EGYPTIAN
DENTAL JOURNAL

Vol. 67, 3617:3627, October, 2021

Print ISSN 0070-9484 • Online ISSN 2090-2360

Fixed Prosthodontics and Dental Materials

www.eda-egypt.org • Codex : 131/21.10 • DOI : 10.21608/edj.2021.85048.1710

EFFECT OF REPEATED HEAT PRESSING AND THERMOCYCLING ON MICRO SHEAR BOND STRENGTH OF POLYETHERETHERKETONE (PEEK) TO RESIN CEMENT

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ABSTRACT

Purpose: This study was aimed to evaluate the effect of repeated heat pressing and thermocycling on micro-shear bond strength of PEEK to resin cement.

Methods: A total of 30 PEEK (Bredent Gmbh &Co.KG, Germany) specimens 10 mm x 10 mm x 2 mm were fabricated and divided to three groups (n=10). Group I was pressed using new PEEK, Group II; 50% new PEEK and 50% reprocessed PEEK, and Group III; 100% reprocessed PEEK. Bonding surfaces were sandblasted and thin layer of bonding agent (visio.link, Bredent Gmbh & Co.KG, Germany) was applied and polymerized. Plastic tygon tubes with a 1.5 mm length and 1 mm inner diameter were fixed on the sample surface and filled with dual polymerized self-etch self-adhesive resin cement (Totalcem, Itena, Paris, France) and cured for 40 seconds. Half the specimens in each group (n=5) subjected to 5000 thermocycles (5–55°C) prior to shear bond strength (μSBS) test. Two-way Analysis of Variance (ANOVA) was used to analyse the effect of PEEK condition, thermocycling and their interaction on mean micro-shear bond strength μSBS. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$.

Results: The PEEK condition (regardless of thermocycling) showed a statistically significant mean μSBS value (P-value = 0.001, Effect size = 0.455). Thermocycling (regardless of PEEK condition) also showed a statistically significant mean μSBS value (P-value = 0.002, Effect size = 0.331). The interaction between the two variables had no statistically significant effect on mean μSBS (P-value = 0.442, Effect size = 0.066).

Conclusion: Both repeated heat pressing and thermocycling had a negative effect on micro-shear bond strength of PEEK to resin cement.

KEYWORDS: PEEK, heat pressing, reprocessing, surface treatment, thermocycling, shear bond strength.

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INTRODUCTION

Polyetheretherketone (PEEK) is the most significant representative of polyaryletherketone (PAEK) family. It is a partially crystalline, high performance, temperature resistant, thermoplastic material. It consists of an aromatic ring connected by ketones to functional groups [(1)] with high melting temperature and has numerous applications in industrial processes. PEEK materials are used in medicine, orthopedics and dentistry. A modified PEEK with incorporation of 20% inorganic fillers are preferred in dental field because of features as stable physical properties, high abrasion resistance, biocompatibility and elastic modulus comparable to bone that allowing it to act as a stress breaker and lessen forces transmitted to restorations and implants [(2-4)].

Additionally, PEEK is more aesthetic than metal and conventional thermoplastic resins, allowing its use in applications such as implant bodies, temporary abutments, implant superstructures, crowns, telescopic crowns, removable partial dentures and fixed partial dentures [(2,5)]. The low specific weight of PEEK allows it to construct considerably lightweight prosthesis with high comfort and satisfaction of the patient. Its hygienic properties easily helps to maintain oral hygiene [(6)]. Moreover, PEEK is radiolucent material making it compatible with different imaging techniques. This allows the diagnosis, examination, and treatment when necessary without need for substructure removal or impairment [(7)]. BioHPP (High Performance Polymer) is a part of the PEEK family. Its good wear resistance, excellent stability, supreme polishing properties, with resulting low plaque accumulation affinity made it ideal choice for precise prosthetic restorations fabrication [(8)].

PEEK restorations are constructed either with computer aided design computer aided manufacturing (CAD CAM) or with pressing technology [(3)]. For milling purposes, blanks of PEEK pressed industrially under standardized criteria are supplied while for compression molding, pre-pressed granular or pellets form is available [(9)].

The manufacturer instructions warn from pressing the material more than once. They claim that it would degrade during another melting process and important physical and mechanical qualities would be lost [(10)]. After pressing, the sprues should be removed, along with the remaining button material and discarded. New material ingots should be used for new pressings. However, this will result in a significant amount of wasted material and it has been found that remaining materials are being reprocessed in some dental laboratories. The issue was thereby raised whether PEEK material could be safely reprocessed. Sufficient data about microstructure, possible degradation, physical and mechanical properties of dental restorations fabricated from reprocessed material is not available. For industrial purpose, Day et al [(11)] tested PEEK produced from the blend by injection moulding and found the test pieces to have greater tensile strength and Young's modulus than a new material containing a similar loading of PEEK composites reinforced with carbon fibres. It was also stated that the viscosity molecular weight of the reprocessed PEEK did not drop greatly after an extra injection moulding cycle.

PEEK blanks have an opaque color, greyish or white and are unsuitable for esthetic monolithic dental restorations, especially for the anterior esthetic region. Therefore, veneering is mandatory, but bonding the veneering composite resin materials to PEEK remains bothersome because of its inert chemical performance, low surface energy, poor wetting capabilities and resistance to surface alteration by chemical treatments [(12)]. Some studies evaluated the adhesion of resin cements to treated and untreated PEEK surfaces and the others tested and compared different surface treatments; sandblasting with alumina and silica coating (Rocatec), chemical

TABLE (1): Materials and equipments used.

Material	Product name	Manufacturer	Composition
PEEK	for 2 press BioHPP (Granulate)	Bredent GmbH & Co KG	PEEK, 20% weight titanium oxide
Investment material	Brevest for 2 Press	Bredent GmbH & Co KG	Phosphate bonded investment
Adhesive system	Visio.link	Bredent GmbH & Co KG	MMA, pentaerythritol triacrylate, photo initiators
Resin cement	Totalcem	Itena	Self-etching and self-adhesive Permanent Nanohybrid Composite resin cement
Aluminium oxide	Cobra	Renfert GmbH	Aluminium 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
Polymerizing unit	Bre.lux Power Unit 2	Bredent GmbH & Co KG	LED Light 370-500 nm

treatment with 98% sulfuric acid, gas inert plasma treatment, silane agents and adhesives. Based on the results, airborne-particle abrasion and an adhesive system could be recommended for reliable bond strength between PEEK substructures and composite resins [5,13-19]. The cementation protocol recommended by manufacturer is to induce micro-roughness using sandblasting with 110 μm alumina particles followed by a special adhesive layer application (visio.link, Bredent Gmbh & Co.KG, Germany) prior to luting system application. Several testing methodologies can be used to assess the bonding properties including shear bond and tensile bond strength tests. As better stress distribution can be fulfilled in smaller specimens, lately more accurate test methods, such as microtensile and microshear tests were introduced [20]. To obtain clinically relevant statements, specimens were exposed to artificial aging in a thermocycler. The aim of this study was to assess the effect of repeated heat pressing (reprocessing) of PEEK on the bond strength with resin cement. As well as to describe the failure modes in newly pressed, partially reprocessed and totally reprocessed material using scanning electron microscopy SEM.

MATERIALS AND METHODS

The details of the materials used in this study are mentioned in Table 1. A total of 30 PEEK specimens (Bredent Gmbh &Co.KG, Germany) each measuring 10 mm x 10 mm x 2 mm were constructed. A pink wax sheet of 2 mm thickness was cut into identical rectangular blocks 10 mm x 10 mm. The wax patterns were subjected to spruing, investing with phosphate bonded investment (Brevest for 2 press investment material, Bredent, Senden, Germany) and divided to three groups (n=10). Group I was pressed using new PEEK, Group II ; 50% new PEEK and 50% reprocessed PEEK, and Group III ; 100% reprocessed PEEK. Half the specimens in each group (n=5) submitted through 5000 thermocycles (5–55°C) before performing shear bond strength (μSBS) test. (Table:2).

TABLE (2): Classification of PEEK specimens in test groups

PEEK composition	Thermocycling
PEEK Control (n=10)	No Thermocycling (n=5)
	Thermocycling (n=5)
PEEK Partially reprocessed (n=10)	No Thermocycling (n=5)
	Thermocycling (n=5)
PEEK Totally reprocessed (n=10)	No Thermocycling (n=5)
	Thermocycling (n=5)

After pressing, the samples were divested then the bonding surfaces of the specimens were polished under running water with 600 and 800 grit silicon carbide paper. To fit the test device, A polyvinyl chloride PVC tube with 25 mm internal diameter for each specimen was filled with autopolymerising acrylic resin (Cold cure special tray material, Acrostone, Cairo, Egypt) and the PEEK specimens were fixed and lodged in the acrylic resin with the bonding surface exposed and in same level with the edge of the PVC tube. Then, the specimens were carefully washed in an ultrasonic cleaner (CD-4820, CODYSON, Guangdong, China) for 10 minutes with distilled water. finally, the specimens were air dried. All bonding surfaces of PEEK received the same surface treatment; sandblasting with 110 µm Al_2O_3 at 2.5 bar at 10 cm distance from the nozzle. Thin , uniform layer of visio.link bonding agent (Bredent Gmbh &Co.KG, Germany) was applied and polymerized at 220 mW/cm2 for 90 seconds (Brelux Power Unit; bredent). Plastic tygon tubes with 1.5 mm length and 1 mm inner diameter were fixed on the sample treated surface where the tube axis was perpendicular to sample surface, filled with automix dual polymerized self etch self adhesive resin cement (Totalcem, Itena, Paris, France). Small disposable brushes were used for excess cement removal from the bonding margin using. The cement material was cured using a light-curing unit (3M ESPE Dental Products, St Paul, USA) for 40 seconds.

Half the samples in every group were stored for 24 hours in distilled water at 37˚C. An automated thermocycling machine (100 SD Mechatronic Thermocycler, Germany) was used to expose the other half to 5000 thermocycles (between 5 and 55°C) with a dwell time of 20 second- and 10-seconds resting time in between by using before the μSBS test. Before testing, samples were checked with light microscope (MA100, Nikon, Japan) at 30x magnification to exclude samples with gaps or air bubbles at PEEK/cement interface. Differences in μSBS between PEEK specimens and resin cement were tested with different composition and thermocycling as variables. For shear bond testing, samples were mounted in lower fixed head of a universal testing machine (Instron 3345, Instron Corporation, England) where the bonding interface of PEEK and resin cement perpendicular to the horizontal plane and A stainless- steel wire (diameter: 0.14 inch) attached to the upper movable head of the testing machine applying shear force at the interface with a crosshead speed of 1.0 mm/min till specimen failure. Machine software (BlueHill 3, Instron, England) was used to calculate μSBS (MPa) by dividing the maximum load (N) by the area of the bonding interface (mm^2).

Failure Analysis

The samples were carefully inspected using a digital microscope (Dino-Lite Pro, Olympus, Tokyo, Japan). To classify failure occurred, images of the PEEK bonded surfaces and resin cement from each specimen were captured, Image identifiers were removed, and images were examined. The failure mode founded was classified as follows: adhesive PEEK/cement (no resin cement remnants on the PEEK surface), cohesive in cement (the fracture was in the cement) and mixed (remnants of resin cement partially found on the PEEK with PEEK surface exposed).

Scanning Electron Microscopy (SEM)

One sample from each group was selected, removing the epoxy resin material used for fixation of PEEK to enable topographical analysis of the bonding surface. The bonding surfaces of PEEK were cleaned with isopropyl alcohol, gold-sputtered then evaluated using scanning electron microscopy (SEM) in high vacuum (JSM-6360LA; JEOL, Tokyo, Japan) operating at 15 kV. Magnification ranged between 50X and 5000X.

Statistical software (SPSS v.23; IBM, Armonk, NY) was used for performing statistical analysis. Numerical data were investigated for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed normal distribution. Data were introduced as mean and standard deviation (SD) values. Two-way Analysis of Variance (ANOVA) was used to assess the effect of PEEK condition, thermocycling and their interaction on mean micro-shear bond strength. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$.

RESULTS

Two-way ANOVA results

The results revealed that PEEK condition (regardless of thermocycling) showed a statistically significant effect on mean micro-shear bond strength (P-value = 0.001, Effect size = 0.455). Thermocycling (regardless of PEEK condition) also showed a statistically significant effect on mean micro-shear bond strength (P-value = 0.002, Effect size = 0.331). The interaction between the two studied variables had no statistically significant effect on mean micro-shear bond strength (P-value = 0.442, Effect size = 0.066). As the interaction between the variables is non-statistically significant, so the variables are independent from each other. (Table.3)

Effect of PEEK condition regardless of thermocycling

Regardless of thermocycling; A statistically significant difference was found between mean micro-shear bond strengths of different PEEK conditions (P-value = 0.001, Effect size = 0.455). Pair-wise comparisons revealed that new PEEK showed the statistically significantly highest mean micro-shear bond strength. There was no statistically significant difference between partially and totally reprocessed PEEK; both showed statistically significantly lower mean values. (Table.4) (Fig.1)

TABLE (3) Two-way ANOVA results for the effect of PEEK condition and thermocycling on mean micro-shear bond strength

Variable	Type III Sum of Squares	df	Mean Square	F-value	P-value	Effect size (Partial eta squared)
PEEK condition	97.326	2	48.663	10.029	0.001*	0.455
Thermocycling	57.561	1	57.561	11.863	0.002*	0.331
PEEK condition x Thermocycling interaction	8.207	2	4.104	0.846	0.442	0.066

df: degrees of freedom = (n-1), *: Significant at $P \leq 0.05$

TABLE (4) The mean, standard deviation (SD) values and two-way ANOVA test results for comparison between micro-shear bond strength (MPa) of different PEEK conditions regardless of thermocycling

New PEEK (n = 5)		Partially reprocessed PEEK (n = 5)		Totally reprocessed PEEK (n = 5)		P-value	Effect size (Partial eta squared)
Mean	SD	Mean	SD	Mean	SD		
15.56 ^{A}	2.82	12.53 ^{B}	2.32	11.26 ^{B}	2.63	0.001*	0.455

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

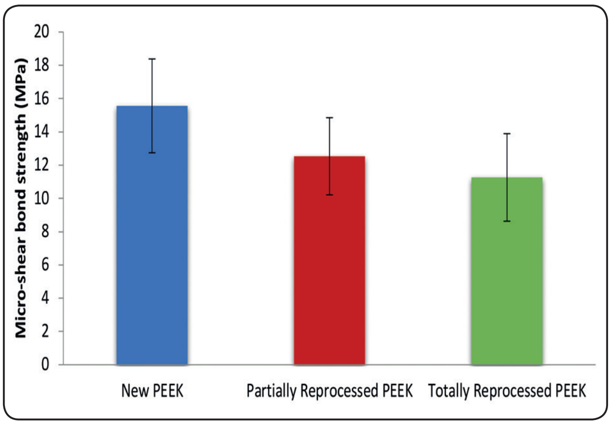

Fig. (1) Bar chart representing mean and standard deviation values for micro-shear bond strength of different PEEK conditions regardless of thermocycling

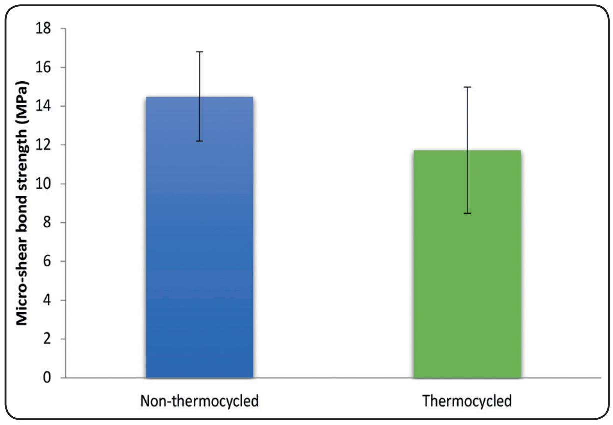

Fig. (2) Bar chart representing mean and standard deviation values for micro-shear bond strength of non-thermocycled and thermocycled specimens regardless of PEEK condition

Effect of thermocycling regardless of PEEK condition

Regardless of PEEK condition; non-thermocycled specimens revealed statistically significant higher mean micro-shear bond strength than thermocycled specimens (P-value = 0.002, Effect size = 0.331). (Table.5) (Fig.2)

TABLE (5) The mean, standard deviation (SD) values and two-way ANOVA test results for comparison between micro-shear bond strength (MPa) of non-thermocycled and thermocycled specimens regardless of PEEK condition

Non-thermocycled		Thermocycled		P-value	Effect size (Partial eta squared)
Mean	SD	Mean	SD		
14.5	2.3	11.73	3.25	0.002*	0.331

: Significant at P ≤ 0.05

Effect of different interactions on micro-shear bond strength

1. Comparison between PEEK conditions

As regards non-thermocycled specimens; statistically significant difference was found between mean micro-shear bond strength of different PEEK conditions (P-value = 0.100, Effect size = 0.175).

While with thermocycled specimens; statistically significant difference was found between mean micro-shear bond strengths of different PEEK conditions (P-value = 0.002, Effect size = 0.41). Pair-wise comparisons between conditions revealed that new PEEK showed the statistically significantly highest mean micro-shear bond strength. There was no statistically significa nt difference between partially and totally reprocessed PEEK; both showed statistically significantly lower mean values. (Table 6) (Fig.3)

2. Comparison between non-thermocycled and thermocycled specimens:

As regards new PEEK; there was no statistically significant difference between mean micro-shear bond strength of non-thermocycled and thermocycled specimens (P-value = 0.321, Effect size = 0.041).

While with partially as well as totally reprocessed PEEK; non-thermocycled specimens showed statistically significantly higher mean micro-

TABLE (6). The mean, standard deviation (SD) values and two-way ANOVA test results for comparison between micro-shear bond strength (MPa) with different variables interactions

Thermocycling	New PEEK (n = 5)		Partially reprocessed PEEK (n = 5)		Totally reprocessed PEEK (n = 5)		P-value	Effect size (Partial eta squared)
	Mean	SD	Mean	SD	Mean	SD		
Non-thermocycled	16.26	1.71	14.01	2.45	13.24	1.85	0.100	0.175
Thermocycled	14.85 A	3.71	11.06 B	0.83	9.29 B	1.52	0.002*	0.41
P-value	0.321		0.045*		0.009*			
Effect size (Partial eta squared)	0.041		0.157		0.251			

****: Significant at $P \leq 0.05$, Different superscripts in the same row indicate statistically significant difference between conditions***

shear bond strength than thermocycled specimens (P-value = 0.045, Effect size = 0.157) and (P-value = 0.009, Effect size = 0.251), respectively. (Table.6) (Fig.3)

The failure analysis showed that the most common mode of failure was adhesive failure followed by cohesive failure and the least common was mixed type. (Fig.4) SEM evaluations of surface topography at 50 X were used to study failure modes of different groups. (Fig.5-7)

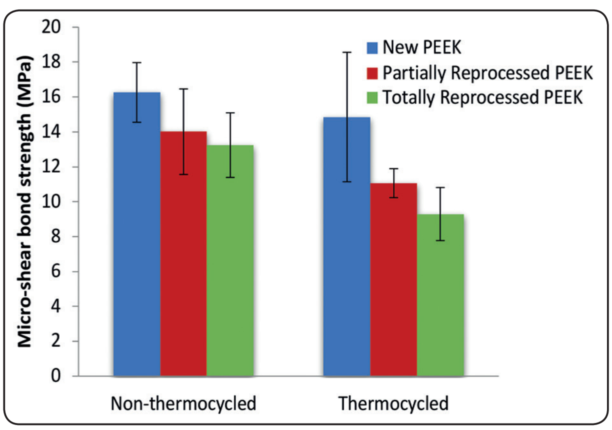

Fig. (3). Bar chart representing mean and standard deviation values for micro-shear bond strength with different interactions of variables

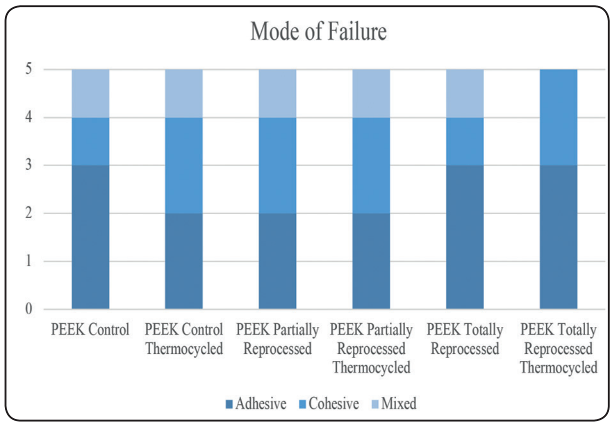

Fig. (4) Bar chart representing mode of failure analysis of all groups.

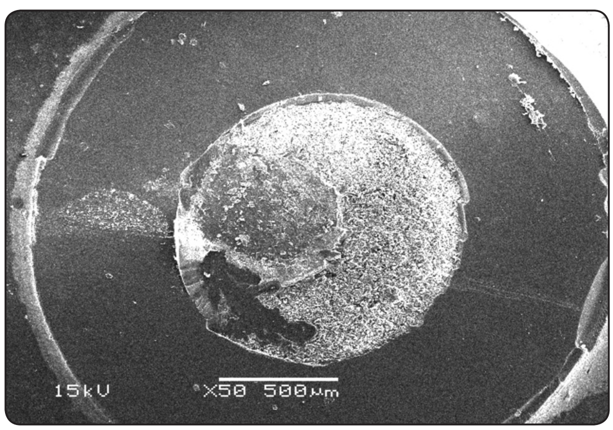

Fig. (5) Topographical analysis of the bonding surface of PEEK partially reprocessed Thermocycled sample showing cohesive mode of failure of cement layer (50X).

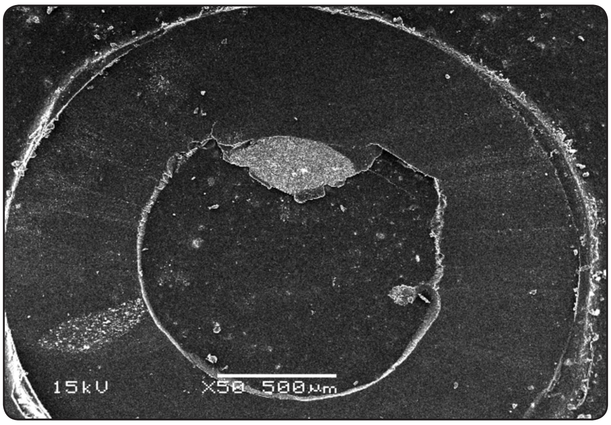

Fig. (6) Topographical analysis of the bonding surface of PEEK totally reprocessed sample showing adhesive mode of failure (50X).

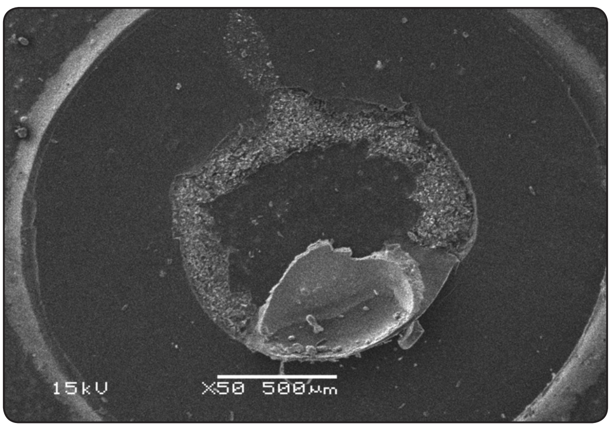

Fig. (7) Topographical analysis of the bonding surface of PEEK totally reprocessed thermocycled sample showing mixed mode of failure (50X).

DISCUSSION

Based on the mechanical and physical properties, PEEK seems to be an appropriate material for dental prosthesis. However, adequate bonding between PEEK and veneering resins is fundamental in ensuring long survival and success rates [21]. It is also obscure whether adherence to PEEK can withstand the hydrolytic effects due to water sorption, which is responsible for reduction of resin-bonding capability to oxide ceramics [19]. Surface topography is a fundamental factor that enables mechanical bonding due to the adhesive penetration inside the pits resulting in resin tags formation. Sandblasting creates surface roughness, cleans organic contaminant from the surface leaving active surface layer promoting micromechanical interlocking with resin-based dental materials [4]. for enhancing the bonding of resin to inert PEEK surface, dental adhesives are used where the adhesive system's content and solvents are fundamental factors to be considered. According to previous studies, MMA monomers containing adhesive systems showed higher values of bond strength between resin and PEEK [15,19,22-24]. It has been recommended to coat PEEK surface with low viscosity adhesive systems before covering it with veneering resins [25,26]. For this purpose, Visio.link bond was selected as an adhesive system in this study [4]. This study evaluated the effect of repeated heat processing 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 μSBS between resin cement and PEEK significantly. To the knowledge of the authors, this is the first study to evaluate the effect of reprocessing of PEEK used for dental restorations on bonding with resin cement. For that reason, the results obtained from this study could not be compared to results obtained from other available studies. The μSBS values of both partially and totally reprocessed PEEK were significantly lower than the new PEEK. The crosslinking between functional group of etched/air abraded PEEK and monomer functional groups of adhesive systems was believed to be the cause of the enhancement of bond strength. Oxidative degradation of PEEK was believed to include two reactions, chain scission and cross-linking. Chain scission results in molecular chains with less length and higher mobility, that allows reorganisation of the crystalline structure, on the other hand cross-linking prompt molecular branching and meshing [27]. Previous studies [28,29] showed that exposing PEEK to longer times and higher temperatures processing procedures in air induce a lower level of re-crystallised material and concluded that the oxidative reactions turn out to precede cross-linking reactions.

Consequently, this may lead to diminished number of bonds available for bonding with the adhesive. μSBS tests are suitable to evaluate the adhesive efficiency of resin-based materials. Any variations in the properties and features of the evaluated material surface may influence the μSBS values, which are attributed to mechanical and chemical adhesion [(4)]. The μSBS test was selected for the measurement of bond strength in this study as it is easy to perform and not technique sensitive allowing decreasing the number of pretest failures [(30)].

As the oral temperatures fluctuate, the long-term bonding stability of PEEK is not guaranteed. In this study, the specimens were thermocycled for 5,000 in a thermocycling machine, approximately corresponds to 6 months of intraoral service in vivo [(31)]. The results showed that thermocycling decreased μSBS between resin cement and PEEK. Thermocycling includes repeated exposure to two temperatures (55 and 5 °C) with 20 sec dwell time to guarantee the specimens are not exposed to extreme thermal stresses. The decreased μSBS might be accounted for thermal loading leading to mechanical stress bonding interface and leading to volumetric changes. Therefore, cracks can be initiated and propagated along the bonding interface, caused by the different dimensional changes of the materials resulting in decreasing the bond strength values [(19)]. In a study by Stawarczyk et al. [(19)] it was confirmed that an adhesive application before bonding to a self-adhesive resin cement enabled establishing of bonding after thermocycling even without etching or air abrasion surface treatment. In another study[(15)] Thermocycling after pretreatment with Visio.link and Signum PEEK adhesives showed no influence on tensile bond strength. On the other hand, thermocycling was proved by other studies to have a detrimental effect on the resin-material bonding due to relaxation of stresses within the composites. These stresses were resulted from polymerization shrinkage process [(32,33)]. A previous study tested the effect of aging conditions on the bond strength of a resin composite to a composite and found that 5000 thermal cycling was the most influential in the degradation of the composite tested between other tested aging methods [(34)]. A previous study [(35)] evaluated the shear bond strength of adhesive system bonded to different pretreated PEEK surfaces using different thermocycling methods. The bond strength after thermocycling was significantly dropped for all groups. They attributed the degradation to the 55°C hot water that might have further stimulated the hydrolysis of incompletely polymerized or unpolymerized resin cements. The failure type's analysis in this study showed no differences in the mode of fracture depending on PEEK reprocessing or thermocycling.

CONCLUSION

1. PEEK reprocessing had a negative effect on micro-shear bond strength with resin cement as new PEEK showed the highest bond strength.
2. Thermocycling had a negative effect on micro-shear bond strength of resin cement with both partially and totally reprocessed PEEK.

Clinical recommendation

1. Only new PEEK should be used for pressing dental restorations as it showed the highest micro-shear bond strength with resin cement even under thermocycling conditions.
2. Using reprocessed PEEK has detrimental effect on its bond strength to resin cement especially after thermocycling.

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