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SURFACE TREATMENTS OVER PEEK: A LITERATURE REVIEW

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ABSTRACT

Introduction:

Aim: The aim of the current study is to review all the literature and evidence supporting PEEK as an acceptable dental material.

Methodology: A thorough search has been performed in all the databases, dating from 1980 to present to gather all the information on the history, properties, modification and application of the PEEK as a medical and dental material. The existing data available in literature was carefully analysed and described under various sections of properties and applications.

Discussion: The available literature on PEEK has very well described the properties of PEEK, which support the use of PEEK as not just a temporary but also a permanent restorative material. The various aspects of material science of PEEK have shown strong evidence of applications of PEEK.

Conclusion: PEEK can be considered as a potential material of choice for individual implant and tooth prosthesis, as well as for full arch prosthetics

Keywords: Polymer, PEEK, Medical and Dental Material

1. INTRODUCTION

Polymer is a material with an unbreakable relationship with dentistry. Before polymers were introduced, Vulcanite rubber was used as a denture base material since 1850. After almost 100years polymers were introduced in dentistry due to the need of a firm and rigid material. Polymers were commercially introduced in 1935 where the material was provided as pre-polymerized beads of PMMA and a liquid monomer.

Polyether ether ketone belongs to the ketone polymer family. It has shown huge application in the medical field, however, it is popularised as a potential material in the field of dentistry since the last 5 years. The properties of PEEK make it a suitable substitute for the conventional prosthetic materials, combined with high esthetics. It shows similar bonding and layering properties to the conventional metal or zirconia substructure using various surface treatment methods.

This review paper aims at analysing and systematically evaluating the available literature on PEEK. The objective of the review is the understand and evaluate the properties of PEEK in comparison to the conventional materials and analyse the various techniques and methods used in the fabrication of PEEK prosthesis.

2. REVIEW OF LITERATURE

2.1. History of PEEK

Polyether ether ketone (PEEK) is from the family of Polyarylether ketones (PAEK) polymers which was invented by Rose in 1978, in collaboration with Imperial Chemical Industries (ICI) **(Rose *et al.* 1986) (Bailly *et al.* 1987)**. It is a semicrystalline polyether made from aromatic nucleophilic substitution of potassium salt of hydroquinone and 4,4’- difluorobenzophenone. It was commercialised in 1981 with the name Victrex PEEK. In 1993, a company was formed, named Victrex which aimed to focus on polymer materials and PEEK predominantly (“Victrex Celebrates 40 Years of PEEK” 2019). The revolution of PEEK in the medical industry came up by introducing PEEK-OPTIMA Natural material which was used to make parts of implantable intervertebral cage **(Zoidis, Bakiri, and Polyzois 2017)**.

2.2. Structure and Composition

0.1 Moles of 4,4-difluorobenzophenone **(Figure 1)** and hydroquinone are condensed at up to 320°C in the presence of diphenyl sulfone and anhydrous potassium carbonate. It has an aromatic molecular backbone with combinations of ketone (–CO–) and ether (–O–) functional groups between aryl rings **(Rahmitasari *et al.* 2017)**.

2.3 Properties of PEEK

PEEK has shown exceptional biological and mechanical properties coming from a polymer family.

Of the various properties of PEEK, the one which is of prime relevance to dentistry is the modulus of elasticity. The modulus of elasticity of PEEK is closest to the human bone compared to any other biomaterial. Young's modulus and tensile properties are close to human bone, enamel and dentin **(Najeeb *et al.* 2015) (Table 1)**.

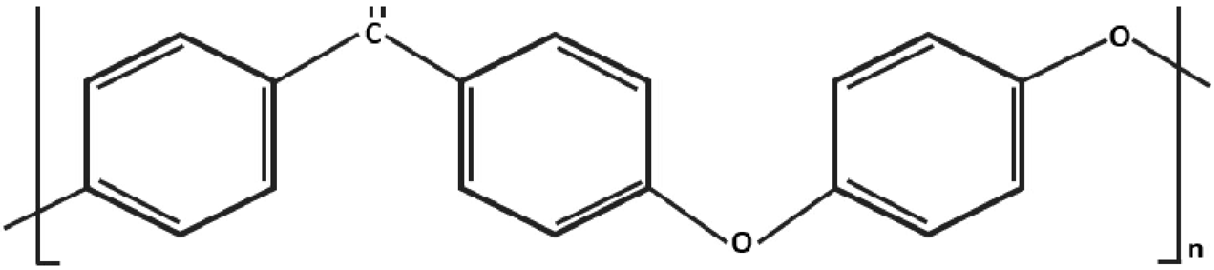

Figure 1: Chemical Structure of PEEK

Table 1: Mechanical properties of various dental materials in relation to bone, enamel and dentin (Najeeb et al. 2016)

Materials	Tensile Strength (MPa)	Young's Modulus (GPa)
PEEK	80	3-4
CFR-PEEK	120	18
Cortical bone	104-121	4
PMMA	48-76	3-5
Dentin	104	15
Enamel	47.5	40-83
Titanium	954-976	102-110

2.3.1 Temperature

PEEK is known for its temperature resistance. It has a melting point of almost 340°C and its glass transition temperature is 143°C (Atkinson 2002). PEEK is much more stable compared to other polymers of its family at higher temperatures. This property to withstand such temperatures made the material to excel exponentially in dentistry. PEEK has good heat aging resistance (May 2008) ("Development and Evaluation of Polyether Ether Ketone (PEEK) Capillary for Electrospray," n.d.).

2.3.2 Mechanical properties

PEEK is both tough and firm. The tensile strength of unfilled PEEK is more than 100MPa, Modulus of elasticity of 3-3.5 GPa and a flexural strength more than 165 MPa with flexural modulus of 40GPa and notched impact strength of more than 6.9 KJ/m^2

(Shrestha, Simsiriwong, and Shamsaei 2015). It has an outstanding creep resistance and can withstand great stress without significant distortion along with time. The tensile modulus of reinforced PEEK is almost 11.4 GPa and flexural modulus more than 9.2 GPa.

2.3.3 Electrical properties

PEEK can retain its electrical properties unchanged substantially for temperatures up to 200 degrees celsius. The dielectric constants of PEEK are 3.2 and dielectric strength ranging from 18.2-20 kV/mm. This balance of properties makes PEEK suitable for all electrical applications with the possible exception of those where electrical loss is a highly critical consideration **(Izamshah et al. 2015)(Giants 1994)**.

2.3.4 Radiation Resistance

PEEK's aromatic structure endows the polymer with excellent resistance to hard radiation. It brittles down on absorption of gamma radiation upto 10-12 MGy (1000-1200 Mrad). Small beta dosage of 0.1-100 MGy does not affect the material (May 2008).

2.3.5 Chemical Resistance

Special chemical structure of PEEK exhibits stable chemical and physical properties: stability at high temperatures (like sterilization processes), resistance to most substances apart from concentrated sulfuric acid and wear-resistance **(Gediminas *et al* 2017)**. PEEK is also known to have least water resorption. Concentrated sulfuric acid dissolves and sulfonates PEEK polymer. Liquid bromine, oxidizing agents such as fumigating nitric acid can severely degrade PEEK but do not dissolve it. Hydrofluoric acid, trifluoro-methanesulfonic acid, or dichlorotetrafluoroacetone monohydrate, and in phenol-1,2,4-trichlorobenzene at high temperatures can make dilute solutions of PEEK which is used for gel-permeation chromatography of PEEK **(Shukla *et al.* 2012)**. PEEK is unusually resistant to attack by hot water or steam even at elevated temperatures and has survived 3000 h of exposure to water at 280°C and 18 MPa (2600 psi) with no significant degradation of flexural or tensile properties **(Berry 2002)**.

2.4 Applications of PEEK

2.4.1 Industrial Applications

Automotive:

Temperature and fatigue resistance and weightless property of PEEK led to its introduction in the automobile industry which made major changes in the components. Weight and noise reduction were primarily focused, followed by bearings, seals, washer, active components of brakes and air-conditioners are the major changes the industry went through **(Izamshah *et al.* 2015)**.

Aerospace:

PEEK and its composites played a major role in replacing the large volumes of metal in the aeronautical industry **(Figure 2)**. Ease of fabrication helped in fabricating huge monolithic components that stay intact and stable compared to fused metals **(Berry 2002)**. Another important property is the tribology interaction of friction, lubrication and wear, which made the part of engine and body work in harmony **(Shekar *et al.* 2009)**. PEEK reduces the metal corrosion due to constant moisture like rain, with high temperatures and when it burns it does not emit any hazardous gases **(Izamshah *et al.* 2015)**.

Electrical:

Lack of electrical conductivity makes PEEK an ideal material for containing high electricity conducting wires **(Ashrafi et al. 2012) (Figure 3)**. This insulating property can handle high power fluctuations which tend to disintegrate any other polymer material used **(Izamshah *et al.* 2015)**.

2.4.2. Medical Application

Applications of PEEK in Medical field:

1. **Orthopaedic Surgery:**

Due to high strength, both filled (CRF-PEEK) and unfilled PEEK are used as implants for femoral bone reconstructions **(de Ruiter *et al.* 2017)**, hip replacements **(Hanasono, Goel, and DeMonte 2009)** and hip resurfacing **(Pokorný *et al.* 2010)**. Braided, laminated and unidirectional fibre-oriented PEEK fracture **(Steinberg *et al.* 2013)** fixation plates **(Steinberg *et al.* 2013)**, as well as extruded PEEK screws can be fabricated in replacement to titanium material **(Neumann, Villar, and França 2014)**. Sternal ZipFix is a new cable-tie system for sternal closure systems studied by Grapow **(Grapow *et al.* 2012)** is a new. It is also used in joint replacement and hip replacement surgeries due to trauma **(Kurtz and Devine 2007)**.

2. **Neuro and Craniofacial Surgery:**

One of the first clinical cases of craniofacial reconstruction is by Scolozzi *et al* **(Scolozzi, Martinez, and Jaques 2007), (Herford *et al.* 2017)** performed an orbito-fronto-temporal reconstruction by designing a Patient Specific implant in CAD. **Kim *et al* in 2009** has done a study on 4 patients with maxillofacial defect with a 16 months follow-up, whose defects were reconstructed with customized PEEK implants which showed promising results **(Kim, Boahene, and Byrne 2009)**. PEEK entered spinal surgery around 2007 **(Kurtz and Devine 2007)** as an alternative to autologous bone. It compensated for drawbacks like bone resorption, donor site morbidity and subsidence and evolved as a much stable and elastic material with no cytotoxicity and MRI compatibility **(Kurtz and Devine 2007)**. It is also highly corrosion resistant and induces minimal inflammatory response **(Kurtz and Devine 2007)**.

Figure 2: Engine part of Propeller blades for aeroplanes

Figure 3: PEEK insulation for electric wires

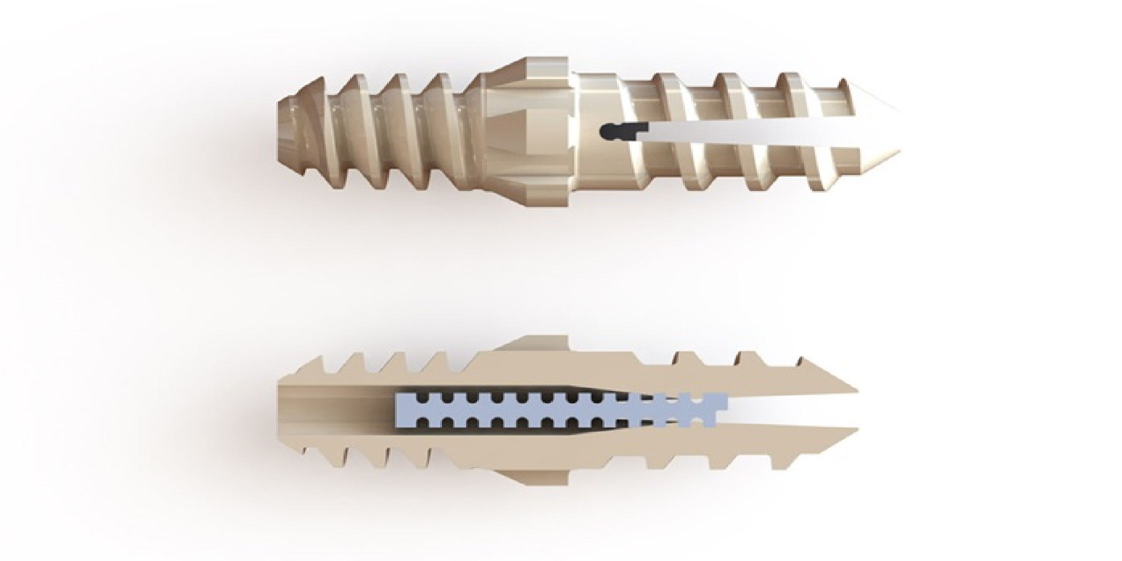

Figure 4: Orthopedic implants used for fracture carpals and tarsals.

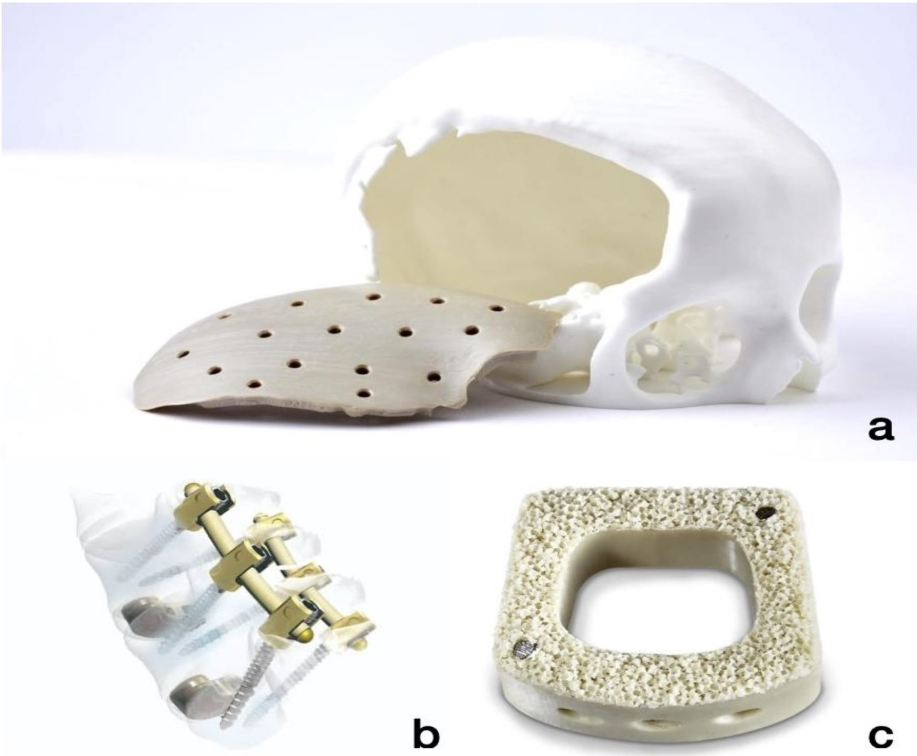

Figure 5: a) Cranio-facial PEEK implants, b&c) PEEK spinal implants

Applications of PEEK in dentistry:

1. Fixed Partial Dentures:

PEEK created its space in crown and bridge prosthetics for almost a decade. Its dimensional stability and highly polished surface makes it an appealing material for fixed restorations **(Taufall *et al.* 2016)**. Full contour crowns are fabricated with PEEK, however due to its un-esthetic gray color, it's not suitable for anterior crowns. Hence it is layered with composites. Bonding agent applications on surface pretreated PEEK frameworks increases the bond strength of PEEK-Composite prosthesis **(Stawarczyk *et al.* 2015) (Cekic-Nagas *et al.* 2018)**. It gives a tough competition to Porcelain fuzed Zirconia and Porcelain fuzed Metal in handling complications such as fracture and veneering material chipping **(Figure 6)**.

2. Implant prosthetics:

PEEK started wandering into implantology as it has a modulus of elasticity of 7.3 MPa which is closest to cortical and cancellous bone.

Many studies are in progress which are testing PEEK as a dental implant. This is due to its modulus of elasticity and aesthetic acceptance **(Najeeb *et al.* 2016)**. PEEK as such does not have any osseointegration or osteogenic properties **(Rahmitasari *et al.***

2017; Mishra and Chowdhary 2019). To promote osseointegration, modifications like surface treatments, surface coating and additives incorporation were done for PEEK to make it an ideal material for dental implant.

Nano-Hydroxyapatite (HA) modified PEEK generates 20-40 nm more abundant layers of nanosized particles having size, configuration and crystallinity similar to anthropoidal bone **(Johansson *et al.* 2015, 2018), (Najeeb *et al.* 2015)**.

PEEK had settled well in implant prosthetics. With the flexure property and reduced weight of PEEK, hybrid bars, locator retentive caps, Malo frameworks **(Maló *et al.* 2018), (Cao *et al.* 2019)**, temporary prosthesis **(Cao *et al.* 2019; Zoidis and Papathanasiou 2016)** are fabricated as it reduces the functional load over the implants which in turn reduces marginal bone loss around the implant and improves the survival. The highly polished surfaces of PEEK make an ideal implant abutment as they dont retain plaque and food debris keeping the surrounding gingiva healthy by preventing peri-implantitis.

- Drawbacks: The major disadvantage with PEEK as an implantable material is its radiolucent nature. It's not possible to evaluate the implants radiographically.

3. **Removable Prosthetics/ Cast Partial Dentures:**

Cobalt-chromium has been the gold stand material in fabrication of removable partial dentures. Though cast partial dentures improved the quality of life of partially dentulous patients, the rigidity of the material tends to impose levering forces onto the abutment teeth which eventually leads to failure. This has also been the same issue with cast partial dentures supported by precision attachments. In recent years PEEK has evolved as a very popular material of choice for cast partial dentures **(Muhsin *et al.* 2019)**.

Advantages of using PEEK as cast partial denture-

PEEK, though a very dimensionally stable material, is also very flexible which helps it dissipate the occlusal forces around the framework and onto the edentulous regions thus reducing the damage to the abutment teeth **(Mohamed, Baraka, and Badawy 2013)**.

PEEK has shown very high dimensional stability even across a larger surface area

Esthetics : The most common complaint with a metal alloy clasp was unaesthetic appearance in the anterior region. However, with PEEK being a material closer to the tooth color, has shown high patient acceptance for removable prosthetics compared to metal cast partial dentures in various cross-over studies **(Sadek 2019)**. The clasps made in PEEK that wrap over the labial and buccal surfaces of the teeth are masked by itself or by layering indirect composites **(Mohamed, Baraka, and Badawy 2013)**.

Another advantage of PEEK removable partial dentures is the weight of the framework **(Zoidis, Papathanasiou, and Polyzois 2016)**. PEEK has a lesser density compared to cobalt-chromium which lightens the prosthesis, especially for the maxilla.

Figure 6: a) First 3 images show the outcomes of fracture testing of Porcelain fused zirconia. Porcelain fused metal and Composite layered PEEK. b) Cross-section of Composite layered PEEK fixed partial denture (Taufall *et al*. 2016)

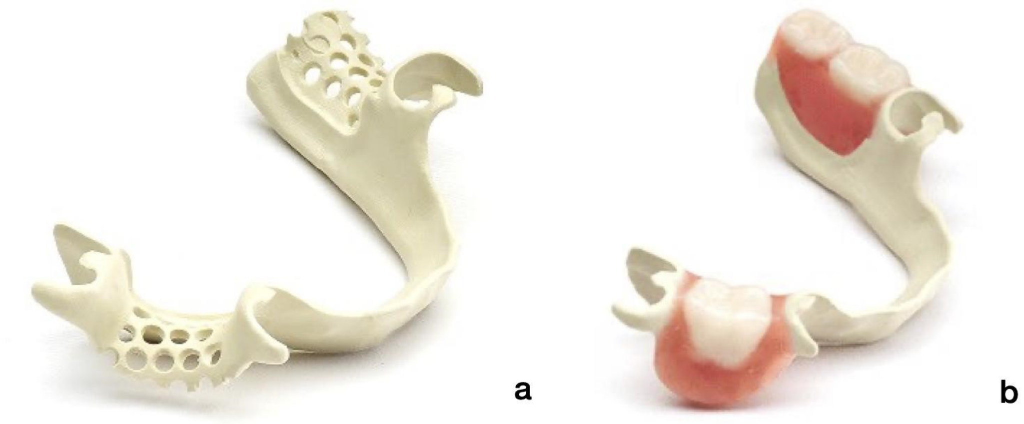

Figure 7: a) CADCAM milled PEEK removable partial denture, b) PEEK framework layered with Heat cured acrylic resin

2.5 Methods of Prosthesis fabrication in dentistry

PEEK is involved in 3 methods of fabrication-

1. Subtractive: PEEK is available in blanks and blocks which can be loaded in milling machines (CAM). Milling is considered to be the closest to the precision method of fabrication of dental prosthesis **(Jeong, Lee, and Lee 2018)**. CAD CAM milled PEEK has higher fracture resistance compared to Lithium disilicate (950 N), alumina (851 N) and zirconia (981-131 N) **(Meshreky, Halim, and Katamish 2020)**.
2. Additive: Rapid prototyping/3D printing PEEK has developed due to the need of manufacturing large sized products and more in number in a single cycle **(Schmidt, Pohle, and Rechtenwald 2007), (Honigmann et al. 2018)**. This is used in fabrication of orthopedic and trauma implants, autoclavable hospital equipment, etc. Selective Laser Sintering (SLS) and Fused Filament Fabrication (FFF) are the 2 techniques assisting in rapid prototyping PEEK.
3. Injection moulding: Before subtractive and additive methods took over, injection moulding the only method for fabrication **(Muhsin *et al.* 2019; Poulsson *et al.* 2014)**. It was used for fabrication of both dental and medical prosthetics and implants.

2.6 Modifications in PEEK

In spite of such promising properties, reinforcements were always done to improve the material. PEEK can be modified either by the addition of functionalized monomers (pre-polymerization) or post-polymerization modifications by chemical processes such as sulphonation, amination and nitration **(Yee, Zhang, and Ladewig 2013)**.

Modifications to improve mechanical properties:

Carbon Fibre Reinforced- PEEK. This increases the modulus of elasticity to 11.4 GPa which is closer to the cortical bone. The strength of the material varies based on the percentage of carbon fibre reinforcement **(Devine *et al.* 2013; Lu *et al.* 2014)**.

Glass Fibre Reinforced- PEEK: An increase in heat resistance upto 75 degrees celsius more than the unfilled-PEEK was seen in GFR-PEEK which improved thermal stability. Also there is 66% higher tensile and flexural strength recorded in GFR-PEEK **(E. Z. Li *et al.* 2013)**.

Modified PEEK containing 20% ceramic fillers known as BioHPP

Modifications to improve Biological properties:

Sulfonation (SPEEK): It is a procedure used to improve the hydrophilicity of the PEEK material. Treatment with >95% sulfuric acid adds a sulfonated group onto the polymer chain, which introduces charged groups that are ion-exchangeable. Sulfonated groups (-SO3-) aids in transport of cations and increases hydrophilicity of PEEK. There are two ways for PEEK sulfonation: Pre-Sulfonation and Post-Sulfonation based on the time of incorporation **(Yee, Zhang, and Ladewig 2013)**.

Hydroxy Apatite for osseointegration (process is given under methods of surface coating) **(Sargon Barkarmo *et al.* 2013)**.

Sand Blasting (nFA/PEEK) to improve osteogenic properties (process is given under methods of surface treatments) **(Li L.-Y. *et al.* 2014)**.

Addition of -COOH, -PO4H2 and -OH on hydroxylated PEEK surfaces gave bane-like apatite formation which improved the cellular adhesion, proliferation and spread of preosteoblast cells **(Li L.-Y. *et al.* 2014; Zheng *et al.* 2015)**.

Methods of Surface Coating:

Melt Blending: PEEK is melted to a temperature of 350-450°C at a pressure of 15 MPa and the solid nano-particles and melted PEEK are blended to make a composite mixture of Bio-active PEEK **(Deng *et al.* 2012; L. Wang *et al.* 2014)**.

Spin Coating: A high speed spinning device, where the implant is spun and heat treated with nano-HA which precipitated in surfactants, organic solvents and aqueous solutions of Ca(NO3)2 and PO4 **(Johansson *et al.* 2014; S. Barkarmo *et al.* 2014)**.

Gas Plasma Nano Etching: Implants are exposed to low power plasma gases like, water vapour, argon/oxygen and ammonia. This method adds more number of functional groups over the surface, increasing the surface hydrophilicity **(Waser-Althaus *et al.* 2014)**. This procedure has the ability to create nano-level roughness which gives a very minimal water contact angle.

Electron Beam Deposition: A highly nano-porous layer of TiO2 is formed on the implant surface with electron beam deposition. In this method, nonvolatile fragments are decomposed and deposited on the substrate. The 2 micron layer promotes the activity of BMP-2 **(Tamilselvan *et al.* 2012)**.

Plasma immersion ion implantation: TiO2 particles are sprayed as plasma which are pulsed at high negative voltages and deposited on the surfaces **(H. Wang *et al.* 2014)**.

Methods of Surface Treatments:

Acid Etching: This is the most acceptable surface treatment for both Filled and Unfilled PEEK. Of all the acids 98% sulfuric acid showed to create the highest surface voids. PEEK when treated with sulfuric acid, swells up and creates a sulfonation reaction and forms interlocking webbed strings with voids **(Silthampitag *et al.* 2016; Zhou *et al.* 2014)**.

Sandblasting: This technique proved to create micro roughness by eroding a thin layer of PEEK with high pressure of aluminium oxide. Studies were done with various particle sizes of aluminium oxide at varying pressures, in which 110 micron particles with 35MPa pressure showed the highest amount of micro roughness **(Ourahmoune *et al.* 2014)**.

Sandpapering: Sandpapering with the lowest grit sandpaper showed surface micro roughness higher than sandblasting, but no study was performed so far comparing both the techniques **(Prochor and Mierzejewska 2019)**.

2.7.Surface Treatments in Dentistry

Unfilled PEEK has a smooth surface with very less surface energy and minimal roughness. This reduces the retentive properties of PEEK to hold onto any veneering material. Six studies **(Zhou *et al.* 2014; Silthampitag *et al.* 2016; Chaijareenont *et al.* 2018; Stawarczyk *et al.* 2013; Younis *et al.* 2019; Keul *et al.* 2014)** were found that used non-surface treated PEEK as a control group comparing various surface treatments like sandblasting, acid etching, plasma sprays (oxygen, helium, argon) etc and showed how surface treatments can affect the bond strength.

Zhou *et al* (Zhou *et al.* 2014) used a PEEK composite blank (reinforced with 7wt%Nano-Sio2) and studied 4 surface treatments which included sandblasting, argon plasma spray, acid etching with 98% sulfuric acid and acid etching with 9.5% hydrofluoric acid. SEM examinations showed voids on sulfuric acid etched surface and very minimal erosion with hydrofluoric acid, sandblasting and argon plasma spray.

Etching with 98% sulfuric acid was done for all the samples after the mechanical surface treatments were performed. Various authors **(Stawarczyk *et al.* 2013; Schmidlin *et al.* 2010; Zhou *et al.* 2014; Sproesser *et al.* 2014)** have performed studies on PEEK surface treatments with 98% sulfuric acid and showed great improvement in bond strength with Composite resins and luting resins cements. **Chaijareenont *et al* (Chaijareenont *et al.* 2018)** had used different concentrations of sulfuric acid ranging from 70-98% without any mechanical surface treatment and showed acceptable results with 90 and 98% sulfuric acid, that agreed with the previous authors. Acid etching with such concentrated acid improves the hydrophilicity of the surface by creating sulfonic acid group (-SO3) into the polymer chains due to sulfonation reaction and gives a webbed/interlocking/sponge appearance as seen in the Scanning Electron Microscope images reported in the previous studies **(Stawarczyk *et al.* 2014; Zhou *et al.* 2014; Chaijareenont *et al.* 2018)**. It also initiates a swelling process which increases porosities **(Rocha *et al.* 2016)**. Higher concentrations of sulfuric acid increased the number of functional groups which improved the shear bond strength of the veneering material.

Sandblasting on PEEK surfaces was studied by **Ourahmoune *et al* (Ourahmoune *et al.* 2014)** with various particle sizes of aluminium oxide and pressures they are sprayed and varied timings. Author concluded that sandblasting more than 5 seconds didn't make a substantial difference in the roughness and wettability of the PEEK surfaces. The study also compared composites of PEEK like CFR-PEEK and GFR-PEEK with unfilled PEEK which showed that no difference in surface roughness, but improved surface wettability due to presence of the fibre particles. 7 studies were found which performed tests by veneering composites resins (n=5) and acrylic resins (n=2) over a sandblasted PEEK surface. All these studies used unfilled PEEK hence even we preferred to use the same in the current study.

Prochor *et al* (Prochor and Mierzejewska 2019) compared various grits of sandpaper on PEEK and Titanium surfaces for surface roughness and osteoblast cells adhesion. 240 grit sandpaper showed the highest amount surface roughness. The grit size of sandpaper was considered in relation to the size of the osteoblast cells. With evidence from this study that 240 grit being roughest on PEEK surface, we choose to go on a lower grit of 80, considering the particle size of resin molecules, to create surface roughness in this study. There was no other evidence found on sandpapering and no study was performed to check material adhesion on PEEK surface after this surface treatment.

CONCLUSION

PEEK has shown promising results with respect mechanical and physical properties. It has been used as a material of choice for temporary prosthesis in the past, but it has proven to have the potential to be used as a final prosthesis.

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