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EVALUATIONS THE EFFECTS OF DIFFERENT SURFACE TREATMENTS ON MICROLEAKAGE OF HEAT CURE ACRYLIC RESIN AT CO-CR ALLOY AND PEEK POLYMER INTERFACE

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ABSTRACT

Background: Microleakage at framework components and acrylic resin junction in removable partial denture may result from poor chemical bonding, PEEK one of the alternative framework materials used to overcome like this drawback. **Objectives:** Evaluate the microleakage of acrylic resin to Co-Cr alloy and PEEK using different surface treatments. **Materials and Methods:** Sixty specimens prepared in regtinguler shap with contained grid and divided according to framework material type into two groups Co-Cr alloy and PEEK (n=30/ group), then each main groups subdivided according to surface teratment type to three subgroups (n=10/ group) control group (no surface treatment), air abrasive group (110 µm alumina oxid) and acid etch group (98% sulfuric acid). The specimens were thermocycled (3000 cycles) after clear heat cured acrylic resin application, then immersed in sodium fluorescein dye solution (0.1 g/1000mL) for 24 hours at room temperature, microleakage assessment done by counting the grids that exhibited dye penetration under ultraviolet-light. Data were analyzed LSD test at ($p<0.05$). Characterizations performed through scanning electron microscope and X-Ray diffraction. **Results:** PEEK acid etch and air abrasive surface treatment groups show the lowest microleakage values, while Co-Cr alloy acid etch and control groups had the highest microleakage values. The results revealed highly significant differences between all groups at $P<0.01$. **Conclusion:** Microleakage reduced with air abrasive treatment for both Co-Cr and PEEK; however, sulfuric acid treatment efficiently improves the microleakage reduction with PEEK whereas exhibited the highest microleakage extension with Co-Cr.

KEYWORD: Co-Cr alloy, PEEK, microleakage, surface treatment.

INTRODUCTION

A stable bonding between two surfaces should exist because it is consider as a primary factor in the prevention of microleakage. Partial dentures are commonly fabricated with acrylic resin and metal framework. Base metals, such as cobalt chromium alloy.[1] Weak adhesion and lack of a chemical bond cause separation of resin denture base from framework in removable prosthodontics due to discrepancies in the coefficient of thermal expansion among these components. Removable partial prostheses are subjected to variations in temperature during oral function; this factor may lead to volumetric shrinkage of acrylic resin that result in deterioration at the interface between metal alloys and acrylic resins, fluid percolation, discolorations, diffusion of bacteria.[2] Because of the drawbacks of metal based frameworks the use of metal free materials including high performance polymers such as polyetheretherketone polymer that recently been considered as an alternative removable partial denture framework material for conventional Cr-Co frameworks in combination with the traditional denture base acrylic resins.[3, 4] This is particularly true mainly because of PEEK owing superior properties like a high biocompatibility, good mechanical properties, high temperature resistance, chemical stability, modulus of elasticity about 4 GPa making it as elastic as bone and can reduce stresses transferred to the abutment teeth. Additional advantages of this polymer material are elimination of allergic reactions and metallic taste, high polishing qualities, low plaque affinity, and good wear resistance.[5, 6] The microleakage is defined as the seepage of oral fluids containing bacteria and debris between a tooth and its restoration or between the materials interfaces of prosthesis; microleakage is a key factor in determining the serviceability of prosthesis[7], and the prevention of microleakage relied primarily on bonding efficiency between materials. Bonding between the framework components and the denture base resin plays an important role in the longevity of the prosthesis; basic bonding systems include mechanical, chemical or combination of both.[2] The hypothesis for present study is there is a correlation between microleakage reduction and framework material type with consideration of surface treatment. This study was designed to evaluate the effect of specific surface treatments on microleakage between a heat activate acrylic resin and different framework materials.

MATERIALS AND METHOD

*Preparation and Casting of the Metal Specimens

To produce thirty Co-Cr specimen, a metal mold was designed to reproduce a wax patterns in rectangular with dimension of 15mm in length, 6mm in width and 1mm in thickness with a grid pattern, each grid´s dimensions are 3mm in length and width and then Co-Cr alloy (Super6, U.S.A.) were cast using a phosphate bonded investment (BEGO, Germany) with centrifugal casting technique, in accordance with the manufacturer's instructions.[1, 8] The specimens surfaces has been finished with silicon carbide papers No. 600 grit (Trojan, China) in the grinding polisher machine (160E, Mapao, China) under abundant 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 let to dried by air.[9, 10]

*Preparation of the PEEK specimens

To produce thirty PEEK specimens, in this procedure use a Co-Cr alloy specimen as metal analog to be scanned by using a fully automated laboratory optical strip light scanner (Zirkonzahn S600 ARTI scanner, Italy)[11], and digitalized with software (Zirkonzahn Modelier & Nesting; Zirkonzahn, Italy) to produce a 3D CAD model with dimension of 15mm in length, 6mm in width and 1mm in thickness with a grid pattern, each grid´s dimensions are 3mm in length and width and locating the thirty PEEK rectangular shape virtual specimens in the virtual blank. Two blanks of dental PEEK (JUVORA™ Dental Disc, UK) were milled under cooled air using a milling unit (M5 Zirkonzahn, Italy) with fallowing the manufacturer's instructions. Each of the specimens was measured and examined twice in three different locations with digital electronic caliper (Powerfix, UK), then all specimens were polished with silicon carbide abrasive papers No.600 grit (Trojan, China) in the grinding polisher machine (160E, Mapao, China) under abundant water for 10 seconds on a 300 rpm in order to provide a standard and uniform surface, PEEK specimens were ultrasonically cleaned for 3 min and then dried with air.[12, 13]

*Application of heat cured acrylic resin

After obtaining thirty rectangular shape specimens for both Co-Cr alloy and PEEK, a layer of modeling wax (Cavex, Holland) adapted all around the Co-Cr and PEEK specimens except one end because from this end the dye penetration had been assed.[7] All Co-Cr alloy and PEEK specimens 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.[14]

Before application of acrylic resin, the specimens of Co-Cr and PEEK divided into three groups according to received surface treatment (n = 10 for each group).

Control groups: without surface treatment.

Air abrasive groups: abraded with alumina oxide 110µm particle size (Renfert, Germany) with an airborne particle abrasive unit (Mestra, Germany) for 15 seconds at 2 bar pressure with distance of 10 mm that standardized by using a specially designed holder and then ultrasonically cleaned (Mestra, Germany) for 10 minutes with deionized water and dried by air.[7, 8, 15]

Acid etch groups: treated with the application of sulfuric acid 98% (Central Drug House, India) for one minute after that rinsing with the deionized water for 30 seconds, then the specimens were air dried for 10 seconds.[15, 16]

Heat cure acrylic resin (SpofaDental, Czech) was mixed and packed according to manufacturer's instructions, then the flask was placed under a hydraulic press (Quayle Dental, UK), and 5MPa pressure was applied slowly.[7] The specimens were cured according to the manufacturer's instructions, and then the flasks cooled slowly at room temperature.[17] After deflasking the specimens were immersed in 37°C water for 24 hours, all specimens were thermocycled (3000 cycles, 5°C and 50°C with a dwell time of 1 minute) in artificial saliva using a thermocycling system (Cooler: HETO, Danmark; Herter: Wincom, china; holder).[8, 17]

*** Microleakage Evaluation**

The thermocycled specimens immersed in sodium fluorescein dye solution (0.1 g/1000mL) for 24 hours at room temperature. The specimens were then ultrasonically cleaned with distilled water for 20 minutes to remove any surface stain, the specimens were allowed to bench dry for 24 hours to ensure that the penetration of the fluorescein dye ended.[1] The depth of the dye penetration at the heat cured acrylic resin and Co-Cr or PEEK denture base materials was assessed under ultraviolet light (with wave length 465nm) and a digital microscope with magnification of 8x as shown in Fig.1.[8] Each square that exhibited any evidence of dye penetration was recorded as positive.

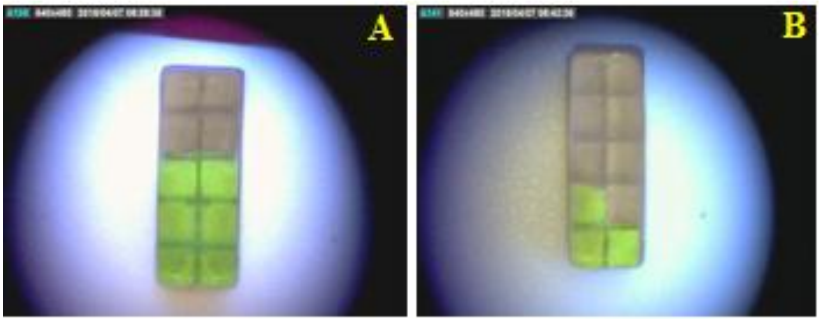

Figure (1): Microleakage specimens under digital microscope.
(A): Co-Cr specimen. (B): PEEK specimen.

*Characterizations

One specimen per each group was prepared and polished for analysis the surface topography after surface treatments by using scanning electron microscope (SEM) (S50, FEI Company, Netherlands) under ×500 magnification. For XRD analysis, addition one specimen per each group was prepared to identify the surface components and elements by using X-ray diffractometer (XRD-6000, Shimadzu, Japan) for the Co-Cr alloy and PEEK polymer specimens before and after sulfuric acid surface treatment.

*Statistical Analysis

The microleakage data had confirmed with normal distribution by using the Kolmogrov Smirnov test. One-way and LSD tests statistical analysis tests used to determine the differences between all the groups.

RESULTS

* Microleakage Observations

The means and standard deviations with One-way ANOVA difference tests for the microleakage means equality of heat-polymerized resin to each Co-Cr alloy and PEEK presented in Table 1. LSD test used to detect differences between all studied groups Table 2.

Table (1): Descriptive statistics with One-way ANOVA test.

Material	Control Group	Air Abrasive Group	Acid Etch Group	One-way ANOVA	
Co-Cr	**4.00±(2.055)**	**3.20±(1.619)**	**6.30±(1.337)**	**F-test**	**Sig.(*)**
PEEK	**3.30±(1.337)**	**1.80±(1.317)**	**1.30±(0.675)**	**14.985**	**0.000 (HS)**
(*) HS: Highly Significant at P< 0.01.					

Table (2): The least significant difference test for all studied groups.

Groups	Co-Co Control	Co-Co Air abrasive	Co-Cr Acid etch	PEEK Control	PEEK Air abrasive	PEEK Acid etch
Co-Co Control	----	N.S	H. S	N .S	H .S	H .S
Co-Co Air abrasive	----	----	H .S	N. S	S	H .S
Co-Cr Acid etch	----	----	----	H .S	H .S	H .S
PEEK Control	----	----	----	----	S	H .S
PEEK Air abrasive	----	----	----	----	----	N .S
PEEK Acid etch	----	----	----	----	----	----
(*) HS: Highly Sig. at P<0.01; S: Sig. at P<0.05; NS: Non-Sig. at P>0.05.						

The SEM observations of Co-Cr specimens for control group (without surface treatment) Fig.2A showed a uniform surface, and the air abrasive group Fig.2B displayed appearance of micro porous and irregularities and a rough surface. For acid etch Co-Cr group SEM Fig.2C demonstrated a similar superficial appearance to the Co-Cr air abrasive group because of micro porosities formation.

The SEM observations Fig.2D of PEEK control group specimen revealed a plain surface with irregular striations. Air abrasive group of PEEK Fig.2E exhibited a pronounced irregularities and a rough texture with peaks and valleys while acid etch PEEK group Fig.2F displayed complex fiber network surface and a sponge like surface with evident porous and pits.

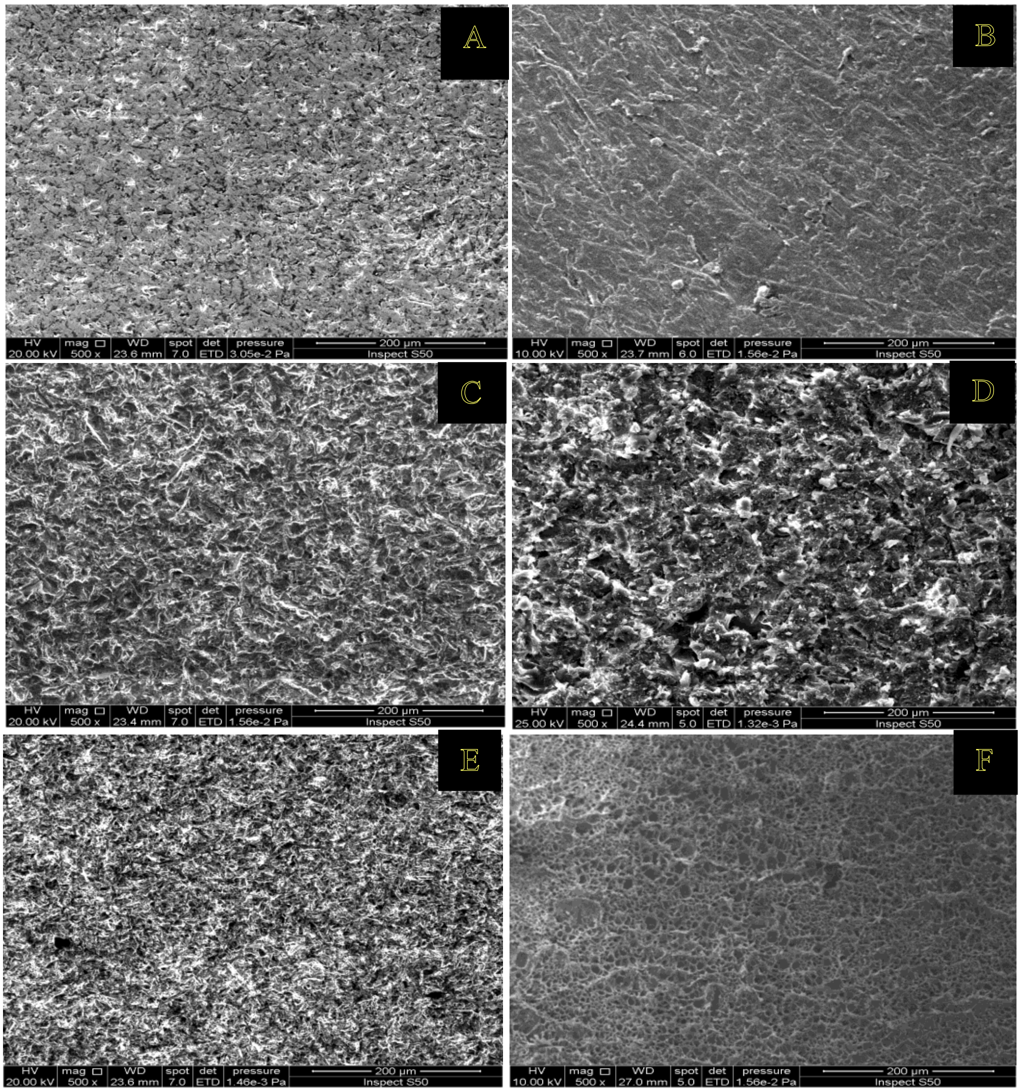

Figure (2): Different surface treatment SEM images at 500× magnification: (A): Co-Cr Control Group. (B): Co-Cr Air abrasive Group. (C): Co-Cr Acids etch Group. (D): PEEK Control Group. (E): PEEK Air abrasive Group. (F): PEEK Acids etch Group.

The XRD pattern of Co-Cr specimen before sulfuric acid surface treatment shown in Fig. 3A, demonstrate the diffraction characteristic peaks of the Co-Cr alloy while Fig. 3B show the XRD pattern after acid surface treatment that revealed a new peaks indexed to chromium sulfate ($CrSO_4.5H_2O$) and cobalt sulfate ($CoSO_4.H_2O$) also showed decrease in the intensity. The XRD pattern results confirm the formation of sulfates salts on the Co-Cr specimen surface through sulfuric acid surface treatment.

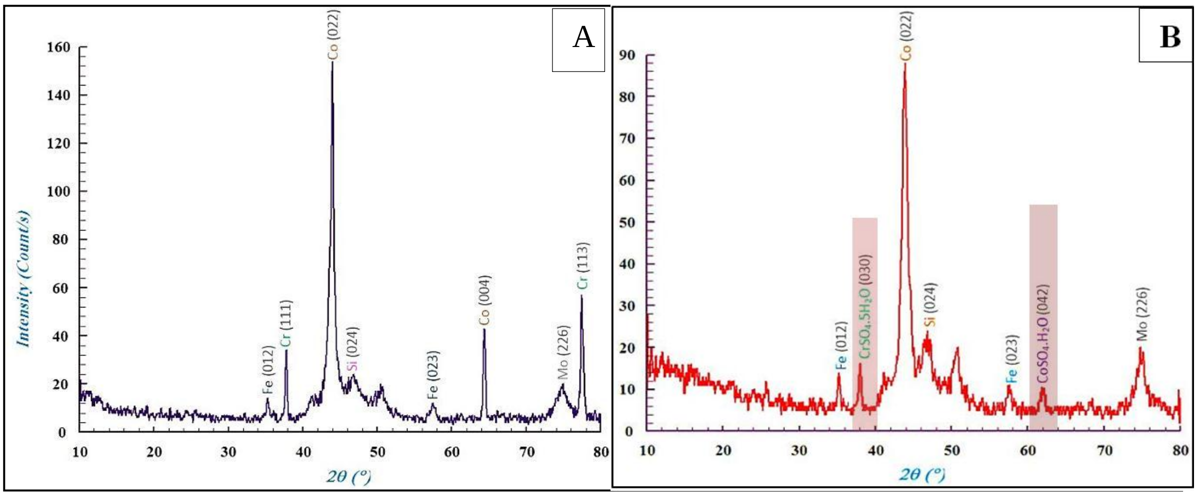

Figure (3): XRD analysis of Co-Cr specimen. (A): Before sulfuric acid surface treatment. (B): After sulfuric acid surface treatment.

The XRD pattern represents the characteristic peaks of PEEK (without acid treatment) Fig. 4A. The XRD pattern of PEEK specimen with acid surface treatment reveals remained characteristic peaks with decrease in intensity, which confirms that there is a notable physiochemical modification had been occurred.

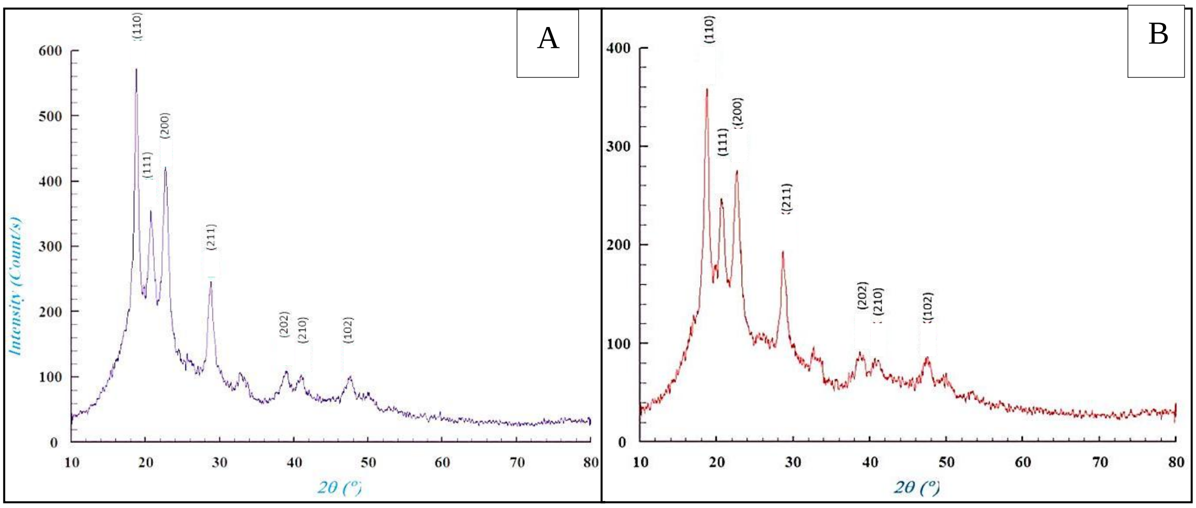

Figure (4): XRD analysis for PEEK specimen. (A): Before sulfuric acid surface treatment. (B): After sulfuric acid surface treatment.

DISCUSSION

Removable partial dentures subjected to various temperatures during oral functions, which may lead to micoleakage, so to simulate the intraoral temperature, thermocycling procedure combination with microleakage studies in order to obtain an initial idea about qualities of a new material or combination of materials. Leakage studies are carried out with most dental material laboratories and used different test techniques including chemical or radioactive tracer molecules infiltration, bacteria and color dye penetration.[7, 18, 19]

Several factors caused leakage like thermal contraction, materials dimensional changes due to polymerization shrinkage, mechanical stress and water absorption. Several studies explain microlaekage due to the discrepancies in coefficient of thermal expansion between framework material and resin denture base that lead to create gap at the materials interface thus induce the microleakage extension.[17, 20, 21]

***Air Abrasion Surface Treatment**

The primary consideration in the prevention microleakage is bond strength[1], with many earlier studies for Co-Cr alloy and PEEK with resins revealed that air abrasion surface treatment had been associated with improving the bond strength.[22-27] Air abrasion process clean the specimen's surface form unfavorable contamination and at the same time increase the surface area, surface wettability and providing micromechanical retention which eventually enhance mechanical bonding.[28, 29]

The SEM images finding shown in Fig.2 B and E of Co-Cr alloy and PEEK polymer that revealed variation in surface morphology after air abrasion surface treatment when compared with untreated specimens of Co-Cr alloy and PEEK polymer Fig.2 A and D. These surface morphology variations influence on surface roughness that considered beneficial on mechanical bonding and attribute in reducing the microleakage extension, the finding SEM images were consistent with other currant studies.[22, 23, 30, 31]

The results in this study for both Co-Cr alloy and PEEK polymer indicate that air abrasion surface treatment decrease microleakage mean values when compared with control groups, similar results with other previous studies support the obtained result[1, 8, 32], therefore air abrasion surface treatment had a positive effect in improvement of microleakage résistance at interference of heat cured acrylic resin to Co-Cr alloy and PEEK polymer.

***Acid Etched Surface Treatment.**

The results of this study indicate that acid etched surface treatment influence was not consistent in microleakage reduction efficiency between Co-Cr alloy and PEEK polymer materials, this difference may be attribute to the chosen materials.

The SEM observation finding of Co-Cr alloy treated with acid etch in Fig. 2C showed an irregular surface with microporous, however the etched Co-Cr specimen show the highest microleakage mean value. On the other hand, the XRD analysis of Co-Cr specimen treated with sulfuric acid in Fig.3B conform there is a reaction between Co and Cr alloy elements with sulfur acid and formed a salts layer of a cobalt sulfate ($CoSO_4.H_2O$) and chromium sulfate ($CrSO_4.5H_2O$) this layer of brittle salts initiate from the alloy surface that act as a separative layer between the polymethylmethacrylate denture base and the Co-Cr metal framework leading to poor bonding efficiency and probably explained the lack of microleakage reduction from this group.

The SEM images observations in Fig.3F for PEEK specimen treated with sulfuric acid showed a micropours and numerous of blister like round cavities over the surface producing a superficial complex network, these surface changes by sulfuric acid provide sufficient surface roughness thus obtaining a good mechanical retention for bonding process[33], while PEEK specimen without surface treatment in Fig.3D shows a plain surface with uneven striations, and these SEM findings of surface topography were supported with other previous studies.[15, 16]

PEEK is inert and apolar polymer with high chemical resistance and low surface energy which need a highly corrosive solutions for surface treatment, anhydrous very polar sulfuric acid is a strong oxidizing agent that can dissolve PEEK at room temperature and causes a swelling process that produce permeable surface with porosities which could act as an anchorages and penetrate easily by bonded material.[15, 34]

In addition, the PEEK specimen XRD pattern after acid etch treatment Fig.4 revealed a decreasing in intensity with remain the same characteristic peaks diffraction of PEEK polymer, this could be attributing to the effect of sulfuric acid that creates remarkable physicochemical modifications on PEEK specimen surface. Many other recent studies investigations also conclude that MMA monomers presence in different adhesive systems or resin varnish seems to act an important factor to increase the chemical bonding of PEEK to composite resins after sulfuric acid surface treatment.[12, 16, 36] Generally, surface topography is important for bonding durability, sulfuric acid surface treatment improve the mechanical bonding by forming micropours on the PEEK surface. The enhancement of bonding efficiency between PEEK polymer and heat cured acrylic interface was important in controlling and restriction of the microleakage extension, therefore in this study PEEK with acid etch surface treatment exhibit the lowest microleakage extension among all groups.

CONCLUSION

Within the limitation of this study, it can conclude that:

1. The use of air abrasion as surface treatment lead to improve microleakage reduction at both Co-Cr alloy and PEEK polymer interface to heat cured acrylic resin.
2. The uses of sulfuric acid etch as surface treatment had achieved a higher microleakage reduction with PEEK and did not cause any microleakage reduction improvement with Co-Cr alloy.
3. The results showed that PEEK polymer might be feasible and safe alternative material for removable dentures framework.

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