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Evaluation of the Effect of Different Surface Treatments on Bond Strength of Heat Cured Acrylic Resin at Co-Cr Alloy and PEEK Polymer Interface

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ABSTRACT

Problem statement: *A significant clinical concern in Co-Cr alloy removable prosthodontics is a failure of bonding with acrylic resins and PEEK is introduced on various materials to overcome this problem. The reason for this study was to examine the denture base acrylic resin bond strength to Co-Cr alloy and PEEK polymer using different surface treatment.* ***Materials and method:*** *Total 60 number of disc-shaped specimens were prepared and divided into 2 main groups according to the type of framework material, 30 specimens for Co-Cr alloy and 30 specimens for PEEK polymer, which were then subdivided into 3 groups each one consist of 10 specimens, according to the type of surface treatments. Each group either had a no surface treatment (control group) or air abrasive group (110 μm of alumina oxide) or acid etch (98% sulfuric acid) group. The specimens were thermocycled (3000 cycles) after applications of heat cured acrylic resin.* ***Results:*** *The results for Co-Cr alloy showed that the air abrasive surface treatments had the highest bond strength mean value followed by the control group (no surface treatment), while the acid surface treatment had the least mean value of bond strength. For PEEK polymer, the results revealed that the acid surface treatment had the highest value of bond strength followed by the air abrasive surface treatment while the control group (no surface treatment) had the least bond strength mean value.* ***Conclusion:*** *Air abrasive surface treatment significantly improved bond strength for both Co-Cr and PEEK, while sulfuric acid treatment show counterproductive result with Co-Cr alloy whereas with PEEK play an important role in bonding mechanism. The use of PEEK framework in combination with heat cured acrylic resin can be recommended as a promising non-metallic alternatives framework material for removable of partial dentures.*

Keywords: Co-Cr alloy, PEEK polymer, Bond strength, Surface treatment

INTRODUCTION

Metal like Co-Cr alloy was commonly used for fabrication of removable partial denture framework because of their favorable mechanical properties. The use of other metal-free materials has been investigated to improve the patient's desire because of the metal-based frameworks drawbacks like the esthetically unsatisfactory appearance of metal clasps, the expanded prosthesis weight and the potential for metallic taste and potential hypersensitivity. The bonding between the acrylic resin and the metal components also plays an important role in the longevity and is essential for the prosthesis success; deficiencies in bonding at the metal-resin interface can become a significant clinical problem [1,2].

Alternatively, a PEEK polymer seems to be a promising material for the removable dentures frameworks because of its high-temperature resistance, sufficient mechanical properties, high biocompatibility and chemical stability [3]. The modulus of elasticity about 4 GPa makes PEEK as bone elasticity, and also PEEK can reduce stresses transferred to the abutment teeth, and other advantages for this polymer material eliminate the allergic reactions and metallic taste, low plaque affinity, high polishing qualities, and good wear resistance [4,5].

The polyether ketone aromatic polymer is an interesting material for dental prostheses applications that can in principle be formed by mill process with CAD/CAM systems like Juvora dental disc (Juvora, UK) or with thermo pressing procedures like BioHPP (Bredent) and final adjustments were accomplished by using dental burs standardized for resin materials [6].

In removable prosthodontics failures attributed to bonding between the metal and acrylic is a common procedure for improving the bonding focused on using different surface treatments. Metal surface treatments can produce retentive aids that were classified into micromechanical retention, chemical bonding [7,8].

The special chemical aromatic structure with ketone provides an inert and low energy surface of PEEK polymer, therefore, surface conditioning like surface roughening was needed for successful bonding on PEEK surface, existing dental methods which were already available for metal or ceramic be adapted to PEEK [9,10].

Nonetheless, the bonding properties of denture base resin to PEEK polymer have not been clearly confirmed. No information is available on the bonding behavior of heat-polymerized resin, which is commonly used for removable prostheses to PEEK polymer.

The hypothesis of the study was that heat polymerized resins exhibit different bonding behavior according to the chemical and physical properties of framework material and surface treatment. The purpose of this study was to evaluate the bonding efficiency between heat-activated denture base resin to Co-Cr alloy and PEEK polymer focused on the different surface treatments.

PATIENTS AND METHODS

Preparation and Casting of the Metal Specimens

For the 30 Co-Cr specimens, a metal mold was designed to reproduce a wax pattern in a disc shape with a dimension of 2 mm thickness, and 10 mm diameter and then cast with Co-Cr alloy (Super6, U.S.A.) using a phosphate bonded investment (BEGO, Germany) and centrifugal casting technique, following the manufacturer's instructions [11]. The surfaces of specimens were ground finished using silicon carbide papers (No. 600 grit) (Trojan, China) in the grinding machine (160E, Mapao, China) under running water on a 300 rpm for 10 seconds in order to provide uniform and flat surface and cleaned ultrasonically for 3 minutes with deionized water and dried with air [12].

Preparation and Casting of the Peek Specimens

For 30 PEEK specimens preparation, the procedure used a Co-Cr alloy specimen as a metal analog to be scanned by using a fully automated laboratory strip optical light scanner (Zirkonzahn S600 ARTI scanner, Italy) [13], then digitalized by using software (Zirkonzahn Modelier and Nesting; Zirkonzahn, Italy) to create a 3D CAD model with dimension of 2 mm thickness, 10 mm diameter and positioning the 30 PEEK disk shape virtual specimens in the virtual blank. One blank of dental PEEK (JUVORATM Dental Disc, UK) was milled under cooling air using a milling unit (M5 Zirkonzahn, Italy) following the manufacturer's instructions. Each of the specimens was measured and examined twice in 3 different locations with an electronic digital caliper (Powerfix, UK). All specimens were polished with silicon carbide paper (No.600 grit) (Trojan, China) in the grinding machine (160E, Mapao, China) under rinsing water for 10 seconds on a 300 rpm in order to provide a standard and uniform surface, PEEK specimens were then ultrasonically cleaned for 3 minutes and then dried with air [14].

Application of Heat Cured Acrylic Resin

After obtaining 30 disk shape specimens for both Co-Cr alloy and PEEK polymer, a special metal split mold was used for all disk specimens to add modeling wax (Cavex, Holland) in a specific area placed centrally with dimensions of 5 mm diameter and 2 mm thickness [11]. These specimens (Co-Cr alloy/PEEK polymer disk modeling wax assemblies) were flasked in a standard flasking technique for acrylic dentures with dental stone (Easydental, Bulgaria), and then the specimens were dewaxed and cleaned using boiled water [15].

Before packing, the specimens of Co-Cr and PEEK were divided into 3 groups according to the surface treatment that they received (n=10 for each group):

- Control groups: Without surface treatment
- Air abrasive groups: Abraded with alumina oxide 110 µm particle size (Renfert, Germany) with an airborne particle abrasive unit (Mestra, Germany) at 2 bar pressure for 15 seconds with 10 mm of distance that was standardized by using a specially designed holder and then ultrasonically cleaned (Mestra, Germany) with deionized water for 10 minutes and dried by air [11,16-18]
- Acid etch groups: Treated with the application of sulfuric acid 98% (Central Drug House, India) for 1 minute after rinsed with the deionized water for 30 seconds, then the specimens were air dried for 10 seconds [16,19]

The heat cured acrylic resin (SpofaDental, Czech) was mixed with powder: liquid ratio of 3:1 and was packed by placing the flask in a hydraulic press (Quayle Dental, UK), and 5 MPa pressure was applied slowly [20]. All the specimens were heat processed according to the manufacturer's instructions, and then the flasks were cooled slowly at room temperature [21]. After deflasking, all the specimens were immersed for 24 hours in 37°C water. The 60 specimens of all groups for each Co-Cr alloy and PEEK polymer were subjected to 3,000 thermocycles in water between 5°C and 55°C with a dwell time of 1 minute using a thermocycling system (Cooler: HETO, Danmark; Herter: Wincom, china; holder) [17].

Characterizations

Scanning electron microscopy (SEM) observations: Three additional specimens per each group were prepared for observing the surface morphology after surface treatments and before acrylic resin application under a scanning electron microscope (SEM) (S50, FEI Company, Netherlands). The SEM photomicrographs was done under 1000X magnification [20,22].

X-ray diffraction analysis: The phase components and elements were identify by using a diffractometer X-ray (XRD-6000, Shimadzu, Japan) for the Co-Cr alloy specimens before and after sulfuric acid surface treatment, and for PEEK specimens before and after sulfuric acid surface treatment and additionally for PEEKs acid treated specimen after acrylic resin application.

Fourier transforms infrared spectroscopy (FTIR): The information of changes in the chemical surface structure for PEEK specimens before and after sulfuric acid surface treatment and for PEEKs acid treated specimen after acrylic resin application were assessed by Fourier transform infrared (FTIR8400S) spectroscopy (Shimadzu, Japan). The measurements was performed from 400 cm^{-1} to 4000 cm^{-1} in the transmission mode.

Bond Strength Test and Failure Analysis

The specimens were embedded in the middle of self-polymerizing acrylic resin blocks by using a special silicon mold in such a way that only the heat acrylic resin portion can be exposed. To obtain the bond strength the specimens were mounted in the specimen holder of universal testing machine in such way that the treated specimen's surface was parallel to the loading piston, and then the load was applied with a crosshead speed of 0.5 mm/min [11,23]. The load at failure was recorded in (N) and dividing by the bonded surface area (mm^2) to obtain the bond strength in (MPa) [16,24]. After the test, the type of failure was defined as adhesive, cohesive, and mixed. The specimens were examined under a stereomicroscope with 20X magnification images of the fracture site to verify the type of failure [19,25].

Statistical Analysis

The Kolmogrov Smirnov test was utilized to confirm that bond strength data were normally distributed; statistical analysis with one-way (ANOVA) tests was used. Games Howell test was used to determine the pair differences.

RESULTS

SEM Observations

SEM observations showed the Co-Cr specimens of the control group (without surface treatment) a uniform surface, and the air abrasive group displayed a clearly rough surface and appearance of micro porous and irregularities. For the acid etch Co-Cr group the SEM demonstrated nearly but not quite similar superficial appearance to the Co-Cr air abrasive group due to the formation of micro porosities. On the other hand, the SEM observation for PEEK specimen of control group revealed a plain surface with irregular striations. Air abrasive PEEK group exhibited pronounced irregularities and a rough texture by peaks and valleys while the acid etches PEEK group displayed a sponge like and complex fiber network surface with evident pits and porous (Figure 1).

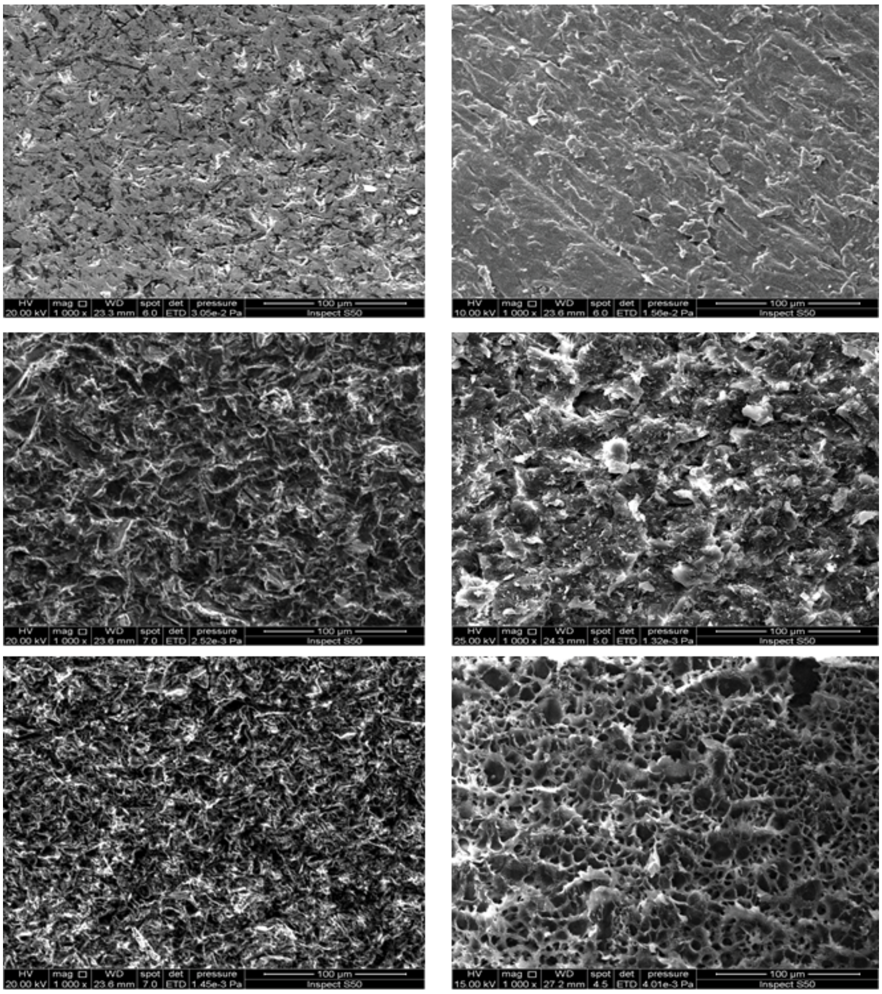

Figure 1 SEM images of the different surface treatment modalities at a magnification of 1000X: a: Co-Cr control; b: Co-Cr air abrasive; c: Co-Cr acids etch; d: PEEK control; e: PEEK air abrasive; f: PEEK acids etch

X-ray Diffraction Analysis

The X-ray patterns of Co-Cr specimen without surface treatment and with sulfuric acid surface treatment are shown in Figure 2 revealed the diffraction characteristic peaks of the Co-Cr alloy without treatment at 2θ found corresponding intensities of (012), (111), (022), (024), (023), (004), (226) and (113) respectively, while after sulfuric acid surface treatment show the formation of (030), (042) peak indexed to chromium sulfate ($CrSO_4.5H_2O$) at 2θ is of 38° and the peak indexed to cobalt sulfate ($CoSO_4.H_2O$) at 2θ is 61.9°, also the pattern showed decrease in the intensity. It is evident from the XRD pattern that the surface of the specimen is well covered with sulfates (salts) as results of acid etch treatment.

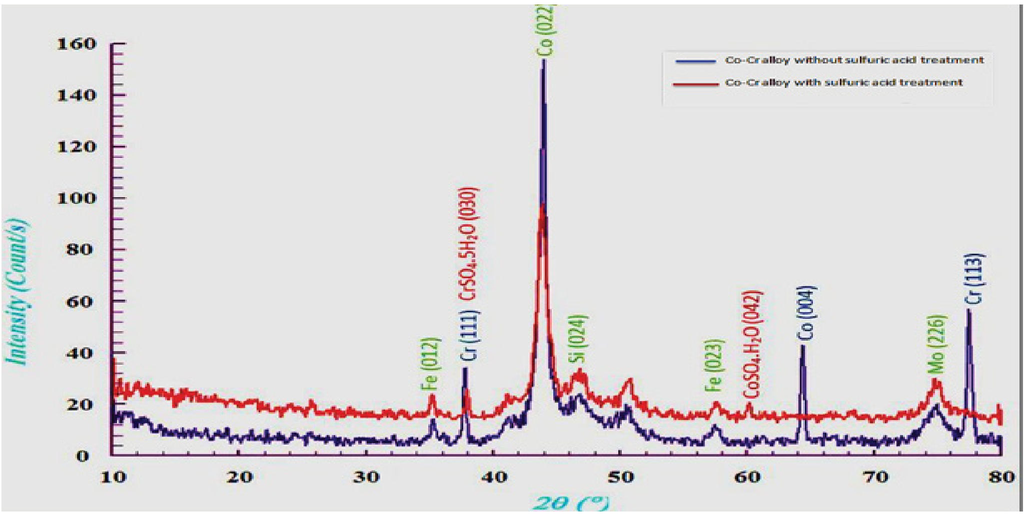

Figure 2 XRD analysis for Co-Cr specimen without and with a sulfuric acid surface treatment

The XRD patterns are shown in Figure 3; it represents the peaks of PEEK polymer without and with sulfuric acid surface treatment, and after application of heat cured the acrylic resin. The characteristic peaks of untreated PEEK polymer at 2θ with intensities of (110), (111), (200), (211), (202), (210), and (102) respectively. The patterns of PEEK polymer with acid surface treatment, and after acrylic resin application shows the characteristic peaks with decreased in intensity, which mean more notably physicochemical modifications, have occurred.

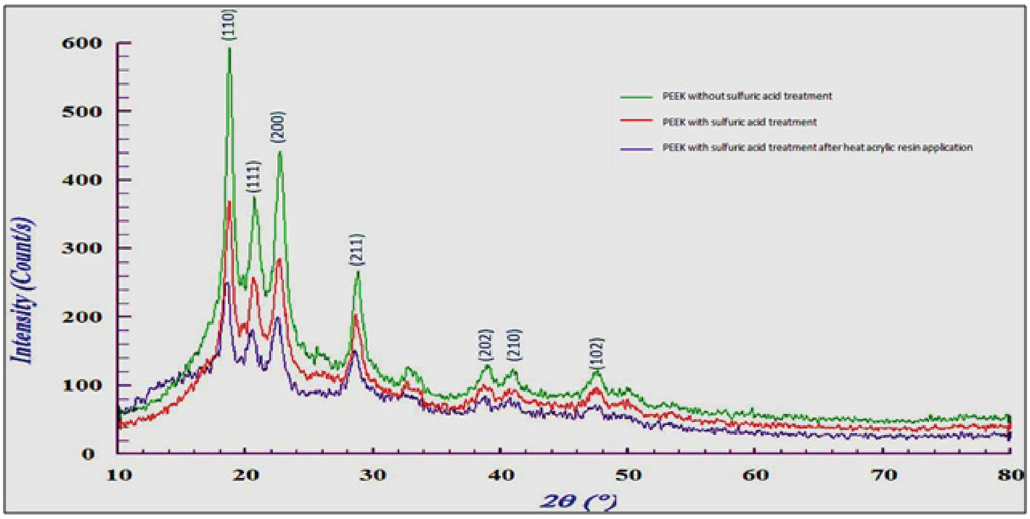

Figure 3 XRD analysis for PEEK specimen without and with sulfuric acid surface treatment and after heat acrylic resin application

Fourier Transforms Infrared Spectroscopy (FTIR)

All various functional groups present are studied through FTIR analysis, the characteristic bands of PEEK polymer main chain are present in the all 3 groups which are the peaks at 3063.06 cm^{-1}, 1593.25cm^{-1}, 1675 cm^{-1}, 1230.63 $cm^{-1,}$ and 1105.25 cm^{-1}. The PEEK specimen after H_2SO_4 treatment revealed new promising peaks at 3412.19 cm^{-1} and 1492.95 cm^{-1} that represent the functional groups that can participate in specific chemical reactions. The FTIR analysis after acrylic resin application detects a new peak around 1728.28cm^{-1} that attributed to the characteristic peak of heat cured acrylic resin, the peak at 1653.05cm^{-1} was clearly indicated to existence for chemical bonding of heat cured acrylic resin to the PEEK polymer surface (Figure 4).

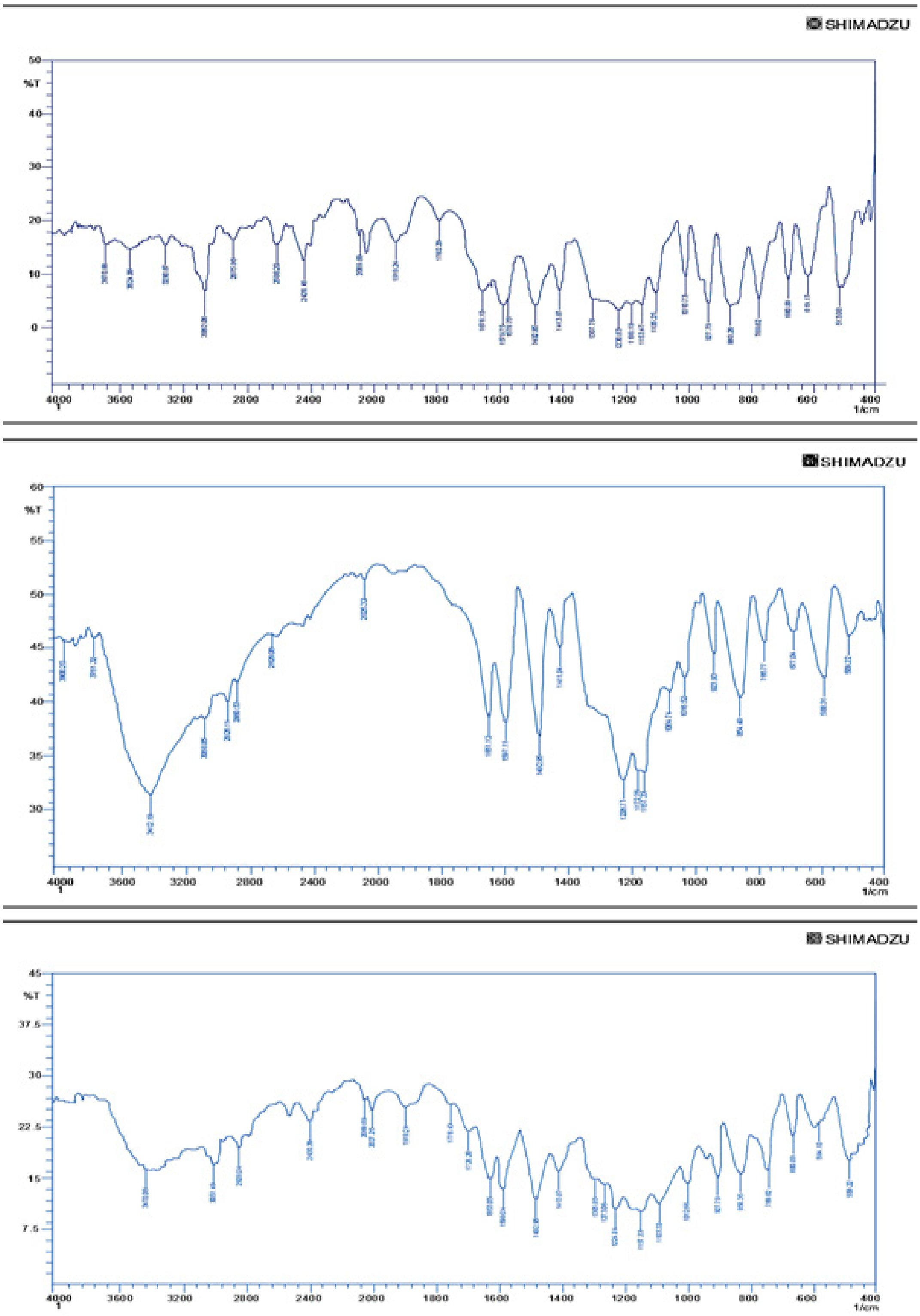

Figure 4 FTIR spectrum of PEEK specimen; a: without acid treatment; b: with acid treatment; c: after heat acrylic resin application

Bond Strength and Failure Type

The bond strength for all tested groups is graphically presented in Figure 5, the results indicate that the mean bond was maximum for PEEK acid etch treatment group (16.450 ± 2.145) and minimum for Co-Cr acid etch treatment group (1.875 ± 0.391). According to the results of one-way ANOVA tests as shown the difference was statistically highly significant among all groups at $p<0.01$ (Table 1).

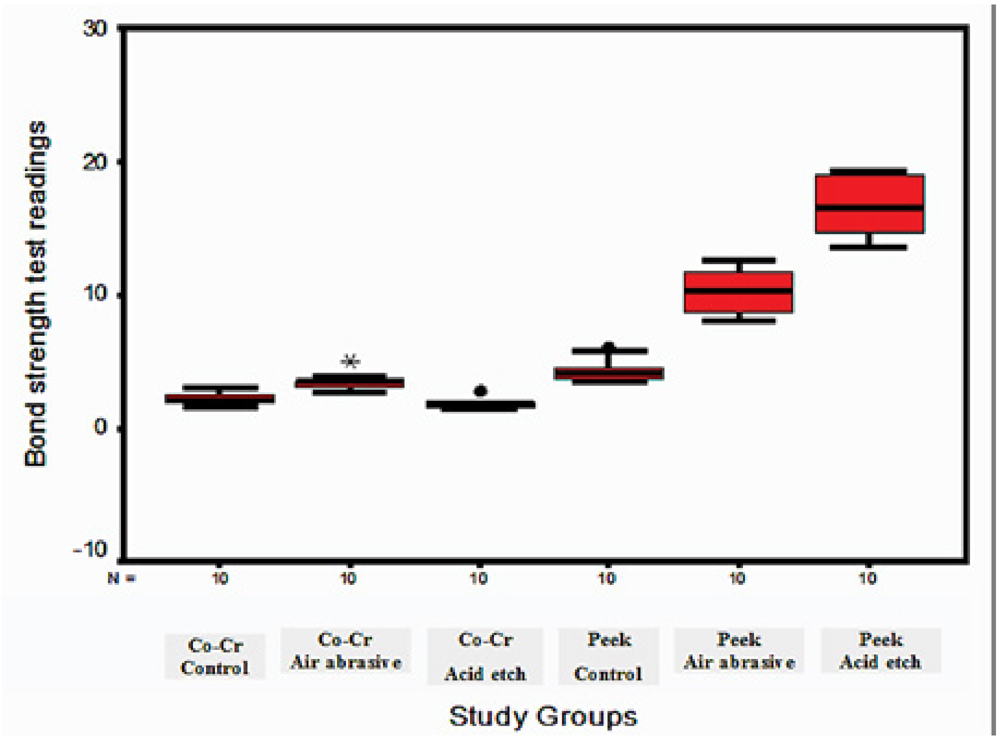

Figure 5 Box plot for bond strength tested groups

Table 1 Descriptive analysis statistics with Levene's and one-way ANOVA tests

Groups	No.	Materials		ANOVA Difference test	
		Co-Cr alloy	Peek polymer	Levene's Test	F-test
		Mean ± SD	Mean ± SD		
Control	10	2.201 ± 0.417	4.361 ± 0.865	p=0.000 HS	p=0.000 HS
Air abrasive	10	3.485 ± 0.626	10.330 ± 1.602		
Acid etch	10	1.875 ± 0.391	16.450 ± 2.145		
*HS: Highly significant at p<0.01					

On applying Games Howell test for multiple pair wise comparisons between tested groups in Table 2, the differences showed a statistically high significance at $p<0.01$ and no significant differences at $p>0.05$.

Table 2 Games Howell Difference Test for multiple comparisons

Groups	Co-Co control	Co-Co air abrasive	Co-Cr acid etch	Peek control	Peek air abrasive	Peek acid etch
Co-Co control	-	H.S	N. S	H .S	H .S	H .S
Co-Co air abrasive	-	-	H .S	N. S	H .S	H .S
Co-Cr acid etch	-	-	-	H .S	H .S	H .S
Peek control	-	-	-	-	H .S	H .S
Peek air abrasive	-	-	-	-	-	H .S
Peek acid etch	-	-	-	-	-	-
*HS: Highly Sig. at p<0.01; NS: Non Sig. at p>0.05						

All groups in this study exhibit predominantly adhesive failure, however, a mixed failure were mostly observed in acid etch group of PEEK polymer, stereomicroscope micrographs are displayed in Figure 6.

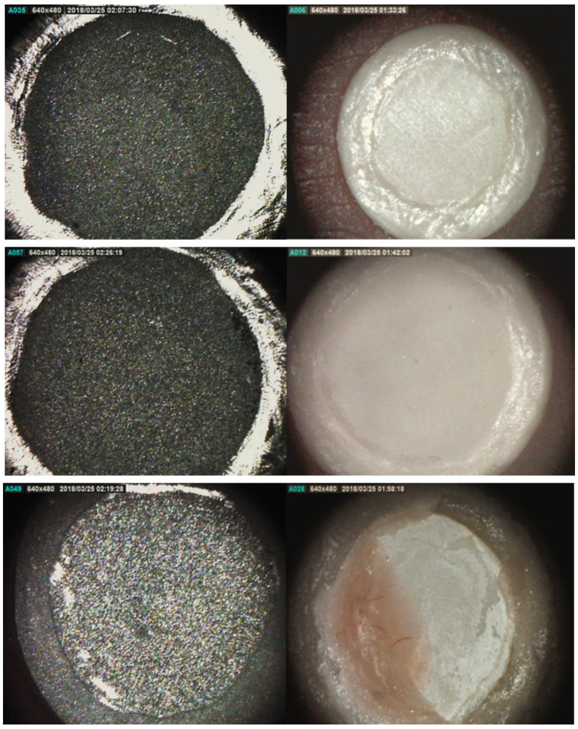

Figure 6 Failure mode images of Co-Cr alloy PEEK specimens; a: control groups; b: air abrasive groups; c: acid etches groups

DISCUSSION

The deficiencies in bonding between acrylic resin and metal, increased the weight of the prostheses and allergic reactions become a significant clinical issue leading to failure of the removable partial denture prosthesis [26]. The older framework materials such as Co-Cr alloy had been replaced by the improved ones which have better properties to overcome these disadvantages, due to the superior biological and mechanical properties of PEEK polymer when compared to Co-Cr frameworks its use has been extended as an alternative for the conventional framework material [5].

Air Abrasive Surface Treatment

The surface treatment by airborne particle abrasion was recommended methods to creates a suitable surface condition to improve the bond strength, this process simultaneously removes the contaminated layer, debris and/or metal oxides and achieved an increase of the surface area by producing micromechanical roughness. Furthermore, this treatment improves the material wettability surface [20,26-28].

The airborne particle abrasion surface treatment improve surface morphology variation [29,30], this can be observed in SEM image of Co-Cr specimen with air abrasive surface treatment that revealed an appearance of microroughness, irregular surface when compared with SEM image of Co-Cr specimen without surface treatment indicates a uniform surface, however the SEM image of PEEK polymer with air abrasive surface treatment show a flaws, streaks and irregularities with accentuated and dispersed surface pattern while SEM image of PEEK specimen without surface treatment reveals generally a plain surface with little irregular striations, this SEM finding agrees with previous studies that show the surface morphology alteration was obtained after sandblasting can significantly increase the bond strength values when compared with untreated specimens [11,20,31-33].

In this study the air abrasive group of Co-Cr alloy and PEEK polymer with 110 µm Al_2O_3 particle size exhibit a significantly higher bond strength than the specimens without surface treatment, this is due to the variation of surface morphology that advances a micromechanical interlocking sites as well as greater wettability, which allows the heat cured acrylic resin be mechanically joined and increase the mechanical bonding.

It is possible that the highly significant difference between Co-Cr alloy and PEEK polymer influences on the effect of bonding by airborne particle abrasion due to the difference in mechanical, physical and thermal properties between Co-Cr alloy and PEEK polymer.

Sandblasting influences on surface roughness, the higher surface roughness is generally considered beneficial for improving the bond strength due to increase the surface area and enhance the mechanical retention [27]. It has been reported that the bond strength of a resins to a base metal alloy was influenced by the size of aluminum oxide particles and they conclude that using 250 µm Al_2O_3 particles result in a significant increase in the roughness of the base metal alloy surface [34], while according to Stawarczyk, et al., the grain size of the air abrasion powder particle did not show an impact on the bond strength to PEEK polymer [28].

Furthermore, the improvement of material surface wettability by air abrasive surface treatment may not be the same on Co-Cr alloy and PEEK polymer, surface wettability depends on the contact angle (smaller contact angle indicates better wettability) and affected by surface morphology, which in turn suggests stronger bonding of the material [35]. The difference in surface contact angle value may be the effect on the influence of air abrasive surface treatment; the finding of the SEM images in that revealed different morphological features between Co-Cr alloy and PEEK polymer can support this.

Thermoplastic resins such as PEEK have a modulus of elasticity of 4 GPa very similar to cortical bone 18 GPa while Cr-Co has a much higher modulus of elasticity 230 GPa; due to the low modulus of elasticity PEEK polymer have a higher flexibility compared to the Co-Cr alloy 211 GPa [36], this could be possible explanation of difference in Co-Cr and PEEK behavior against the stress that is created due to heat cured acrylic polymerization shrinkage by thermocycling effect.

Acid Etched Surface Treatment

Chemical etching with acids such as H_2SO_4 is a conventional and an effective performed way for surface treatment of different materials with considerations of the governing processes parameters such as acid etch concentration, time and temperature. Chemical etching with acids can create surface irregularities to increased mechanical bonding of materials [37-39]. Anhydrous H_2SO_4 is a very polar liquid and a highly corrosive solution which is needed for dental PEEK surface treatment because PEEK is an apolar and inert polymer with high chemical resistance and low surface energy [40]. Sulfuric acid causes a swelling process on PEEK surface and produces porosity which could act as an anchorage site that is penetrated by bonded materials [16].

The SEM image of Co-Cr specimen with acid etch surface treatment revealed a surface morphology with microporous structure, whereas our results showed the lowest bond strength value in Co-Cr specimens conditioned with sulfuric acid, however the XRD analysis results clearly revealed the presence of a cobalt sulfate ($CoSO_4.H_2O$) and chromium sulfate ($CrSO_4.5H_2O$) along Co-Cr specimen surface after sulfuric acid treatment caused by reaction of sulfuric acid with Co and Cr elements of alloy. These sulfates (salts) act as a separative layer between Co-Cr metal and heat cured acrylic resin which probably explained the poor bond strength for this group.

The SEM images for PEEK specimen treated with sulfuric acid display numerous round cavities blister like micropours over the entire surface producing a complex network (Figure 1). This finding is consistent with the SEM observation of Stawarczyk, et al., who observed round cavities and a different surface topography created after etching PEEK specimen with sulfuric acid [19].

In the current research, the characteristic peaks found XRD spectrum for PEEK polymer in agreement with studies published previously [41,42] (Figure 3). The X-ray pattern revealed a decrease in intensity for the diffraction peaks of PEEK specimen after sulfuric acid treatment, this could be attributed to the sulfuric acid effect that creates remarkable physicochemical alterations on PEEK surface, the physical alterations can be supported by SEM finding of PEEK specimen with acid etch treatment and the chemical alterations can be explain by alternation of the chemical characteristics of the PEEK structure by attacking the functional carbonyl and/or ether groups between the benzene rings of PEEK polymer thus lead to increase in the number of available functional groups on PEEK surface, as a result PEEK surface polarity is obtained and enhance its reactivity [43,44], also the X-ray pattern show considerable decrease in intensity of diffraction peaks with presence of new characteristic peak of the acrylic resin in an amorphous region that could be a confirmation of acrylic resin linking. Moreover, the fact that characteristic peaks of PEEK are still present in PEEK specimen without teatment, after acid treatment, and after acrylic resin application suggest that PEEK'S backbone is not broken.

FTIR analysis in Figure 4 is used to identify the chemical mechanism of sulfuric acid with PEEK polymer specimen, also to observe the interaction between heat cured acrylic resin and PEEK specimen treated with sulfuric acid (Figure 4). The characteristic bands of PEEK polymer main chain were indicated in the 3 groups, while the introduction of new bands (polar functional groups) revealed after sulfuric acid treatment for PEEK specimen. The chemical mechanism started when H_2SO_4 acid open up the double bond in keton (carbonyl) group and produce a carbocation ion within the PEEK specimen surface which grants the surface polarity. This carbocation positive ion reacts with the double bond in the methyl methacrylate monomers of heat cured the acrylic resin and produce a cross-linked polymer, this bonding is represented by (C=C) stretching bond that is revealed in FTIR analysis and explains the highest bond value in the PEEK acid group.

These results is supported by investigation of many other recent studies that concludes the existence of MMA monomer seems to be a decisive factor to increase the chemical bonding between PEEK and composite resins and they discovered that the best bonding to PEEK was achieved by using different adhesive systems or resin varnish that contains methyl methacrylate monomers (-MMA) [14,19,44]. Since the bond strength depends heavily on the materials composition, the heat cured acrylic resin consider polymer of methyl methacrylate that could be preferred material of choice for bonding to PEEK.

The highly significant difference that is shown between Co-Cr alloy and PEEKS polymer acid etch surface treatment groups could be explanted due to the different behavior of sulfuric acid with metals than polymers in genaral. Sulfuric acid as a surface treatment is considered as a highly corrosive acid which can release hydrogen gas when comes in contact with metals and at high concentration shows a strong oxidizing character which raises significantly the corrosion attack on metal materials, this enables some metal alloys to protect them by forming a protect layer, while with plastic materials this oxidizing properties is highly corrosive [45,46].

All Co-Cr alloy specimens were noted with dominant adhesive failure, this might attribute to the poor of chemical bonding between the polymethylmethacrylate denture base and the Co-Cr metal framework [25]. However, the PEEK control (without treatment) and air abrasive groups display a predominantly adhesive failure when compared with acid etch group of PEEK polymer that show predominately mixed failure.

PEEK polymer has a very inert surface which leads to poor bonding with other dental materials so surface treatment should be performed to accomplish a sufficient bonding, however, the bonding mechanism varies that could be attributed to the chemical bond resultant by acid etch treatment which was stronger because acid etch provides PEEK surface modified chemically by augmenting the functional features in PEEK polymer, whilst the mechanical bond provided by air abrasive treatment produce a mechanically modified PEEK surface with increased surface areas; also a higher effect from the thermalcycling may be shown with mechanical bonding that confronted mechanical stress on the bonding area, leading to volumetric changes, therefore the formation of cracks on the bonding area rised due to the difference in dimensional changes of the materials [47,48].

CONCLUSION

Within the limitations of this study, it can be concluded that:

- Air abrasive surface treatment revealed a comparable effect in enhancing the bond strength of heat cured the acrylic resin to both Co-Cr alloy and PEEK polymer
- Acid etch surface treatments demonstrated an adverse effect according to the framework material, type sulfuric acid with Co-Cr alloy show a significant decrease in bond strength to heat cured acrylic resin, while for PEEK polymer demonstrate significantly superior bond strength with heat cured the acrylic resin
- Heat acrylic resin showed statistically higher bond strength with PEEK polymer than Co-Cr alloy, which probably makes PEEK polymer feasible to use for removable prosthodontics and considered as a promising material as an alternative removable partial denture framework

DECLARATIONS

Conflict of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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