

## Influence of polyetheretherketone short span Bridges surface treatment on their flexural strength

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### Abstract:

**Background:** This *in vitro* study was conducted to evaluate the influence of surface treatment on flexural strength of computer aided design/computer aided manufacturing (CAD-CAM) fabricated polyetheretherketone short span Bridges using different surface treatment method.

**Materials and Methods:** Twenty-one 3-unit FDPs frameworks replacing a first molar with abutments on 2nd premolar and 2nd molar and were prepared in a standardized method by a steel model was used with 2 abutments, which will also make of steel to minimize their residual deformation during loading. 21 impressions of the model with steel abutments were taken by replisil silicone impression material (REPLISIL 22N, dent-e-con, Germany) then poured in epoxy resin to produce the epoxy resin models. After scanning of the steel model using CAD/CAM optical scanner (SHERA Werkstoff-Technologie GmbH & Co. KG Espohlstrasse Lemförde, Germany). Three group of standardized three-unit FDPs framework was made of Bio HPP blank using (CAD/CAM) milling machine (Imes-Core GmbH Im Leibolzgraben, Eiterfeld / Germany) with flat occlusal surface (n=7/group) were fabricated. Cementation of the specimens were done using dual cure resin adhesive (Panavia F2). The specimens were divided into 3 groups (n = 7) to receive different surface treatment materials. Group I Consists of seven samples (n=7) of peek framework without any surface treatments as a controlled group. Group II Consists seven samples (n=7) which were abraded by airborne particles with a mean particle size of 110 µm Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) at 2.5 bar air pressure at a working distance of 7 mm for 15 second. Group III: Consists of seven samples (n=7) which were etched with (98%) sulfuric acid for 60 seconds followed by washing in running water for one minute. Scanning electron microscopy was performed to analyze morphological examination only. Flexural strength values were evaluated by three-point bending test using "Instron testing machine". Data were analyzed for statistical significance using one-way ANOVA analysis of variance.

**Results:** Different techniques used in surface treatment had an effect on the flexural resistance. Regarding these techniques, the initial flexural resistance of all test groups showed statistical significance difference. Sulfuric group had the highest value (893.429 ±153.314 Mpa) and Controlled group had the lowest value (330.408 ± 19.183 Mpa). **Conclusion:** Bio-HPP Framework which treated by sulfuric acid showed the highest flexural strength.

**Key Word:** Framework, 3-unit short span bridges, flexural resistance, CAD/CAM, BioHPP, surface treatment, sulfuric acid (98%), Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>), Panavia F2.

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### I. Introduction

Many ceramic materials have been introduced and proposed for use in the posterior region. A new generation of material in dentistry, known as Bio HPP (High Performance Polymer) may be a suitable alternative with comparable wear properties in the range of ceramics. Bio HPP is biocompatible and resistant to nearly all organic and inorganic chemicals. It can withstand high temperatures as it has high mechanical properties and high dimension stability<sup>1</sup>.

The elasticity of the material lies within the range of bone which makes it a more natural material because of its ability to compensate for the torsion of bone upon occlusal forces especially in the case of larger implant work and long framework. Also it has no abrasive effect on the remaining teeth<sup>2</sup>. Based on the excellent physical and biological properties of this material, it seems to be suitable for superstructures in dentistry field, i.e. for provisional abutments, dental implants and FDPs frameworks<sup>1</sup>.

Surface modification of biomaterials plays a significant role in determining the outcome of biological materials interactions. Surface treatments increase surface area and create microporosities on peek material surface which enhance the potential mechanical retention of the material also promote surface reactivity of material to veneer or dentin<sup>3</sup>. Chemical surface treatment of peek include use of sulfuric acid<sup>4,5</sup>. Acid treatment leads to hydrolysis of the connecting ether and ketone links<sup>6</sup>. Also, it provides more functional groups to which the components of adhesive systems can bond through the emerging carbon-oxygen compounds<sup>7</sup>.

On the other hand, Mechanical surface modification such as airborne-particle abrasion (sandblasting) creates micro undercut areas, increases the surface roughness and improve the micro-mechanical retention of bonding agent which increase the surface energy and wettability<sup>8,9</sup>.

The fracture mechanics approach is considered a reliable indicator of the performance of brittle materials<sup>10</sup>. Therefore, the purpose of this study is to evaluate influence of surface treatment on the flexural strength of polyetheretherketone bridge.

## II. Material And Methods

This prospective comparative study was carried out on steel models and Approval for this research was obtained from Research Ethics Committee, Faculty of Dentistry, Tanta University. The design and procedures of the present study were accomplished according to the research guidelines published by Research Ethics Committee, Faculty of Dentistry, Tanta University, from November 2018 to February 2020. A total 21 Bio-HPP framework specimens were for in this study.

**Study Design:** Prospective open label observational study.

**Study Location:** Fixed Prosthodontics Department, faculty of dentistry/ Tanta university, Egypt.

**Study Duration:** November 2018 to February 2020.

**Sample size:** 21 specimens of 3-unit Bio-Hpp frameworks.

**Subjects & selection method:** The study was done between from November 2018 to February 2020. This study consists of twenty-one Bio-HPP framework (N=21) and these specimens were randomly divided into 3 groups according to surface treatment techniques, Table (1), Each group include seven specimens for each group (n=7) as follows:

Group I(N=7 frameworks) .

Group II (N=7 frameworks).

Group III (N=7 frameworks).

**(Table 1):** Materials used in the study are presented with their main composition, manufacturer.

Material	Product Name	Manufacturer	Composition
Peek	bre.CAM Bio HPP	Bredent GmbH & Co. KGt, Senden, Germany	1.31 g of 4,4'- difluoro benzophenone, 0.66 g of hydroquinone, and 1.24 g of K <sub>2</sub> CO <sub>3</sub> has to be dissolved in a mixture containing 15ml of solvent and 35ml of toluene
Resin cement	Panavia f 2.0	Kuraray, Noritake Dental Inc, Japan.	Dental Dual-Cured Adhesive Resin Cement
Adhesive system	Visio.link	Bredent GmbH & Co KG	MMA, pentaerythritol triacrylate, photo initiators
Surface treatment	Particle abrasive(cobra)	Renfert, GmbH, Germany	Airborne Particles abrasion with a mean particle size of 110 µm Aluminium oxide and 2.5 bar pressure at a working distance of 10 mm for 15 second.
	Sulfuric acid	Laboratory	98% H <sub>2</sub> SO <sub>4</sub> for 60 sec

### Procedure methodology

A model with two steel abutments simulating a FDPs between a second premolar and a second molar was fabricated using a standardized computer numerical control machine (CNC)<sup>11</sup>. The abutments of this model were milled to be cylindrical (diameter: 7 mm premolar; 8 mm molar) (height:4 mm for the premolar; 5 mm for the molar) with a 1-mm circular shoulder and 6 degree of taper<sup>1,12</sup>. The abutments were anchored in acrylic block with the aid of a surveyor (Bredent GmbH & Co.KGt,Senden,Germany) to ensure parallelism<sup>13</sup>.

21 impressions of the model with steel abutments were taken by replisil silicone impression material (REPLISIL 22N,dent-e-con,Germany), then poured in epoxy resin (Kemaboxy 150 Chemical industries of construction CIC-Egypt) to produce the epoxy resin models<sup>14</sup>.

### **Construction of FDPs framework:**

After scanning of the steel model using CAD/CAM optical scanner (SHERA Werkstoff-Technologie GmbH & Co. KG Espohlstrasse Lemförde, Germany). Three group of standardized three-unit FDPs framework was made of Bio HPP blank using (CAD/CAM) milling machine (Imes-Icore GmbH Im Leibolzgraben, Eiterfeld / Germany) with flat occlusal surface (n=7/group) were fabricated. The wall thickness of the framework was 0.7 mm and the connectors had an almost rectangular cross-section of 7.36 mm<sup>2</sup> surface area, an occluso-gingival height of 3.2 mm, and a bucco-lingual width of 2.3 mm.<sup>12</sup>

### **Milling procedures of Bio HPP frameworks:**

- 1)The disk was taken from the packaging and confirmed that the disk did not have a crack or other damage.
  - 2)The disk is placed into the milling machine, then the milling process (Dry milling) was started according to the standardized design following the milling system technical instructions using the breCAM. cutter (a milling tool especially matched with the properties of the material).
  - 3)Milling chips were collected during dry processing using suction device connected to the milling (CAM) system.
  - 4)After milling, the frameworks were removed from the disk with a diamond bur.
  - 5)The cutting waste or dust, which were attached to the restorations, were removed with gentle air steam.
- After removing the Bio HPP frameworks, they were checked on the steel model and on the epoxy resin models.

### **Surface treatment:**

This study consists of twenty-one Bio-HPP framework (N=21) and these specimens were randomly divided into 3 groups. Each group include seven for each group (n=7)

- Group I:** Consists seven samples of Bio-HPP framework (n=7) without any surface treatments as a controlled group.
- Group II:** Consists seven samples of Bio-HPP framework (n=7) which were abraded by airborne particles with a mean particle size of 110 µm Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) at 2.5 bar air pressure at a working distance of 7 mm for 15 second.
- Group III:** Consists seven samples of Bio-HPP framework (n=7) which were etched with (98%) sulfuric acid for 60 seconds followed by washing in running water for one minute.

### **Examination of Morphological surfaces:**

One sample from each group was used for morphological examination only and are not subject to complete of the research. The surface structure topography of each pretreatment group was examined under a scanning electron microscope (JEOL JSM-5200LV scanning microscope, JAPAN). For this purpose, specimens were ultrasonically cleaned then coated with gold-palladium (SPI-MODULE VAC/Sputter coater, USA).

### **Scanning electron microscopy (SEM) observations:**

Scanning electron microscopic images at high magnifications after the different surface treatment methods are depicted in the following data.

Non treated Bio HPP showed a plain and homogeneous surface. While the sandblasting group exhibited irregular, fissured surfaces with polygonal-shaped alumina oxide embedded in them.

Etching with sulfuric acid displayed round cavities on the Bio-HPP surface showing a complex network characterized by a sponge-like porous fiber network and sub-surface corrosion.

### **Cementation of FDPs framework on the epoxy resin models:**

All frameworks firstly were coated with Visio.Link primer and polymerized in ultraviolet light curing unit for 90 sec before cement application.

Equal amount of Paste A and B were mixed, then was applied to the fitting surface of Bio HPP framework according to manufacturer's instructions. The frameworks were held in place over their corresponding epoxy dies by the using of a specially designed and fabricated holding device which securely held the specimen to insure marginal adaptation. The frameworks were cemented to the corresponding epoxy resin models then exposed to a brief light curing for only 2 seconds and excess cement was removed with a scaler(52, 5353). After removing excess cement, Oxyguard was applied to allow self-curing of the cement and to prevent oxygen inhibition of polymerization.

A specially designed cementation device was machined in order to aid in load (3 kg) application via Instron testing machine during cementation procedure to ensure even flow of the cement<sup>15</sup>.

**Flexural strength, Brief Report**

These tests were performed using Bluehill Lite Software from Instron®.

**Test procedure**

All samples were individually mounted on a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) with a load cell of 5 kN and data were recorded using computer software (Instron® Bluehill Lite Software). Samples were secured to the lower fixed compartment of testing machine by tightening screws.

Fracture test was done by flexure (bending) load applied occlusally at the center of the pontic using a metallic rod with round tip (5.6 mm diameter) attached to the upper movable compartment of testing machine traveling at cross-head speed of 1mm/min with tin foil sheet in-between to achieve homogenous stress distribution and minimization of the transmission of local force peaks.

The load at failure manifested by an audible crack and confirmed by a sharp drop at load-deflection curve recorded using computer software (Bluehill Lite Software Instron® Instruments). The load required to flexure fracture resistance was recorded in Newton and flexural strength recorded in megapascal.

Loading was continued until bridge failure occurred. The maximum loads were recorded. Flexural strength values were calculated from the following formula: <sup>16</sup>

$$\text{Flexural strength} = \frac{3F_{\max} l}{2bh^2}$$

Where: Fmax= maximum load before fracture; I = distance between supports; b = width of specimen; h = height (thickness) of specimen

**Statistical analysis**

Mean values for each group were calculated, and differences between the groups were tested for statistical significance using one-way ANOVA analysis of variance.

**III. Result**

Data analysis was performed in several steps. Numerical variables are expressed by descriptive statistics as mean and standard deviation that all were recorded in MPs.

Statistical analysis using three-way ANOVA showed that there was a significant statistical difference between the groups of surface treatment and its influence on the flexural strength and the interaction between them.

P-value <0.001(\*) was considered significant difference in all tests. Statistical Package for Social Sciences (SPSS version 24; chichago, IL, USA) were used to analyze data.

**Flexural strength**

Measurements showing mean values, standard deviations (SD), range (minimum & maximum) and 95% confidence intervals (CI) limits (lower and upper) for flexural strength measured in megapascal (Mpa) recorded for three groups summarized in table (2) and graphically represented in (Fig 1).

The flexural strength mean±SD values recorded for Bio HPP controlled group were (330.408 ± 19.183 MPa) with minimum value (304.3 Mpa) and maximum value (360.6 Mpa).

While The flexural strength mean±SD values recorded for Bio HPP with airborne abrasive group were (485.820 ± 72.148 Mpa) with minimum value (392 Mpa) and maximum value (589 Mpa).

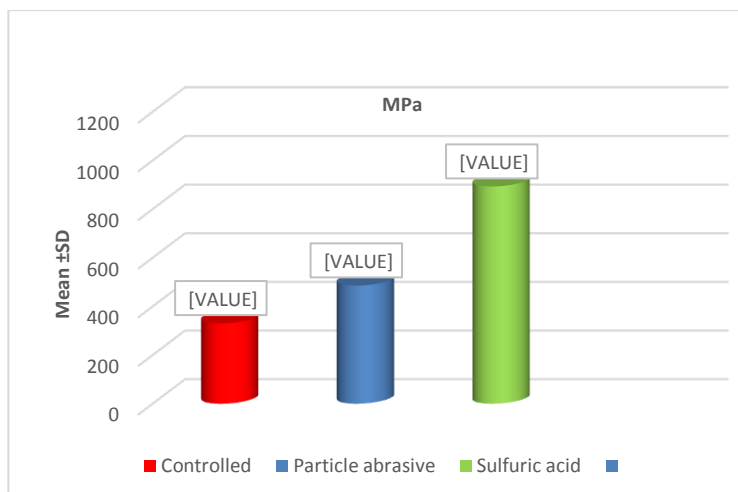
The flexural strength mean±SD values recorded for Bio HPP sulfuric acid group were (893.429 ±153.314 Mpa) with minimum value (664.5 Mpa) and maximum value (1102.2 Mpa).

It was noted that Bio HPP with Sulfuric acid group recorded statistically significant (p<0.01) higher flexural strength mean values than other groups.

It was noted that Bio HPP with Sulfuric acid group recorded the highest flexural strength mean values (893.429 ±153.314 Mpa) ,While Controlled group recorded the lowest mean values (330.408 ± 19.183 MPa).

**Table (2)** Measurements of flexural strength (Mpa) results (Mean values ±SD) for all groups.

Groups	MPa						ANOVA	
	Range			Mean	±	SD	F	P-value
Controlled	304.3	-	360.6	330.408	±	19.183	87.228	<0.001*
Airborne Particle abrasive	392	-	589	485.820	±	72.148		
Sulfuric acid	664.5	-	1102.2	893.429	±	153.314		
TUKEY'S Test								
C&P			C&S			P&S		
<0.001*			<0.001*			<0.001*		



**Fig. 1** Column chart showing flexural strength mean values for surface treatment groups

#### IV. Discussion

This in vitro study evaluated the flexural resistance of CAD/CAM 3unit Bio HPP FDPs framework (tested material) by using two different kinds of surface treatment techniques on the fitting surface of the specimens and compare the effect of each one with the controlled group which has no surface treatment. In vitro studies offer standardized and optimized conditions in the experimental performance which may not be possible to achieve in vivo<sup>17,18</sup>. Clinically, many factors such as tooth preparation, impression and cementation techniques can complicate the testing process and deviate from the ideal situation<sup>19</sup> making the in vivo measurements more difficult than in vitro ones<sup>20</sup>.

Therefore, this in vitro was conducted to study the Influence of polyetheretherketone short span bridges surface treatment on their flexural strength.

In 1999, **Beschmidt & Strub**<sup>21</sup> reported the difficulties of using natural teeth in laboratory studies as they present a great variation considering the age, individual structures and time of storage making the standardization of the abutments difficult. Also, extracted human teeth are very difficult to obtain due to recent progress in conservative dental treatment, complicated restrictions and the research ethics followed in studies using human substrates<sup>22</sup>.

Therefore, several authors have employed metallic or resin models for the measurements in various studies<sup>23</sup>. In this study, metallic master models were currently used to achieve standardization for all samples<sup>24</sup>.

The flexural resistance test of the Bio HPP frameworks was done on the epoxy resin models, which have an elastic modulus closer to dentine than that of metal<sup>1</sup>.

Polyether ether ketone (PEEK) has been used in the present study as a framework material in FPDs. Materials with a lower modulus of elasticity such as PEEK and composite resins have been proved to reduce occlusal stress by acting as stress breakers<sup>25</sup>. PEEK exhibits a modulus of elasticity of 4 GPa, which could dampen force transmission, thereby preventing the tooth and subsequently the root from overloading and breakage. PEEK have good polishing properties, wear resistance, low plaque and is radiolucent, which may facilitate recurrent caries detection<sup>26</sup>.

In our study BioHPP is manufactured within a framework. As It is not esthetic, the material cannot be processed in an overall shape<sup>12,27</sup>.

In addition, PEEK as a core material further reduces the elasticity of the composite resin veneering material from 8 to 10 GPa to 4 GPa<sup>28</sup>.

Secondly, flexural strength and elastic modulus could have an important effect. The flexural strength of BioHPP (165 MPa) and elastic modulus of (4 GPa)<sup>29</sup>. So that, the flexural strengths of the materials in this study seem to reflect the fracture strengths of the frameworks<sup>30</sup>.

Restoration clinical evaluation showed good retention and appearance with no sign of microleakage, better marginal fit and good fracture strength with very high patient comfort and acceptability. So that PEEK could be considered an alternative framework material for FPDs restorations<sup>31</sup>.

So, using PEEK (BioHPPBredent, GmbH & Co. KG, Germany) as a framework material, the restoration not only gains retention but further protecting the tooth and the root structures.

In other study, the PEEK three-unit FDPs showed significant higher fracture resistance (1626.31±191.9 N). It can therefore be assumed that PEEK blank has increased mechanical properties. This is may be due to:

- 1) Nature of the material: PEEK is a semi crystalline polymer that has a considerable ductility and can accommodate a wide range of plastic deformation in both uniaxial tension and compression. The

crystalline content of PEEK enhances its hardness and it varies depending upon its thermal processing. Increasing crystallinity increases tensile modulus and yield strength.<sup>32</sup>

2) Behavior of the material during high rate testing: Semi crystalline polymers undergo a hardening mechanism during deformation caused by strain induced crystallization (SIC) in which the alignment of polymer chains gives rise to an increase in overall crystalline content in the material and hence an increase in density and hardness<sup>33</sup>. This behavior is not fully understood till now but, in any event, it seems that PEEK does work harden at large strains under static compressive loading.<sup>34</sup>

3) Industrial fabrication of CAD/CAM blanks under optimal conditions display a reduced risk of porosities within the restorations and therefore show improved mechanical properties<sup>1</sup>.

4) Better marginal adaptation of Bio HPP because of the absence of a sintering process and therefore, of contraction<sup>27</sup>, which increases the fracture resistance of the material under functional loading<sup>35</sup>.

The original model with the steel abutments was scanned, and a fixed design with the same dimensions - the wall thickness was 0.7 mm and the connectors had an almost rectangular cross-section of 7.36 mm<sup>2</sup>, an occluso-gingival height of 3.2 mm, and a bucco-lingual width of 2.3 mm - was used to produce the Bio HPP framework using CAD/CAM system following the manufacturer's instructions to ensure standardization<sup>12</sup>.

**Stawarczyk et al**, assessed the influence of different fabrication methods of three-unit PEEK FDPs on fracture load. The milled CAD/CAM FDPs showed a mean fracture resistance of (2,354±422 N)<sup>1</sup>. An increased connector surface area would have probably additionally increased the fracture resistance<sup>36</sup>.

The connector represents a relatively thin section of an irregularly shaped construction and will bend more easily than the thicker sections, such as in the pontic and abutment areas. During loading, the connector will reach its critical strain before the thicker portions. To ensure optimum strength, the FPD framework, in particular the connectors, must be of adequate dimensions<sup>37</sup>.

In other study, Five different connector dimensions were used ranging from 2.0 to 4.0mm<sup>38</sup>. As the connector is a comparatively thin section of the FPD, it will bend more easily than the pontic and abutment areas and will reach its critical strain before the thicker sections. Increasing the dimensions of the connector would have a beneficial effect on the strength of an all-ceramic reconstruction because stress concentrations would be reduced in the critical section of the beam. Even a small increase in the dimensions of the connector can have a substantial effect on strength<sup>39</sup>.

**Gotzen et al**, found that when the connector height increased from 3 to 4 mm, stress would decrease by 50%, thus increasing the flexural strength<sup>38,40</sup>.

Connector diameters below 3.0mm are insufficient, especially for FPDs that replace molars like the pontic in the present study. In such cases range from 2.0 - 4.0mm could be sufficient. In cases where excessive forces are expected - as in patients with a deep overbite, patients who brux, or patients who have experienced fractured reconstructions, it may be necessary to use dimensions >4.0mm to avoid all-ceramic FPDs failure. For shorter FPDs as well as anterior ones, however, smaller dimensions might be adequate<sup>41</sup>.

In general, the industrially pre-pressed and consequently milled CAD/CAM FDPs showed higher fracture loads than those pressed from granular PEEK. It can therefore be assumed that the industrial pre-pressing process for the CAD/CAM blanks and for the pellets increases the mechanical properties. Additionally, CAD/CAM blanks, after industrial fabrication under optimal conditions display a reduced risk of porosities within the restorations and therefore show improved mechanical properties<sup>1,42</sup>.

It can be supposed that an additional industrial pre-pressing of PEEK blanks/pellets does not only increase the flexural strength of the material, but also reduces its elastic deformability. In spite of its relatively rigid molecular chain structure, thermoplastic PEEK material demonstrated considerable ductility and can accommodate a wide range of plastic deformation, in both uniaxial tension and compression<sup>1,42</sup>.

**Arslan et al**, showed that The Flexural Strength and hydrophobicity of the CAD/CAM PMMA-based polymers were higher than the conventional heat-polymerized PMMA<sup>43</sup>.

Flexural strength is defined as materials ability to resist deformation under load. Also it is a combination of tensile strength, compressive strength, and shear strength. It is measured as the highest stress experienced within the material at its moment of rupture. Strength is given as a general mechanical term, but what we are really measuring are stresses within the resin. In the three-point bending analysis, the samples underwent compression forces on the occlusal side and tension forces on the gingival side<sup>44</sup>.

In this vitro study axial forces were applied to the center of the occlusal pontic area. Clinically, axial forces in addition to lateral forces and fatigue loading on short span FPDs should be considered. These may have an additional effect on the mechanical properties of FPDs<sup>44</sup>.

There are several ways in cementation techniques such as uncontrolled finger pressure or overfilling of the crown with cement can cause uneven flow of cement with one axial wall having a thick film and the opposite wall having a thin film<sup>17</sup>.

In our study cementation was performed in a controlled manner under a standard load of three pounds by the cementing device in order to ensure even flow of cement on the axial walls of the specimens and simulates the amount of forces generated by the jaw on the restorations intra-orally<sup>45</sup>.

A constant seating force was applied in order to prevent rebounding of the cemented restorations thus, obtaining uniformly distributed cement layer over the epoxy resin dies<sup>46</sup>.

The "specially designed holding device" was used by several investigators with some modifications<sup>23,47,48</sup>. These standardizations helps in fair comparison between different techniques of surface treatment<sup>49</sup>.

In our study, bonding of the Bio-HPP frameworks were done by using a self-etch dual cure resin cement to simulate the clinical situation<sup>50</sup>. Panavia F2.0 as recommended by the manufacture for the used materials as it contains adhesive monomer MDP in the primers.

The evidence that supports the use of adhesive monomers containing methacryloyloxy decyl dihydrogen phosphate (MDP) is that it provides strong chemical bonds to hydroxyapatite and metal oxides which resulted in long-term durable resin bond to high-strength ceramic materials and ensures a reliable adhesion to both enamel and dentin<sup>51,52</sup>.

**Piowarczyk et al.** concluded that cements containing phosphoric-acid methacrylates (in their study RelyX Unicem and Panavia F) provide a strong physical interaction, such as hydrogen bonding, with the airborne particle-abraded surface and eventually copolymerized with the industrially polymerized CAD/CAM resin. SO, bond strengths were higher than other cementing agents<sup>53</sup>.

**Stawarczyk et al.**, proved that All tested cements showed no bonding when polymeric crowns were untreated. This phenomenon can be explained by the fact that free radicals were not sufficient to achieve adhesion between the studied cements and the untreated surfaces of the crowns. While, Pretreatment with alumina increased tensile strength results<sup>54</sup>.

**Uhrenbacher et al.**, Crowns that were untreated had the lowest retention strength. The adhesion of the tested PEEK crowns to dentin was satisfactory after treatment with airborne-particle abrasion or etching with sulfuric acid and when additional adhesive systems<sup>55</sup>.

The result of the present study reported that BioHPP Sulfuric acid group had the highest value of flexural strength followed by airborne particle-abrasive group, While BioHPP controlled group without surface treatment showed the lowest value of flexural strength. Several factors could be considered to explain this result.

Firstly, Adhesively cemented restorations usually show higher flexural strength and survival rates<sup>56</sup>. The improvement of mechanical properties was attributed to the high interfacial shear strength between the filler and resin matrix which fills the irregularities and defects of the pretreated surface.

Complete wetting of the fillers by resin prevent propagation of crack and improving the mechanical behavior which in turn lead to increase the fracture resistance as volume of filler increased<sup>47,57,58</sup>.

This mechanical interlocking cannot found in The BioHPP controlled group which had the lowest mean values of flexural strength in this current study. This is in agreement with **Stawarczyk**, who reported that groups without pretreatment showed decreased fracture loads of 737 N than other groups<sup>59</sup>.

This is supported by SEM observations. It can be seen that the control group with no treatment showed a basically regular, smooth and homogenous surface.

Secondly, Previous studies reported a significant increase in fracture resistance values after sandblasting compared to non-treated PEEK due to variation of surface morphology. In agreement with these results, in the current study, the specimens sandblasted with 110  $\mu\text{m}$  Al<sub>2</sub>O<sub>3</sub> showed significantly higher flexural resistance than the controlled group<sup>60,61,62,63</sup>.

This is due to increase the irregularities of the bonding area and increases surface roughness to clean and activate the surface by thoroughly removing organic contaminants from the composite material and create micro-mechanical interlocks with the luting cement hence improving biological activity of the material<sup>64,65</sup>.

SEM images verified this view that showed an irregular fissure pattern with larger grooves which is more suitable for the flow of both adhesive and resin cement.

**Stawarczyk et al** reported visio.link (Bredent) as an ideal adhesive system to increase the bond strength to PEEK surface and recommended sandblasting as one of the best initial pretreatment options for PEEK surfaces. Thus, that support the finding of the current study<sup>54</sup>. This also in agreement with **Hallmann et al**, who proved that<sup>62</sup>.

Based on these results, the adhesion between the polymeric framework and the resin cements could be considered as mechanical retention<sup>65,66</sup>.

Some studies have shown that the strength of ceramic materials decreases with the use of large abrasive particle size, but the strength of BioHPP in this experiment does not decrease significantly with the increase of sandblasting particle size<sup>54,63</sup>.

**Garcia Fonseca et al.** showed that air abrasion in Y-TZP creates surface microcracks around which the grains exhibit a volumetric increase resulting from the tetragonal to monoclinic phase transformation. This

outward expansion due to a plastic deformation of the surrounding zirconia provides compressive stresses that counteract the crack propagation. This process, known as transformation toughening, may increase the bulk strength of zirconia<sup>67,68,69</sup>.

**Çulhaoğlu et al**, Highest mean shear bond strengths were observed for 98% sulfuric acid-etched PEEK surfaces followed by airborne particle abraded and untreated PEEK surfaces<sup>70</sup>.

Regarding sulfuric group which recorded the highest flexural strength mean values this may be due to the carbonyl and ether groups of peek were attacked from sulfuric acid and the atomic oxygen released during the this reaction, reacted with benzene ring. This leads to the oxidation of PEEK polymer, the increase of surface polarity, the opening of aromatic ring and subsequently more functional groups are established, which are able to react with adhesive<sup>71</sup>.

**Zhou et al**, proved that The mechanical bonds, which depend on the morphology of the pretreated surface, are in addition an important factor on the tensile bond strength. The reaction between functional groups of etched PEEK and resin cement enhance the material strength. Additionally improved the diffusion of adhesive inside the polymer, increase the micro-roughness and the contact surface between PEEK and adhesive resulting in enhances the mechanical interlocking and improvement of the bond strength<sup>61,71</sup>.

The fact that the H atom on the plastic polymer chain may be replaced by sulfate does not cause weight loss, but some properties of the plastic change. The reaction in which this H atom is replaced by -SO<sub>3</sub>H is called a sulfonation reaction, and it has been experimentally confirmed that a sulfonation reaction does occur on the surface of PEEK etched by concentrated sulfuric acid<sup>71</sup>.

As shown in SEM observations. Sponge-like porous and complex fiber networks were displayed through the surfaces etched with 98% concentration of sulfuric acid. Differences in irregularity of PEEK surfaces after sulfuric acid etching were due to the dissolution of the PEEK matrix by sulfonation reaction which improve hydrophilicity of the surface by introducing sulfonic acid group (-SO<sub>3</sub>) into the polymer chains of PEEK.

The application of 98% sulfuric acid etching for 60 s in this study showed higher flexural strength due to the micro-topographical changes of PEEK surface after treated with the acid which enhance the penetration of micro-retentive resin tags into the etched PEEK surface.

These resin tag penetrations confirmed the existence of micromechanical interlocking. High concentration of sulfuric acid promoted deeper and more evident pits and porous surface for bonding, consequently, higher resin tag length and the Shear Bond Strength increased<sup>71,72,73</sup>.

**Silthampitag et al**, supported that surface topography affected the adhesion due to mechanical bonding. The complex fiber network with porosities of etched PEEK surface with 98% sulfuric acid attributed to the higher Shear Bond Strength than the other groups<sup>72</sup>.

The sulfonation reaction is an important step in improving the bonding performance of PEEK, but its mechanical properties are reduced by sulfonation. The degree of sulfonation of PEEK varies depending on the concentration of the acid, the reaction time, and the reaction temperature<sup>55,74,75</sup>.

**He Guangji , Zhang Wenyun** showed that BioHPP mechanical properties are reduced by sulfuric acid etching and airborne abrasion with 110 μm Al<sub>2</sub>O<sub>3</sub> resulting in significantly decrease in its the flexural strength<sup>76</sup>.

The difference in the results obtained in the current study can be attributed to a difference in methodology between our current study and the previous study.

From a methodological point of view, a shortcoming of the previous study is the lack of cementation with resin cement, that explain the rule of improvement the diffusion of resin into the polymer matrix and failure, leading to complete wetting of the fillers by resin, preventing propagation of crack and improving the mechanical behaviours<sup>56,57,58</sup>.

**Rocha et al**, showed that traditional methods of surface treatment such as sandblasting with aluminum oxide are safe and well established in prosthetic laboratories and offices, we should used it instead of sulfuric acid. Both physical and chemical surface treatments were effective in promoting similar results<sup>77</sup>.

At the end, the results of the current study accept in part the first null hypothesis as showed with **Uhrenbacher, J., Schmidlin**, airborne-particle abrasion and sulfuric acid etching of PEEK crowns or bridges should be recommended before luting PEEK crowns which had a significant effect on flexural and fracture resistance for all pretreated groups and increasing its mechanical properties<sup>55,60</sup>.

Regarding the second null hypothesis, the result of the current study rejected it. That surface treatment of peek reduce the strength of the restoration and shorten the service life. While, this current study revealed that chemical and mechanical surface treatment and resin cement showed a significant advantage in term of fracture strength.



## V. Conclusion

Based on the results and within the limitation of this study –including no aging the following conclusions were made:

- Bio HPP framework treated with sulfuric group showed significant higher flexural strength values than Bio HPP framework treated with Aluminium oxide 110 µm while untreated framework showed the lowest mean values.
- Bio HPP can be potentially used as crown and bridge material even in posterior area.
- Fracture load resistance of PEEK as a framework seems sufficient for clinical application.

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