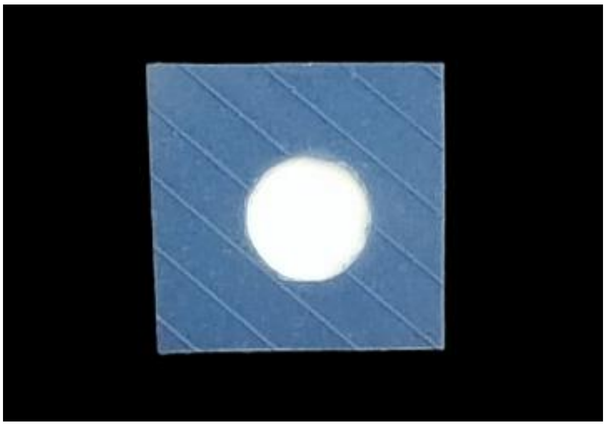

Fig 3: A customized blue tape with a central hole of 3 mm diameter on a PEEK sample

2.3 Adhesive resin cement application

A specially customized split mold cylinder which has an inner diameter 3 mm and a height 2 mm was constructed, Figure (4) on the bonded areas of PEEK samples. The G-CEM capsule (GC, Japan) was activated and used immediately and mixed in an amalgamator for 10 seconds according to the manufacturer's instructions and then the mold was filled with the resin cement and polymerized by light curing device (woodpecker ILED Plus, China) by a visible light of wavelength between 420 nm – 480 nm for 40 seconds to ensure optimal polymerization of the resin cement and then the mold is removed. All the polymerization procedures were performed according to the manufacturers' instructions.

Fig 4: A customized split mold cylinder with an inner diameter of 3 mm and a height of 2 mm

Shear bond strength measurement

Each sample was fixed in a PVC (polyvinyl chloride) tube with internal diameter 25 mm tube previously filled with acrylic resin to be held with during samples' testing. Before testing, samples were checked with a light stereo-microscope (Nikon MA100 Japan) at 30× magnification to discard sample with air bubbles or gaps at the interface.

The acrylic block with the sample was attached to the lower fixed head of the universal testing machine (Instron model 3345 England). A unibeveled chisel with 0.5 mm width blade was attached to the upper movable head of the testing machine, Figure (5), compression mode of force applied via the chisel blade which was placed as close as possible to the resin cement/slice interface at a crosshead speed of 1.0 mm/min up to sample failure.

The force required for failure (Newton) was divided by the surface area (mm^2) to calculate the shear bond strength in MPa by machine software BlueHill 3345 Instron England. EDX was then repeated.

Failure analysis

A stereo-microscope (Nikon MA100 steriomicroscope Japan) with a 20X magnification was used to study the debonded areas. Four failure types were considered: 1) Cohesive failure mode within PEEK, 2) Cohesive failure mode within the resin cement, 3) Mixed failure mode within PEEK and the resin cement (composite remnants partially left on PEEK with PEEK surface exposed), and 4) Adhesive failure mode between PEEK and the resin cement (no resin cement remnants left on the PEEK surface). Two examiners who were not aware of the group allocation examined the failure types.

Statistical Analysis

Statistical analysis was performed using SPSS 20®, Graph Pad Prism® and Microsoft Excel 2016. Data was represented as mean and standard deviation. Data were explored for normality by using Shapiro Wilk and Kolmogorov-Smirnov normality tests and comparison between groups & between three sections were performed by ANOVA test followed by Tukey`s post hoc test for multiple comparisons.

Results

Surface roughness

Control group (PEEK with no surface treatment)

In PEEK with no surface treatment group, Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) were presented in table (1).

Table 1: Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) regarding PEEK with no surface treatment Group

Control group (PEEK with no surface treatment)	
Minimum	12.6104 µm
Maximum	16.6679 µm
Mean	15.1635 µm
Median	16.2122 µm

Group A (3 bar)

In 3 bar pressure group, Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) were presented in table (2).

Table 2: Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) regarding 3 bar pressure group

Group A (3 bar)	
Minimum	26.8035 µm
Maximum	35.9847 µm
Mean	30.2691 µm
Median	29.1441 µm

Group B (2.5 bar)
In 2.5 bar pressure group, Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) were presented in table (3).

Table 3: Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) regarding 2.5 bar pressure group

Group B (2.5 bar)	
Minimum	23.3845 µm
Maximum	28.5342 µm
Mean	26.315725 µm
Median	26.6721 µm

Group C (3.5 bar)
In 3.5 bar pressure group, Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) were presented in table (4).

Table 4: Minimum, maximum, mean, median and standard deviation of surface roughness average (Ra) regarding 3.5 bar pressure group

Group C (3.5 bar)	
Minimum	28.6974 µm
Maximum	35.0521 µm
Mean	30.85528 µm
Median	30.0497 µm

Comparison between surface roughness of all groups
Mean and standard deviation of all groups were presented in **figure (5)**. Comparison between the groups was performed by using One Way ANOVA test which revealed significant difference between them as $P < 0.001$.
Multiple comparisons were performed by using Tukey`s Post Hoc test and revealed that:

- **Group A & Group B**: Group A (30.27±3.976) was insignificantly higher in surface roughness values than group B (26.32±2.431).
- **Group A & Group C**: Group A (30.27±3.976) was insignificantly lower in surface roughness values than group C (30.86±2.592).
- **Group B & Group C**: Group B (26.32±2.431) was significantly lower in surface roughness values than groups C (30.86±2.592).
- **Group A & Control**: Group A (30.27±3.976) was significantly higher than control group (15.16 ± 2.22).
- **Group B & Control**: Group B (26.32±2.431) was significantly higher than control group (15.16 ± 2.22).
- **Group C & Control**: Group C (30.86±2.592) was significantly higher than control group (15.16 ± 2.22).

(PEEK with no surface treatment group was significantly the lowest, then Group B was significantly the lowest, while there was insignificant difference between other groups)

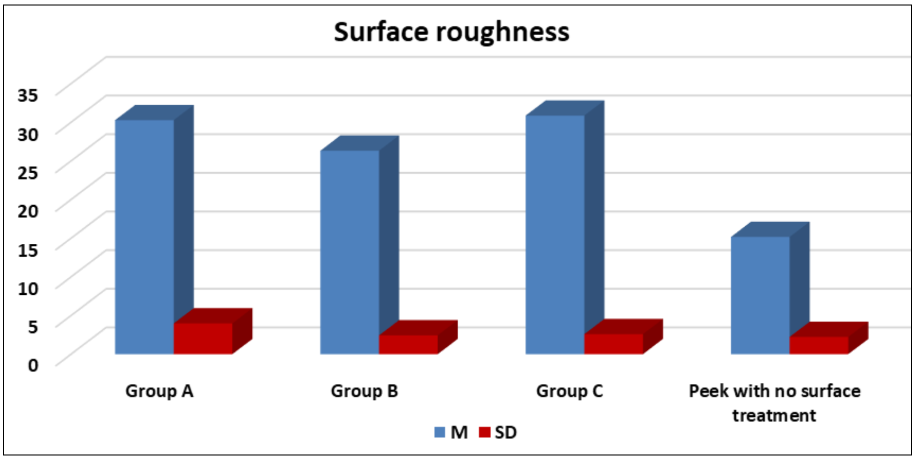

Fig 5: Bar chart showing Mean and standard deviation of surface roughness at maximum load for Group A, Group B and Group C and the control group

Shear bond strength at maximum load
Group A (3 bar)
In 3 bar pressure group, Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load were presented in table (5).

Table 5: Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load regarding Group A (3 bar)

Group A (3 bar)	
Minimum	8.0307 MPa
Maximum	14.81258 MPa
Mean	12.6864 MPa
Median	13.76724 MPa

Group B (2.5 bar)
In 2.5 bar pressure group, Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load were presented in table (6).

Table 6: Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load regarding Group B (2.5 bar)

Group B (2.5 bar)	
Minimum	8.59369 MPa
Maximum	13.50423 MPa
Mean	11.2842 MPa
Median	11.28419 MPa

Group C (3.5 bar)
In 3.5 bar pressure group, Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load were presented in table (7).

Table 7: Minimum, maximum, mean, median and standard deviation of shear bond strength at maximum load regarding Group C (3.5 bar)

Group C (3.5 bar)	
Minimum	6.73424 MPa
Maximum	15.06365 MPa
Mean	10.1024 MPa
Median	9.99857 MPa

Comparison between shear bond strength of all groups

Mean and standard deviation of all groups were presented in **figure (6)**. Comparison between the groups was performed by using One Way ANOVA test which revealed insignificant difference between them as P = 0.057**.**

Multiple comparisons were performed by using Tukey`s Post Hoc test and revealed that:

- **Group A (3 bar) & Group B (2.5 bar):** Group B (11.28±1.555 MPa) was insignificantly lower than group A (12.69±2.486).
- **Group A (3 bar) & Group C (3.5 bar):** Group A (12.69±2.486 MPa) was significantly higher than group C (10.10±2.317).
- **Group B (2.5 bar) & Group C (3.5 bar):** Group B (11.28±1.555 MPa) was insignificantly higher than group C (10.10±2.317).

(There was only significant difference between Group A (3 bar) and Group C (3.5 bar))

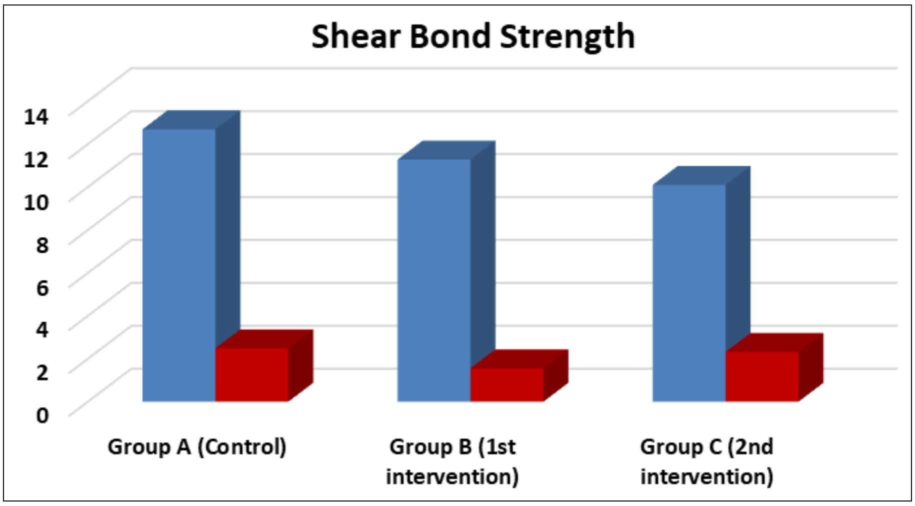

Fig 6: Bar chart showing Mean and standard deviation of shear bond strength at maximum load for Group A, Group B and Group C

Failure analysis

The majority of failure in 2.5 (67%) and 3 bar (88%) pressure groups was mixed failure, while that in the 3.5 bar (62%) pressure group was adhesive failure.

Elemental analysis

There was an obvious increase of Carbon and an increase in the Silica weight percentage in all the PEEK samples after the shear bond strength test compared to that before. They were the least in the 3.5 bar pressure group.

However, a decrease of Oxygen and Aluminum weight percentages was noted. A comparison between the weight percentages of elements C, O, Al and Si before and after the shear bond strength test was shown in table (8).

Table 8: EDX analysis comparing the weight percentages of elements C, O, Al and Si before and after the shear bond strength test

	No surface treatment	2.5 bar pressure group	3 bar pressure group	3.5 bar pressure group
Carbon (C)	35.99% - 36.55%	43.24% - 46.93%	45.94% - 47.04%	45% - 46.77%
Oxygen (O)	30.37% - 30.9%	26.04% - 29.04%	25.5% - 29.3%	24.9% - 30.2%
Aluminum (Al)	11.02% - 11.26%	2.82% - 6.16%	0.89% - 1.19%	3.74% - 7.43%
Silica (Si)	0.99% - 1.04%	3.09% - 4.71%	1.41% - 6.21%	1.24% - 4%

Discussion

Adhesive bonding is very important for the stability and long term clinical success of the prosthesis (6). Surface treatment is regarded as a necessary step for establishing adhesion of restorations since it impacts the surface's wettability, roughness, and area and so improves the bonding with resin cement.

Air blasting is the surface treatment of choice, since it is a safe, easy, available, economic method of surface. The exact parameters of air-borne particle abrasion were mentioned in previous studies where we use 110 µm aluminum oxide particles at a distance 10 mm for 10 seconds but the exact optimum pressure was not mentioned clearly in literature [11, 12].

Respectively the current study was conducted to evaluate the effect of different air blasting pressure on surface roughness of PEEK and bond strength to adhesive resin cement.

The study design of the current study was invitro study, since we are trying new pressure values and also bond strength tests cannot be performed intraorally.

The material of choice to obtain PEEK samples in this study was BioHPP which is a reinforced PEEK material with ceramic fillers as it's widely used in fixed dental prosthesis production [2].

The size of aluminum oxide particle chosen in this study was 110 µm as the manufacturer and other research claimed that this size provides favorable surface roughness and the best shear bond strength [13].

We used orthogonal projection (air blasting with angle 90 degrees) because in the inclined projection, the abrasion of the material is greatly affected by complicated phenomena that result in grooves from sliding and rotating [11].

The pressure values used were 2.5 bar and 3 bar pressure where the manufacturer of BioHPP recommended pressure value from 2.5-3 bar pressure, and 3.5 bar pressure was chosen as a new intervention [14].

A dry air blasting treatment was carried out in order to prevent surface fluid adsorption transmitted by particles in the case of wet air blasting, which causes morphological variations (such as changing crystallinity) that change the material's rheological behavior [11].

After air blasting, cleaning was done only by dry air to remove remaining aluminum oxide particles on the surface since if steam is used, water residue will be retained in the PEEK which may affect the bond strength [15].

Visio-link primer was used. It contains methylmethacrylate (MMA) monomers which has great importance in bonding with PEEK. Also, it contains dimethacrylates (DMA) and pentaerythritol-triacrylate (PETIA) which has a high capacity to modify the PEEK surface. Visiolink primer showed high results for bond strength and survival rates as it helps to build a durable bond of PEEK with resin cement. The use of resin cement alone is not recommended, (9,16); [17].

The resin cement of choice was self-adhesive dual-cured resin cement (G-CEM resin cement "GC, Japan") was, which has an organic matrix made of multifunctional phosphoric-acid methacrylates. The tooth surface is conditioned by these phosphoric-acid groups, which also aid in adhesion. The cementation process is straightforward and lessens postoperative sensitivity [18].

Shear bond strength (SBS) test was done to evaluate the bond strength since it has many benefits, including ease of sample preparation, a straightforward testing protocol, a lower incidence of pretest failure, ease of sample alignment with the loading device, and overall non-technique sensitivity. It is

One of the most popular methods for measuring bond strength. It is more ideal for assessing the bonding capabilities of resin cements to PEEK since it could easily replicate the clinical condition [19, 20].

A scanning electron microscope was employed for surface roughness measurement since it can produce images of sufficient quality and resolution to detect fine details in surface topography to a level of nanoscale [21].

EDX analysis was done to know the change in elemental composition before and after the shear bond strength test which may be an indicative to the penetration of the visiolink adhesive and resin cement to the PEEK surface.

The null hypothesis, which claimed that altering the air blasting pressure has no effect on the surface roughness of PEEK and shear bond strength with resin cement, was rejected.

When the pressure was raised from 2.5 to 3 bar, the shear bond strength increased, indicating that greater pressure could improve the bond to PEEK material because it increases the surface area with which the adhesive can make contact when creating a bond and adds more mechanical interlocking at the interface. But when the air-abrasion pressure increased from 3 bar to 3.5 bar, shear bond strength decreased as this pressure increase improves shear bond strength only to a certain point. These outcomes could be explained by the severe surface flaws and defects that occur at higher pressures, where extreme roughness might prohibit the adhesive from flowing, resulting in reduced total surface contact and trapped air that weakens the bond [22, 23].

All the pressure values used gave clinically acceptable shear bond strength values for the resin based materials in the oral environment which is 10 to 12 MPa according to ISO 10477. However, the 3.5 bar pressure group showed a mean of 10.1024 MPa which is very near to the minimum clinically acceptable shear bond strength.

The results of shear bond strength were inconsistent with Stawarczyk *et al*. who examined the impact of increasing pressure value from 0.5 bar to 3.5 bar on the shear bond strength between PEEK and veneering resin composite and proved that samples treated with air blasting pressure 3.5 bar showed the higher survival rate than that treated with 0.5 bar pressure [9].

Although there are no enough studies evaluating the effect of increasing bar pressure in PEEK and its effect on shear bond strength with resin cements but the process was carried out on zirconia which has the same criteria as PEEK in being inert [24].

Zhang *et al*. studied the effects of air blasting pressure on the shear bond strength between resin cement and translucent zirconia. They increased air blasting pressure from 1 bar to 5 bar and the results showed that shear bond strength increased significantly in the 2 bar pressure group but there was insignificant increase in the 3 bar pressure group and then the SBS significantly decreased in the 4 and 5 bar pressure groups. These findings were supported by SEM photos, where severe surface flaws and defects occurred at higher pressures [23].

On the contrary, Lümkemann *et al*. who investigated the impact of increasing air-abrasion pressure from 0.5 bar to 2 bar and finally to 4 bar and found that changing the air abrasion parameters didn't affect the results of the tensile bond strength values of the three groups when increasing pressure and using Visio link adhesive. This may be due to the different bond strength test used which is tensile not shear bond strength and the use 50 μm aluminum oxide particles which were proved to give significantly lower surface roughness values than 110 μm aluminum oxide particles [16].

The findings of shear bond strength were correlated with the images of the scanning electron microscope, where 3.5 bar air abrasion group images showed severe surface flaws and defects, while 3 bar pressure group showed more favorable surface irregularities, multiple grooves and deeper pores than 2.5 bar group images.

This was in agreement with Lümkemann *et al*. who found that the parameters of the surface roughness which are average roughness "Ra" and ten-point height of irregularities "Rz" are increased by increasing the pressure of air abrasion [16].

The failure analysis for the groups showed adhesive and mixed modes of failures. According to EL-Wassefy *et al*., it was found that the degree of substrate fracture is frequently predictive of the retentive adhesive strength. The mode of failure occurred supports the findings of the bond strength values obtained in this study where adhesive failure occurred mainly in the 3.5 bar group, which had the least shear bond strength results while mixed failure occurred in the other two groups where there was more than 50% of resin on the PEEK surface in the examined sample of 2.5 bar pressure group, and more than 75% of resin on the PEEK surface in the examined sample of 3 bar group which had the highest shear bond strength results [25].

The elemental analysis showed that the increase in the percentage of carbon and silica in the samples after the shear bond strength test means that they most probably share in the formation of a chemical bond between resin cement and PEEK surface while the decrease of Oxygen and Aluminum percentage means that they most probably don't share in the formation of chemical bond between resin cement and PEEK. Carbon and silica are the main constituents of Visio link adhesive and resin cement so EDX is a good indication of the penetration of Visio link adhesive and resin cement into PEEK surface. The least increase in the percentage of carbon (the main constituent of the resin cement) was in 3.5 bar pressure group which showed the adhesive failure.

A general limitation of this study is that *in vitro* experiments can't completely represent the mouth's true environment, but they may suggest a reliable bond formation of PEEK restorations in dentistry. To describe the long-term bonding robustness, more in-vivo research is required.

Conclusions

Within the limitations of this study, the following could be concluded

1. The air blasting pressure is an important parameter in the surface treatment of PEEK material before PEEK cementation with resin cement.
2. Increasing the air blasting pressure, increases the surface roughness of PEEK material.
3. 3 bar air blasting pressure showed the highest shear bond strength of PEEK to adhesive resin cement.
4. 3.5 bar air blasting pressure increases surface roughness of PEEK but adversely affects its shear bond strength to adhesive resin cement.

Recommendations

- Further in-vivo studies are recommended to simulate the oral environment.
- Further studies are recommended to test the effect of different air abrasion pressure values on the shear bond strength with resin cement after thermo-mechanical aging.

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Conflict of Interest

The authors declare that they have no conflict of interest regarding the publication of this paper

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