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FIXED PROSTHODONTICS AND DENTAL MATERIALS

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EFFECT OF REPEATED HEAT PRESSING ON FRACTURE RESISTANCE AND MODE OF FAILURE OF POLYETHERETHERKETONE (PEEK) THREE-UNIT FIXED DENTAL PROSTHESIS (FDP)

Ahmed Mohamed Arafa* and Marwa Emam**

ABSTRACT

Purpose: to evaluate the effect of repeated heat pressing on fracture resistance and mode of failure analysis of new and reprocessed PEEK either partially or totally 3-unit FDPs.

Methods: 15 PEEK FDPs were manufactured and divided into 3 groups (n=5). **Group I** was pressed using new PEEK, **Group II**; 50% new PEEK and 50% reprocessed PEEK, and **Group III**; 100% reprocessed PEEK. The FDPs were vertically loaded till failure in a universal testing machine. Failed specimens were examined for mode of failure and microstructural analysis with SEM and XRD.

Results: There was a statistically significant difference between mean fracture resistance values of different PEEK conditions (P-value = 0.006, Effect size = 0.577). Pair-wise comparisons between conditions revealed no statistically significant difference between new (1719.6±315.7) and partially reprocessed PEEK (2010.4±444.4); both showed statistically significant higher mean fracture resistance than totally reprocessed PEEK (1097.2±319.5). Mode of failure analysis showed only bending in the new PEEK group bending (100%). While in the partially processed PEEK group 40% of the samples showed visible cracks at the connector area. Finally, in the totally processed PEEK group 80% showed complete fracture.

Conclusion: Totally reprocessed PEEK 3-unit FDPs had the lowest fracture resistance compared to new and partially reprocessed PEEK although all exceeded maximum occlusal forces recorded in the molar area. Incorporating reprocessed PEEK material in 3-unit FDPs increased brittleness and decreased their ability to deform and absorb stresses created during force application.

KEYWORDS: PEEK, fixed dental prosthesis, heat pressing, reprocessing, fracture resistance, microstructure, SEM, XRD.

* Lecturer, Department of Fixed Prosthodontics, Faculty of Dentistry, Beni-Suef University, Beni-Suef, Egypt.
** Lecturer, Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.

INTRODUCTION

The advanced polymer composite with thermoset matrix resins have actually found widespread industrial applications in the defense, automotive, marine, sports communities, aircraft, and aerospace. After curing, they achieve a crosslinked insoluble or infusible rigid three-dimensional molecular structure[1,2]. In contrast to irreversible cured thermosets, thermoplastics on cooling harden but retain their plasticity, so if heated above their processing temperature they can be reshaped again. In early 1980's, recent thermoplastic composites based on aromatic polymers were introduced with an excellent thermal stability, low moisture absorption, high toughness damage tolerance and indefinite shelf life. The short processing cycles with subsequent low manufacturing costs offer reprocessing, remelting, and repairing damaged structures by pressure and heat application. the introduced so-called high temperature and high-performance thermoplastics[3]. Because of the forementioned advantages, high-performance thermoplastic composites gained much interest in the last few years. Among many, Polyetheretherketone (PEEK), Polyetherketoneketone (PEKK), Polyphenylene sulphide (PPS) and Polyetherimide (PEI) perform well in high temperature environments and neither absorb nor degrade when exposed to moisture. The reinforcement with high performance fibers, such as Carbon exhibits exceptional impact resistance and vibrational damping, although they present processing challenges as well due to their high viscosity[4]. Additionally, unlike their cross-linking counterparts, the mechanical properties of these polymer systems can be manipulated through the processing cycle due to variations in levels and types of crystallinity. It is composed of a chain of 100 linear monomer units with an average molecular weight 80000-120000 g/mol. The composition and length of molecular chain have a great effect on the properties of temperature resistance and deformation[5].

Polyetheretherketone (PEEK) is the most significant representative of high-performance thermoplastics family with low melting temperature of 343°C, PEEK can be formed by thermo-pressing procedures, milled with computer-aided design/manufacturing techniques[6] or 3D printing technology[7]. It shows resistance to hydrolysis, chemical wear and deterioration at high temperatures. Also, it offers superior mechanical properties[8]. Additionally, it is biologically inert material[9] with a very light density ($1.32/cm^3$)[10-11] and low modulus of elasticity (3-4 GPa) being close to human bone[12]. All these attractive properties have allowed PEEK applications in various medical applications such as spine implant or bone substitute in traumatology[13]. In order to obtain a higher rigidity of the material for dental applications, pure PEEK material has been modified by filling, blending and fiber reinforcement[14-17].

Innovative material or ceramic reinforced High-Performance Polymer (BioHPP) has recently been introduced in dentistry as a modified PEEK containing 20% ceramic fillers having grain size of 0.3 to 0.5 μm equally distributed in the partially crystalline polymer matrix. It is non allergic and highly biocompatible with low plaque affinity. It exhibits excellent stability, good wear resistance, optimal polishable properties and aesthetic shade. Consequently, BioHPP has become a viable alternative biomaterial replacing conventional polymers, metals, and ceramics overcoming problems faced with porcelain fused to metal restorations such as corrosion of metals which may cause allergies or metallic taste and core opacity preventing light permeability[18-21]. Furthermore, PEEK material is easier to repair than ceramics and more friendly to opposing natural dentition[18,22-25]. With modulus of elasticity resembling that of the bone, a Shock-absorbing effect is allowed reducing the forces transmitted to the restoration and the tooth root accordingly[26]. PEEK has significant applications in dentistry as superstructures for implants, implant- supported bars, provisional abutments, removable partial denture frameworks and fixed dental prosthetic frameworks[27-29].

The manufacturer instructions claim that pressing PEEK more than once would cause it to degrade during another melting process and important physical and mechanical qualities would be lost[30]. PEEK is however an expensive polymer, so the considerable amount of leftover material (the remaining button and sprues) tempts some dental laboratories to repress it from economic point of view[31].

Evidence is lacking regarding whether PEEK material could be safely reprocessed and the impact of repeated pressing on the crystallinity and mechanical characteristics of PEEK restorations is not clear up till now. The aim of this study was to test the null hypothesis that there is no difference in fracture resistance between new and reprocessed PEEK either partially or totally regarding full contour three-unit fixed dental prostheses (FDPs) fabricated with pressing technique.

MATERIALS AND METHODS

According to Stawarczyk et al[32], a sample size calculated, the minimally accepted sample size was 5 per group, when the response within each subject group was normally distributed with standard deviation 439, the true mean difference was 850, when the power was 80 % & type I error probability was 0.05. The details of the materials used in this study are mentioned in Table 1.

In this study, the fracture resistance of BioHPP full contour 3-unit fixed dental prostheses (FDPs) were tested. A total of 15 three-unit fixed dental protheses (FDPs) were fabricated from pressed PEEK (for 2 press BioHPP Granulate, Bredent GmbH & Co KG, senden, Germany). The specimens were divided into 3 groups 5 specimens each (n=5) according to the weight percentage of new and reprocessed (repeated pressing) material used as following: the first group was 100% new PEEK. The second group was partially reprocessed PEEK, in which 50% new and 50% reprocessed PEEK were used. The third group was 100% reprocessed PEEK (Table.2).

TABLE (1): Materials and equipments used

Material	Product name	Manufacturer	Composition
PEEK	for 2 press BioHPP (Granulate)	Bredent GmbH & Co KG	PEEK, 20% weight ceramic filler
Investment material	Brevest for 2 Press	Bredent GmbH & Co KG	Phosphate bonded investment
Aluminum oxide	Cobra	Renfert GmbH	Aluminum oxide sand (110 μm mean particle size)
Sandblaster	Basic Classic, 70-250μm, 220-240 V	Renfert GmbH	1 x 70–250 µm, incl. nozzle 1.2 mm

TABLE (2): The experimental groups

Group I (Control)	Group II (Partially reprocessed PEEK)	Group III (Totally reprocessed PEEK)
100% new PEEK n=5	50% new + 50% reprocessed PEEK n=5	100% reprocessed PEEK n=5

Master dies preparation

In order to obtain standardized FDPs, two stainless steel dies were designed simulating a prepared second lower premolar and second lower molar to receive 3-unit FDP. They were machine milled to the dimensions of 5 mm height (assuming flat occlusal reduction with a uniform 120 degrees circumferential chamfer finish line of 1 mm width, and a total occlusal convergence angle of 12 degrees). The diameter for premolar die was 6 mm while for molar die was 8 mm. The stainless-steel dies were placed in pairs in a stainless-steel holder. The distance between the centers of the master model dies was adjusted to 17.5 mm to reproduce an average distance from the center of second premolar to that of the second molar. Each master die was tightened in its place using tightening screw to ensure complete stabilization during fracture testing procedures (Figure 1).

Construction of resin patterns

The stainless-steel dies representing prepared abutments for FDP were scanned using AutoScan-DS-EX (Shining 3D, Hangzhou, China). The stainless-steel holder with tightened dies in place was fixed at the jig of the scanner and the metal dies were sprayed with scan spray (Renfert-Scanspray, Renfert GmbH, USA) for optimal scanning accuracy. After the 3D model of stainless-steel dies was obtained, the FDPs design was constructed utilizing a designing software (Exocad Dental CAD; exocad GmbH, Germany) where all the "Biogeneric" preparation margins and base lines for FDP had been selected. Full anatomical FDPs were constructed where axial area had 1.5 mm thickness, occlusal area had 2 mm[33] and connectors with a cross-section area of 16 mm^2 [32], a bucco-lingual width of 3.6 mm and an occluso-gingival height of 4.45 mm (Figure 2).

The STL file of the design was exported to print preparation software (PreForm software, formlabs, Massachusetts, USA) to create resin frameworks utilizing 3D printer (Form 2, Formlabs SLA 3D printer, Massachusetts, USA). The printed castable resin patterns were washed and dried using automated wash station (Form Wash, FormLabs, Massachusetts, USA) before the unneeded supports were removed.

Fabrication of FDPs by pressing technology

The 3D printed resin patterns were transferred into PEEK using lost wax and heat pressing techniques. The 3D printed resin patterns were sprued and invested using phosphate bonded investment material (Brevest For2press, Bredent, Germany) according to manufacturer's instructions. After

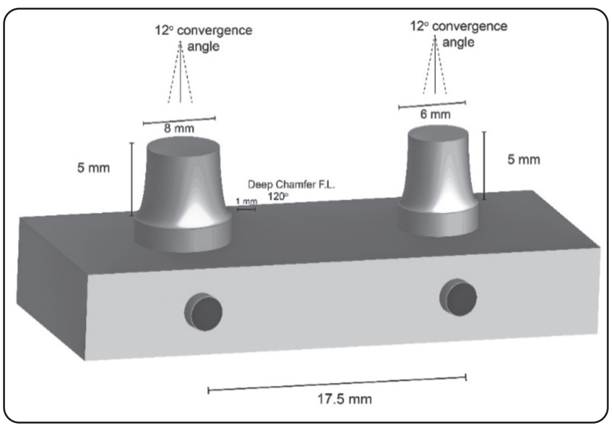

Fig. (1): Schematic diagram of the fabricated stainless-steel dies in a stainless-steel holder.

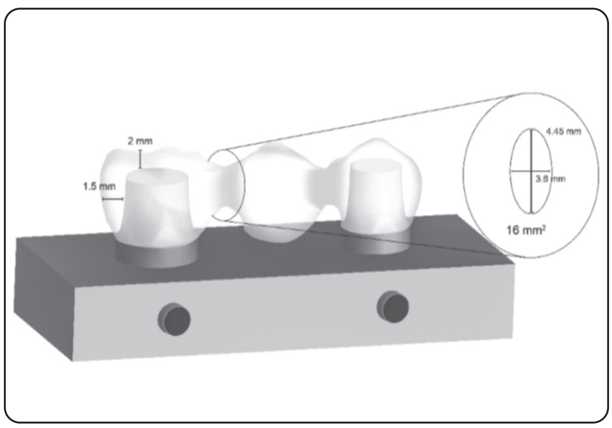

Fig. (2). Schematic diagram of 3-unit FDP seated on the stainless-steel dies.

resin elimination, Pressing of Granulate PEEK (for 2 press BioHPP, Bredent GmbH & Co KG, senden, Germany) using vacuum press device (For2press, GmbH & Co KG, senden, Bredent, Germany) was performed. A fine blasting unit (Basic Classic, Renfert GmbH, Germany) with 110 µm alumina (Cobra Abrasive, Renfert GmbH, Germany) at 3 bar pressure was used for the divesting process then the specimens were finished and polished following manufacturer instructions.

Fracture resistance test

All specimens of tested groups were loaded compressively in a universal testing machine (Instron Model 3345, England) at cross head speed 1 mm/min [33]. Each specimen was individually secured by tightening screws on the lower fixed compartment of the universal testing machine. The loading piston, a vertically movable rod with a semi-spherical head 5 mm in diameter, was mounted directly over the central fossa of occlusal surface of pontic sample. Even stress distribution was ensured by placement of a tin foil between the load piston and the specimen. The load at failure was recorded in Newton as soon as load decreased by 10% of the maximum load [33] using computer software program (BlueHill 3 software version 3.3). (Figure 3).

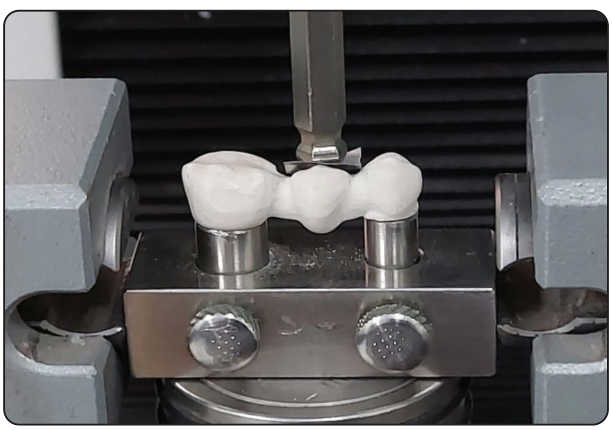

Fig. (3). Full contour 3-unit FDP during fracture load measurement.

Scanning electron microscopy (SEM) examination

Fractured and deformed surfaces were inspected and cleaned with isopropyl alcohol, gold-sputtered then evaluated using SEM in high vacuum (JSM-6360LA; JEOL, Tokyo, Japan) operating at 15-20 kV. Magnification ranged between 150, 1000 and 5000 X.

X-ray diffraction analysis (XRD)

X-ray diffraction analysis to characterize the crystalline properties was performed to a representative part taken from a specimen of each group. Using a diffractometer (D8 ADVANCE, Bruker, Germany).

Statistical analysis

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed normal (parametric) distribution. Data were presented as mean and standard deviation (SD) values. One-way Analysis of Variance (ANOVA) was used to compare between different PEEK conditions. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY:IBM Corp.

RESULTS

Fracture resistance

There was a statistically significant difference between mean fracture resistance values of different PEEK conditions (*P*-value = 0.006, Effect size = 0.577). Pair-wise comparisons between conditions revealed that there was no statistically significant difference between new and partially reprocessed PEEK; both showed statistically significantly higher mean fracture resistance than totally reprocessed PEEK. (Table.3) (Figure 4)

TABLE (3). The mean, standard deviation (SD) values and results of one-way ANOVA test for comparison between fracture resistance (N) of different PEEK conditions

New PEEK (n = 5)		Partially reprocessed PEEK (n = 5)		Totally reprocessed PEEK (n = 5)		*P*-value	Effect size *(Eta squared)*
Mean	SD	Mean	SD	Mean	SD		
1719.6 ^{A}	315.7	2010.4 ^{A}	444.4	1097.2 ^{B}	319.5	0.006*	0.577

: Significant at $P \le 0.05$, Different superscripts are statistically significantly different

Mode of failure analysis

Mode of failure analysis of the failed samples revealed that in the new PEEK group bending was the dominant mode of failure (100%) with zero samples showing cracks or evident signs of fracture. While in the partially reprocessed PEEK group two out of five samples (40%) showed visible cracks at the connector area. Finally, in the totally reprocessed PEEK group four out of five (80%) showed complete fracture. (Figure 5)

Microstructure and Scanning Electron Microscopy SEM

SEM images of the investigated groups are illustrated in (Figure 6) at two different magnifications 150 and 1000 X showing the deformation suffered by the polymer at the areas of deformation and fracture. The cracking and fracture lines seem to increase in the reprocessed groups.

SEM images of the investigated groups are illustrated in (Figure 7) at 5000 X showing crystals size. The crystals size ranged between 1.3 to 1.7 μm in the new PEEK group, 548 to 872 nm in partially reprocessed PEEK group and 2.9 to 3 μm in totally reprocessed PEEK group.

X-Ray Diffraction (XRD) Analysis

The results obtained from the X-ray diffraction analysis are displayed in (Figure 8) showing well crystalline peaks with crystallinity percentage decreasing with increased reprocessing.

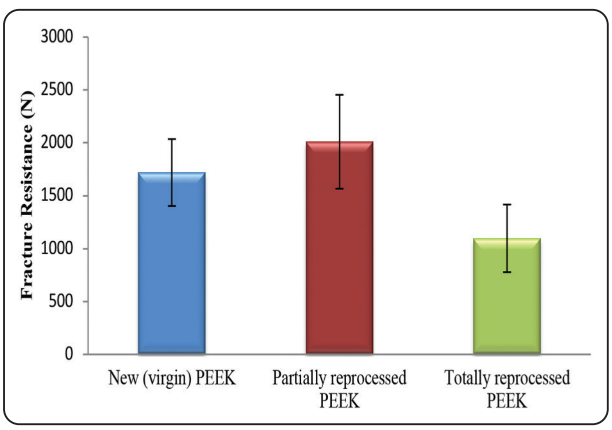

Fig (4). Bar chart representing mean and standard deviation values for fracture resistance (N) of different PEEK conditions.

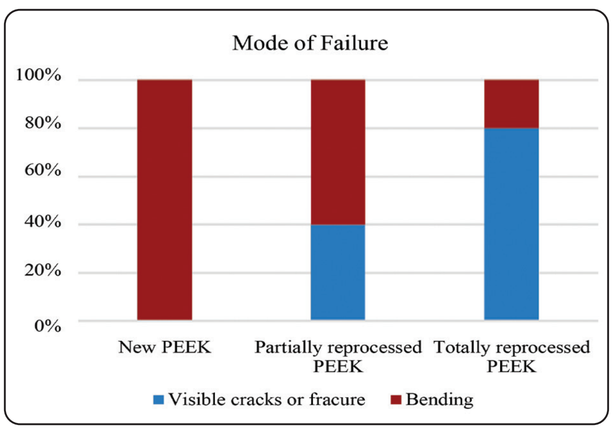

Fig (5). Bar chart showing percentage (%) of each failure type in the tested group.

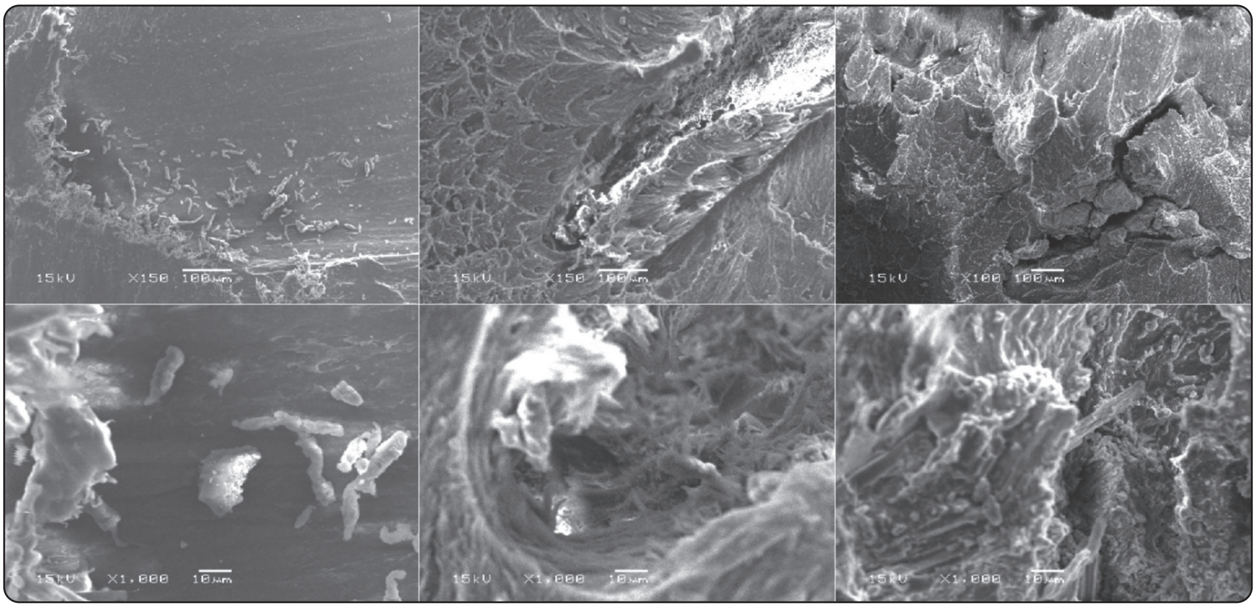

Fig (6). SEM images in two magnifications of the microstructure of tested groups in crack and deformation areas. Left: New PEEK, Middle: Partially reprocessed PEEK, Right: Totally Reprocessed PEEK

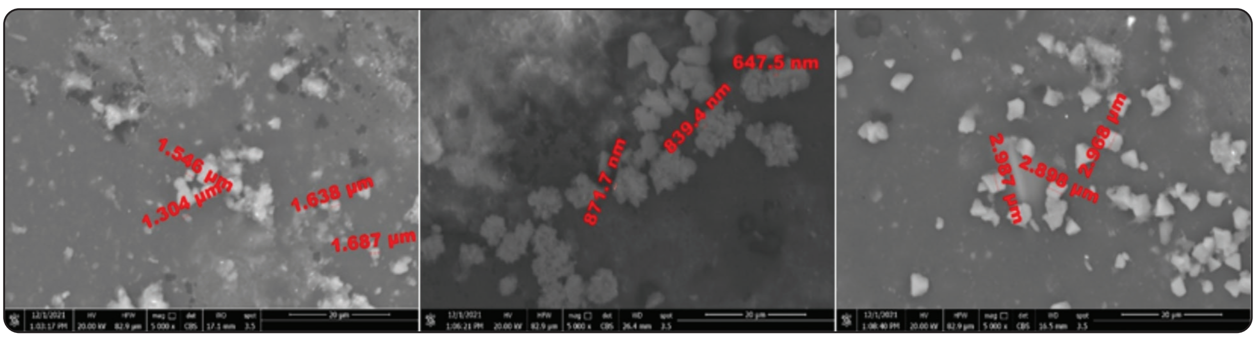

Fig (7). SEM images of crystal size. Left: New PPEK, Middle: Partially reprocessed PEEK, Right: Totally reprocessed PEEK (5000 X).

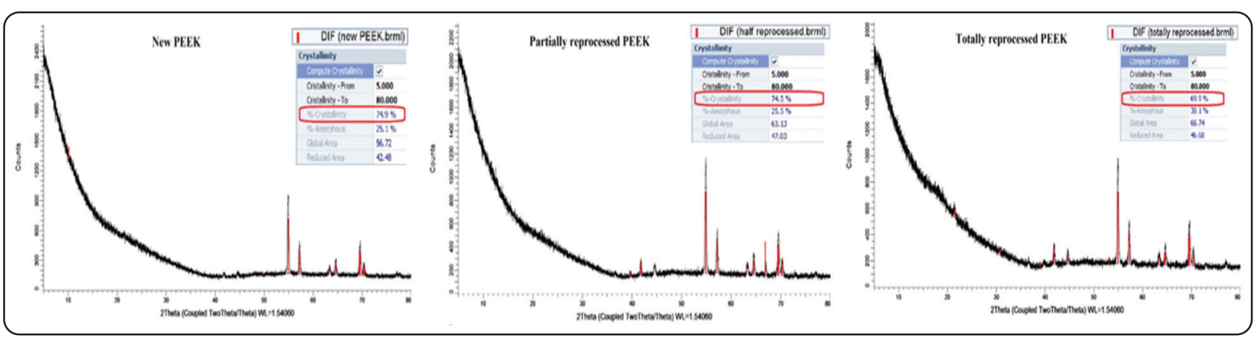

Fig (8). XRD spectra with crystallinity percentage: left: New PEEK, middle: Partially reprocessed PEEK, right: Totally reprocessed PEEK.

DISCUSSION

Fixed dental prostheses (FDPs) constructed from different materials represent a viable prosthetic treatment choice for the replacement of missing teeth. In the past, full metal or porcelain fused to metal frameworks have been used whereas metallic FDPs have often been suffered from esthetic shortcomings. Several different materials available for FDPs have been analyzed. In literature, the FDP framework material with a low E-modulus has been proved to result in a more even stress distribution within the framework[34]. The rapid evolution of restorative materials in dentistry led to introduction of a relatively new generation of composites namely high-performance polymer (HPP) [32].

Polyetheretherketone (PEEK) which is good representative member of this family, is semi-crystalline high-performance polymer which combines excellent resistance to chemicals, as well as good mechanical properties and high temperature resistance [6,35]. It has an off-white color and offers many advantages. It is located at the top of the thermoplastic polymer pyramid with capability of melt processability which allows that the material can be repaired and reprocessed many times at temperatures above the melting or softening point [36].

High performance polymer (BioHPP) is a PEEK variant strengthened with 20% ceramic filler of 0.3-0.5 μm. This allows better polishing ability of the restorations resulting in less plaque retention and more color stability [37,38]. Based on these excellent biological and physical properties, BioHPP is considered ideal for superstructures in dental field as provisional abutments, dental implants, fixed and Removable prostheses [32].

PEEK has been thoroughly investigated in literature as an alternative for single crowns and fixed dental protheses (FDPs) [13,39-42] However, none have investigated the effect of reprocessing and repressing the material either partially or totally on mechanical properties of PEEK FDPs. Thermal processing history of this polymer can be considered a very important determinant of its properties due to its semicrystalline thermoplastic properties. The dramatic influence of the thermal history on percent crystallinity, microstructure, and resulting ultimate properties, has not yet been reported.

In order to closely imitate clinical condition of replacing one missing tooth with fixed prosthesis, three-unit FDPs were selected [43]. In literature, steel or resin dies have been used for fracture testing of ceramic crowns and FDPs [44,45]. In this study using natural teeth was avoided due to invisible cracks or inconsistent dentin structure that may cause the tooth to fracture during fracture testing and may result in some restrictions in the reproducibility and comparability between specimens [46]. Stainless steel dies with elastic modulus 200 MPa were used. They can be standardized with the same physical properties and dimensions, such as taper and finish line [47].

It has been showed that abutment mobility is a decisive clinical factor in the fracture resistance of FDPs when a small amount of abutment rotation occurs; failure is more probable [48]. For that reason, in our study, the abutments were secured in stainless-steel holder at each accurate position so that no mobility would occur during testing procedures to ensure consistency. The fracture loading was performed using a standardized protocol. The fracture loading Parameters were within the range of similar studies with a crosshead speed of 1 mm/min and a 5 mm ball diameter size [49].

In the present study, the null hypothesis was partially rejected as there was no statistically significant difference between new (1719.6 ± 315.7) N and partially reprocessed PEEK groups (2010.4 ± 444.4) N; but both showed statistically significantly higher mean fracture resistance than totally reprocessed PEEK group (1097.2 ± 319.5) N. To the knowledge of the authors, this is the first study to estimate the effect of reprocessing of PEEK used for dental restorations on fracture resistance of FDPs. For that reason, the results obtained from this study could not be compared to results obtained from other available studies.

In an in vitro study by Stawarczyk et al [32] that investigated monolithic PEEK three-unit fixed dental prostheses (FDPs) fabricated by CAD/CAM technology from pre-pressed PEEK blanks and by pressing from granular form in an in vitro study, the fracture loads were 2354 N and 1738 N respectively. In another study, three-unit PEEK framework demonstrated deformation of the FDPs at 1200 N and connector fracture of FDPs at 1383 N [39].

Regarding effect of reprocessing on bond strength, another study [50] evaluated the effect of repeated heat pressing and thermocycling on bond strength of PEEK to resin cement. The results manifested that incorporation of previously processed PEEK either partially or totally and thermocycling decreased micro-shear bond strength between resin cement and PEEK significantly.

Evaluation of the results of this study showed a significant decrease of the fracture resistance of three-unit FDPs between the new PEEK group and the totally reprocessed PEEK group. This may be attributed to molecular deterioration and degradation mechanisms of PEEK from repeated heating of polymer starting with molecular scission in which components of the polymer chain backbone begin to separate together to shorter molecular chains with cross-linking of these fragments with other polymer chain elements and higher mobility. These molecular changes create structural defects resulting in significant deterioration of the mechanical properties [36]. Partially reprocessed PEEK showed insignificant increase in the fracture resistance of three-unit FDPs when compared with new PEEK group. This may be attributed to that the repressed material might have acted as nuclei of crystallization for new polymer chains with increased branching and netting. Therefore, causing increase in fracture resistance.

All groups in this study showed failure at loads beyond maximum occlusal forces recorded in the molar area so they are all considered clinically accepted. Occlusal loading during mastication varies individually and have been reported to be highest in the posterior area for adult male 600-900 N [49,51-53].

Scanning electron microscopic observation of a sample from each group showed that largest crystal size was found in totally reprocessed PEEK group. This may be explained by presence of some unmelted crystals which preferentially nucleate crystallization allowing rapid crystal growth. In Partially reprocessed PEEK, the crystal size was the smallest. This may be explained by presence of nuclei of crystallization from totally reprocessed part and those from new PEEK which lead to multiple nucleation centers. hence, did not allow for crystal growth and maturation.

Moreover, XRD results showed decrease in the crystallinity with increase of amorphous part of PEEK at temperature above melting point. From a micro-structural point of view, semi-crystalline polymers are combination of two phases: amorphous regions (amorphous polymer chains or fibrils) and crystalline regions (crystallites). The relative amount of crystalline regions can be influenced by thermal processing conditions such as cooling rate during crystallization from the melt. Molecular degradation of PEEK can be manifested clinically with increased brittleness and lower shock absorption ability which was proved with the mode of failure analysis findings.

The dominant mode of failure in the new PEEK samples was deformation (100%) while in totally reprocessed PEEK was fracture (80%) which indicated lower mobility of polymeric chains. Scanning electron microscopic observation of the fracture specimens confirmed the results findings as the evident cracking was evident on microstructural level with increasing the reprocessed PEEK material in the FDP.

All specimens tested in this study were not aged so further investigations with additional aging in chewing simulator or thermocycling are required to support the application of reprocessed PEEK for long-term restorations.

CONCLUSION

1. Totally reprocessed PEEK three-unit FDPs had the lowest fracture resistance compared to new and partially reprocessed PEEK although all exceeded maximum occlusal forces recorded in the molar area.
2. Incorporating reprocessed PEEK material in three-unit FDPs increased brittleness and decreased their ability to deform and absorb stresses created during force application.

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