



The Effect of Different Times of Plasma Treatment on the Bond Strength of Polyether-Ether-Ketone to Composite Resin

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Research Article

History

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ABSTRACT

Objective: This study aimed to investigate the effect of plasma treatment of varying durations on the surface roughness of PEEK and its bond strength to veneering composite.

Materials and Methods: 36 samples (7x7x2 mm in size) were prepared using a PEEK sheet, sanded with a silicon carbide sandpaper to standardize the surfaces sandblasted with Al₂O₃. They were divided into four groups in terms of surface treatments: (A)sandblasting (control), plasma treatment for 1 minute(B), 2 minutes(C), and 4 minutes(D). According to adhesive application shear bond strength (SBS) values of specimens were measured. The data were evaluated using the analysis of variance and the Tukey multiple comparison test, as well as the Pearson Correlation Coefficient between surface roughness and bond strength.

Results: The analysis of variance showed no significant effect from varying durations of plasma treatment on roughness (p=0.182) and bond strength (p=0.345). The highest bond strength was found in the samples of Group D. The lowest value was found in the group of samples that underwent plasma treatment for 1 minute (Group B). Limitations of this study are that it is an in vitro study and cannot reflect the inside of the mouth.

Conclusion: The highest and lowest roughness values were found in the control group (A) and the 2-minute treatment group (C), respectively. The highest and lowest bond strength values were found in the 4-minute treatment group (D) and the 1-minute treatment group (B), respectively. No correlation was found between surface roughness and bond strength.

Keywords: PEEK, veneering composite, plasma, surface roughness, bond strength.

Polieter-Eter-Ketonun Kompozit Rezine Bağlanma Dayanımına Farklı Sürelerdeki Plazmanın Etkisi

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Öz

Amaç: Bu çalışma, farklı sürelerdeki plazma işleminin polietereterketonun yüzey pürüzlülüğü ve kompozit rezine bağlanma dayanımı üzerindeki etkisini araştırmayı amaçladı.

Gereç ve Yöntemler: Al₂O₃ ile kumlanan yüzeyler, silisyum karbür zımpara kâğıdı ile zımparalanarak standardizasyonu sağlanan 36 adet numune (7x7x2 mm boyutunda) PEEK hazırlandı. Örnekler rastgele dört gruba ayrıldılar: (A)kumlama (kontrol), 1 dakika plazma(B), 2 dakika plazma(C) ve 4 dakika plazma(D). Kompozit rezinle adezyonu sağlanan numunelerin bağlanma dayanımı değerleri ölçüldü. Veriler varyans analizi ve Tukey çoklu karşılaştırma testinin yanı sıra yüzey pürüzlülüğü ile yapışma mukavemeti arasındaki Pearson Korelasyon Katsayısı kullanılarak değerlendirildi.

Bulgular: Varyans analizi ile değişen plazma tedavisi sürelerinin pürüzlülük (p=0,182) ve bağlanma kuvveti (p=0,345) üzerinde anlamlı bir etki göstermedi.

Sonuç: En yüksek ve en düşük pürüzlülük değerleri sırasıyla kontrol grubunda (A) ve 2 dakikalık tedavi grubunda (C) bulundu. En yüksek ve en düşük bağlanma mukavemeti değerleri sırasıyla 4 dakikalık tedavi grubunda (D) ve 1 dakikalık tedavi grubunda (B) bulundu. Yüzey pürüzlülüğü ile bağlanma dayanımı arasında herhangi bir korelasyon bulunamadı.

Anahtar Kelimeler: PEEK, kompozit rezin, plazma, yüzey pürüzlülüğü, bağlanma dayanımı

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Introduction

The recent rise in aesthetic expectations has added momentum to research studies in materials science.¹ In this context, polyether-ether-ketone (PEEK) has attracted a lot of attention in dentistry due to the issues associated with the aesthetics of a metallic reflection observed with metal-ceramic restorations, as well as the related issues with marginal integrity, a lingering metallic taste, and possible allergic reactions to metal materials.² PEEK is less reactive, insoluble in water, and corrosion resistant.³ It also offers additional features including high failure resistance and low elastic modulus. Owing to all these features, PEEK has come to the foreground as a good alternative abutment material for both fixed and removable prostheses.^{4,5} However, all this superiority in mechanical and physical properties will not be able to help overcome the aesthetic issues associated with the greyish-brown and opaque colour of the material. Therefore, PEEK must be veneered with composite material before it can be used as an abutment material in fixed prosthetic treatments.⁶ This composite veneering process for PEEK, however, must secure the desired level of adhesion, which requires an improvement on the naturally low surface energy of the material. PEEK is considered to suffer from issues with adhesion as a result of its inherent resistance to chemicals. This adhesion issue must be addressed before PEEK can be put to widespread clinical use.³ The relevant literature has primarily focused on achieving a reliable improvement in the bonding between PEEK and veneering composite.² These studies have mainly attempted to improve the bond strength of composite to micro-retentive areas created through increased surface roughness.⁷

Long-term adhesion can only be achieved through a combination of chemical and micro-mechanical retention. To secure micro-mechanical retention, steps are taken to widen the surface area by increasing surface roughness and to improve the wettability by decreasing surface tension.⁸ At this point, plasma treatment comes forward as an alternative approach to improving wettability of materials in general and the bond strength of veneers more specifically.⁹⁻¹¹ Gas plasmas are partially ionised gases at room temperature comprising ions and reactive particles such as electronically excited neutrals and free radicals.^{12,13}

Changes to surface energy and improved bond strength have been reported with non-thermal atmospheric plasma (NTAP) treatment.¹⁴ Plasma has been shown to improve the wettability and adhesive properties of tooth structures such as enamel and dentin, as well as of veneering composites.¹² In addition, studies have found that plasma treatment improves the bond strength of primer and adhesive materials to the dentin.¹⁵⁻¹⁸ Argon plasma treatment on PEEK itself has also been found to improve bond strength.¹⁹ Non-thermal atmospheric plasma (NTAP) is an ionized gas that can improve the surface wettability and energy of polymers.²⁰⁻²² Positive results have been reported with low-pressure argon and

oxygen plasma treatment on the bonding between veneering composites and PEEK.^{23,24} Studies aiming to improve bond strength have attempted to do so through various durations of plasma surface applications. However, no consensus has been reached concerning the optimum duration for such treatments.

This study aims to investigate the effect of various durations of plasma treatment on the surface roughness of PEEK samples and their bond strength to veneering composite. To this end, the study hypothesises that various durations of plasma treatment on the surfaces of PEEK samples will increase surface roughness, and thus lead to improved bond strength.

Material and Methods

The G*Power software package (G*Power Ver. 3,0,10, Franz Faul, Universität Kiel, Germany) was used to determine the sample size. According to the results, a total of 32 samples were needed with a type I margin of error of $\alpha=0,05$ for 80% power at an effect size of 25%.

Polyether-ether-ketone (PEEK, Whitepeaks Dental Solutions) samples used in the study were sized to 7 x 7 x 2 mm with a K5 Vhf milling device (K5, Vhf, software version DentalCAM 7). The surfaces of the samples were standardized using 200-600-grit SiC sandpaper. Then, all samples were treated with a sandblaster (Zhermack, Rovigo, Italy) by the same operator using Al₂O₃ under 2.8 bar pressure from a working distance of 10 mm at an angle of 45° perpendicular to the surface for 15 seconds. The samples were cleaned in an ultrasonic bath with 96% ethanol for 5 minutes and then dried with compressed air.

The samples of the control group were set aside, and non-thermal atmospheric plasma (NTAP) (Kinpen, Neoplas, Germany) containing argon was applied on the bonding surfaces of the samples of the other three groups for various durations. The gas flow rate was set to 5 L/min and the gas pressure to 2.5 bar throughout the application. The plasma device was powered at 21 kHz frequency with 5 kV voltage (Figure 1). The plasma was applied to the sample groups for 1, 2, and 4 minutes, respectively, with care taken to leave 10 mm of a distance between the applicator tip and PEEK materials. The samples were grouped as indicated in Figure 2.

Surface roughness was measured for each sample using a stylus profiler (Kla Tencor Stylus Profiler P7).

For an examination of surface topography, an additional sample had been prepared for each group and these extra samples were taken onto a scanning electron microscope (Zeiss Sigma300, Carl Zeiss Microscopy GmbH 07745 Jena, Germany) at 5kV to obtain images magnified by x500, x2000, and x5000.

An adhesive (Visio.link, Bredent, GmbH & Co KG, Senden, Germany) was applied on the bonding surfaces of all samples with the help of a micro brush for 5 seconds. The samples were then polymerised for 90 seconds with a polymerisation device powered at 220 mW/cm² with a wavelength of 370-500 nm as per the recommendation of the manufacturer.

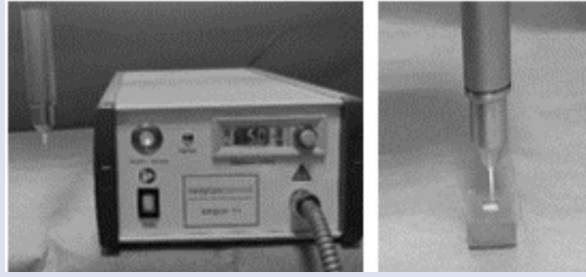


Figure 1. Non-thermal atmospheric plasma treatment

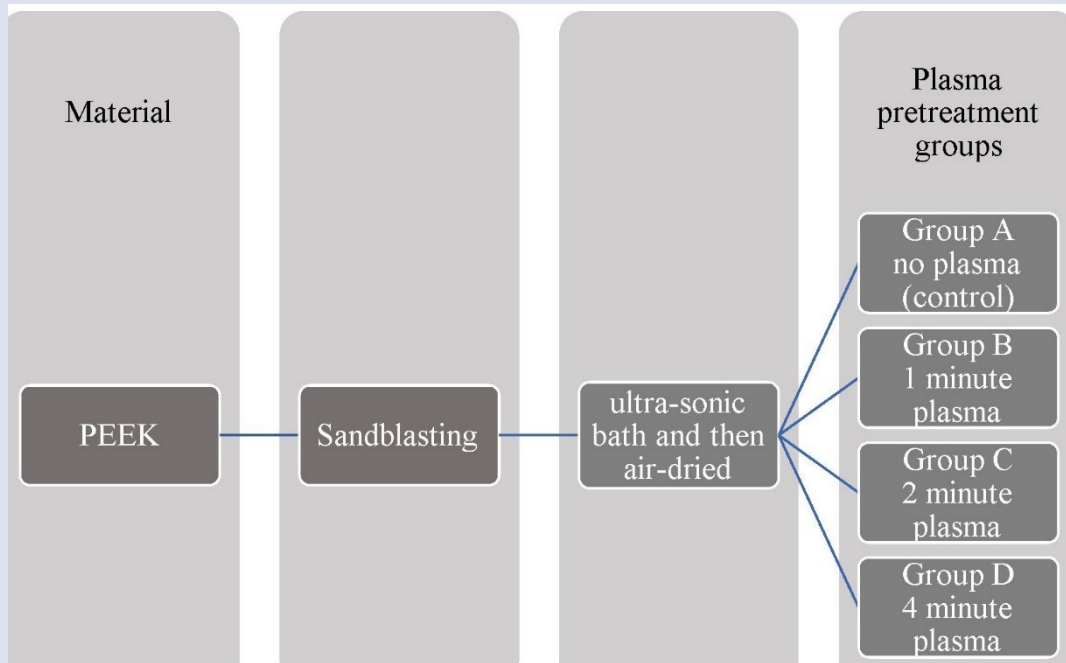


Figure 2. Grouping of study samples by surface treatment (n=8)

For the veneering composite application (Crea.lign Bredent, Germany), Teflon moulds were prepared using an internal diameter of 3 mm and a depth of 3 mm. Veneering composites were placed into the moulds with the PEEK sample kept at the centre of each mould and they were then polymerised in line with the recommendations of the manufacturer. The samples were then laid into acrylic blocks through cylindrical moulds of 15 mm in diameter and 20 mm in depth for subsequent placement into the mechanism prepared for the Universal Tester. All samples were kept in distilled water at 37°C for 24 hours before being subjected to shear bond strength testing in the Universal Tester (Instron 3340, Wycombe, United Kingdom) at the Research Laboratory of the Faculty of Dentistry of Recep Tayyip Erdogan University. To perform the test, the samples were fixed on the device holder and then, the metal end, shaped like the edge of a knife, was placed parallel to the bonding interface between PEEK and composite material, and force was applied on the sample at a head speed of 0.5 mm/min. The maximum force value achieved at the moment of separation of the composite material from the PEEK surface was measured in Newton (N). The breaking load

was divided by the bonding area to calculate the bond strength value in Megapascal (MPa).

Shear resistance (MPa) = Load (N) / Area (mm²), Area = (mm²), r = radius of the bonding surface, π=3.14

The types of failure were assessed on a stereomicroscope (Carl Zeiss AG, Oberkochen, Germany).

The data obtained were evaluated through an analysis of variance, the Tukey multiple comparison test, and the "Pearson Correlation Coefficient".

Results

The results of the analysis of variance undertaken to evaluate the data obtained for the study indicated that any variation in the duration of plasma treatment was not significant in terms of its effect on roughness (p=0.182) and bond strength (p=0.345).

A comparison of surface roughness values found the highest value in the control group (Group A) and the lowest value in the group of samples that were subject to 2 minutes of plasma treatment (Group C) (Table 1 and Figure 3).

The data on bond strength values are given in Table 2.

In terms of bond strength, the highest value was found in the group of samples that underwent plasma treatment for 4 minutes (Group D), and the lowest value was found in the group of samples that underwent plasma treatment for 1 minute (Group B) (Table 2). Mean graph of the bond strength values to veneering composite among groups is given in Figure 4.

The Tukey multiple comparison test undertaken to compare the groups showed that the differences between the groups in terms of roughness and bond strength were not significant ($p>0.05$).

When compared in terms of bond strength, all groups except for Group B (6.34 MPa) performed better than the control group, while the highest bond strength was found in the samples of Group D (11.08 MPa).

Profiler and SEM images are given in Figures 5 and 6, respectively.

The Pearson correlation test found no significant correlation ($p>0.05$) between the groups in terms of their average scores in roughness and bond strength ($r=1$).

Considering the types of failures observed, there were adhesive failures in 28 samples and mixed failures in 4 samples.

Table 1. Average, minimum, and maximum values and standard deviation of roughness (n=8)

	Mean (Ra)	Minimum (Ra)	Maximum (Ra)	Standard deviation
Group A (Control)	7.45	4.04	16.48	5.11
Group B	6	3.19	9.92	2.77
Group C	4.25	3.22	5.29	0.76
Group D	7.37	3.61	11.89	3.23

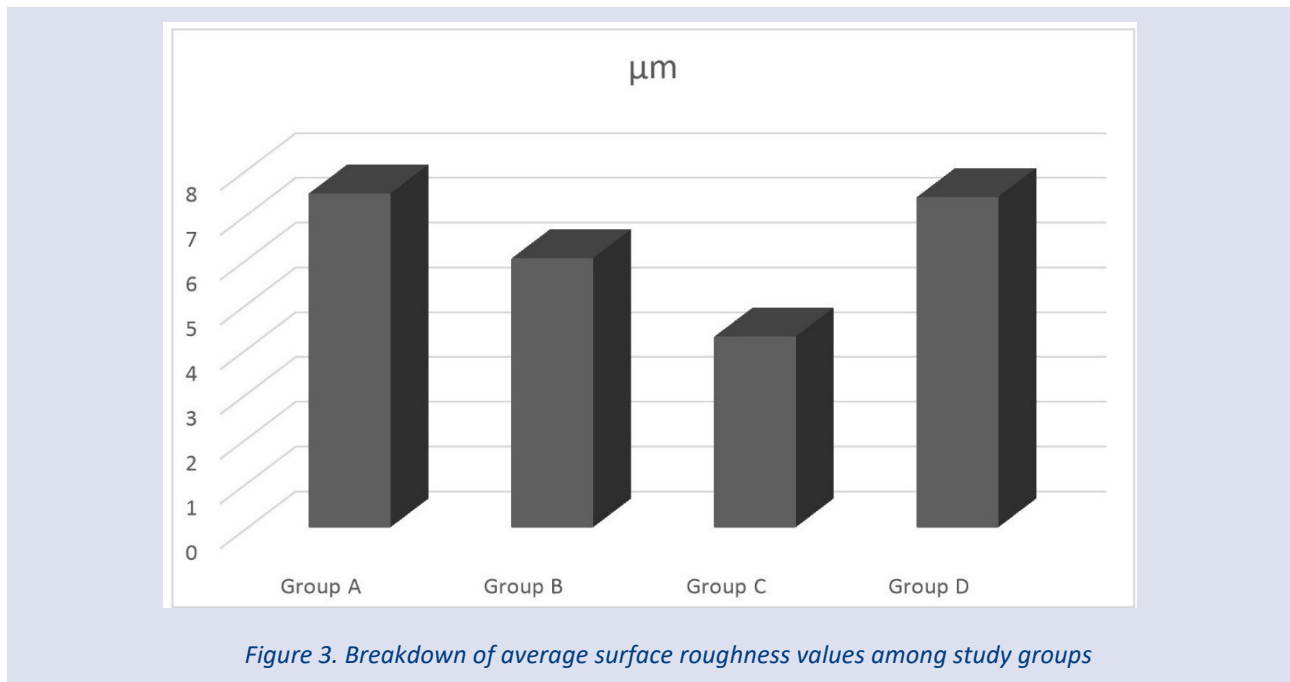


Figure 3. Breakdown of average surface roughness values among study groups

Table 2. Breakdown of bond strength data (n=8)

	Mean (MPa)	Minimum (Mpa)	Maximum (Mpa)	Standard deviation
Group A (Control)	7.52	6.83	8.35	0.62
Group B	6.34	3.92	9.52	2.56
Group C	9.7	5.84	16.15	4.26
Group D	11.08	6	19.91	5.23

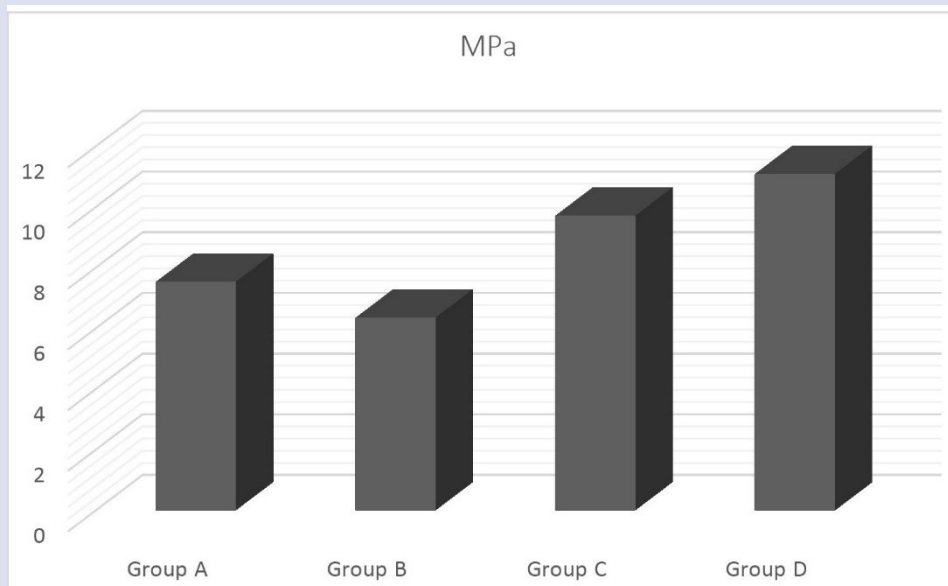


Figure 4. Bond strength to veneering composite among groups

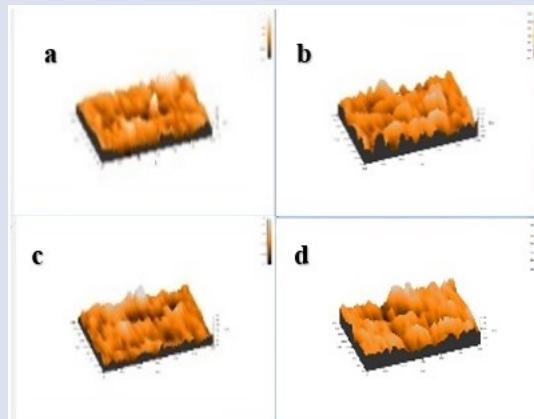


Figure 5. Post-Treatment Profiler Images of PEEK Samples for (a) Group A, sandblasting (control); (b) Group B, 1 min Plasma Treatment; (c) Group C, 2 min Plasma Treatment, and (d) Group D, 4 min Plasma Treatment

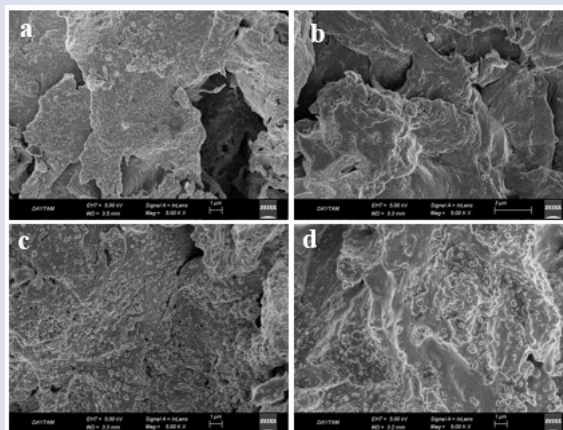


Figure 6. Post-Treatment SEM Images of PEEK Samples at x5000 Magnification for (a) Group A, sandblasting (control); (b) Group B, 1 min Plasma Treatment; (c) Group C, 2 min Plasma Treatment, and (d) Group D, 4 min Plasma Treatment

Discussion

PEEK has several features for its use in prostheses; however, its disadvantage of having an unsuitable colour for monolithic use means that composite veneering must be added to the procedure.^{7,11} As an inert material, PEEK also suffers from an unsuitable composition and low surface energy, which complicates its adhesion to veneers.⁷ In fact, PEEK is resistant to almost all organic and inorganic chemicals, as well as any surface changes created by a variety of chemical processes. Therefore, the question of increasing surface energy to improve bond strength to veneering composite brings forward an important challenge that needs a solution as it may shed light on the entire range of the material's benefits. This challenge is currently attempted by way of trials of various methods of surface modification. The studies report that surface treatment on PEEK is a prerequisite for adequate adhesion with veneering composite, and its absence will lead to inadequate bond strength values.^{3,26}

Plasma treatments find increasingly more uses in medicine and dentistry.²⁷ The literature on the effects of plasma treatments surface modification reports this application of improving surface energy by making the material hydrophilic as a possible way to increase bond strength to other materials.²⁸ The studies have attracted the attention of the dental community to plasma treatments.²⁹ In this vein, this study attempted to establish the optimum duration of plasma treatment for maximum bond strength by treating samples with plasma for different durations.

Jha *et al.*³⁰ reported atmospheric-pressure plasma to be more effective than low-pressure plasma for improved surface energy in PEEK. Iqbal *et al.*³¹ compared atmospheric-pressure plasma and low-pressure plasma in terms of effectiveness when used with high-performance polymers and found that treatment with atmospheric-pressure plasma increased surface energy significantly. Built on similar work^{30,31}, the present study entailed surface treatment of samples in plasma groups with a compact cold plasma device using atmospheric pressure. This device system is also known as non-thermal atmospheric plasma (NTAP).

ISO 11405³² requires the use of Teflon moulds or perforated plasters in the application of restorative materials on surfaces. Accordingly, this study was conducted with the use of appropriate Teflon moulds for adhesion between PEEK and veneering composites in line with similar studies.^{23,24}

In this study, roughness measurements were taken and SEM images of extra samples in all groups were obtained to allow for a detailed examination of the effects of the surface morphology of PEEK material on bond strength following varying durations of plasma treatment. As a result, the highest roughness value was found in the samples of the control group (7.45 Ra), and the lowest value was found in the samples of the group that was subjected to 2 minutes of plasma treatment (4.25 Ra).

Schwitalla *et al.*²⁴ evaluated the effects of a range of surface treatments including sandblasting, low-pressure argon/oxygen (Ar/O₂) plasma treatment, and a combination of these treatments on the roughness, contact angle, and shear bond strength of PEEK surfaces and found a decrease in surface roughness in plasma treatment groups. The authors attributed this phenomenon to the thermal impact created by plasma treatment at 70°C or to the smoother surface structure resulting from plasma acting more abrasively on the most prominent peaks. In a similar vein to the findings of Schwitalla *et al.*²⁴ and Türkkal³³, the findings of this study indicate that surface roughness is reduced more significantly in plasma-treated groups than among controls and yet, the difference between plasma-treated samples and samples in the control group is not significant. The specific plasma used for the study (NTAP) not creating any thermal impact in its use, combined with the abrasive effects of the plasma treatment, is thought to account for the reduction in surface roughness.

The studies report that surface treatments on PEEK (sandblasting and plasma treatment) create the conditions necessary for a strong bond between PEEK material and veneering composite.^{23,24} In this study, the results obtained in all plasma treatment groups except for Group B were compared with the results in the control group, and it was found that bond strength increased in all groups with even the control group and Group B giving values higher than the minimum 5 MPa threshold specified in ISO 10477.²³ The hypothesis of the study was that plasma applied on PEEK surfaces for various durations would increase surface roughness and thus bond strength, which is partially accepted by way of the above finding.

The studies indicate that the highest bond strength was found in the group treated with Ar gas for 4 minutes among plasma treatments performed with Ar, nitrogen (N₂) and O₂ gases for different durations (0.5, 1, 2, 3, and 4 minutes). Similarly, the present study found the highest bond strength in Group D which was treated with plasma for 4 minutes. There was no significant link between surface roughness and the increase in bond strength after plasma treatment, whereas roughness values were consistent with the SEM images.

The improvement in bond strength following plasma treatment has also been posited to stem from the removal of organic residues by plasma.²⁵ For the purposes of this study, it is considered that this mechanism can explain the finding of improved bond strength.

One study combined sandblasting and plasma treatment and established that this combination improved bonding strength between PEEK material and composites but specified the ideal parameter for bond strength as 35 minutes of combined treatment.²³ Clinicians surely cannot keep to this timing for simple repairs.

Schwitalla *et al.*²⁴ reported higher bond strength values in PEEK samples treated with low-pressure Ar/O₂ plasma than in the control group, in a finding similar to those of this study.

Zhou *et al.*¹⁹ evaluated the effects of different surface treatments (98% sulfuric acid for 60 seconds, 9.5% hydrofluoric acid for 60 seconds, Ar gas plasma treatment, and sandblasting with 50µm Al₂O₃ particles) on the bond strength between PEEK material and veneering composite and found the highest bond strength in the sulfuric acid group with 8.7 ± 0.2 MPa, while the bond strength in Ar plasma group was 6.8±0.7 MPa. This study also found consistent values with PEEK samples (7.52 MPa) when plotted against the data reported in the literature (6.72 ± 3.66).²⁴

Stawarczyk *et al.*¹¹ evaluated the effect of helium plasma treatment on the bond strength between PEEK and veneering composite and found that adhesion between PEEK and veneering composite improved with the use of adhesive agents containing methyl methacrylate and that helium plasma treatment had no effect on adhesion. The authors attributed these findings to an inadequate stock of groups being exposed to form chemical bonds. They also added that the type of PEEK material used and the type of gas preferred for plasma treatment may affect bond strength.

In this vein, Zhang *et al.*¹⁴ reported that the types of gases used for plasma treatment of PEEK surfaces and the treatment duration affected the adhesion force, reporting the highest adhesion force with the use of Ar gas for the plasma treatment.

This study covered an examination of types of failure following a bond strength test on a stereomicroscope. The examination indicated adhesive failure to be more common, which represents a finding similar to that from Tukkal.³³

Adhesion between PEEK and veneering composite is known to improve through surface treatment with non-thermal atmospheric plasma (NTAP) following sandblasting on the PEEK material.²⁴ In this study, the optimum process parameter was found to be the use of Argon gas for 4 minutes. It is suggested that the long-term performance of the bonding of veneering composite with PEEK be evaluated by *in vitro* and *in vivo* studies under different ageing conditions.

The methods implemented for this study represent a combination of surface treatments that may be preferable to many other methods owing to their ease of clinical use.

The stronger the bonding of two different materials in intraoral conditions, the longer the life of the restoration can be. For this reason, sandblasting and non-thermal atmospheric plasma (NTAP) to the PEEK polymer surface to be bonded with composite resin can increase clinical success.

Conclusions

Improving the surface energy of PEEK and increasing its bond strength with veneering composite represents an important challenge that needs to be addressed to gain a complete understanding of potential benefits. Surface treatment of PEEK with non-thermal atmospheric plasma

(NTAP) following sandblasting has a positive effect on the adhesion between PEEK and composite material.

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Conflict of Interests

The authors declare no competing interests.

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