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ASSESSMENT OF VARIOUS SURFACE TREATMENTS ON RETENTION OF POLYETHERETHERKETONE PREMOLAR CROWN

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ABSTRACT

Objectives: The primary aim of this research is to evaluate the durability of premolar crowns made from Polyetheretherketone (PEEK) after undergoing various surface treatment methods.

Materials& Methods: A set of twenty-five Polyetheretherketone (PEEK) crowns with full coverage were manufactured and thereafter affixed onto human maxillary premolars that had been appropriately prepped. The 25 crowns were classified into five distinct types according on the surface treatment utilized. The notion of control pertains to the capacity to manage or regulate variables with the intention of exerting influence over outcomes or preserving stability within a given system. Hydrofluoric acid (HF) with a concentration of 9.5% is being referred to. The solution under consideration is sulfuric acid with a concentration of 98%. The piranha solution is composed of sulfuric acid (H2SO4) at a concentration of 98% and hydrogen peroxide (H2O2) at a concentration of 30%. The experimental agent employed in this investigation was nitric acid, and each trial consisted of a sample size of 5. Following that, every crown sample was securely attached to its corresponding abutment using self-adhesive resin cement G-CEM. Following that, the specimens were subjected to a thermocycling protocol comprising of 5000 cycles, wherein the temperature oscillated between 5°C and 55°C. The pull-off test was performed utilizing a universal testing apparatus. The data was collected, systematically arranged into tables, and subsequently subjected to rigorous statistical analysis.

Results: The mean pull off values were ordered in the following manner:

Conclusions: It is recommended to employ Piranha solution and 98% sulfuric acid for the purpose of treating the surface of PEEK, since this approach has been shown to result in a substantial improvement in retentive strength.

KEYWORDS: Premolar, peek, retention

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INTRODUCTION

Polyetheretherketone (PEEK) is a polymer-based material commonly referred to as Bio HPP, which has been augmented by the incorporation of ceramic fillers. The engineering plastic material demonstrates uniform physical properties and exhibits high resistance to abrasion, making it suitable for a wide range of industrial applications. [(1)] PEEK demonstrates advantageous mechanical properties, chemical stability, and hydrolysis resistance in comparison to alternative plastic materials[(2)]. Furthermore, PEEK exhibits the potential to be utilized in a wide range of dental applications, encompassing, but not restricted to, the production of dental crowns, bridges, superstructures for dental implants, and orthodontic wires [(3-6)].

Furthermore, it is noteworthy to mention that the utilization of PEEK material can also be employed for the examination of removable partial denture clasps. Nevertheless, in order to attain a more realistic tooth aesthetic, the inherent opacity of PEEK restorations requires the application of veneering material to ensure enough coverage. The PEEK material exhibits numerous advantages in the context of routine dental applications [(7)]. Nevertheless, a significant challenge in the clinical setting was the attainment of a durable and universally acknowledged adhesion between dental substances. Current scholarly investigations have focused on augmenting the reactivity of the polyetheretherketone (PEEK) surface with resins, with the aim of achieving optimal adhesion [(8-12)].

The importance of the luting cementation process cannot be overstated, as it plays a pivotal role in determining the clinical effectiveness of permanent dental prostheses. The main methodology utilized in these experiments centered on improving the PEEK material through surface modification employing an adhesive system conditioning technique, hence promoting chemical interactions. A series of evaluations have been carried out by researchers to assess the binding strength between PEEK and resin materials. These evaluations involved the application of different surface treatments, such as sandblasting, silica coating, etching with piranha solution, etching with sulfuric acid, and other plasma procedures. The authors' conclusion implies that the application of air abrasion has a beneficial impact on the adhesive characteristics between PEEK and resin materials. It is advisable to consider air abrasion as a key alternative for the surface treatment of PEEK surfaces.Various chemical surface treatments were utilized to treat the surface, employing etching agents like sulfuric acid etching and hydrofluoric acid etching (HF), which are commonly employed in the field of dentistry. In addition, a micromechanical surface treatment was performed using air abrasion [(12-19)]. The incorporation of various surface treatments augments the capacity for adhesive bonding. The pull-off test is often regarded as more advantageous in comparison to the bond strength test due to its capacity to accommodate the complex geometry of an abutment preparation [(20,21)].

Nevertheless, it is important to acknowledge that the existing body of research on the evaluation of pull-out tests for PEEK crowns is relatively scarce. Consequently, the objective of this in vitro investigation was to assess the pull-off values subsequent to different surface treatments.

The hypothesis proposed in this study postulated that PEEK crowns, when treated with Piranha solution and 98% Sulfuric acid, would demonstrate the highest value in the pull-off test.

MATERIALS AND METHODS

A sample of twenty-five freshly extracted teeth was collected, establishing their absence of cavities or any crown-related issues. The teeth were chosen according to their mean crown dimensions. The extraction was required because to periodontal issues. Subsequently, the teeth underwent a meticulous cleansing process to remove any accumulated dirt, employing an ultrasonic scaler called the Cavitron GEN-119. This device is produced by SpsTM and Dentsply, a company based in York, Pennsylvania. The teeth were stored in distilled water at ambient temperature until they were prepared for utilization.

The procedure for generating holes with a depth of 1 mm in the roots was executed. Following this, the specimens were meticulously inserted into a split mold composed of chemically cured acrylic resin known as Acrostone, originating from Egypt. The preparation of the mixture was conducted in line with the instructions provided by the manufacturer. The teeth were centrally positioned within the mold and were aligned in parallel to the long axis of the tooth using a parallel meter (PFG 100, CendresMétaux, Biel-Bienne, Switzerland). Following that, the molds were disassembled after they had completely hardened.

In order to achieve uniform reduction, a rubber silicon index (specifically, Speedex C-Silicone Impression Material - Putty 910 ml - Coltene/ Whaledent, Patterson Dental Supply, Inc., USA) was acquired for each tooth prior to initiating the tooth preparation process. In order to maintain consistency, the preparation process was carried out by a sole operator. The prescribed preparation criteria encompassed a 5 mm axial reduction, 1.5 mm incisal reduction, a 1 mm thick shoulder finish line, and an approximate taper of 6°. In order to evaluate the thickness of the preparation, a periodontal probe fitted with a rubber silicon index was utilized. Prior to cementation, the specimens under investigation were submerged in distilled water at a temperature of 37 °C for a period of 24 hours.

The specimens were divided into five groups, with each group containing five specimens, based on the surface treatment given in a random manner. Group I refers to the control group.

In Group II, the utilized sample consists of hydrofluoric acid at a concentration of 9.5% (HF). The hydrofluoric acid in question, referred to as Ultradent Porcelain Etch, is produced by a firm situated in South Jordan, Utah, United States. The time period is 20 seconds.

In this study, a solution of 98% sulphuric acid (RCI Labscan, Samutsakorn, Thailand) from Group III was employed for the purpose of treating the crowns. The treatment duration lasted for a period of 60 seconds. Before undergoing the aforementioned procedure, the crowns underwent a rinsing process with distilled water for a duration of 10 seconds.

Within Group IV, the crowns were treatment with a solution comprising 98% sulfuric acid and 30% hydrogen peroxide for a period of 60 seconds. In the past, the crowns underwent a 10-second rinsing process using pure water.

The specimens in Group V had a 60-second exposure to nitric acid. Historically, crowns underwent a rinsing procedure utilizing distilled water for a period of 10 seconds.

The experimental procedure consisted of subjecting the surface to a pressure of 120 pounds per square inch (psi) for a period of 5 seconds. The instrument was situated at a distance of approximately 2-3 millimeters from the surface. After the implementation of surface treatments, the specimens underwent a 30-second water spray to eradicate any residual acid particles. Following this, the utilization of oil-free compressed air was implemented to aid in the facilitation of the drying procedure.

In order to produce a CAD/CAM PEEK premolar crown utilizing the inEosX5 system (Dentsply Sirona, Milford, USA), an optical impression was acquired. In order to enhance the quality of the digital impression, the tooth underwent treatment with occlutec optical spray (Renfert, Giesswiesen, Hilzingen, Germany). The precision of the scanning procedure is of utmost importance in order to obtain a comprehensive digital depiction of the teeth that is devoid of any flaws. The restorations were designed utilizing the Sirona inLab MC X5 CAD program, which was produced by Dentsply Sirona, a Milford-based firm in the United States. Each crown was intentionally designed with two retentive arms located at the incisal one third of the crown. This particular design characteristic facilitates the application of a pull-off test on the crown by utilizing a universal testing machine. All the crowns were manufactured utilizing computer-aided design and computer-aided manufacturing (CAD/CAM) technology and were constructed from ceramic Polyetheretherketone (Bredent GmbH, Co KG, Senden, Germany).

Each specimen was securely attached to its corresponding tooth using the manufacturer's recommended procedures, ensuring a uniform static stress was maintained throughout. The utilization of G-CEM (capsule A2, GC Co., Japan) was employed to accomplish this. The specimens were subjected to a dwell time of 20 seconds for a total of 5000 thermocycles, spanning a temperature range of 5°C to 55°C. This was accomplished using Robota, an automated thermal cycling apparatus. The focus of this conversation is Robota BILGE, a company situated in Turkey. Based on ISO/TS 11405, the utilization of cycles in this investigation can be equated to a timeframe of two years of clinical treatment. [22-25].

Following this, all crown specimens underwent a crown pull-off test utilizing a Zwick/Roell Z010 universal testing machine (Zwick, Ulm, Germany). The cemented PEEK crowns were removed by adhering to the direction of insertion and utilizing a crosshead speed of 0.5 mm/minute until the debonding procedure was successfully accomplished. The pressures causing dislodgment were recorded at the designated position N, as seen in Figure 1.

Fig. (1) Application of Pull off test

Following this, all relevant descriptive data were collected, arranged in a tabular format, and assessed for conformity to a normal distribution using a statistical test. Afterwards, the data that was gathered was subjected to analysis using a one-way analysis of variance (ANOVA) followed by a post-hoc test. The statistical program employed for the present investigation was SPSS version 15.0, which was developed by SPSS Inc. in Chicago, IL, USA. The findings of this research are displayed in Table 1 and Figure 2.

RESULTS

The findings of the one-way analysis of variance (ANOVA) revealed a statistically significant difference in retention rates across the five groups ($p<0.001$). The post-hoc LSD analysis yielded statistically significant findings, indicating a notable disparity in retention rates between the control group and the remaining groups ($p<0.001$). Significant variations in retention rates were seen among the different treatment groups, with a p-value of less than 0.001.

TABLE (1) Effect of different surface treatments on retention

		Control	HF	H2SO4	Piranha	HNO3	P value
		N=5	N=5	N=5	N=5	N=5	
Retention	Range	(9.32-10.54)	(11.93-12.55)	(34.32-36.9)	(41.82-42.76)	(17.92-18.17)	*<0.001**
	Mean ± SD	9.98±0.46	12.12±0.25	35.79±0.96	42.41±0.38	18.02±0.09	
P value between each two groups							
Control			*<0.001**	*<0.001**	*<0.001**	*<0.001**	
HF				*<0.001**	*<0.001**	*<0.001**	
H2SO4					*<0.001**	*<0.001**	
Piranha						*<0.001**	

One Way ANOVA test for quantitative data between the 5 groups followed by post hoc LSD analysis between each two groups

****: Significant level at P value < 0.05***

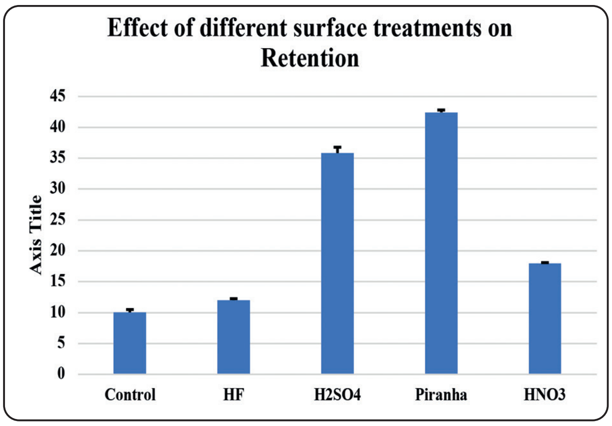

Fig. (2) Bar chart showing average Maximum pull out load (N) for different surface treatments within each tested material

DISCUSSION

PEEK has gained significant attention as a highly sought-after dental restorative material owing to its advantageous biocompatibility and remarkable mechanical properties. In a recent study, it was demonstrated that the PEEK three-unit fixed dental prosthesis (FDPs) had a significant deformation of 1200 N, exceeding the average mastication pressures exerted in the posterior region, which typically range up to 600 N. The efficacy of utilizing PEEK in fixed dental prosthesis (FDPs), specifically in areas subjected to load-bearing, has been substantiated. The present work focuses on the examination of a composite material comprising polyetheretherketone (PEEK) reinforced with 7 weight percent of nano-sized silicon dioxide (Nano-SiO2). The addition of Nano-SiO2 to PEEK results in a significant improvement in its biomechanical properties and a noticeable decrease in its thermal expansion coefficient. As a result, the modified PEEK material demonstrates enhanced compatibility for dental applications.

In order to ensure sufficient mechanical adhesion, it is crucial to achieve a reasonable degree of surface roughness for PEEK throughout the bonding process. PEEK, or polyetheretherketone, is a thermoplastic polymer renowned for its exceptional mechanical strength characteristics. Hence, the surface roughening phenomenon is limited because to the exceptional hardness and strength exhibited by PEEK.

The present work utilized four various surface treatment methodologies in order to augment the adhesive strength of the PEEK composite material. Etching is a frequently utilized method for surface modification. The adhesive demonstrates the capability to penetrate the surface pores of the pretreated PEEK material, leading to the creation of an adhesion layer that efficiently achieves micro-mechanical retention. The utilization of concentrated sulfuric acid has the capacity to induce erosion in PEEK material. In a previous investigation, the surface of polyetheretherketone (PEEK) was subjected to treatment with 98% concentrated sulfuric acid. This treatment led to the formation of a surface characterized by a notable level of porosity and permeability to adhesives. As a result, the connection's robustness was increased.The user has provided a set of coordinates, particularly [13,16].

The results of this current study align entirely with the previously posited concept, as both Piranha acid and Sulfuric acid 98% had much higher pull-off mean values in comparison to the other examined groups. Nevertheless, the disparity discovered demonstrated a substantial degree of statistical significance. In order to enhance the adherence to cement, it is imperative to apply a thin coating of Visio link, as the low surface energy of PEEK [13] poses a challenge in achieving satisfactory adhesion.

The crowns utilized in the experiment were manufactured using CAD/CAM technology under wet conditions, adhering to the manufacturer's published instructions. The utilization of self-adhesive cement offers several benefits as a result of its ability to establish a robust adhesion to diverse surfaces. This is primarily attributed to the inclusion of methacrylate monomers with phosphoric acid groups. Moreover, this particular cement demonstrates outstanding mechanical properties, enduring color stability over extended periods, and convenient attributes for mixing and handling.

Air abrasion with AL_2O_3 is a commonly employed technique for conditioning polymeric ceramic materials. The utilization of this method, which is commonly applied for the purpose of modifying surfaces, has promise for inducing changes in the surface morphology of polyetheretherketone (PEEK). These modifications enhance the infiltration of cement into the resin composite, hence strengthening the micro-mechanical interlocking and improving the retentive strength [15].

Furthermore, it is important to acknowledge that hydrofluoric acid exhibits selective reactivity towards the PEEK silicon phase, leading to the creation of tetrahedral fluorosilicate. The elimination of this waste can be achieved efficiently through the utilization of water [26].

The phenomenon of HF acid etching results in the disintegration of the filler components that are exposed on the surface. Consequently, the absorption of acid into the resin matrix can lead to the subsequent weakening of this matrix.

The utilization of sulfuric acid etching in therapeutic contexts is currently a subject of ongoing contention, primarily attributable to its notable oxidizing characteristics. Currently, the available literature offers limited insights into the effects of sulfuric acid etching, specifically at optimum concentrations, on the surface properties and adhesive strength of PEEK [27].

Based on the findings presented in the research article, it was determined that the application of sulfuric acid solutions with concentrations of 90% and 98% led to the attainment of the most pronounced surface roughness, following an etching process lasting no less than 60 seconds [21].

Based on the findings presented in the paper, it has been observed that Pianha acid, a compound consisting of 98% sulfuric acid and 30% hydrogen peroxide, exhibits the highest degree of surface roughness and possesses a notable abundance of pores characterized by diverse diameters and widths. The acidity level of piranha acid is comparatively higher when compared to other acid tests. As a result, the bond strength that demonstrated the highest level of magnitude was recorded.

An advantageous aspect of the pull-off test is in its ability to include the surface bonded area in the calculation, hence improving the precision of the obtained outcomes. In the current study, the crowns that were being analyzed underwent controlled force application utilizing a universal testing equipment, with a cross head speed of 0.5 mm/min. The force was exerted until either the crowns were separated or the tooth or crown suffered a fracture, as evidenced in prior research [17,18].

Previous studies have shown evidence that the application of sandblasting methods can lead to modifications in the surface morphology of polyetheretherketone (PEEK) composites. This modification enables the penetration of luting cement into the composite material, hence improving the micro-mechanical interlocking and ultimately leading to an increase in bond strength [15].

The objective of this work was to evaluate the adhesive characteristics of PEEK composites after exposure to different acid treatments, namely Hydroflouric acid 9.5%, Sulfuric acid 98%, Piranha acid, and Nitric acid. The utilization of Piranha and Sulfuric acid 98% solutions has been found to be efficient in eliminating the grooves and imperfections observed on the surface of the polished PEEK composite material, as indicated by the analysis conducted using scanning electron microscopy (SEM). The absence of pore formation is observed on the surface of PEEK when treated with HF 9.5% or nitric acid.

The application of hydrofluoric acid or nitric acid in conjunction with G-Cem did not result in any observable adhesion in the respective groups.

Previous studies have provided evidence indicating that a dry, hydrophobic adhesive surface is effective as a primer for water-based applications[28].

PEEK's surface displays hydrophobic characteristics and showcases chemical inertness. The major solvent utilized in G-Cem Bond is water, which facilitates the efficient penetration of the porous structure of a PEEK composite material. The evaporation of the solvent is not detected until the substitution of the surface pores and fissures in the PEEK composite by water and air, hence aiding the penetration of the monomer HEMA. In this study, the utilization of Hydroxyethyl methacrylate (HEMA) as a co-solvent was investigated to improve the wetting characteristics of polyetheretherketone (PEEK) composites, specifically in relation to compounds that are insoluble.

Improved wetting promotes the efficient infiltration of the luting cement into the pores of the PEEK composites, hence enhancing the bond strength [29]. The shear strength of a material can be affected by modifications in its surface treatment, leading to potential changes in both mechanical and chemical adhesion [30].

However, it is important to acknowledge that instances of cohesive failures frequently occur due to the uneven distribution of stress at the bonding contact during loading circumstances [40]. As per the findings of the investigation, adhesive failures were identified as the prevailing fracture type. The observed phenomenon can be ascribed to the inadequate adhesive strength between the composite material and the luting cement [31]. The occurrence of mixed failure can be attributed to the non-uniform distribution of stress at the contact interface.

However, previous studies have indicated that the surface morphology of PEEK stays unaltered when exposed to hydrochloric acid and nitric acid, regardless of the concentration used. The application of scanning electron microscopy (SEM) facilitated the confirmation of the porosity formation on the surface of the PEEK composite material following its exposure to Piranha solution and 98% sulfuric acid. The results indicate that the material can be effectively etched through the utilization of a combination of Piranha solution and 98% sulfuric acid. Therefore, the application of Piranha solution and 98% sulfuric acid etching has been identified as a highly effective method for enhancing the adhesive properties of the material.

The utilization of hydrofluoric acid gel as a preliminary treatment for PEEK surfaces yielded no substantial improvement in bond strength. Researchers have observed a decrease in the bonding strength between a luting cement and an indirect composite material that has been treated with a 9.6% hydrofluoric acid (HF) gel for etching. The potential impact of HF gel involves the complete dissolution of filler particles that are exposed on the surface. The acid possesses the capacity to undergo absorption into the resin matrix, resulting in the material's softening [33,34] .

PEEK is a thermoplastic polymer renowned for its remarkable performance attributes. Hydrofluoric acid exhibits the capacity to selectively react with the silicon constituent of a PEEK composite material, leading to the creation of tetrahedral fluorosilicate. The chemical equation provided illustrates the reaction as follows: 6 moles of hydrogen fluoride (H2F2) combined with 2 moles of silicon dioxide (SiO2) yield 2 moles of hexafluorosilicic acid (H2SiF6) and 4 moles of water (H2O). The principal techniques employed for plasma polymer surface modification encompass plasma surface treatment, plasma polymerization, and plasma graft polymerization. These techniques enable the occurrence of diverse physical and chemical modifications in surface properties. As a result, improvements can be implemented to increase the properties of the material.

The surface of the PEEK composite material, which underwent pretreatment with Piranha treatment, had a multitude of grooves and cracks as observed through scanning electron microscopy (SEM) analysis. The application of this treatment led to an increase in the surface roughness of the material. As a result, the resin permeated the pores, leading to an increase in mechanical adhesion.

In a study conducted by **Zhou et al. (2014),** [26] alterations in the chemical and physical characteristics of the PEEK surface were examined through the application of 98% sulfuric acid. This phenomenon leads to an increase in the formation of bonds.

The results of our investigation were incongruous with the findings of **Spitznagel FA, et al (2014)**[35], which indicated that mechanical surface treatment, particularly air abrasion, had more effectiveness in improving bond strength when compared to alternative chemical surface treatments.

Moreover, our results were in line with the investigations carried out by **Schmidlin PR et al. (2010)**[13], **Stawarczyk B et al. (2013)**[16], and **Silthampitag P et al.** (2016)[8], all of which recommended the utilization of 98% sulfuric acid for the purpose of surface modification of PEEK.

In contrast, **Sproesser et al. (2014)**[19] conducted a study which revealed that the application of a 98% sulfuric acid surface treatment had a detrimental effect on the adhesive infiltration process, leading to the formation of weakened regions at the bond surfaces. The proposed recommendation entails the utilization of sulfuric acid etching technique at a reduced concentration.

CONCLUSION

The findings of this in-vitro investigation suggest that the surface treatment had an effect on the PEEK pull-off test, within the limitations of the study's constraints.

The utilization of Piranha solution and 98% Sulphric acid yielded superior pull strengths in comparison to alternative surface treatments that were examined.

The lowest average pull-off values were seen when hydrofluoric acid was combined with nitric acid at a concentration of 9.5%.

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