

ASSESSMENT OF SHEAR BOND STRENGTH OF DIFFERENT VENEERING MATERIALS TO POLYETHERKETONKETON (PEKK)

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ABSTRACT

Aim: is to assess the bond strength of different veneering materials to polyetherketonketon (PEKK).

Material and Methods: Thirty PEKK discs (n=30) were fabricated from PEKK blanks. According to the kind of the veneering materials used, they were assigned into three major groups: Group A: PEKK discs veneered with hybrid ceramics (Vita Enamic), Group B: PEKK discs veneered with Lithium disilicate (IPS E.max CAD), Group C: PEKK discs veneered with zirconia (BruxZir). The surfaces of PEKK specimen in all groups were subjected to sandblasting according to the manufacture instructions. After the surface treatment method had been completed, visio.link was applied to all treated PEKK specimens. The veneering materials in each group were adhered onto the surface of the treated PEKK specimens using universal dual cure self-adhesive resin cement (TheraCem™). Following the creation of the specimens, thermocycling (5°C to 55°C at 10,000 cycles) was applied to half of each group (n=5) before testing. A universal testing machine was used to assess the shear bond strength, and the failure modes were evaluated using stereomicroscope. Data were collected, tabulated and statistically analyzed.

Results: The highest shear bond strength values were observed for BruxZir shaded zirconia (27.4±2), followed by Vita Enamic and IPS E.max CAD (21.2±2.2 & 21±1.5 respectively). After thermocycling, IPS E.max Cad showed a marked decrease in the shear bond strength value (6.5±0.8), followed by BruxZir and Vita Enamic (15.3±2.2, 17±2.2 respectively).

Conclusions: material type and thermo-cycling significantly affected the shear bond strength values of the three veneering materials to PEKK specimens.

KEYWORDS: Polyetherketoneketone, Veneering materials, Shear bond strength test.

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INTRODUCTION

Ceramics are widely used materials for implant prosthetics and ordinary dental restorations. The use of ceramics for production of the crowns provide better translucency for the implant restorations, and diminish the gray color related to the metal components that is transmitted through the peri-implant tissues.

Despite their long-term survival rates, biocompatibility and excellent esthetics, fracture of the framework especially in the connector area and chipping of the veneering ceramic are considered the main drawback of these restorations. In addition evidence suggests that oxide and glass-ceramics, when used as crowns restoring dental implants, transfer more stresses to the peri-implant bone.¹

Recently biocompatible high performance polymers, PolyEtherKetoneKetones (PEKKs) were introduced to the dental field. It's highly rigid, light weighted, dimensional stable in high temperatures, and can be either "pressed" or "milled" from blocks.

High performance polymers are being introduced as an alternative to metal and glass ceramics for dental application due to their acceptable fracture resistance, superior stress distribution, and shock-absorbing capabilities.²

Despite its numerous advantages, the grayish color and low translucency of the PEKK material restrict its application as a monolithic dental restoration. Therefore, machine-milled or heat-pressed PEKK material is usually veneered with light-curing resin composites, prefabricated veneers and ceramic crowns to achieve high esthetic demand.³

Moreover, many in-vitro researches focused on the impact of various surface treatment procedures using micromechanical and chemical conditioning technique, in order to enhance the bonding potential of the PEKK polymer to veneering resins. It was also noticed that, the air type non-thermal plasma in combination with sandblasting enhanced the bond

durability between PEKK and resin cement.⁴ In addition, sulfuric acid in a concentration of 90% for 5s, and 95% vinyl sulfonic acid for 30 s experienced a significant increase in SBS values of the veneering composite to PEKK.⁵ Favorable bonding has also been demonstrated with tribochemical silica coating and sandblasting.⁶

The recently introduced PEKK polymer is still restricted by limited information concerning the bonding performance. Therefore, the surface treatment protocol for this study was selected according to the manufacture instructions. Furthermore, lack of information about the bonding performance of lithium disilicate and zirconia to PEKK must also be eliminated.

Hence, this study was conducted to evaluate the shear bond strength of different veneering materials (lithium disilicate, zirconia, hybrid ceramics) to PEKK.

The null hypothesis of this study was that the bond strength to PEKK material wasn't influenced by the type of the veneering material used.

MATERIALS AND METHODS

Sample size estimation

Calculation of sample size was performed using G-power 3.1.9.4 windows software (Heinrich-Heire, Dusseldorf, Germany). ANOVA F test was used: fixed effect, omnibus and one way. Based on the previous study,⁴ the three main tested groups were divided into two subgroups that contained 4 samples at least. Each subgroup had an extra sample for result confirmation. Beta error β of up to 20%, and an 80% study power were acceptable with alpha level α set at 5% and a 95% significance level.

Ethical approval

This research was accepted by the members of the Research Ethics Committee, faculty of dentistry, Minia University (RHDIRB2017122004) with protocol number (489/2020) at meeting number (78).

PEKK discs preparation

Thirty PEKK discs were fabricated from PEKK blank (Pekkton Ivory, Cendres+Métaux, Biel/Bienne, Switzerland), measuring $7 \times 7 \times 4$ mm of each PEKK disc. Specimens were obtained from cutting and milling of PEKK blank in the shape of bar using CAD/CAM technique (Roland DWX50, Roland DGA Corp. USA). A low speed diamond saw (Isomet 4000 precision cut, buehler, USA) with a 0.6 mm thick blade (running at 2500 rpm) was used to cut a bar of PEKK perpendicularly under copious amount of water to obtain a uniform thickness of each PEKK disc (final thickness 4.0 mm). Each one of the PEKK discs was then embedded and fixed in auto-polymerizing acrylic resin (Acrostone, Egypt) with the aid of cylindrical mold (25 mm diameter and 2mm height) with 2 mm of its thickness was outside the acrylic resin. The bonding surface of each prepared disk was polished using 600-grit silicon carbide abrasive paper (microcut Silicon Carbide grinding papers Buehler Inc) under water for 40 second. All specimen surfaces underwent a 10 minute ultrasonic cleaning in an ultrasonic bath (Codyson Digital Ultrasonic CD-4820) containing alcohol before initiating the specimens to the subsequent surface treatment procedures.

Grouping of samples

The thirty PEKK discs were separated into three major groups (each group $n=10$) depending on the type of the veneering materials used:

Group A: polyetherketonketon(PEKK) discs veneered with hybrid ceramics.

Group B: polyetherketonketon(PEKK) discs veneered with Lithium disilicate.

Group C: polyetherketonketon(PEKK) discs veneered with zirconia.

Preparation of the veneering materials

Vita Enamic (Vita Zahnfabrik, Bad Sackingen, Germany), IPS e.max CAD (Ivoclar Vivadent Schaan, Liechtenstein), and BruxZir shaded zirconia

(prismatik dentalcraft inc USA) were selected for this study. A cylinder was designed using the suitable digital software (3D Builder, Microsoft, WA, USA) with intended dimension ($\text{Ø} = 3\text{mm}$, and height = 14 mm.). A total of thirty cylindrically shaped specimens ($n=10$ for each type) were obtained from cutting of the designed cylinders using a low speed diamond saw under copious amount of water. Slicing machine with a low speed diamond blade (Isomet 4000 precision cut, buehler, USA) was used to obtain uniform thickness of each ceramic specimen (final thickness 2.0 mm).

Vita Enamic: no sintering or firing needed for this type of the specimens. IPS E-max CAD specimen was crystallized at a temperature (840 °C to 850 °C) in a ceramic furnace (Programat P 310 ceramic furnace, Ivoclar Vivadent, AG, schaan/Liechtenstein) using a pre-programmed setting limited to the polished ceramic. While, BruxZir shaded zirconia specimens was sintered at temperature of 1580 °C for about 8 hours inside the sintering furnace (TABEO-1/5 ZIRKON-1000-Germany) following the recommended sintering temperature chart.

Surface treatment of the specimens

PEKK discs were subjected to sandblasting (Al_2O_3 with a mean particle size of 110 μm at a continuous pressure of 2 bar for 10 second at a distance of 15 mm and at 90 degree angle to the disk surface) ⁶. After that, the treated PEKK surfaces were then coated with visio.link (bredent, senden, Germany) and light cured for 90 seconds using a laboratory-use light polymerization device (bre.lux Power Unit 2W/LED 370-500NM, bredent, Senden, Germany). Vita Enamic specimens were etched with 5 % hydrofluoric acid gel (Vita Ceramics Etch, Vita Zahnfabrik, Bad Sackingen, Germany) for 60 second, then rinsed for 60 second and air-dried. Coat of Silane for 60 seconds was then applied to the bonding surface of the specimens. IPS.Emax CAD specimens were treated with hydrofluoric acid 9.5%(BISCO Inc. Schaumburg,

U.S.A) for 40 second, followed by application of silane coupling agent (Porcelain primer, BISCO Inc)⁷. BruxZir shaded zirconia were subjected to Air particle abrasion (Al₂O₃ 50µm at pressure 2 Bar, at a distance of 10 mm for 20 second), and then z prime plus (BISCO Inc. Schaumburg, Illinois, U.S.A) was then applied on the internal surfaces of samples.⁸

Bonding of the veneering materials to pekk specimens

Universal dual cure self- adhesive resin cement (TheraCem™, BISCO Inc) was applied to the bonding surface of the pekk specimens. After that, discs of the veneering materials were seated and cemented to their corresponding PEKK substrate as shown in **figure (1)** under a standardized static load (constant seating load of 10 N)⁹. The specimens were then cured for 40 sec using a wireless LED light curing device (with wavelength 420-480 nm and light intensity 1000-1200 m W/cm²).

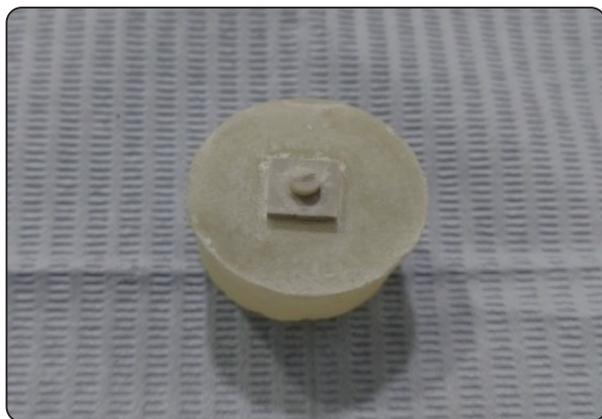


Fig. (1) Veneering materials adhered to PEKK substrate

Thermo-cycling

Each group of samples (PEKK samples veneered with Vita Enamic n=10, lithium disilicate n=10, and zirconia n=10) was randomly assigned into two subgroups depending on either subjecting the specimens into thermo-cycling or not. The selection of samples in each subgroup was carried out using blind randomization with the help of a non-dental person who was asked to distribute the tin samples

in each group into two subgroup (n=5) without knowing the aim nor the intervention that will be carried in the samples.

Subgroup AI,BI,CI (control group) : five discs (n=5) of each subgroup were immersed in distilled water at 37 °C for 24 hours.

Subgroup AII,BII,CII : the other halves of the specimens (n=5) in each group were subjected to thermocycling (SD Mechatronik Thermocycler, Westerham, Germany) for 10000 cycles from 5°C to 55°C with 1 minute dwell time.

Shear bond strength (SBS) testing

A universal testing device (INSTRON, CAT. NO:2710-115.USA) was used to calculate the shear bond strength (SBS) values at room temperature. Testing was performed using knife-edge-shaped equipment with a crosshead speed of 1 mm/min as shown in **figure (2)**. Following that, the specimens were examined under a 50 x optical stereomicroscope to identify the failure mode. Three sets of failure types (adhesive, cohesive, and mixed) were examined and identified following fracture of the specimens.

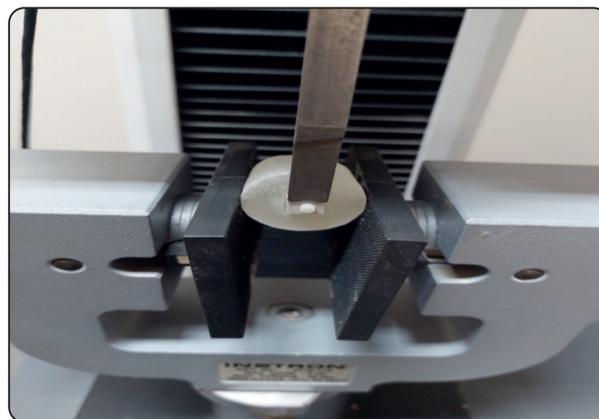


Fig. (2) Shear bond strength using universal testing machine

Statistical analysis

SPSS version (25) was used to validate, code, and analyze the data. The mean differences across groups were examined using the one-way ANOVA test. For pairwise comparisons between the two research groups, post-hoc LSD analysis was utilized. The Independent Samples T test was used to analyze parametric quantitative data between the two groups. For the study of qualitative data between groups, the chi square test was employed.

RESULTS

Effect of thermo-cycling on the shear bond strength for each veneering materials to PEKK:

Subgroup (AI) recorded a mean value of shear bond strength (21.2 ± 2.2) before they have been subjected to thermo-cycling, while subgroup (AII) recorded a mean value of shear bond strength

(17 ± 2.2) following thermo-cycling. There was a statistical significant difference at P value (**0.017**) between the two subgroups.

Subgroup (BI) recorded mean values of shear bond strength (21 ± 1.5) before they have been subjected to thermo-cycling, while subgroup (BII) recorded mean values of shear bond strength (6.5 ± 0.8) following thermo-cycling. There is a statistical significant difference at P value **<0.001** between subgroup (BI) and subgroup (BII).

Subgroup (CI) recorded mean values of shear bond strength (27.4 ± 2) before thermo-cycling, while subgroup (CII) recorded mean values of shear bond strength (15.3 ± 2.2) after they have been subjected to thermo-cycling. There is a statistical significant difference at P value **<0.001** between the two subgroups (CI&CII) as shown in table (3)

TABLE (1) The shear bond strength of different veneering to PEKK before they have been subjected to thermo-cycling:

		Vita enamic (Sub AI)	E max (Sub BI)	Zirconia (Sub CI)	P value
		N=5	N=5	N=5	
Shear bond strength	Range	(19.2-24.9) ^a	(19-22.8) ^a	(24.7-29.1) ^b	<0.001*
	Mean \pm SD	21.2 \pm 2.2	21 \pm 1.5	27.4 \pm 2	

One way ANOVA test for quantitative data between the 3 groups followed by posy hoc LSD analysis between each two groups
Superscript with different small letters refer to significant difference between the 2 groups

**: Significant difference at P value < 0.05*

TABLE (2) The Shear bond strength of different veneering to PEKK after they have been subjected to thermo-cycling:

		Vita enamic (Sub AII)	E max (Sub BII)	Zirconia (Sub CII)	P value
		N=5	N=5	N=5	
Shear bond strength	Range	(15.5-20.9) ^a	(5.5-7.5) ^b	(13-18.3) ^a	<0.001*
	Mean \pm SD	17 \pm 2.2	6.5 \pm 0.8	15.3 \pm 2.2	

One way ANOVA test for quantitative data between the 3 groups followed by posy hoc LSD analysis between each two groups
Superscript with different small letters refer to significant difference between the 2 groups

**: Significant difference at P value < 0.05*

TABLE (3) Shear bond strength between before and after thermo-cycling in different materials

Material	Shear bond strength	Before thermocycling	After thermocycling	P value
		(I) N=5	(II) N=5	
Vita enamic (Sub A)	Range	(19.2-24.9)	(15.5-20.9)	0.017*
	Mean \pm SD	21.2 \pm 2.2	17 \pm 2.2	
E max (Sub B)	Range	(19-22.8)	(5.5-7.5)	<0.001*
	Mean \pm SD	21 \pm 1.5	6.5 \pm 0.8	
Zirconia (Sub C)	Range	(24.7-29.1)	(13-18.3)	<0.001*
	Mean \pm SD	27.4 \pm 2	15.3 \pm 2.2	

Independent Sample T test for quantitative data between the two groups.

**: Significant difference at P value < 0.05*

TABLE (4) Type of Failure between different material before and after thermo-cycling

Thermocycling	Type of failure	Vita enamic	E max	Zirconia	P value
		N=5	N=5	N=5	
Before thermocycling	Adhesive	1(20%)	3(60%)	3(60%)	0.534
	Cohesive	3(60%)	1(20%)	2(40%)	
	Mixed	1(20%)	1(20%)	0(0%)	
After thermocycling	Adhesive	1(20%) ^a	4(80%)	5(100%) ^b	0.088
	Cohesive	1(20%)	0(0%)	0(0%)	
	Mixed	3(60%)	1(20%)	0(0%)	
<i>P value (before v after)</i>		0.368	0.565	0.114	

Chi square test for qualitative data between groups

Superscript with different small letters refer to significant difference between the 2 groups

Significant difference at P value < 0.05

DISCUSSION

In comparison to PEEK (Polyetherketoneketone), PEKK had a higher melting point, and an 80% increase in compressive strength. The large proportion of ketone groups in its chemical structure offered a stronger solidification between the glass and polymer which in turn leading to enhancement in its compressive strength and its melting point.¹⁰ Due to its lower elastic modulus (5.1GPa)¹¹, better stress distribution, shock absorbing ability, and good biocompatibility, the PEKK material could be the

material of choice for construction of the crowns, post and core, fixed partial dentures, and also as a framework for implant supported prosthesis.⁴

Grayish colors of the PEKK material restrict its application as a monolithic dental restoration. Improved adhesive properties of the resin cement to PEKK are essential to reduce micro leakage, enhance retention, and increase prosthesis stability. Suitable surface treatment is considered a significant factor in achieving the highest bond strength of the veneering material to PEKK.

According to the manufacture, sandblasting (Al₂O₃ 110 µm at 2 bar) create a suitable adhesive potential of the veneering material to PEKK. However **Labriaga, W et al (2018)** reported that air-type-non-thermal plasma in combination with sandblasting enhanced adhesive potential between PEKK and resin cements ⁴.

The veneering materials were also surface treated in order to enhance and achieve the highest shear bond strength of the materials to PEKK. In hybrid ceramics, Micromechanical interlocking and chemical bonding considered the two most crucial techniques for creating a reliable connection between cement and ceramics. Micromechanical method is achieved by HF acid application, while chemical bonding is achieved by silane coupling agent. Several studies reported that HF acid enhance bonding to resin content by dissolving the silica containing glassy matrix portion. **Bayındır et al (2020) and Schwenter et al (2016)** claimed that, HF acid etching and silane coupling agent exhibited a favorable increase in shear bond strength values within Vita Enamic¹²⁻¹³.

For lithium disilicate, **Aboushelib et al (2014) and El-Damanhoury et al (2018)** reported that the best method for generating a favorable bond reliability to lithium disilicate glass ceramics is still hydrofluoric acid etching combined with silane priming ¹⁴⁻¹⁵ **Sudré et al (2020)** experienced that, acid concentration and exposure time are a significant factor in surface treatment of lithium disilicate. So 10 % HF acid concentration and exposure time from 20 to 40 second achieved the most suitable results with lithium disilicate¹⁶.

For zirconia specimens, **de Lucena Pereira et al (2015) and Joukhadar et al (2020)** concluded that air borne particle abrasion followed by a primer application showed a favorable increase in bond strength of the resin cement to zirconia. In addition, air abrasions enhance wettability of the primer on zirconia ¹⁷⁻¹⁸.

Several studies focused on the role of application of composite primer (visio.link). The pentaerythritol triacrylate in visio.link was reported to act as solvent, which play a significant effect in enhancing bonding to PEKK. **Stawarczyk et al (2014)** reported that, adhesive system (visio.link) should be applied in order to achieve a durable bond when veneering a PEEK restoration.¹⁹

In this study, we used dual cure self-adhesive resin cement (thera cem) as it contains a hydrophilic monomer (*methacryloxydecyl-dihydrogen-phosphate*). It also had a feature that would help reduce or prevent secondary caries. In addition, it has many advantages such as bond strength and stability, antimicrobial activity, alkaline PH, as well as calcium release.²⁰

Regarding thermo-cycling, the materials in this study were exposed to 10000 cycle. **Morresi AL et al 2014** claimed that, 10000 cycle equivalent to 1 year clinically. Each water bath had a 25-second dwell time, with 10-second lag time. 5°C was the low-temperature point. The maximum temperature was 55°C ²¹. The ISO standard stated that temperatures between 5°C and 55°C were appropriate for testing dental materials since they were thought to be the most closely related value to the physiology of the oral cavity. **Galea MS et al (1999)** reported that, the mean low-temperature point was 6.6°C (range 0–36°C, median 5.0°C). The mean high-temperature point was 55.5°C (range 40– 100°C, median 55°C) ²².

One of the most crucial techniques for determining bond strength is the shear bond strength test (SBS). This test was chosen because to its many benefits, including its non-technique sensitive nature, ease of specimen preparation, straightforward testing methodology, lower frequency of pretest failure, and ease of specimen alignment with loading device.²³

Regardless thermo-cycling, **the results** of vita Enamic and IPS E-max Cad (21.2±2.2&21±1.5 respectively) could be attributed to the type of the surface treatment applied to the tested specimens. Hydrofluoric acid (HF) etching modifies surfaces

of vita enamic and lithium disilicate by partial dissolution of the glassy and crystalline phases of the restorative material. HF acid etching created micro porosities on the surface the restorative material, which in turn increase surface area, and enhance micro-mechanical interlocking with the luting resin cement.²⁴⁻²⁵

Silane is bi-functional monomers that contain two essential groups: a) Silanol group that reacts with ceramic surfaces. b) metha-acrylate group that co-polymerizes with composite organic matrix. silane play a significant role in facilitating the chemical interaction between restorative materials and resin cement. *Cinar et al (2019)* reported that salinization after HF acid etching improved shear bond strength values of Vita Enamic and lithium disilicate.²⁵

On the other hand, BruxZir shaded zirconia showed a marked increase in shear bond strength values (27.4 ± 2) compared with other veneering material.

It was reported that, airborne particle abrasion with 110 μm aluminum oxide revealed a significant increase in shear bond strength values of polyether ketone ketone to veneering materials. Airborne particle abrasions increase surface roughness on the PEKK surfaces and thus enhance its mechanical retention with the suitable adhesive. *Stawarczyk et al (2013) and Gouveia et al (2021)* reported that airborne particle abrasion with 110 μm aluminum oxide showed a significant increase in shear bond strength values of PEKK to the veneering material.²⁶⁻²⁷

In addition, the resin cement (thera cem) contains a hydrophilic monomer (*methacryloxydecyl-dihydrogen-phosphate*). The high shear bond strength values achieved in zirconia could be attributed to the interaction between MDP and zirconia. *Nagoka et al (2017), and Mahrous et al (2020)* outlined three different hypotheses for how MDP and zirconia interact: a) Hydrogen bonding between the P=O (oxo group) and Zr-OH group, which causes the 10-MDP monomer to adsorb on

the surface. (b) Zirconia and the 10-MDP monomer forming an ionic connection.(c) The adsorbed 10-MDP monomers engage with zirconia via P=O hydrogen-bonding interactions in addition to ionic bonding.²⁸⁻²⁹

Shear bond strength showed significant different before and after thermo-cycling with vita Enamic specimens. *Cekic-Nagas et al and Campos et al* reported that resin bonding to hybrid ceramics significantly decreased following thermo-cycling. This could be attributed to penetration of water into small spaces between polymer chains or functional groups owing to small molecular size and high molar concentration of the water. As a result thermal stability of the polymer was decreased, and led to its plasticization. It is possible to say that, Polymer present in the material was unable to endure changes in humidity and temperature.³⁰⁻³¹

The results of IPS E-max Cad specimens experienced a marked decrease in shear bond strength values (6.5 ± 0.8) compared with other two materials (vita enamic and BruxZIR shaded zirconia). This could be attributed to hydrolytic cleavage of siloxane bonds in the siloxane interfacial layer that occur as result of exposure silanized interface to water. In addition there is a significant water sorption owing to exposure of the adhesive with water. As a result, these lead to plasticized polymers and lower their mechanical properties, resulting in lower bond durability.³²⁻³³⁻³⁴

Several studies reported that, the interactions that occur between silane and MDP, which noticeably affect in the bond durability with E max. silane become unstable when combined with bis-GMA and MDP. In the acidic environment induced by MDP, self-condensation reaction may occur in the silane.³²⁻³⁵⁻³⁶

Another possible explanation is that, the more HEMA presented in the resin, the more water absorbed. This action leads to hydrolysis of siloxane and weaken the adhesive bond at the adhesive interface. *Takahashi et al (2011)* reported

that HEMA presented in the adhesives significantly affected by increasing water sorption and decreasing ultimate tensile strength.³⁷

Another possible explanation could be related to the concentration of the 10 MDP presented in the resin cement. Higher concentration of 10 MDP experienced a noticeable increase in water sorption of the adhesive resin cement. *Shibuya et al (2019)* reported that water sorption in the experimental resin cements increased with increase concentration of 10-MDP, which in turn lead to impairment of bond durability.³⁸

Prochnow et al (2018) assigned that, thermo-cycling significantly diminished adhesive resistance of the cement. The negative effect of thermal cycling on adhesion of the resin cement could be attributed to different liner coefficient of thermal expansion of the material which inherent a variable degree of expansion and shrinkage. This process induced a fatigue phenomenon of the material which in turn leads to deterioration (rupture) of the bond and interface.³⁹

The cause for bond deterioration between polyetherketoneketone and lithium disilicate might be due to the unreactive nature of the peek surfaces. This was in accordance with Sloan et al (2021) that attributed the reason for bond disparity between peek and lithium disilicate to the nature of bonding to PEEK substrate. Adhesion to peek substrate was thought to be almost entirely micromechanical.⁴⁰

It was previously mentioned, application of dual cured resin cements and light cured adhesives significantly affected by lowering the bond strength. This diminished bond was attributed to incompatibility between the acidic resin monomer presented in the adhesive and the peroxide amine catalyst infiltrated in the dual cured resin.⁴¹

In recently published data, Mustafa et al (2023) stated that, indirect laboratory composite experienced a significant increase in SBS values compared to lithium disilicate. This could be

attributed to MMA-PETIA content of the Visio.link. The chemical interaction between MMA-PETIA monomer and resin polymer provided a durable and better bonding with composite than other functional polymers.⁴² Based on the aforementioned data, it was expected that, Vita Enamic specimens had the ability to withstand the thermal aging to the extent greater than Ips E-max cad specimens.

The results of this study experienced a significant decrease in shear bond strength (15.3±2.2) after thermo-cycling with zirconia specimens. This was in accordance with Aboushelib et al, and Oyagüe et al who reported that aging was associated with lowering bond strength owing to deterioration of zirconia resin bond⁴³⁻⁴⁴. *BAN (2019)* reported that tetragonal to monoclinic phase transformation was accompanied with a noticeable increase in crystal volume, which in turn leading to development of micro and macro-cracks. They claimed that low temperature degradation based on generation of hydroxyl ions that react with oxygen following adsorption of water on zirconia grain surface.

Several studies focused in the role of water in deterioration of MDP-mediated zirconia resin bond. *Chen et al (2017)* reported that, the surface of the zirconia resin interface was incorporated by water which in turn led to violation of marginal seal. They supported their finding that MDP-based primers did not successfully stop the penetration of the methylene blue dye. Also, it was shown that at least a molecular fragment of MDP was released following water sorption by the presence of leached phosphorous in a soaking solution. So water negatively weakens resin bonding of MDP-conditioned- Y-TZP.

Salem et al (2019) reported that thermo-cycling significantly decrease SBS values of zirconia with all adhesive system used. This could be attributed to the degradation composite resin cement. Moisture absorption caused by thermo-cycling increased the coefficient of thermal expansion and lowered the glass transition temperature.⁴⁵

In Vita Enamic group, predominantly mixed type of failure were observed at disc/ cement interfaces, after they have been subjected to thermocycling. This was in accordance with finding of Moftah et al (2018)⁴⁶ and Beyabanaki (2022)⁴⁷. However, several researches have documented the interactions that occurred between water and the epoxy network upon subjecting the resinous based material to thermocycling. The reaction between positive ions in the filler particles and water causes a hydrolytic degradation which thus alters the charge equilibrium of the silica matrix. The (Si-O-Si) links in the silica matrix disintegrate as more hydrogen ions fill the empty spaces; leading to surface deterioration.⁴¹ Therefore, thermo-cycling negatively influenced the SBS value of vita enamic specimens.

Moreover, adhesive mode of failure was observed in Ips-emax cad specimens. The negative effect of thermal cycling on adhesion could be attributed to the different in liner coefficient of thermal expansion, which in turn led to varying degree of shrinkage and expansion. This phenomena generated a fatigue stresses on the material, leading to disparity of the bond and interface.³⁹ Another factor that might have attributed to the deterioration of the bond is the small dimension of the bonded area, which made the impact of thermocycling on its surface more noticeable. Shono et al ⁴⁸ stated that, adhesive joint in samples with small areas experienced a deterioration of the bond strength after similar periods of storage and thermal aging. In Bruxzir shaded zirconia, the role of water in deterioration of MDP-mediated zirconia resin bond might be the exact reason for adhesive failure associated with thermal aging.⁴³⁻⁴⁴

CONCLUSIONS

Material type and thermo-cycling significantly affected shear bond strength values of the veneering material to polyetherketoneketone (PEKK).

Zirconia specimens experienced a marked increase in shear bond strength values compared

with hybrid ceramics and lithium disilicate regardless thermo-cycling.

Thermo-cycling significantly affected shear bond strength values in all specimens regardless the material type. Vita Enamic specimens was the least veneering materials affected when subjected to thermo-cycling. While Ips- emax cad showed a marked decrease in shear bond strength values.

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