The Effect of Different Surface Treatments on The Shear Bond Strength of Resin Composite Attached to Polymer-Infiltrated Ceramic-Network Material

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Abstract

Dental ceramic materials are subject to damage when in use. A conventional surface treatment used to repair dental material include etching with hydrofluoric acid, sandblasting, and griding by diamond bur. However, those methods face limitations and various difficulties as well as can be hazardous. This study, therefore, aimed to present an experimental research design to compare the shear bond strength between resin composites and polymer-infiltrated ceramic-network material (PICN) using varying surface treatments to simulate the repair of PICN. The PICN blocks were cut for forty specimens and sorted randomly into five groups. The PICN surface was pre-treated with hydrofluoric acid (HF) and self-etched ceramic primer, followed by both the application of silane and forgoing silane. In the next step, the resin composite was bonded to the PICN surface using a putty index. The specimens were tested for their shear bond strength. Data were analyzed using One-Way ANOVA and Tukey's Test ($\alpha = 0.05$). Modes of Failure were examined under a stereomicroscope at ×40 magnification. The surface prior to silane application was examined using a scanning electron microscope. The PICN surface that was treated with HF followed by silane yielded the best results because HF etched the surface greater than MEP and silane had a chemical reaction with the PICN, resulting in a greater bond. However, there was no significant difference between the MEP and the MEP followed by silane ($p < 0.05$). All groups undergoing surface treatment yielded optimal repair bond strength. Hydrofluoric acid followed by silane is still the gold standard to repair the PICN but self-etching ceramic primer offers another good alternative.

Keywords: *hydrofluoric acid, polymer-infiltrated ceramic-network material, PICN, repair ceramic, shear bond strength, self-etching ceramic primer*

1. Introduction

Nowadays, ceramic is a popular material for dentistry. As a result, there are a variety of ceramics used in the industry. One novel method is categorizing ceramic restorative materials into three groups, namely 1) Glass-Matrix Ceramics, 2) Polycrystalline Ceramics, and 3) Resin-Matrix Ceramics. Resin-Matrix Ceramics could be further divided into three categories, namely 1) Resin Nanoceramics such as Lava Ultimate, 2) Polymer-infiltrated ceramic-network materials such as Vita Enamic, and 3) Zirconia-Silica Ceramics in a resin interpenetrating matrix such as Shofu Block HC (Gracis et al., 2015; Mainjot et al., 2016). Polymer-infiltrated ceramic-network material (PICN) is the ceramic network formed from 3-dimensional scaffolds of sintered ceramics. Other resin-matrix ceramics are composed of composite blocks with dispersed filler materials (Gracis et al., 2015; Lambert, Hugo, Durand, Jacquot, & Fages.. 2017). Resin-matrix ceramics, when compared with typical ceramics, better resemble the modulus of elasticity of dentin. Accordingly, they are easy to mill and adjust. Besides, a resin matrix can facilitate the repair through the use of composite resin (Dirxen, Blunck, & Preissner 2013; Gracis et al., 2015).

Restoration fractures or damage are typically caused by trauma or exhaustion from masticatory activity. Repairs using a composite resin are less expensive and less invasive compared with a replacement. Furthermore, repair procedures are easier and take less time (Federation 2017; Marquillier et al., 2018). Repaired ceramic surfaces can be treated with different methods such as sandblasting with aluminum oxide, acid etching with hydrofluoric, or roughening with diamond burs. Then, one can apply a primer composed of silane to the ceramic surface, followed by the adhesive and resin composite (Reston et al., 2008). However, intraoral sandblasting requires the use of a specific device. During sandblasting, the control pressure, time, distance, and size of the aluminum oxide must be tightly controlled. It is important to use caution while sandblasting since improper sandblasting might result in microcracks (Tekçe, Tuncer, & Demirci 2018; Yoshihara et al., 2017).

The conventional technique for treating ceramic surfaces is etching with hydrofluoric acid followed by the application of silane. Hydrofluoric acid improves bond strength through chemical reactions. Hydrofluoric acid etching not only creates a micromechanically retentive surface but also stimulates the production of hydroxyl groups on the ceramic surface (Matinlinna & Vallittu, 2007). Silane reacts with a hydroxyl group on the ceramic surface, promoting bond strength. In addition, the silanization of the ceramic surface after the surface treatment increased the bond strength. According to research, combining the methods of etching with hydrofluoric acid and the application of silane provided an optimal bond strength(Kalavacharla, Vamsi, Lawson, Ramp, & Burgess, 2015; Vargas, Marcos, Bergeron, & Arnold 2011; Elsaka & Shaymaa, 2014).

However, hydrofluoric acid is a harmful inorganic acid, and it can cause severe burns. Therefore, one needs to exercise proper caution in using hydrofluoric acid in the oral cavity (Bertolini, 1992). A ceramic primer with self-etching properties (Monobond Etch & Prime, Ivoclar Vivadent) was launched as a single-component ceramic primer and was considered an alternative treatment to the common usage of hydrofluoric acid etching/silane. The self-etch ceramic primer creates a roughness pattern but eliminates the toxicity of hydrofluoric acid and salinization occurs in one step. This technique is safer and less time-intensive and produces less sensitivity during the surface treatment when compared with the conventional method (Thomas, Volkel, & Erik., 2015).

Some studies report that a self-etching ceramic primer creates a similar bond strength between ceramic and resin cement to the conventional method (Alshihri, & Abdulmonem. 2019; Donmez, Mustafa, Okutan, & Yucel, 2020). However, according to some other research, conventional treatment resulted in stronger bond strengths than a self-etching ceramic primer (Damanhoury, Hatem, & Gaintantzopoulou, 2018; Levartovsky et al., 2021). Uses of self-etching ceramic primer combine sandblasting and adhesive bonding to create higher bond strength (Yildirim, Recen, and Paken 2019; Guimarães et al. 2018). Few studies are using a self-etching ceramic primer between a repaired lithium disilicate and glass-ceramic with a resin composite (Ueda et al., 2021; Yildirim, Recen, & Paken 2019) while no prior research studies the bond strength of self-etching ceramic primer repairing resin-matrix. For this reason, this paper explores this topic in further detail.

2. Objectives

1) To compare the shear bond strength of resin composite attached to polymer-infiltrated ceramic-network material (PICN) using varying surface treatments

2) To compare the surface characteristics of PICN subjected to varying surface treatment

The hypothesis was that there is no difference in shear bond strengths of resin composite attached with PICN using varying surface treatments.

3. Materials and Methods

3.1 Specimen Preparation

Vita Enamic shade 2M2-HT (VITA Zahnfabrik, Bad Säckingen, Germany) blocks were used in this study. A total of forty specimens were cut by a low-speed cutting machine (ISOMET 1000, Buehler, Illinois, USA) under water irrigation, to a size of 5 mm x 5 mm x 3 mm. The specimens were embedded into acrylic resin (Unifast TRAD, GC Dental Products Corp, Japan) in a rigid polyvinyl chloride (PVC) tube with a diameter of 22.0 mm and height of 15.0 mm, then left for 24 hours, as shown in Figure 1. The specimens' test areas were polished with waterproof abrasive paper (DCC; TOA Paint Co., Thailand) 600, 800, 1,000, and 2,000, respectively, using a grinding and polishing machine (Nano 100T Grinder-polisher, PACE Technologies, Thailand) at 100 rpm counterclockwise for 5 minutes under each roughness. After polishing, the specimens were soaked in distilled water and cleaned using an ultrasonic cleaning machine

(L&R Ultrasonics Quantrex 650, L&R Ultrasonics, New Jersey, USA) for 5 minutes to eliminate any surface debris, then dried by blowing oil-free air at them.

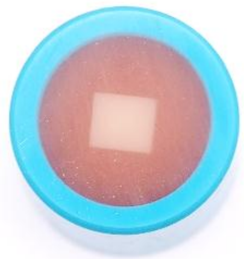

Figure1 The specimens embedded into acrylic resin

Table 1 Materials Used in This Study

Material	Manufacturer	Composition
Vita Enamic	VITA Zahnfabrik, Bad Säckingen, Germany	polymer network (14 wt%): UDMA, TEGDMA feldspar ceramic network (86 wt%): SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO
IPS Ceramic Etching Gel	Ivoclar Vivadent, Schaan, Liechtenstein	5% hydrofluoric acid, water
RelyX Ceramic Primer	3M ESPE, St.Paul, USA	ethyl alcohol, water, 3-methacryloxypropyltrimethoxysilane
Monobond Etch & Prime	Ivoclar Vivadent, Schaan, Liechtenstein	butanol,trimethoxysilylpropyl methacrylate bis (triethoxysilyl) ethane, tetrabutylammoniun dihydrogen trifuoride,methacrylated phosphoric acid ester, colorant
Adper Scotchbond Multipurpose Adhesive	3M ESPE, St.Paul, USA	bisphenol A diglycidyl ether dimethacrylate (BISGMA) 2-hydroxyethyl methacrylate (HEMA)
Filtek-z350 (Nanofill)	3M ESPE, St.Paul, USA	matrix: Bis-GMA, UDMA, Bis-EMA, TEGDMA filler: SiO_2 nanofiller,ZrO_2 nanofiller,ZrO_2/ SiO_2 nanocluster

3.2 PICN Surface Treatment

Forty specimens were randomly selected and divided into 5 groups, each consisting of 8 specimens whose surfaces would be prepared according to each test group. Material compositions are shown in Table 1. They were listed as 1) Control (n = 8): No treatment, 2) HF (n = 8): The PICN (Vita Enamic) surface was etched using 5% HF gel (IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein) for 60 seconds, rinsed with a strong water spray for 60 seconds, and dried using oil-free air for 10 seconds until dry, 3) HFS (n = 8): The PICN surface was etched using 5% HF gel (IPS Ceramic Etching Gel) according to the protocol above, then applied RelyX™ Ceramic Primer (3M ESPE, St. Paul, USA), blown until no further material movement, 4) MEP (n = 8): Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein) was applied onto the PICN surface with a microbrush and agitated for 20 seconds, left for the reaction to take place for 40 seconds, then washed off with a strong jet of water spray, and dried with air for another 10 seconds, 5) MEPS (n = 8): Monobond Etch & Prime applied according to the protocol above, then the RelyX™ Ceramic Primer applied, and then blown until no further material movement was observable.

3.3 Scanning Electron Microscope (SEM)

Specimens were prepared according to control/HF/MEP group, 1 piece per group, with the surface prepared according to the methodology in the previous section, and ultrasonically cleaned in distilled water for 5 minutes. Gold was then coated at the thickness of 20 µm to the test area by an Auto Fine Coater (JFC-1600, JEOL Inc., Tokyo, Japan). A scanning electron microscope (JEOL-6510 LV, JEOL Inc., Tokyo, Japan) was used to observe the results. The microstructures were investigated by scanning electron microscope under ×1,000 and ×3,000 magnifications, under accelerating voltage of 15 kV, and at a distance of 13-15 mm.

3.4 Composite Cylinders Production

After the preparation was completed, Adper Scotchbond Multipurpose Adhesive (3M ESPE, St. Paul, USA) was applied using a microbrush, then blown until no further material movement could be observed. A light cure was applied for polymerization for 20 seconds at the intensity of 1000 mW/cm^2. A hollow silicone mold with a hole of a diameter of 2.0 mm and a depth of 2.0 mm was prepared and placed on the top of the treated PICN specimen, paying special attention that the surface must be aligned with the PICN's surface. The resin composite shade A1 (Filtek-z350, 3M ESPE, St. Paul, USA) was used on the silicone mold until it was full as shown in Figure 2. A light cure was applied for polymerization for 40 seconds at an intensity of 1000 mW/cm^2. The mold was then carefully removed, and an additional light cure was applied for 40 seconds. Each silicone mold was cleaned with ethyl alcohol and oil-free air was blown between the specimens until dry. Specimens were soaked in 37-degree Celsius distilled water for 24 hours in an incubator.

Figure 2 Demonstrating the application of resin composite into silicone mold

3.5 Shear Bond Strength Test

The shear bonds of specimens were tested in a universal testing machine (universal testing machine: EZ Test Series, Shimadzu, Japan) with a crosshead speed of 0.5 mm/min. The specimens were mounted on the machine while the loading unit was set at the same level and parallel to the material interface as shown in Figure 3. The maximum fracture load was recorded in newton and divided by the surface area in mm^2 to calculate the shear bond strength in MPa.

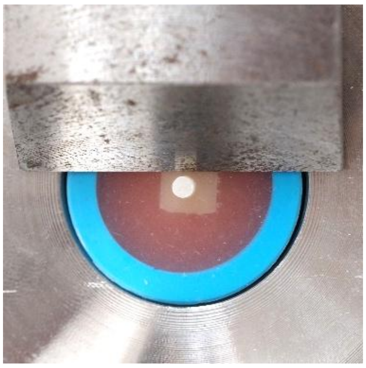

Figure 3 shear bond strength test

3.6 Modes of Failure

The de-bonded specimens were examined under a stereomicroscope (JEOL-6510 LV, JEOL Inc., Tokyo, Japan) at ×40 magnification to determine the failure modes.

The modes of failure could be categorized into 4 types (Damanhoury et al., 2018; Kraisintu et al.,2019)

Type 1: Adhesive Failure, No resin composite on the PICN

Type 2: Cohesive Failure in resin composite, Resin composite covers all surfaces of PICN

Type 3: Cohesive Failure in PICN, PICN covers all surfaces of resin composite

Type 4: Mixed Failure or A Combination of Adhesive and Cohesive Failure, mixed failure with the combination of all types of failures

3.7 Statistical Analysis

The analysis was done using IBM SPSS Statistics 21.0 software for Windows, a test of normality (normal distribution) was conducted by Kolmogorov-Smirnov test, and a test of homogeneity of variance was conducted by Levene's test. For the test of normality and homogeneity of variance, One-Way ANOVA was used and an in-group analysis was done through multiple comparisons with Tukey's test ($\alpha = 0.05$).

4 Results and Discussion

4.1 Results

The shear bond strengths were normally distributed and had homogeneity of variance. Mean and standard deviations of shear bond strength were reported in Table 2 and Figure 4.

Table 2 Mean and standard deviations of shear bond strength for each group (n=8)

Surface Treatments	Mean ± SD (MPa)
Control	5.83 ± 0.86^{A}
HF	16.99 ± 1.66^{C}
HFS	20.29 ± 1.30^{D}
MEP	14.96 ± 1.19^{B}
MEPS	15.07 ± 0.87^{B}

The same superscript indicates no significant difference (Tukey's test, α<0.05)

The results showed significant differences in shear bond strength values between different surface treatments ($p < 0.0001$). The HFS group had the highest mean SBS value (20.29 ± 1.30 MPa), followed by the HF group (16.99 ± 1.66 MPa), the MEPS group (15.07 ± 0.87 MPa), the MEP group (14.96 ± 1.19 MPa), and finally the Control group (5.83 ± 0.86 MPa). However, there was no significant difference between the MEP and MEPS ($p < 0.05$).

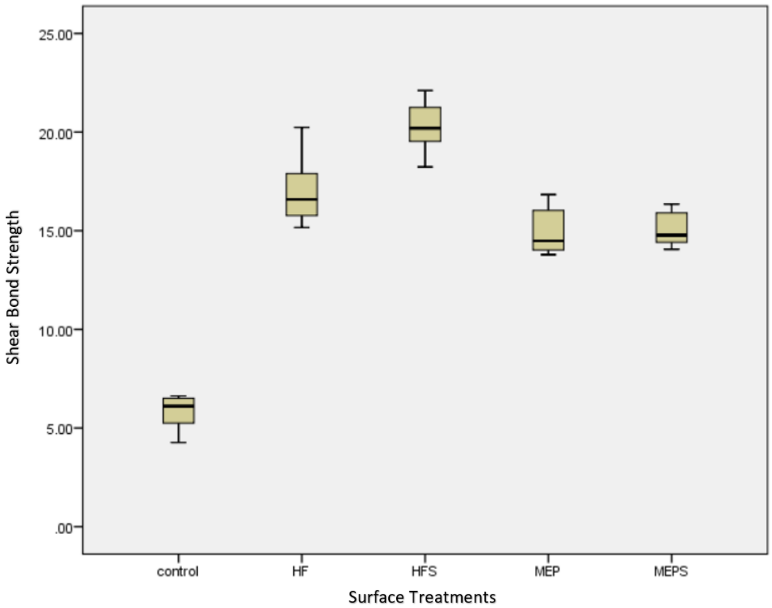

Figure 4 Shear Bond Strength Boxplot for Each Surface Treatment

Table 3 Failure types for each surface treatment

Surface Treatments	Failure types% (n=8)			
	Adhesive	**Cohesive(composite)**	**Cohesive (PICN)**	**Mixed**
Control	100	-	-	-
HF	25	-	-	75
HFS	62.5	-	-	37.5
MEP	75	-	-	25
MEPS	62.5	-	-	37.5

Failure types are shown in Table 3. The control groups demonstrated a 100% adhesive failure at the PICN and composite interface. In comparison, the failure analysis of all surface-treated groups revealed adhesive and mixed failures: only a combination of adhesive and cohesive failure in resin composite is observed, as shown in Figure 2.

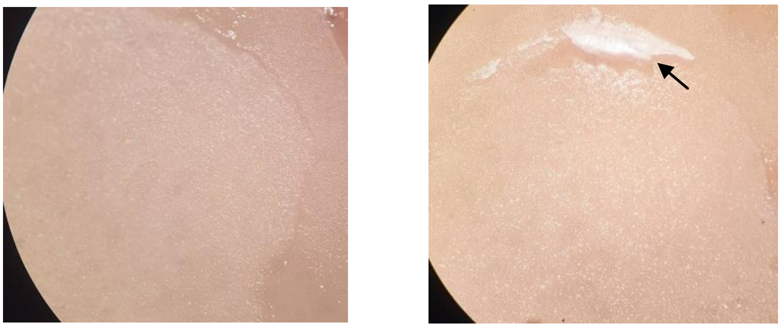

Figure 2 The stereomicroscope images at ×40 Magnification Represent Modes of Failure (a) adhesive failure and (b) mixed failure (black arrow presents resin composite)

The SEM images at ×1000 and ×3000 magnification of the sample from each surface treatment group are presented in Figures 3 and 4. The non-surface treated group showed the ceramic network with a 3-dimensional scaffold of sintered ceramic, which formed a real skeleton. The group which was etched with HF for 60 seconds resulted in a honeycomb-like structure with a porous surface. In comparison, the MEP generated a less porous surface than the hydrofluoric acid.

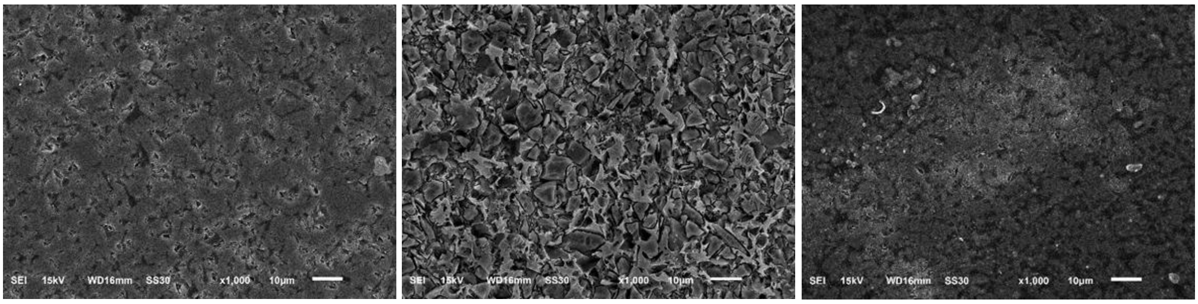

Figure 3 The scanning electron microscope images of the Vita Enamic with different surface treatments at ×1000 (a) no treatment, (b) Hydrofluoric 60 seconds, and (c) Monobond etch and prime 60 seconds

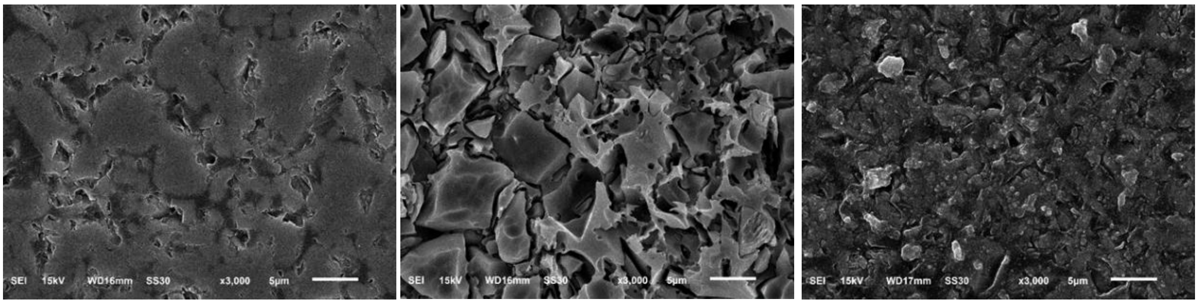

Figure 4 The scanning electron microscope images of the Vita Enamic with different surface treatments at ×3000: (a) no treatment, (b) Hydrofluoric 60 seconds, and (c) Monobond etch and prime 60 seconds

4.2 Discussion

This study offers an experimental research design to study the shear bond strength between PICN and resin composite. The surfaces were prepared by applying HF, with silane or without silane. A self-etching ceramic primer, MEP, was also applied, with or without silane. The modes of failure were then examined with a stereomicroscope. The surfaces to which HF and MEP were applied were examined before applying silane using SEM.

The study found that there is a significant difference in the shear bond strength of the resin composite attached to PICN by various surface treatments. Besides, HF and HFS provided a better bond strength compared with MEP and MEPS. In Vita Enamic (PICN), HF is more corrosive than MEP, resulting in better micromechanical interlocking that corresponded to SEM Photo. The SEM Photo showed that HF extremely promoted irregularity and a porous surface for Vita Enamic, better than MEP. Also, El-Damanhoury and Gaintantzopoulou (2018) presented that surface roughness generated from HF was more than MEP. Vita Enamic is composed of a feldspathic ceramic network, which has 58-60 % SiO_2 (Gracis et al., 2015; Lambert et al., 2017; Mainjot, Dupont, Oudkerk, Dewael, & Sadoun, 2016). HF reacts with the matrix that contains the silica, resulting in silicon tetrafluoride (SiF_4). Silicon tetrafluoride reacts with HF, resulting in hexafluorosilicate (SiF_6). Finally, hexafluorosilicate reacts with protons, resulting in tetrafluorosilicic acid (H_2SiF_6) that could be washed away by water (J. Matinlinna & Vallittu, 2007). The reaction generates a micromechanically retentive surface and promotes hydroxyl group formation on the ceramic surface(Vargas et al., 2011). However, HF is highly hazardous and toxic (Bertolini & John, 1992; Wang et al., 2014). Therefore, self-etching ceramic primer or MEP, which combines etching and silane into one step, was invented. In contrast, MEP which contains ammonium polyfluoride as an etching acid and trimethoxypropyl methacrylate as silane demonstrated less effectiveness to etch the ceramic surface than HF. This result could be confirmed by an SEM image of MEP that showed less roughness than other conditioning methods (Thomas et al., 2015). There still is no comprehensive study that clarifies the reaction process. The surface etched by MEP was less than HF, according to the SEM images in this study, which is consistent with prior studies (Alshihri & Abdulmonem, 2019; Donmez et al., 2020; Levartovsky et al., 2021; Lyann et al., 2018).

HFS provided a better bond strength, compared to HF, which corresponds to various studies stating that ceramics with sandblasted or etched surfaces with silane resulted in better bond strength than those without silane. The reason is that silanization improves the surface energy and chemical bond (Brentel et al., 2007; Elsaka & Shaymaa, 2014; Flury, Simon, Dulla, & Peutzfeldt, 2019). Silane is bondable with silica, glass, and quartz. First, silane is activated by an acidic catalyst to become silanol (SiOH). Silanol will be attached to an inorganic surface. After the etching reaction, a hydroxyl group (-OH) is produced. The hydroxyl group will react with silanol, resulting in siloxane (-Si-O-Si-) (Matinlinna & Vallittu, 2007; Matinlinna, Pekka, Lung, & Tsoi, 2018). Many studies found that HF followed by the application of a silane provided the gold standard bonding strength for surface preparation of ceramics with glass composite (Kalavacharla et al., 2015; Miranda et al., 2020; Vargas et al., 2011), which corresponded to the product manual of Vita Enamic that recommended an application of 5% HF to the inner surface for 60 seconds and followed by an application of silane to the etched surfaces. However, in this study, it was found that using MEP and MEP followed by silane provided equivalent bond strength since MEP and silane both contain the same trimethoxypropyl methacrylate that can react with glass-ceramics (Thomas et al., 2015; Matinlinna & Vallittu, 2007). Therefore, additional silane did not increase the bond strength after surface treatment with MEP.

Furthermore, in this study, PICN with MEP surface attached to the resin composite provided less shear bond strength compared with the HF surface, which corresponded to the study that the repair of feldspathic glass-matrix ceramic by MEP produced less shear bond strength when compared with HF (Yildirim et al., 2019). The result also corresponded to various other studies stating that MEP provided less bond strength between ceramic and resin cement than the conventional method (Damanhoury et al., 2018; Levartovsky et al., 2021). However, some research stated that the bond strength is equivalent to the conventional method (Alshihri & Abdulmonem, 2019; Donmez et al., 2020). Therefore, using MEP to repair ceramic remains a controversial issue. Although the repair bond strength was not clearly stated, the repair bond strengths referred from the other research were around 10-20 MPa (Kiomarsi et al., 2017; Matsumura, et al., 2001; Teixeira, Bayne, et al., 2005). All groups' surface preparations, namely HF, HFS, MEP, and MEPS, were greater than the control and were in the acceptable range to repair the bond strength. Furthermore, it was found that various surface treatments resulted in more mixed failure modes. From this experiment, repairing PICN with HF followed by silane gave the best bond strength. Nonetheless, MEP was still a good alternative, which means that further study is required. In the next study, thermocycling is recommended to simulate long-term oral usage.

5. Conclusion

From the findings of this present study, the shear bond strength could be respectively ordered as HFS > HF > MEP = MEPS > control. The result also conformed to the SEM image that the surface irregularity and porosity created by HF were more than MEP. Therefore, Hydrofluoric acid followed by silane is still the gold standard for the repair of PICN. Self-etching ceramic primer remains an alternative with clinically acceptable to repair the PICN without any other necessary silane.

6. Acknowledgement

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