Comparative analysis of surface characteristics and hardness of three dimensional printed PEEK vs PEKK - as implant biomaterial

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ABSTRACT

Aim The aim of this study is to conduct a comparative analysis of surface characteristics and hardness of three dimensional printed PEEK and PEKK.
Materials and methods Total sample size was 60, in which 30 were PEEK and the other 30 was PEKK. The 10 mm diameter and 2 mm thickness circular disc was designed in CAD software and it was 3D printed for manufacturing. PEEK and PEKK were compared using AFM, wettability and contact angle test, SEM and Vicker's microhardness test to know their surface characteristics when used as an implant material. SPSS software version 22 was used to evaluate the independent t test values for the average of contact angle, microhardness and surface roughness in order to determine their significance.
Results The material is hydrophobic in nature both PEEK and PEKK, the materials hydrophilic property can be increased by using various surface treatments. PEKK had more Vicker's hardness numbers. The material seems to be less porous. The surface roughness characterics of PEEK and PEKK were statistically significant ($P<0.05$).
Conclusions PEEK and PEKK are polymers that have good microhardness and surface roughness. These both materials are highly aesthetic and can be used in aesthetic zones. Both PEEK and PEKK can be used as implant materials.

KEYWORDS: PEEK, PEKK, surface characteristics, microhardness, High-performance polymer, Dental implants, restorations, dental prosthodontics, innovation.

INTRODUCTION

The placement of cosmetic implants is guided by both a biological and restorative perspective. The implant should be positioned aesthetically to suit the contour requirements so that the restoration looks good. In order to maintain the architecture of both hard and soft tissues, it should be positioned biologically(1). There are many clinical uses for prosthetic dental treatment, including fixed, removable, and implant-supported prostheses made of different materials(2). New goods are continually being introduced, and material qualities are always being improved. In recent years, polyether ether ketone (PEEK) has become a material of choice in dentistry. It is a poly(aryl ether ketone) polymer based on ultra-high molecular weight polyethylene and has the chemical formula (-C6H4-O-C6H4-O-C6H4-CO-)n. It is a semi-crystalline thermoplastic biomaterial(3). This material, which was first created in 1978, has been employed as an alternative to metal substructure treatments in orthopedic, maxillofacial, and dental procedures. PEEK offers a wide range of uses in dentistry, including as an aesthetic substitute for metal systems in implants, braces, temporary abutments, and fixed and removable prostheses(4). Its outstanding chemical, thermal, and mechanical qualities as well as its great biocompatibility are to blame for its widespread use. PEEK is also a strong, long-lasting material that deforms less under high temperatures than other thermoplastics(5). Thermoplastic polyaryletherketone, or PEKK, is a kind of PAEK. It is comparable to

PEEK, which is mostly used in dentistry for abutments for temporary dental implants(6). It is being offered as a metal substitute for dentures and bridges that are supported by implants. Recently, dental labs have started employing it in place of metal for frameworks.

A material needs to be least impacted by oral fluids and have the least amount of plaque buildup on it in order to be used in the mouth for a long time. Its surface must be finely polished in order to achieve this. Surface roughness can result in tooth abrasion, plaque buildup, coloring, or discoloration(7). On the other hand, the surface roughness increases the cell adhesion when used as implant material. For a material to produce its maximum yield, surface topography, roughness, hardness, and abrasion properties are crucial. Prior to being introduced into the mouth, a restoration might also need additional procedures (like proximal contact control and occlusion control). In these situations, the restoration's surface finish deteriorates as the material is taken off of it(7,8).

Vickers hardness measurements and surface roughness (profilometer) analyses are tests used to look at a material's surface characteristics. The surface structure of the material is quantified by these mechanical tests, although the material's surface topography is not entirely covered(2). To clearly see changes on the surface of the material and evaluate its topography, scanning electron microscopy (SEM), atomic force microscopy (AFM), three-dimensional (3D) optical profilometers, and confocal laser scanning microscope (CLSM) are utilized(3). The natural circumstances and microscopic characteristics of a material without any surface coatings are examined using environmental scanning electron microscopy (ESEM)(6). PEEK's semi-crystalline polymer chain sections do align into a crystalline structure as it cools, giving it a sensitive cooling process akin to that of Yttria-stabilized tetragonal zirconia polycrystal (3-YTZP)(1). The crystalline structure will experience higher thermal stress and deformation if it cools too quickly. In contrast, PEKK is likewise a semi-crystalline polymer, but the key distinction is that PEKK crystallizes at a far slower rate than PEEK, allowing it to be handled similarly to an amorphous polymer. This indicates that PEKK has better layer adhesion and less bending because it is less impacted by cooling in a lower-temperature build chamber(4). Our team has extensive knowledge and research experience that has translated into high quality publications(9–18,19–24).

The aim of this study is to conduct a comparative analysis of surface characteristics and hardness of three-dimensional printed PEEK and PEKK. Where null hypothesis stated that there is no difference between PEEK and PEKK when compared for surface characteristics and hardness.

MATERIALS AND METHODS

Sample size estimation

In the prosthodontics department of a university hospital, an in vitro study was conducted. G power software was used to estimate the sample size, and the sample included 60 samples (30 PEEK and 30 PEKK).

Ethical Approval

The ethical approval was obtained from the institutional ethics committee. Number: IHEC/SDC/UG-1801/22/PROSTHO/635.

Sample preparation

The 10 mm diameter and 2 mm thickness circular disc was designed in CAD software and it was 3D printed for manufacturing. The 3D printed disc was cleaned with deionized water thoroughly, keeping it on a magnetic stir for 10 mins.

Wettability and water contact angle

Contact angle measurements were used to analyze the surface characteristics. When a liquid droplet is deposited on a solid surface, the contact angle is the angle between the solid surface and the liquid-vapor interface. Here, we gauge the wettability of the PEEK and PEKK samples by measuring the contact angle between water and the material. Based on contact angle measurements made using the static sessile drop method and deionized water at 22°C, PEEK surface wettability was calculated. Deionized water in the amount of 67 3 1 was suspended from a Kruss needle (model NE62, OD = 1 mm, ID = 0.82 mm) and allowed to fall freely to the substrate surface. The freefall motion was captured by a high-speed camera. DSA10-Mk2 drop shape analysis contact angle system, Ossila goniometer (Fig.1A) was used to analyze the angle formed between the surface and the water droplet. Each sample's surface was measured three times, and a circular algorithm technique was used to calculate the droplet angle.

Vicker's microhardness test

In Vickers microhardness testing, a surface is impressed by a pyramidal diamond indenter with a 136° facing angle under a specified load for a predetermined amount of time. The imprint width that remains after the diamond has been removed is used to compute the area of the indentation that remains. The machine used in this study was the Shimadzu HMV-G31DT Micro Vickers Hardness Tester(Fig.1B). The force used for this microhardness test was HV0.3 (2.942N) and the holding time was 20 seconds. The following equation yields the Vickers microhardness number: $HV= 0.1891 \times F/d2$. Where F is the applied load and d is

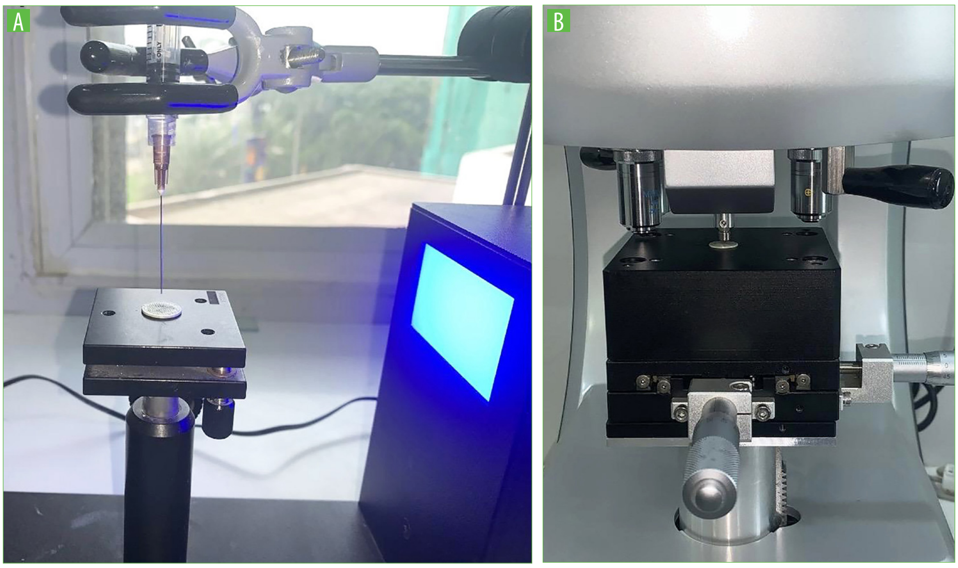

FIG. 1 The above figure is a machine used for analysis of contact angle and microhardness. A contact angle tester (ossila goniometer) B Shimadzu HMV-G31DT Micro Vickers Hardness Tester.

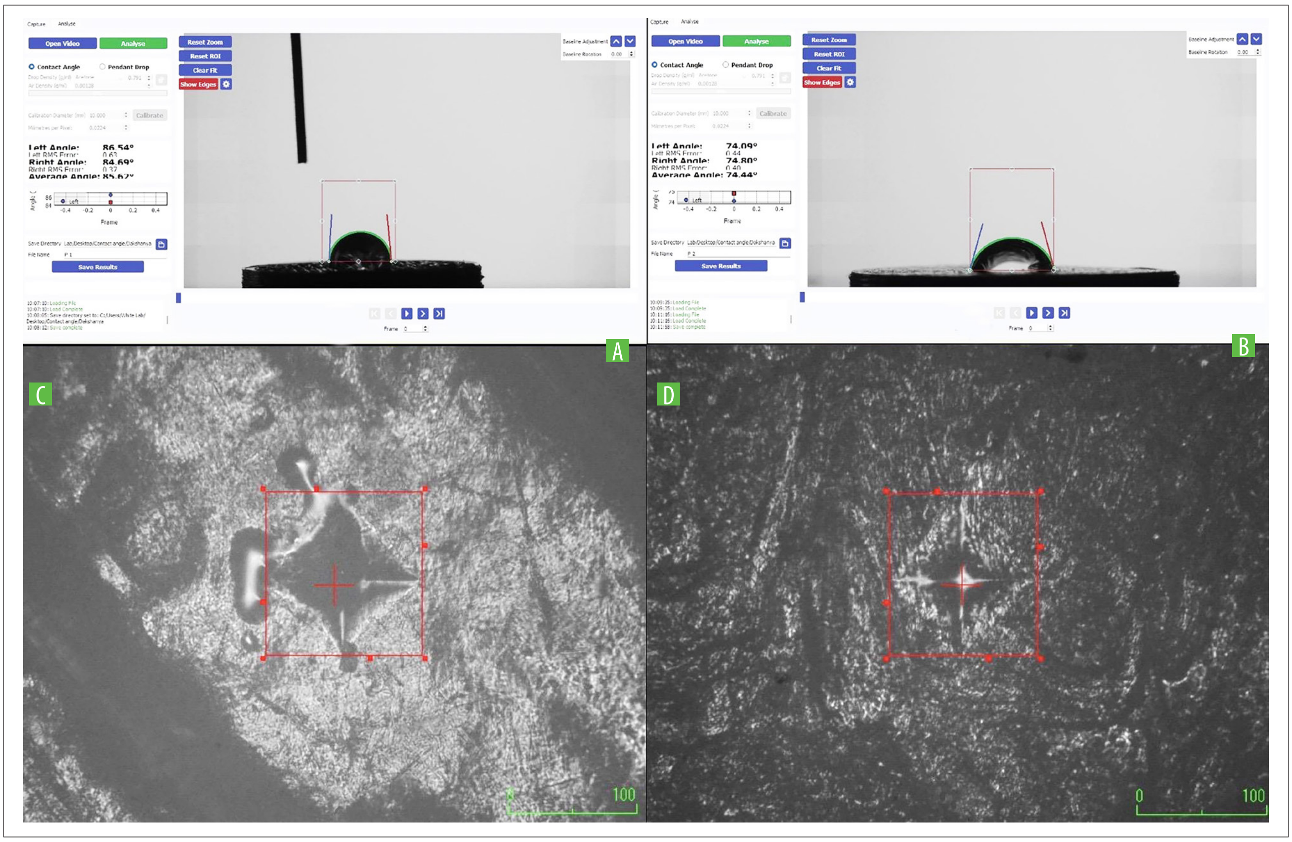

FIG. 2 The above Fig. shows the results of contact angle and Vicker's microhardness. A-B represent the contact angle values of PEEK and PEKK respectively. C-D represent the Vicker's microhardness number of PEEK and PEKK.

the average of the imprint's two diagonals.

Sem analysis

The specimen was examined with an FEI XL-30 FEG high resolution SEM (FEI, Hillsborough, OR, USA) employing secondary electron imaging under a 7 kV acceleration voltage (SE). Digital images were acquired directly as TIFF grayscale files with a resolution of 1424 x 968 and 8 bits per pixel. The identical specimen was then polished flat, sectioned along its longitudinal axis with a high precision diamond-coated disc, and implanted in glycol methacrylate (Technovit 7200 VLC, Heraeus Kulzer, Hanau, Germany).

Afterward, the specimen was mounted as before, coated with carbon using an Emitech K250 flash evaporator (Emitech, Montigny Le Bretonneux, France), and examined using the same FEG SEM that was running at an acceleration voltage of 15 kV using backscattered electron (BSE) imaging.

SAMPLE	NUMBER OF SAMPLES	MEAN±SD	STANDARD ERROR	95%CI (Upper)	95%CI (Lower)	t value	P value
PEEK	30	85.56 ± 2.87	0.52453	-11.43	-20.31	-8.497	0.00*
PEKK	30	75.6 ± 2.81	0.51327	-11.43	-20.31	-8.497	0.00*

TABLE 1 The values obtained were statistically estimated with an independent t test for the wettability and contact angle, the obtained result was statistically significant, P<0.05;P value was derived from independent t test.

SAMPLE	NUMBER OF SAMPLES	MEAN±SD	STANDARD ERROR	95%CI (Upper)	95%CI (Lower)	t value	P value
PEEK	30	27.00 ±0.83	0.151	-1.570	-2.429	-9.327	0.00*
PEKK	30	29.00 ±0.83	0.151	-1.579	-2.429	-9.327	0.00*

TABLE 2 The values obtained were statistically estimated with an independent t test for microhardness, the obtained result was statistically significant, P<0.05;P value was derived from independent t test.

AFM for surface roughness

A tip attached to a flexible cantilever will travel across the sample surface using atomic force microscopy (AFM) to evaluate the surface shape at the atomic level. By observing the cantilever's deflection during scanning, the forces between the tip and the sample are calculated. This force depends on the material qualities of the tip and the sample as well as the tip-sample separation. Other properties of the sample, the tip, or the medium in between can be investigated using additional interactions that emerge between the tip and the sample. We were able to gauge the roughness of PEKK and PEEK surfaces that had undergone plasma activation using AFM measurements. The measurements were conducted using silicon cantilevers with a Si3N4 coating, a tip radius of 20 nm, a spring constant of 40 N/m, and a resonance frequency of 325 kHz (NSC15/A1BS, Mikromasch, CA, USA) on a DimensionTM 3100 instrument (Veeco, Mannheim, Germany) in Tapping Mode® in ambient air under dry conditions. More information about the nanostructures created by the plasma may be provided by tips with a radius of less than 20 nm. 2 x 2 m2 and 1 x 1 m2 of the scan area were chosen, respectively. The software Nanoscope 6.13R1 was used for the data processing and roughness evaluation (Veeco Instruments Inc., Santa Barbara, CA, USA).

Statistical analysis

The statistical analysis done was SPSS software version 22. P value was derived using an independent t test. If the p<0.05, shows the statistical significance between PEEK and PEKK.

RESULTS

The contact angle results of PEEK, the average angle was 85.62 degrees (Fig. 2A) and the contact angle results of PEKK, the average angle was 74.44 degrees (Fig. 2B). Both the materials are hydrophobic in nature. From the results of independent t test to assess the hydrophilicity of PEEK and PEKK, both the materials were statistically significant (p<0.05). The mean VHN of PEEK and PEKK was 27 and 29 respectively (Fig. 2C-2D). The p value was less than 0.05 and statistically significant for the microhardness of both PEEK and PEKK (Table 2). SEM analysis of PEEK had fewer pores after-cross section (Fig. 4 A-B) and SEM analysis of PEKK was also less porous (Fig. 4 A-B). The average AFM value for PEEK was 89.808 (Fig. 4A) and for PEKK it was 118.04 (Fig. 4B). The independent t test results for surface roughness values of PEEK and PEKK respectively, the p value was less than 0.05 which shows that both are statistically significant (Table 3).

DISCUSSION

PEEK and PEKK has poor osseointegration due to its bio-inertness and relative hydrophobicity, which hinders its long-term clinical success. A successful orthopedic implant is primarily determined by osseointegration, which occurs when the implant surface and bone tissue bind together(25,26). In order for an implant to become osseointegrated, the surface of the implant material must allow osteoblast cells that help create mineralized bone to adhere effectively. Studies have shown that the hydrophilicity, roughness, porosity, and presence of bioactive groups on the implant surface have a significant impact on the adhesion, proliferation, and differentiation of osteoblasts. Therefore, surface modification is a key step to activate the PEEK implant's surface for osteoblast adhesion(27). From our study results the wettability and contact angle average was 85.62 degrees and 74.44 degrees for both PEEK and PEKK respectively which shows the poor wettability property. It is important to

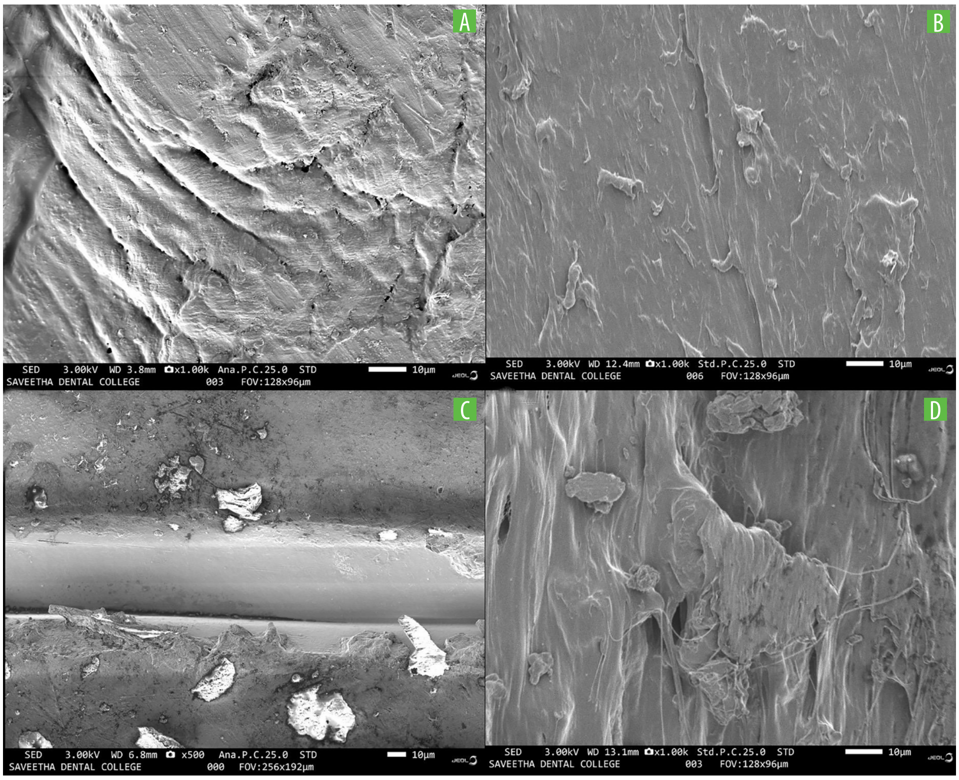

FIG. 3 The above figure represents the results obtained from SEM analysis, showing the morphology of the sample.
A represents the SEM results of PEEK before breaking.
B is SEM cross section results.
C-D represents the SEM analysis results of PEKK before and after breaking.
The pictures were taken in 10 micrometers, using 3kv current.

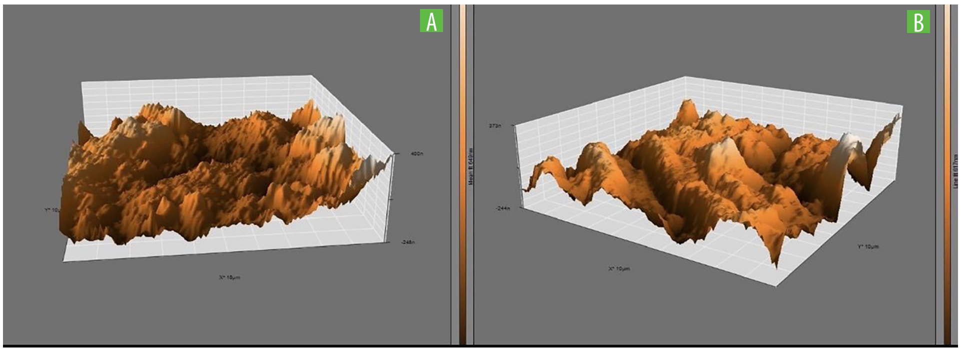

FIG. 4 The above Fig. represents the analysis of surface roughness using the AFM test.
A represents the AFM of PEEK.
B represents the AFM of PEKK.

do a few surface treatments in order to increase the hydrophilicity of the above material.
When test samples are unsuitable for macro-hardness, microhardness testing is a method of determining a material's hardness or resistance to deformation. A material's individual phases, very small/thin samples, complex shapes, and surface coatings/platings can all be tested for hardness using microhardness methods(28). When test samples are unsuitable for macro-hardness, microhardness testing is a method of determining a material's hardness or resistance to deformation. A material's individual phases, very small/thin samples, complex shapes, and surface coatings/platings can all be tested for hardness using microhardness methods(29). The Vicker's microhardness value for PEEK and PEKK was 97.9 and 99.7 respectively. As an implant material, PEEK and PEKK demonstrate sufficient flexural strength, extending its clinical longevity. Improved toughness prevents implant fracture while increased hardness reduces the likelihood of implant material wear.
Values for pore size, porosity, and interconnectiv-

SAMPLE	NUMBER OF SAMPLES	MEAN±SD	STANDARD ERROR	95%CI (Upper)	95%CI (Lower)	t value	P value
PEEK	30	86.76±4.21	0.769	-15.22	-20.31	-13.981	0.00*
PEKK	30	104.53±0.83	0.011	-15.21	-20.31	-13.981	0.00*

TABLE 3 The values obtained were statistically estimated with an independent t test for surface roughness, the obtained result was statistically significant, $P<0.05$;P value was derived from independent t test.

ity that have been shown to permit vascularization, nutrient transport, and cell migration, all of which are necessary for successful bone-implant integration. Both PEEK and PEKK are porous before cutting and in cross section the porosity of both materials is reduced(28,30). Additionally, the results show that PEEK and PEKK does not lose its specific strength (strength/density), indicating that the addition of porosity using this processing technique only spreads the material out rather than weakening it. The AFM values for both PEEK and PEKK were statistically significant, the average value of PEEK was 86.808 and PEKK was 118.04.

PEKK has been introduced as a possible substitute material for titanium in long-term orthopedic applications because of its outstanding biocompatibility. For spinal surgery and oro-maxillofacial surgery, it has FDA approval. PEEK is also widely utilized as a prosthetic and implant biomaterial in dentistry. It provides metal-free restorations and is useful for allergy sufferers. Yuan et al. studied the chemistry and surface microstructure of osseointegration in PEKK as an implant material(29). According to a report, the PEKK's other ketone group enhances the capacity for surface chemical alteration. The presence of -SO3H will be more prevalent on PEKK than PEEK as there are more ketone groups(28). This results in a complex surface topography, a larger surface area, and a micro-rough surface, all of which have an impact on the behavior of the cells and the rate of osseointegration on the PEKK surface. The osseointegration property was improved by the surface modification by increasing porosity and incorporating HA(31). By altering the surface with different bioactive ceramics like beta-tricalcium phosphate (-TCP), hydroxyapatite (HA), and bioactive glasses, bioactive PAEK material can be produced (BG). Converse et al. created HA whisker reinforced porous PEKK by combining several techniques, including compression molding, particle leaching, and powder processing. Walsh et al. reported that coating PEEK with plasma-sprayed titanium improved the histological and mechanical characteristics of the bone-implant interface after implantation in comparison to uncoated PEEK(26). In terms of antibacterial activity, Wang asserts that PEKK exhibits lower bacterial adhesion on its surface than PEEK used in the orthopedic industry. Staphylococcus epidermidis adhered to the PEKK surface with 37% less adhesion. After five days of culture, they discovered a roughly 50% reduction in Pseudomonas aeruginosa adhesion and development on PEKK compared to PEEK without the use of antibiotics. PEEK has advantages over metal parts due to its high heat tolerance, excellent chemical resistance, and ability to withstand mechanical and physical stress. PEEK is suitable for the automotive, aerospace, and other general industrial settings thanks to these characteristics. In our study PEEK had more VHN when compared to PEKK which was 29.7 and 27.9 respectively. There was another study supporting this, The hardness of PEEK and PMMA was 24 VHN and 19.4 VHN, respectively(32).

The PEKK has been used as a prosthesis and implant biomaterial with great success in dentistry. Due to PEKK's good mechanical, fracture resistance, shock-absorbing, and superior stress distribution properties, it has recently been used in many dental applications(33). Because it allows metal-free restorations and is regarded as an alternative to metal and ceramics, the PEKK has high biocompatibility(34). Low elastic modulus, high strength, and good wear resistance are all characteristics of the PEKK. It might be suitable for use as a restorative component in fixed prosthodontics(35). Modern restorative and prosthetic materials can now be created with greater accuracy thanks to computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies. The wear resistance of entire dentures can be increased by adding individual ceramics made using CAD/CAM technology. Recently, PEKK prosthetic restorations have been made using CAD/CAM technologies. With an indirect composite veneer, Pekkton® ivory (PEKK) is used for monolithic and bi-layered materials(36). Potential uses for PEKK's high-performance, isoelastic properties included in the field of oral implantology. The benefits of PEKK include adequate strength, lightweight, resistance to wear, and an elastic modulus that is comparable to dentin. The percentages of bone contact for dental implants made of thermoplastic resins have likewise yielded respectable results(37). PEKK and PEEK is a versatile material that can be used in oral implantology as implant abutments, implant prosthesis framework, implant biomaterial, and prosthetic crown materials. The non-metal PEKK and PEEK material offers a titanium implant substitute(38). The PEKK and PEEK abutments' advantages include adjustability, compatibility with a range of veneering materials, and the ability to serve as the framework for an implant-supported prosthesis. A material that can offer long-term retention in implant prostheses is titanium combined with the PEKK and PEEK attachment system(39).

CONCLUSION

Since both PEEK and PEKK are inert and nonallergenic polymeric biomaterials recommended as substitutes for metal alloys in various types of prostheses and orthoses, biological requirements are not an issue. In SEM analysis, both substances were capable of forming apatite and shared a similar interconnected macroporous structure. These polymers are also biocompatible and have an elastic modulus that is comparable to that of natural bone and dentin. Wider uses in clinical dentistry may also result from modifications and improved material qualities. As PEEK and PEKK have only lately been used in dentistry and there is little research available, long-term evaluations are required but the only limitation was the material was hydrophobic which has to be overcome by doing various surface treatments. Overall PEKK is having better surface morphology and microhardness than PEEK for implant biomaterial.

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Conflict of interest

The author declares no conflict of interest.

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