ACID ETCHING SURFACE TREATMENT OF PEEK MATERIAL: A MICRO-MORPHOLOGICAL SEM ANALYSIS

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Abstract

Back ground: PEEK has been presented as a potential replacement for metal, zirconia, and glass ceramics. It is a thermoplastic partly crystalline high-performance polymer (HPP) made of an aromatic benzene molecular chain joined by functional groups from ether and ketone.

Objectives: To use a scanning electron microscope (SEM) examination to determine how various acids affect the surface topography of PEEK material.

Materials and Methods: Twenty-five PEEK discs with dimensions of 1.5 mm in thickness and 15 mm in diameter were created. According to the ingredients used for the acid etchant, all samples were sorted into five groups (n=5). The first group underwent SEM analysis as a control and received no surface treatment. Hydrofluoric acid gel 9.5% was administered to the second group. The third group received 98% sulfuric acid. The fourth group received Piranha (a 98% sulfuric acid and 30% hydrogen peroxide combination). Nitric acid was administered to the fifth group. After that, SEM analysis was performed on all samples. Each sample was captured in 5000X magnification photographs.

Results: Our study showed that the material can be successfully etched by concentrated sulfuric acid. Therefore, etching with 98% sulfuric acid is a useful technique for improving the material's bonding capabilities. The surface of the PEEK composite material had numerous holes, which enhanced its roughness, as seen by the SEM picture of the material after piranha treatment.

Conclusions: The surface roughness and pore development of PEEK discs were greatest after treatments with piranha solution and 98% sulfuric acid.

Key words: Acid etching surface, PEEK, SEM.

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1-Introduction

PEEK has been presented as a potential replacement for metal, zirconia, and glass ceramics. The most widely used kind of polyaryletherketone (PAEK) resin is known as [1,2] PEEK. It is a thermoplastic partly crystalline high-performance polymer (HPP) made of an aromatic benzene molecular chain joined by functional groups from ether and ketone. [3] Using pressing or computer-aided design and computer-aided manufacturing (CAD-CAM) methods, PEEK has been utilized in prosthetic dentistry to create interim implant abutments, endo crowns, interim restorations, fixed dental prostheses, and partly removable dental prostheses.[4-7]

One of the most well-liked high performance engineering polymers now on the market is polyetheretherketone (PEEK). Because of its appealing mechanical properties, including heat resistance, solvent resistance, excellent electrical insulation, good wear resistance, and high fatigue resistance, PEEK is highly advantageous for applications in a variety of industries, including aerospace, automotive, electronics, and medical equipment [8–10].

PEEK is mostly used in orthodontic bite sticks, healing caps, and transitional abutments in dentistry [11,12]. Excellent physical and biological qualities of PEEK satisfy fundamental prosthetic needs in clinical settings.

However, PEEK's opaque and drabby color restricts its use in full-coverage restorations. PEEK must thus be combined with veneering or composite resin. However, due to PEEK's inert chemical performance, low surface energy, and resistance to surface modification, the material's bond strength is poor when mixed with composite resin [13]. As a result, one of the hottest areas of study is enhancing PEEK's surface characteristics. The surface treatment and luting cement have an impact on the adhesive characteristics, which are crucial for the durability of prostheses in dental applications.

Due to their aesthetic and biomechanical qualities, dental ceramics are biocompatible, natural-looking indirect restorative materials in contemporary restorative dentistry [14–16]. Ceramics are often employed, and there is rising interest in applying them in several dental specialties [4-6]. Clinicians can now provide accurate, high-quality ceramic restorations because to advancements in computer-aided design and machining (CAD/CAM) technology [17]. A major problem over the years has been the poor tensile strength and absence of direct chemical bonding with natural enamel.

By raising the surface free energy, two different materials may adhere better in dentistry [19,20]. The wettability of the surface for resin cement bonding is enhanced by increasing the surface free energy [21]. Therefore, it is advised to use hydrofluoric acid to condition the inside surface of the ceramic repair. In accordance with a number of recent studies, etching ceramic with 5% hydrofluoric acid for 2–3 minutes is sufficient to selectively dissolve the glassy phase and create pores on the surface that allow for the penetration of resin composite cement [18,22,24,25]. In addition to increasing the surface area available for resin bonding [8], this porous ceramic surface also exposes and produces hydroxyl groups that are necessary for chemical bonding via silane coupling agents [22,26,27].

In comparison to traditional luting cements, the adhesive can offer greater retention, better edge seal, and a more permanent bond effect [28]. The adherend's porous surface can be penetrated by the resin cement to create a resin nail, which has apparent implications on micro-mechanical retention.

The goal of the current study is to analyze surface roughness of PEEK material using a scanning electron microscope (SEM) to determine the effects of 9.6% hydrofluoric acid, 98% sulfuric acid, Piranha (98% sulfuric + H2O2 30%), and nitric acid.

2-Material and method:

Material used:

1-PEEK (bredent breCAM BioHPP: 80% PEEK and 20% nanoceramic filler)

2- Hydrofluoric acid 9.5%, 98% sulfuric acid, Piranha (98% sulfuric acid, 30% H_2O_2)and Nitric acid.

Fabrication of PEEK Disc using CAD/CAM technology:

1-Designing the ceramic material cylinder

Using CAD system software (Germany), a PEEK cylinder with dimensions of 15 mm in diameter and 16 mm in length was designed. The program was used to import the cylinder design from the common template library, thus there was no need to scan any templates. The imported design didn't need to be modified. The cylinder's form was verified before being sent to the CAM system.

2-Milling of the cylinders

A dental milling device (Sirona milling machine, Germany), a five-axis dental milling device used for PEEK material, was utilized to mill the cylinders. When the milling machine imported the cylinder design, a dialog window to select the type of material to be milled appeared, and peek blanks were selected. Cylinders were milled in accordance with the imported design after the blanks were placed into the milling machine. The milling machine was then given the command to grind the cylinders. The cylinders were remained attached to the main blank after the milling operation was complete. The cylinders were separated from the main blank using a particular finishing bur. To get rid of any leftovers, all cylinders were then submerged in a distilled water ultrasonic bath for ten minutes.

3-Acrylic PEEK Cylinder Construction & Cutting to Discs

To lengthen the length of the ceramic cylinder before holding it in the cutting machine, place it in an acrylic mold.

According to the manufacturer's instructions, the correct powder to liquid ratio for self-curing acrylic resin was blended. In a split cylindrical copper mold and ring, it was then poured. Each ceramic material's cylinders were placed into the metal mold's cylinder (20 meters in diameter and 25 meters in length) until they completely covered the cylinder surface area. With a metallic wax carver, extra acrylic resin was quickly removed. To make it easier to hold the ceramic cylinder in the cutting machine (IsoMet 5000, Buehler, Illinois, USA), each peek cylinder was held in an acrylic resin mold.

The cylinders were cut into disks using the precision cutting machine (IsoMet 5000, Buehler, Illinois, USA) while being irrigated with water. The saw used a fine 0.3 mm diamond thread with embedded diamonds (diameter approximately 60 m), and the applied pressure was roughly 50 g. A precision sectioning saw, the Buehler IsoMet Low Speed cutting machine is made for cutting a variety of materials with little distortion. The finished disk was 15mm in diameter and 1.5mm thick.

Specimen preparation and treatment:

Prior to surface treatment, the surfaces were cleaned for 10 minutes in an ultrasonic water bath (Euronda, Italy). The specimens (n = 25) were allocated at random into five test groups based on the following surface treatments:

Group A: No treatment

Group B: (Hydrofluoric acid etching): For 60 seconds, 9.5% hydrofluoric acid (Sino-Dentex Co., Ltd., China) was used to etch the bonding surfaces of PEEK composite materials. The gel was washed with distilled water for 60 seconds, then compressed air devoid of any oil was used to dry it.

Group C: (Sulfuric acid etching): Beijing Chemical Works in China's 98% sulfuric acid was used to etch the PEEK composite materials' bonding surfaces for 60 seconds. The acid was removed using distilled water for 60 seconds, and it was then dried using compressed air without any oil.

Group D (Piranha): Piranha solution was used to treat the PEEK composite materials' surfaces. The acid was removed using distilled water for 60 seconds, and it was then dried using compressed air without any oil.

Group E (Nitric acid): The surfaces of the PEEK composite materials were treated using Nitric acid solution. The acid was rinsed off with distilled water for 60 s and then dried with oil-free com-pressed air.

Following a 60 second cleaning with distilled water, the specimens were dried with oil-free compressed air.

An extra specimen was created and examined by scanning electron microscopy (SEM, Quanta600, FEI, Netherlands) in order to conduct micro-morphologic assessment of each surface treatment group. Each group received a gold sputter coating before being examined at a 5000x magnification.

3. Results

SEM allows for the visualization of the surfaces following various pretreatments. Although the structure of the polished PEEK materials (Group A) was regular, SEM investigation revealed that they caused minor surface scratches (Fig. 1).

Hydrofluoric acid Topography was somewhat altered by etching. The surfaces still had evidence of the abrasive tracts left behind from grinding, although they were less noticeable than on the untreated surface. This outcome demonstrates that undercuts were created that removed and disintegrated the fillers (Fig. 2).

Sulfuric acid Etching caused the PEEK material to dissolve in a porous, blister-like manner (Fig. 3).

Piranha etching caused the PEEK material to dissolve into many porous blister-like structures (Fig. 4).

Nitric acid had a negligible impact on the topography. The surfaces still had evidence of the abrasive tracts left behind from grinding, although they were less noticeable than on the untreated surface. This outcome demonstrates that undercuts were created that disintegrated and removed the fillers (Fig. 5).

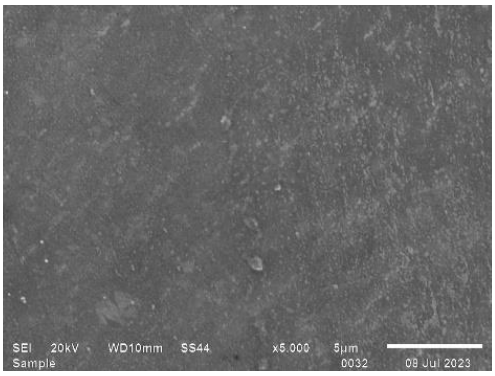

Fig. 1 – SEM image of PEEK composite material without pretreatment.

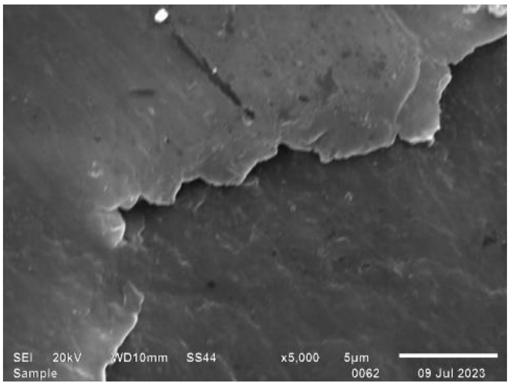

Fig. 2 – SEM image of PEEK composite material with pretreatment of 9.5% HF

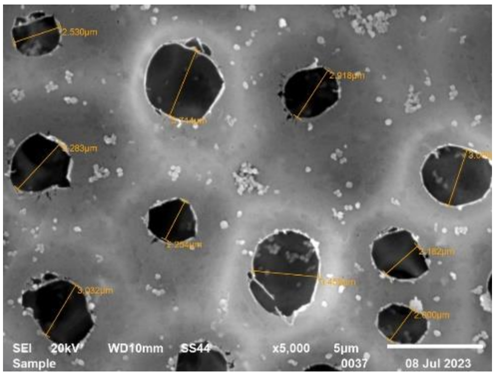

Fig. 3 – SEM image of PEEK composite material with pretreatment of 98% sulfuric acid.

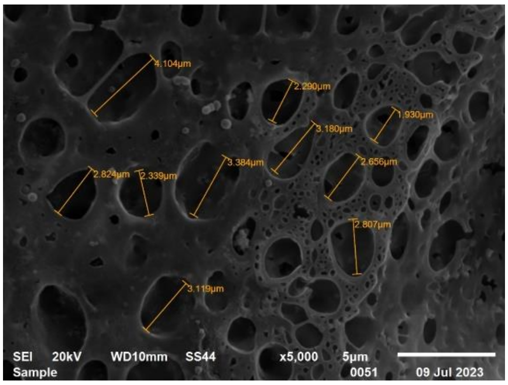

Fig. 4 – SEM image of PEEK composite material with pretreatment of piranha

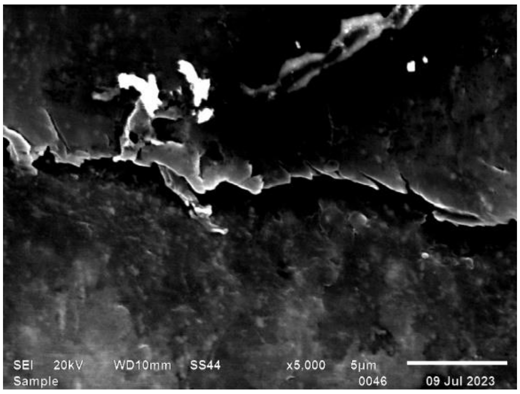

Fig. 5 – SEM image of PEEK composite material with pretreatment of Nitric acid.

4-Discussion

Due to its high mechanical qualities and strong biocompatibility, PEEK makes a perfect material for dental restorations. The three-unit PEEK FDPs demonstrated a visible deformation of 1200 N in a recent investigation, which was greater than the mastication forces of up to 600 N in the posterior area. PEEK has been shown to be appropriate for fixed dental prosthesis (FDPs), particularly in places that bear weight [8]. The biomechanical characteristics of the PEEK composite material used in this work, which is reinforced with 7 weight percent Nano-SiO2, are dramatically enhanced. The thermal expansion coefficient is also significantly reduced, making PEEK more suited for use in dental applications.

To ensure enough mechanical retention during the bonding process, the PEEK surface needs to be sufficiently rough. PEEK, on the other hand, is a strong resin substance. Thus, the surface roughening process is constrained by PEEK's high hardness and strength. In this work, four surface treatment techniques were used to increase the PEEK composite material's binding strength. Etching is a common technique for treating surfaces. The adhesive can create an adhesion collateral that performs micro-mechanical retention by penetrating the pretreatment PEEK's surface pores. Sulfuric acid at a high concentration can degrade PEEK. According to earlier reports, sulfuric acid at a concentration of 98% was used to treat the PEEK surface, creating a highly porous and adhesivepermeable surface. The binding strength thus increased [29,30].

However, regardless of the concentration utilized, neither nitric acid nor hydrochloric acid can alter the surface morphology of PEEK.

After being treated with 98% sulfuric acid, a SEM picture (Fig. 3) showed that the surface of the PEEK composite material had become porous. This outcome shows that the material can be successfully etched by concentrated sulfuric acid. Therefore, etching with 98% sulfuric acid is a useful technique for improving the material's bonding capabilities.

The binding strength was not increased by hydrofluoric acid gel pretreatment of the PEEK surfaces. Researchers discovered a weaker binding between a luting cement and an indirect composite that had been etched with 9.5% HF gel. Possible effects of HF gel include the complete dissolving of surface-exposed filler particles. The resin matrix may also absorb the acid and soften as a result [31,32]. PEEK is a resin that performs quite well. In order to create tetrahedral fluorosilicate, hydrofluoric acid can also specifically react with the silicon phase of a PEEK composite material (6H2F2+ 2SiO2 2H2SiF6+ 4H2O).

The surface of the PEEK composite material had numerous holes, which enhanced its roughness, as seen by the SEM picture of the material after piranha treatment (Fig. 4). As a result, the resin entered the pores and improved mechanical retention.

Conclusions

It is possible to draw the conclusion, within the constraints of this investigation, that piranha solution treatments have a greater impact on surface roughness and many porous forms than 98% sulfuric acid treatments. Surface roughness on PEEk material was not significantly affected by the etchant with hydrofluoric and nitric acid.

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