



Effect of Different Surface Treatments on Shear Bond Strength of Repaired Hybrid Ceramic

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Abstract

Hybrid ceramic is a polymer-infiltrated ceramic network (PICN) that has been widely used in recent years due to its esthetic properties and biocompatibility. Moreover, the possibility of less invasive restorations. Achieving higher bond strength in ceramic restoration is required in mechanical and chemical approaches. In case repaired hybrid ceramic is needed, resin composite is a material of choice because it can be directly repaired in the oral cavity and is tooth-like color. Surface treatment is recommended to provide better bond strength. This study compares the effect of different surface treatments on the shear bond strength of repaired hybrid ceramic with resin composite. Forty specimens (6x11x2 mm) were prepared from VITA Enamic. All specimens were divided randomly into 4 surface treatment subgroups (n=10). Group 1: Control group, Group 2(HF): Etching with 5% hydrofluoric acid for 60 seconds, Group3(SB): Sandblasting at specimen surface for 15 seconds, Group 4(SC): Grinding with sandpaper grit 120 for 4 seconds. Then, all groups were applied with a silane coupling agent followed by placing a resin composite on the treated specimen surface using a metal split mold and then storing all specimens at 37 °C for 24 hours. The specimens were tested for shear bond strength using a Universal testing machine and analyzing the failure mode with a stereomicroscope. The mean shear bond strength data were analyzed using One-way ANOVA followed by Tukey's test at a confident level of 95%. The result revealed that the HF group promoted the highest bond values while the control group showed the least values of shear bond strength. There were no significant differences between HF and SB groups or SB and SC groups.

Keywords: Shear strength, VITA Enamic, Surface treatment

1. Introduction

In recent years, all-ceramic restorations have been highly utilized in dentistry due to their biocompatibility, high mechanical properties, and optimal esthetics. Resin composites are also used to restore teeth for an esthetic reason, but they also have disadvantages. Ceramic properties are high in flexural strength and great in color stability. However, it causes antagonistic tooth wear and extensive loss of tooth structure because it requires a 1.5-2.0 mm minimum in thickness (Bajraktarova-Valjakova et al., 2018). Resin composite is easy to wear and does not cause antagonistic tooth wear. Afterward, there was a development of a material that combined the advantages of both ceramic and resin composite. The material was named "resin-matrix ceramic" and classified as one type of ceramic. Resin-matrix ceramic was classified into polymer infiltrated ceramic network (PICN) and resin-nano ceramic (Ceren et al., 2016).

This study was based on a polymer infiltrated ceramic network (PICN) consisting of ceramic and resin composite advantages. The compositions of PICN are feldspar ceramic 86%(wt) and dimethacrylate polymer 14%(wt). So, this type of ceramic has lower brittleness than conventional ceramic and has better abrasion behavior than resin composite. However, all materials have their lifespan. Besides replacing the complete prosthesis, repairing some parts should be considered. In the case of repairing hybrid ceramic, resin composite is the material of choice because of its ability to directly repair in the oral cavity, mechanical properties, and tooth-like color.

Surface treatment is recommended before ceramic repair with resin composite to provide better bond strength (Wahsh & Ghallab, 2015). Since resin composite bonding relies on micromechanical interlocking and chemical bonding, roughening and cleaning the treatment surface is crucial (Pirat et al., 2019). Various methods of preparing the surface by mechanical bonding are available regarding advancing

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adhesive technology. Some techniques are hydrofluoric acid etching, tribochemical silica coating, and grinding with silicon carbide paper (Blatz et al., 2003). Chemical bonding was achieved from silane coupling agents and adhesive. Silane coupling agents have been applied as adhesion promoters (Lung & Matinlinna, 2012). It contains two different reactive functional groups: the methacrylates group that reacts with resin composite and other reactive compositions toward silica in ceramic structures (Matinlinna et al., 2004).

The adhesive plays an important role in bonding between different interfaces. A Single Bond Universal adhesive is commonly used due to its easy application and it can be applied to various materials. Also, it has silane in its composition. One study found that using Single Bond Universal adhesive with silane on ceramic samples achieves higher bond strength than applying silane solely (Thong-ngarm & Cheewakriengkrai, 2016). There are various surface treatments tested on PICN, but the results are conflicting (Campos et al., 2016, Silva et al., 2018). Therefore, this study investigates which surface treatments are appropriate for repairing PICN with resin composite.

2. Objectives

To evaluate the effect of different surface treatments on shear bond strength of repaired hybrid ceramic with resin composite

3. Materials and Methods

Forty specimens (size 6x11x2 mm) were prepared by using a hybrid ceramic block (VITA ENAMIC[®]). All specimens were cut using a water-cooled diamond blade (Diamond Wafering blade, Buehler, USA) with a low-speed cutting saw (Isomet machine, Buehler, USA). Each specimen was fixed in a PVC mold (diameter 10 mm x height 10 mm) with self-cure acrylic resin (Formatray). All specimens were polished with silicon carbide paper grit 400, 600, 800, 1000, and 1200 for one minute with a polishing machine until the surface area was flat and smooth (Figure 1).

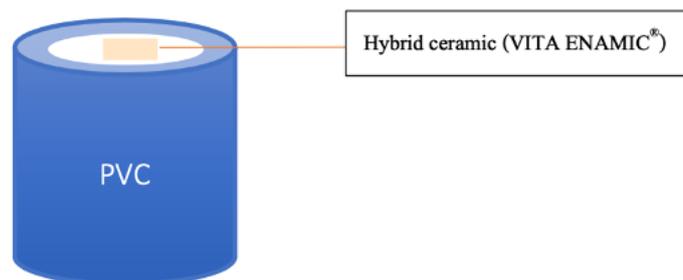


Figure 1 VITA ENAMIC[®] size 6x11x2 mm in the PVC mold

Each specimen was cleaned using an ultrasonic cleaner for 5 minutes in distilled water and air-dried before applying clear plastic tape that was punched 4 mm in diameter centrally (Figure 2).

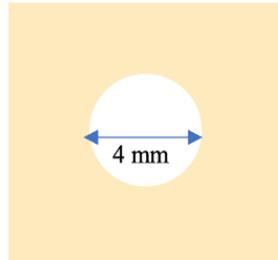


Figure 2 Area on a ceramic block of clear plastic tape punched centrally 4 mm in diameter

All Specimens were randomly divided into four surface treatment subgroups (n=10) (Figure 3):

Group 1 (control group): No surface treatment

Group 2 (HF): 5% Hydrofluoric acid was applied to the ceramic surface for 60 seconds with a micro brush, and rinsed with distilled water for 60 seconds, then air-dried.

Group 3 (SB): Sandblast air abraded with 50 μm Al_2O_3 particles for 15 seconds from a 10 mm distance with blasting pressure 0.2-0.3 MPa perpendicularly to the specimen surface then blew the extra sand off the restoration and ultrasonic for 5 minutes.

Group 4 (SC): Grinding with silicon carbide paper grit 120 under copious air and water irrigation in one direction for 4 seconds on each surface using a polishing machine, then rinsed with distilled water for 15 seconds, then air-dried

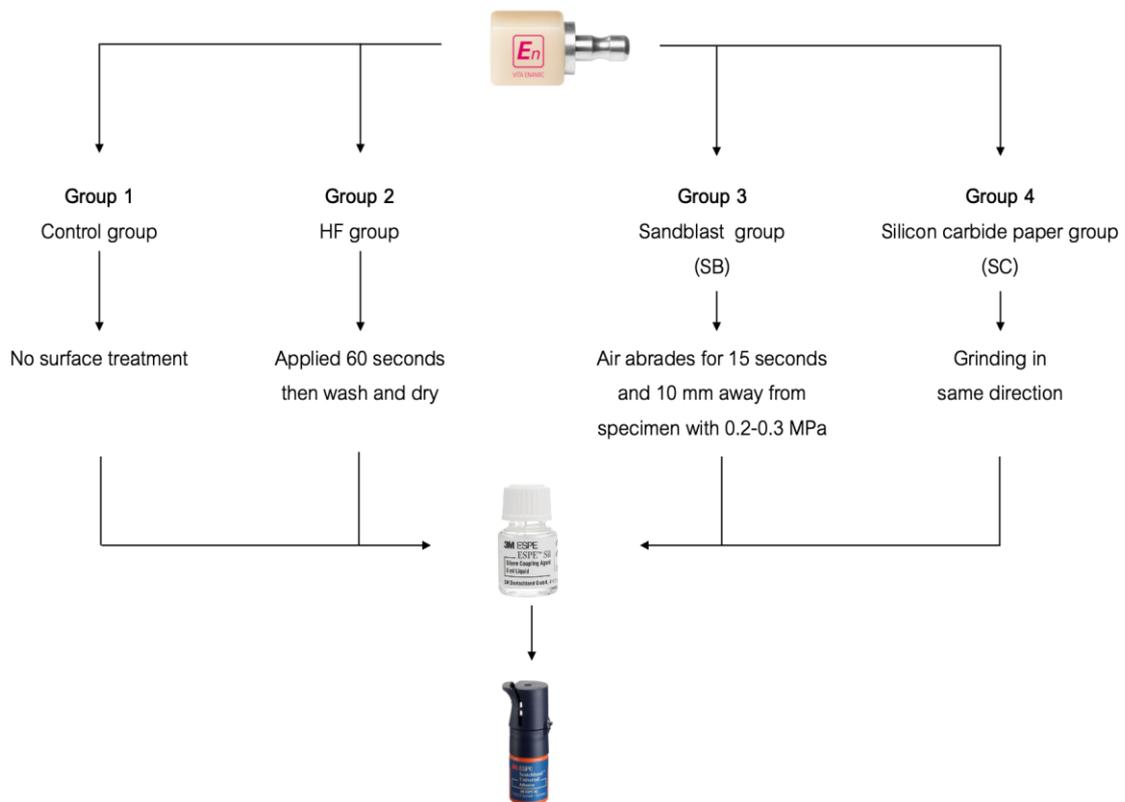


Figure 3 Flow chart of different surface treatment groups



The brands, manufacturers, chemical compositions, and manufacturers of the materials used in this study are listed in Table 1.

All subgroups were applied with hydrolyzed silane for 60 seconds and lightly air-dried. Then, the adhesive materials (Single Bond Universal Adhesive, 3M™) are applied for 20 seconds, air-dried for 5 seconds, and light-cured for 20 seconds using an LED light-curing unit (Demi™ Plus, Kerr). Finally, the resin composite (Filtek™ Z350 XT, 3M ESPE) was filled on the treated surface using a metal split mold (size 2x4 mm) to standardize the dimension of the composite and light-cured for 40 seconds (Figure 4).

Table 1 Materials used in this study

Material	Composition	Manufacturer
VITA ENAMIC®	Shade 3M2: 86 wt% feldspar ceramic, 14 wt% polymer (UDMA, TEGDMA)	Vita Zahnfabrik, Bad Sackingen, Germany
3M™ Silane Coupling Agent	Stabilized 97-100% ethyl alcohol and 1-3% MPS	3M™ ESPE™, St. Paul, MN, USA
3M™ Single Bond Universal Adhesive	MDP Phosphate Monomer, DMA, HEMA, Filler, Ethanol, Water, Initiators silane	3M™ ESPE™, St. Paul, MN, USA
Filtek™ Z350 XT	Shade A1: Bis-GMA, UDMA, Bis-EMA, ZrO ₂ / SiO ₂	3M™ ESPE™, St. Paul, MN, USA
Hydrofluoric acid	5% buffered hydrofluoric acid	Vita Zahnfabrik, Bad Sackingen, Germany
Sandblast	50 μm Al ₂ O ₃ airborne particles	RENFERT, Thailand

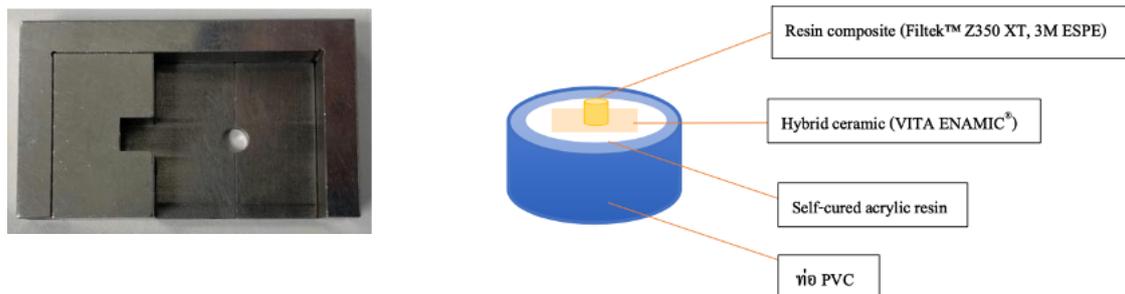


Figure 4 Resin composite (Filtek™ Z350 XT, 3M ESPE) size 2x4 mm filled on the treated surface using a metal split mold

All bonded specimens were stored in distilled water at 37°C for 24 hours before shear bond strength testing with a universal testing machine (EZ-S, SHIMADZU, Japan) (Figure 5). The interface between the VITA ENAMIC® specimens and resin composite was loaded by force with a crosshead speed of 0.5 mm/min until fracture occurred. Then, shear bond strength was recorded in MPa.

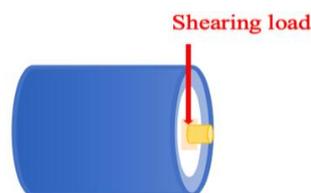


Figure 5 Shear bond strength test



Stereomicroscope (Olympus corp., Tokyo, Japan) was used to analyze failure mode. Then, it was evaluated by Scanning electron microscopy (SEM) (JEOL Ltd, Tokyo, Japan) with 100, 500, and 2000 magnification to study the surface of hybrid ceramic on various kinds of treatments after the failure of bonding. The failure mode was classified as follows:

- 1) Adhesive failure: The failure occurred between resin composite and ceramic.
- 2) Cohesive failure: The failure occurred within resin composite and resin composite or ceramic and ceramic.
- 3) Mixed failure: The failure occurred in both ceramic or resin composite and the interface.

All statistical analyses were performed using SPSS program version 26.0 (SPSS INC, Chicago, IL, USA). Shear bond strength (MPa) data were submitted to the Shapiro-Wilk test to check the normal distribution of the data. Normal distribution was found in all subgroups. One-way ANOVA followed by Tukey's test was performed to analyze the differences between groups. In all tests, the level of confidence was set at 95%

4. Results and Discussion

4.1 Results

The mean and standard deviation of shear bond strength values of different surface treatments to repaired hybrid composite are summarized in Table 2. The lowest bond strength values were found in the control groups (5.80 MPa). The highest bond strength values were found in HF (13.00 MPa). One-way ANOVA and Post hoc Tukey's HSD test showed that the mean shear bond strength of HF and SB was significantly higher than control groups and SC. Still, there was no significant difference between HF and SB or SB and SC (Table 2).

Table 2 Mean shear bond strength (MPa) \pm SD of the tested group

Tested group	Mean shear bond strength (MPa) \pm SD
Group 1: Control	5.80 \pm 1.60 ^C
Group 2: HF	13.00 \pm 3.09 ^A
Group 3: SB	10.85 \pm 2.14 ^{AB}
Group 4: SC	10.18 \pm 2.10 ^B

Values are mean \pm standard deviations. A group with the same uppercase superscripts is not significantly different ($p > 0.05$)

After testing shear bond strength, all specimens' failure mode was evaluated using a stereomicroscope. The failure modes were classified into 3 types: adhesive failure (between ceramic and resin composite), cohesive failure of the ceramic, and mixed (adhesive and cohesive).

In all test groups, the predominant failure mode was mixed failure except in the control group (Figure 6). There was adhesive and mixed failure seen in the HF group, SB, and SC groups.

One sample from each group was examined under a scanning electron microscope (SEM) to determine the treatment surface at magnifications of 40x and 2000x, respectively (Figure 7 and Figure 8). The HF, SB, and SC groups showed surface irregularities with several distributed micropores and gaps. In contrast, the control group showed only micropores.

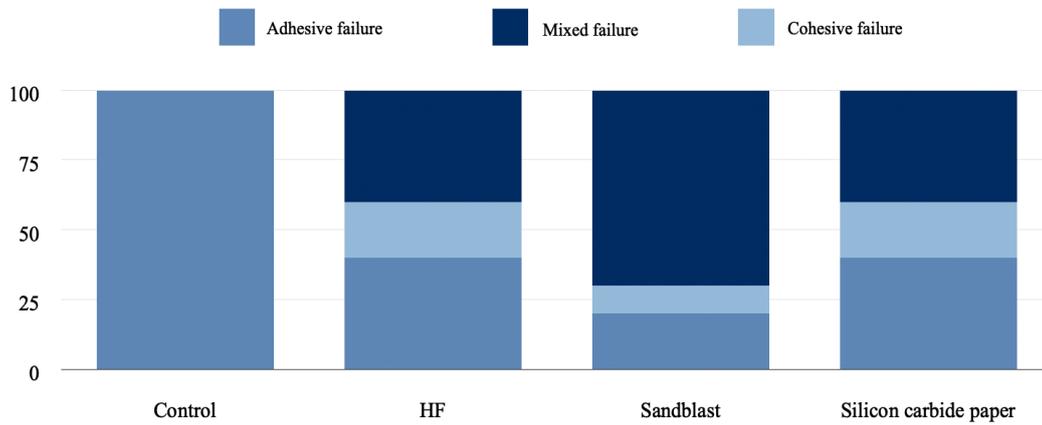


Figure 6 The percentage of the mode of failure in the tested group

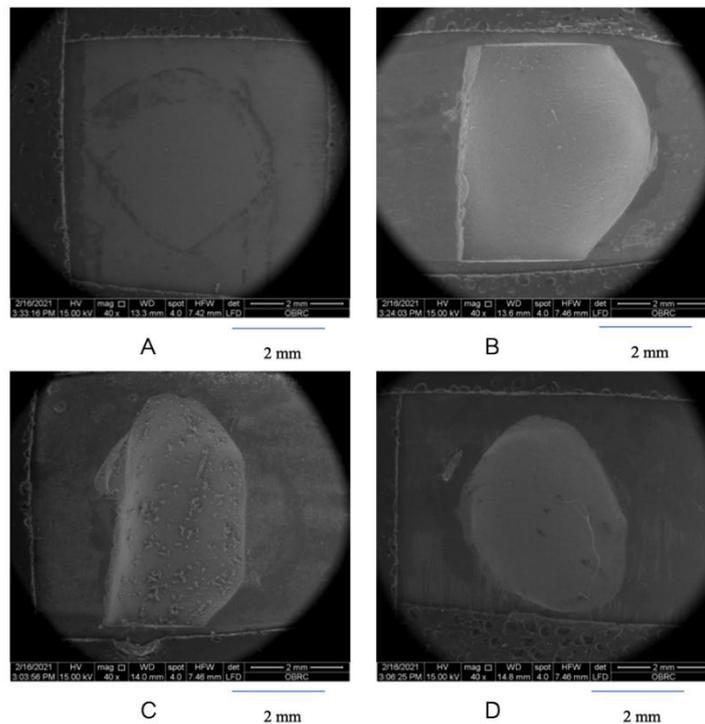


Figure 7 SEM micrographs of representative hybrid ceramic surfaces.

A: control, B: HF, C: SB, D: SC (At magnification 40X)

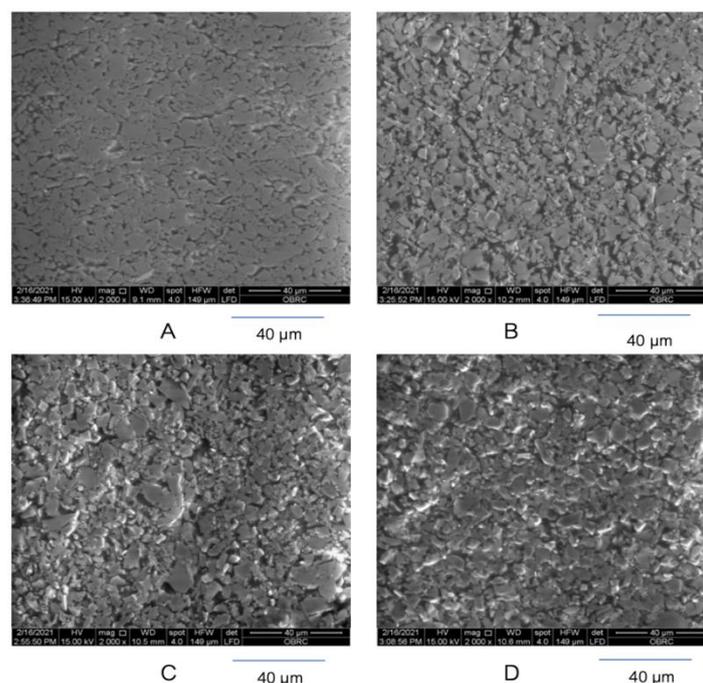


Figure 8 SEM micrographs of representative hybrid ceramic surfaces after surface treatment.
A: control, B: HF, C: SB, D: SC (At magnification 2000X)

4.2 Discussion

This study examined the impact of different surface treatments after 24 hours of storage in distilled water. It demonstrated that surface treatment by HF promoted the highest bond strength values.

VITA ENAMIC® (Polymer-infiltrated ceramic network) was used in this study. It is composed of a ceramic matrix (86% in weight/75% in volume) and a polymer matrix, which are UDMA and EGDMA (14% in weight/25% by volume). According to the manufacturer's instruction, the recommended surface treatment for VITA ENAMIC®, as stated in studies, was an application of 5% hydrofluoric acid for 60 seconds, rinsing for 60 seconds, and drying. Then, the silane coupling agent was applied for chemical surface treatment.

The relationship between the micro-mechanical retention and bond strength had been investigated by the study of Duzyol et al., 2016. It demonstrated that bond strength increases with increasing surface roughness. Bonding between ceramic and resin composite may occur by two distinct mechanisms: 1) chemical bonding with the organic matrix and the exposed filler particles and 2) micro-mechanical retention to the treated surface.

According to Eliane Placido et al., 2007, the shear test is the most widely used in measuring bond strength. The shear bond strength test is a fast and easy method for reliable results. Anyway, non-homogeneous stress distribution in the test process might cause erroneous interpretation of the results. Then, in this study, smaller specimens were then prepared to reduce the bonded area and ensure loading of the direct shear stress on the adhesion site.

In this study, the shear bond strength values of the control group were statistically significantly lower than the other groups because it was treated with chemical bonding by silane and adhesive only (Single Bond Universal Adhesive). VITA ENAMIC®, which HF treats, attained the highest bond strength values because HF reacts with the glassy matrix that contains silica and selectively removes the glassy or crystalline phases of the restorative material and forms microporosity on the ceramic surface. Consequently, the ceramic surface becomes rough and promotes micro-mechanical interlocking with resin composite. In



addition, The HF changes the surface energy and the bonding potential of ceramic to resin by enhancing the bond between ceramic and resin composite (Tekçe et al., 2018).

Sandblast particle size 50 μm for 15 seconds was used in this study. As seen in the result in Table 2, the mean shear bond strength values of SB are not significantly different from HF. Sandblasting caused roughness and deep crevices on surfaces if the particles exceeded 120 μm and the duration was more than 30 seconds (Matinlinna et al., 2018). According to Tecke et al. 2018, superficial cracks may form over 30 seconds of sandblasting, expanding up to 3 μm into the material mass. Therefore, consideration of the size of particles and duration of the sandblast before repairing VITA ENAMIC® is recommended.

Silicon carbide paper was used to imitate the effect made by diamond bur (medium-coarse) in the clinical situation. The mean shear bond strength values are not significantly different from the SB group. Surface preparation with silicon carbide paper created deep grooves and streaks that form macro- and micro-retentive areas (Duzyol et al., 2016). Thus, grinding with silicon carbide paper is accessible and easy to process. Furthermore, the adhesive can intrude into these retentions to form a siloxane bond between the fillers and the polymer matrix.

Besides, adhesive bonding agents and salinization enhance bonding when repairing hybrid ceramic restorations (Lung et al., 2021). In this study, the surface treatment that achieves greater surface roughness shows better mean shear bond strength values. Therefore, this study affirms the importance of chemical and mechanical preparation in repairing VITA ENAMIC® with resin composite.

According to the shear bond strength test and failure mode analysis performed in this study, each group of resin composite, surface treatment, and ceramic material predominantly showed mixed failure between ceramic and resin composite. In comparison, the cohesive failure was less frequent inside the ceramic. It indicates that the bond between the ceramic and resin composite seemed to exceed the strength of the material itself (Campos et al., 2016).

HF is the most appropriate surface treatment method for glass-ceramic. Still, the effects of HF on oral tissues are potentially harmful such as the potential for systemic intoxication that can cause eye lesions and irritate soft tissues. Clinical use of HF must be cautious. HF removes the glass matrix, keeping only the polymer component. On the other hand, the different treatment groups such as SB and SC only create a rough surface, maintaining both the glass matrix and the polymer. Thus, at the interfaces, the residue polymer alone might lead to weaker bond strengths. Possibly, this is the main difference between the acid-etched hybrid material and conventional feldspar-based ceramic (Placido et al., 2007).

Furthermore, The VITA ENAMIC® microstructure imaged through SEM clearly distinguished the control group and others. While in HF, SB and SC were slightly different. However, HF appeared in deeper crevices than the other groups. Thus, HF, SB, and SC groups had higher mean shear bond strength results when compared with the control group.

This study only investigates the shear bond strength within 24 hours for the immediate bond test of each surface treatment of resin composite bonding on VITA ENAMIC®. Further studies investigating the effect of other types of ceramic and thermocycling are recommended.

5. Conclusion

Within the limitations of this study, the authors would like to conclude that the surface treatment is suggested since it significantly affected shear bond strength values. Among 3 groups of mechanical surfaces, treatments were not significantly different. Thus, the SB and SC groups are safer to use in the oral cavity under rubber dam applications than HF.

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