รายงานการวิจัย

Research report

เรื่อง

การศึกษาความแข็งแรงยึดเฉือนของไฮบริดเซรามิกด้วยการเตรียมพื้นผิวที่แตกต่างกัน

Effect of different surface treatment on repair shear bond strength of hybrid ceramic

โดย

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ชื่อเรื่อง: การศึกษาความแข็งแรงยึดเฉือนของไฮบริดเซรามิกด้วยการเตรียมพื้นผิวที่แตกต่างกัน

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วัตถุประสงค์: เพื่อเปรียบเทียบประสิทธิภาพของความแข็งแรงยึดเฉือนหลังการซ่อมแซมผิวไฮบริดเซรามิกด้วยวัสดุเรซินคอมโพสิทเมื่อเตรียมพื้นผิวด้วยวิธีที่แตกต่างกัน

วิธีการวิจัย: เตรียมชิ้นทดสอบขนาด 6x11x2 มม จำนวน 40 ชิ้นจากบล็อกไฮบริดเซรามิก (VITA ENAMIC®) สุ่มแบ่งชิ้นทดสอบออกเป็น 4 กลุ่มย่อย กลุ่มละ 10 ชิ้น ตามการเตรียมพื้นผิวที่แตกต่างกัน กลุ่มที่ 1 เป็นกลุ่มควบคุม ไม่มีการเตรียมพื้นผิว กลุ่มที่ 2 เตรียมพื้นผิวด้วยกรดไฮโดรฟลูออริก 5% กลุ่มที่ 3 เตรียมพื้นผิวด้วยเครื่องพ่นทราย กลุ่มที่ 4 เตรียมพื้นผิวด้วยกระดาษทรายเบอร์ 120 จากนั้นทุกกลุ่มทาด้วยไซเลนและตามด้วยซิงเกิ้ลบอนด์ยูนิเวอร์แซลแอดฮีซีฟ ทำการซ่อมผิวไฮบริดเซรามิกด้วยเรซินคอมโพสิท (Filtek Z350 XT) นำตัวอย่างทั้งหมดแช่ในน้ำกลั่นที่อุณหภูมิ 37 องศาเซลเซียส เป็นเวลา 24 ชั่วโมง และนำไปทดสอบค่าความแข็งแรงยึดเฉือน โดยใช้เครื่องทดสอบสากล นำค่าเฉลี่ยแรงยึดเฉือนที่ได้ไปวิเคราะห์ความแปรปรวนแบบทางเดียว ($p<0.05$) และหาความแตกต่างระหว่างกลุ่มด้วยการเปรียบเทียบเชิงซ้อนชนิดทูกี้ และจำแนกพื้นผิวการแตกหักของแต่ละกลุ่มตัวอย่างด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด

คำสำคัญ: โพลีเมอร์อินฟิลเตรทเซรามิก, ความแข็งแรงยึดเฉือน, การซ่อมแซมไฮบริดเซรามิก, การเตรียมพื้นผิว

Abstract

The objective of this study was to compare the effect of various surface treatment on shear bond strength of repairing hybrid ceramic with resin composite. Forty specimens (size 6x11x2mm) of VITA ENAMIC® were used in this study. All specimens were randomly divided into 4 surface treatment subgroups(n=10). Group 1 Control: No surface treatment, Group 2 treated with 5% Hydrofluoric acid, Group 3 treated with sandblast and Group 4 Grinding with sandpaper grit 120. All subgroups were applied with silane and single bond universal adhesive. Filtek Z350 XT was used as repair resin composite. All specimens were stored in water at 37°C for 24 hours.

The shear bond strength of specimens was performed using universal testing machine and failure mode were evaluated by using Stereomicroscope. Data were statistically analyzed with one-way analysis of variance(ANOVA) and Tukey multiple comparison test ($p<0.05$).

Keywords: Polymer Infiltrated Ceramic Network, Shear bond strength, Repair hybrid ceramic, Surface treatment

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CHAPTER 1

Introduction

Background

Currently, various tooth-color restorative materials are developed. Ceramic and composite are often used to restore teeth for esthetic reason but both of them also have many 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 1.5-2.0 mm minimum in thickness. While composite does not cause antagonistic tooth wear, it worn easily. Currently, there is a new material coming up which combined the advantages of both material into one, called “Hybrid Ceramic”. There are currently 2 types of hybrid ceramic, nanoceramic and Polymer Infiltrated Ceramic-Network (PICN).

In case repairing hybrid ceramic is needed, resin composite is a material of choice, because it can directly repair in oral cavity and it is tooth-liked color. To repair with resin composite, surface treatment is recommended to provide better bond strength. The surface treatment methods include etching with hydrofluoric acid, grinding with diamond bur, airborne particle abrasion by using aluminum oxide and tribochemical silica coating. Despite its benefit and usefulness in clinical application, the PICN is still vaguely known. Therefore, it's mostly important to study on this material further to achieve the most effective application on this material.

Research question

Does the different surface treatments affect to shear bond strength of Polymer Infiltrated Ceramic Network when repaired with resin composite.

Objective

To compare the effect of various surface treatments on shear bond strength of repair hybrid ceramic with resin composite

Hypothesis

H0: shear bond strength of different surface treatments on repairing PICN with resin composite, are not different

H1: shear bond strength of different surface treatments on repairing PICN with resin composite, are different

Keywords

- Polymer Infiltrated Ceramic Network
- Shear bond strength
- Repair hybrid ceramic
- Surface treatment

Expected benefits

To find out which surface treatments are appropriated for repairing PICN with conventional resin composite.

CHAPTER 2

Materials and method

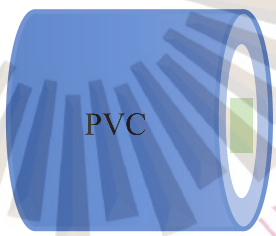
Table1 Materials used in this study

Material	composition	Manufacturer
VITA ENAMIC®	Shade 3M2: 86 wt% feldspar ceramic, 14 wt% polymer (UDMA, TEGDMA)	Vita Zahnfabrik, Bad Sackingen, Germany
Sandblast	50 μm Al_2O_3 airborne particles	RENFERT, Thailand
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
Sandpaper	Silicon carbide paper	TOA, Thailand
Demi™ Plus curing light	Intensity 1,000 mW/cm^2	Kerr Demi™, CA, USA
Polishing machine-MINITECH 233	MINITECH 233	PRESI, France
Universal testing machine	EZ-S, SHIMADZU	SHIMADZU, Japan
SEM	JSM-6610LV, Oxford X-Max 50	JEOL Ltd, Tokyo, Japan
Stereomicroscope	Olympus SZ61standard type	Olympus corp., Tokyo, Japan

Microbrush	Nylon superfine microbrush	GZ sunshine Dental Instruments Co.,LTD. Guang Dong, China
Metal mold	Hole diameter 2x4 mm^2	Custom made
PVC mold	Polyvinyl chloride tube	SCG Ltd, Bang sue, BKK
Clear plastic tape	Clear plastic tape	3M, Minnesota, USA
Self-cured acrylic resin		Formatray™, California, USA

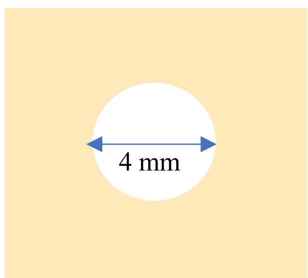
Method

Forty specimens (size 6x11x2 mm) were prepared using 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 PVC mold size diameter 10 x height 10 mm with self-cured acrylic resin. Then polished with sandpaper 400, 600, 800, 1000 and 1200 with polishing machine until the surface area is flat and smooth. (as shown in Fig.1)



(Fig.1) VITA ENAMIC® size 6x11x2 mm

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



(Fig.2) Clear plastic tape punched centrally 4 mm in diameter.

Specimens were divided randomly into 4 surface treatment subgroups (n=10).

group 1 : No surface treatment (control group)

group 2 : 5% Hydrofluoric acid – applied to the ceramic surface for 60 seconds, and rinsed with distilled water for 60 seconds. Then air dried.

group 3 : Sandblast air abraded with 50 µm Al_2O_3 particles for 15 seconds from 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 rinse.

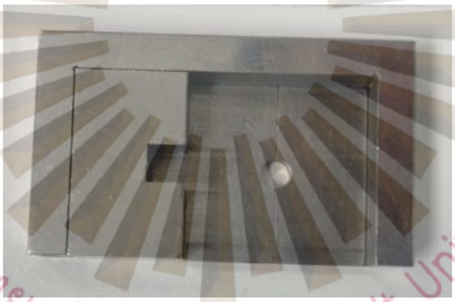
group 4 : Grinding with sandpaper grit 120 under copious air and water irrigation in one direction for 4 seconds on each surface using polishing machine then rinsed with distilled water for 15 seconds then air dried.

(Fig.3) Flow chart of method

All subgroups were applied with silane 60 seconds and lightly air dried. Then, an adhesive material (Single Bond Universal Adhesive, 3M™) applied for 20 seconds and air dried for 5 seconds then light cured for 20 seconds using an LED light curing unit (Demi™ Plus, Kerr).

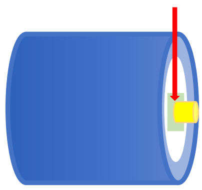
Finally, a resin composite (Filtek™ Z350 XT, 3M ESPE) was placed onto the treated surface (as shown in Fig.4) using a metal split mold with a disc-shape cavity (2x4 mm) (as shown in Fig.5) to standardize the dimension of the composite.

(Fig.4) Resin composite (Filtek™Z350XT, 3M ESPE, USA) size 2x4 mm^2 placed onto the treated surface



(Fig.5) Metal split mold

All bonded specimens were stored in distilled water at 37°C for 24 hours before shear bond strength testing. Specimens were tested with universal testing machine (EZ-S, SHIMADZU, Japan) (as shown in Fig.6) The interface between the VITA ENAMIC® specimens and resin composite was loaded by force using a crosshead speed of 0.5 mm/min until fracture occurred. Then, shear bond strength was recorded in Newton/mm^2 = MPa.



(Fig.6) Specimens were tested with universal testing machine (EZ-S, SHIMADZU, Japan)

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

1) Adhesive failure : between resin composite and ceramic.

2) Cohesive failure : within resin composite and resin composite, ceramic and ceramic.

3) Mixed failure : failure in ceramic or resin cement and the interface

CHAPTER 3

Data analysis

Shear bond strength (MPa) data were submitted to Shapairo-Wilk test to check normal distribution of the data. Normal distribution was found in all subgroups. One-way ANOVA followed by Tukey test were performed to analyze the differences between groups. (SPSS version 26 , SPSS INC, Chicago, IL, USA)

In all tests, level of significance was set at $P<0.05$

CHAPTER 4

Results

The data was analyzed by Shapairo-Wilk test and presented normal distribution in all groups. Thus, One-way ANOVA and Tukey tests were used for the comparison test at confident level 95%.

Table 2 presents the mean shear bond strength and standard deviations (SD) of the tested materials. The HF group promoted the highest bond values (13.00 MPa) when compared with control and sandpaper group. One-way ANOVA showed that different surface treatment influenced the result of shear bond strength. Which control group has mean shear bond strength value significantly different from other groups ($p<0.05$). Among these 3 groups, the HF group has mean shear bond strength value higher than sandpaper group significantly ($p<0.05$) but the HF and sandblast groups are not significantly different ($p>0.05$).

After shear bond strength tested, all specimens were evaluated by stereomicroscope. The failure modes were classified into 3 types (Fig.8, Fig.9)

1. Adhesive failure (between ceramic and cement),
2. Cohesive failure of the ceramic
3. Mixed failure (both adhesive and cohesive).

The predominant failure modes were adhesive. There were cohesive and mixed failure seen in HF, sandblast and sandpaper groups. (Table 3)

Representative SEM images of the tested specimens in Fig.10 and Fig.11 at magnification 40x and 2000x respectively. The HF, sandblast and sandpaper exhibited similar irregularities surface whereas control group showed lowest irregularities surface.

Table 2. Statistic test between mean shear bond strength of each factor

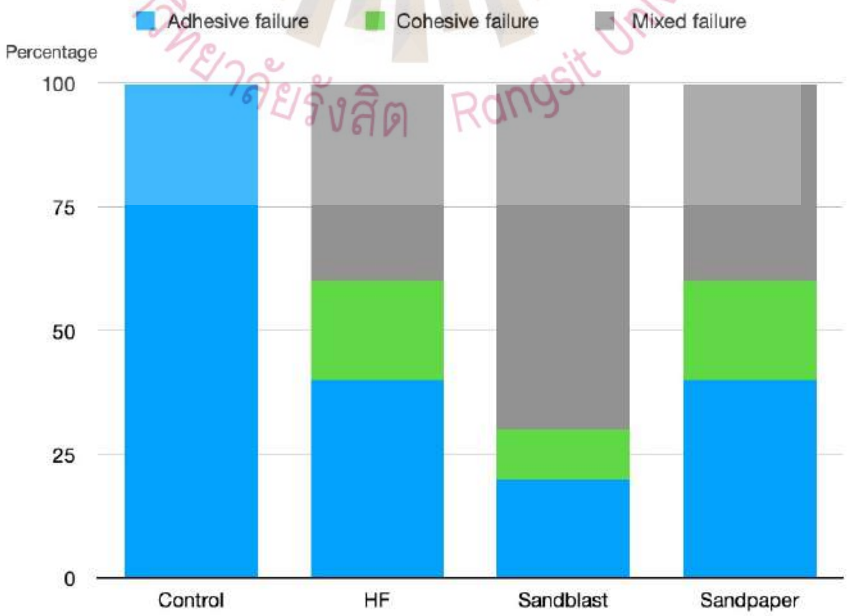
	Mean	Std. Deviation
Control	5.80[A]	1.60
HF	13.00[B]	3.09
Sandblast	10.85[BC]	2.14
Sandpaper	10.18[C]	2.10
Total	9.96	3.45

.

The results were considered statistically significant for $p<0.05$. The different superscript letters indicate statistically significant differences between surface treatment.

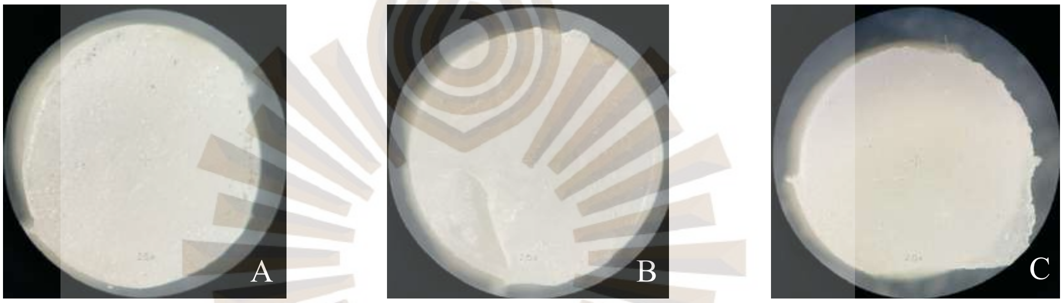
Table 3. Failure modes of each surface treatment after 24 hours storage in temperature 37^{o} C.

	Control	HF	Sandblast	Sandpaper
Adhesive	100%	40%	20%	40%
Cohesive	0	20%	10%	20%
Mixed	0	40%	70%	40%

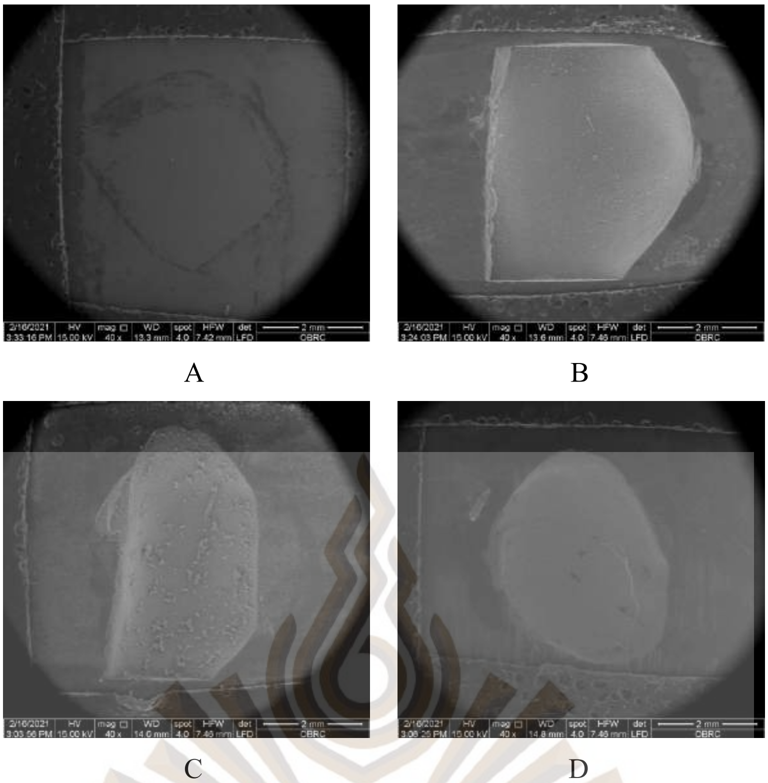


(Fig.7) Mode of failure in stacked column chart

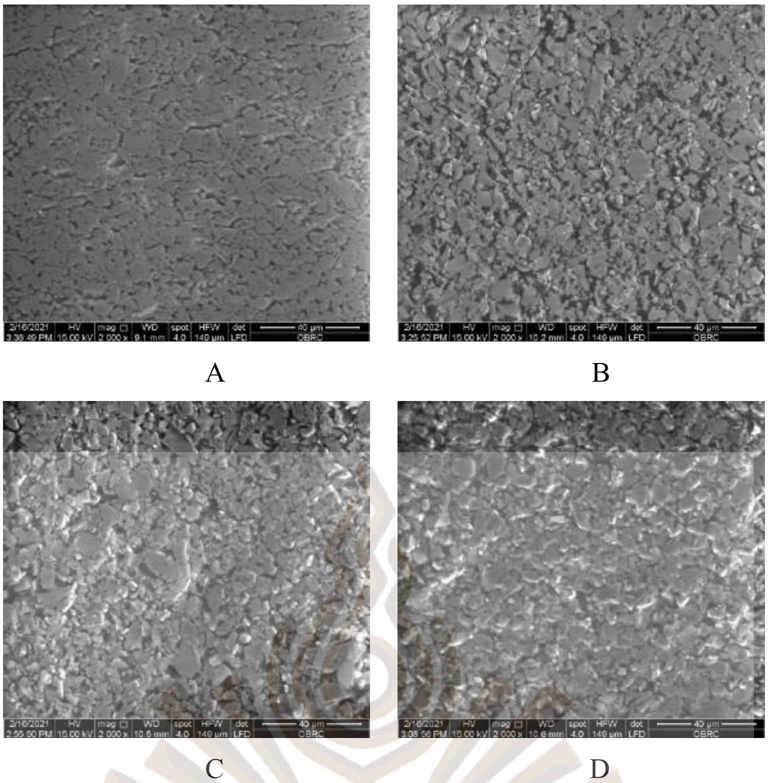
(Fig.8) The mode of failure. The stereomicroscope photographs above legends show representative failure modes for each corresponding type of failure on ceramic side. A: Adhesive failure, B: Mixed failure and C: Cohesive failure



(Fig.9) The mode of failure. The stereomicroscope photographs above legends show representative failure modes for each corresponding type of failure on composite side. A: Adhesive failure, B: Mixed failure and C: Cohesive failure



(Fig.10) SEM micrographs of ceramic surfaces. Representative images: A: Control, B: HF, C: Sandblast and D: Sandpaper (Original magnification: 40X)



(Fig.11) SEM micrographs of ceramic surfaces. Representative images: A: Control, B: HF, C: Sandblast and D: Sandpaper (Original magnification: 2000X)

CHAPTER 5

Discussion

This study examined the impact of different surface treatments after 24 hours storage in distilled water. It demonstrated that surface treatment by etching with HF promoted highest bond strength values. Thus, the null hypothesis is not accepted.

VITA ENAMIC® (Polymer-infiltrated ceramic network) were used in this study, composing of ceramic matrix (86% in weight/75% in volume) and polymer matrix (UDMA and EGDMA) (14% in weight/25% by volume). (VITA, 2014) The recommended surface treatment for VITA ENAMIC® as stated in studies was application of 5% hydrofluoric acid for 60 seconds, rinsed for 60 seconds and dry. Then, the silane coupling agent was applied for chemical surface treatment. Similar to the surface treatment method for the silica-based ceramics. (Pollyanna Silva et al., 2018). The relationship between the micromechanical retention and bond strength had been investigated by a previous in vitro study, and demonstrated that bond strength increases with increasing surface roughness. In this study, The HF group promoted the highest bond values when compared with control and sandpaper group, which is in line with the study. (Merve Bankoglu Güngör et al., 2016) Bonding between ceramic and composite may occur by two distinct mechanisms: (1) chemical bonding with the organic matrix and the exposed filler particles, (2) micromechanical retention to the treated surface (Al smar, 2017).

Shear bond strength values of control group showed statistically significantly lower than that of other 3 groups. Control group was treated only chemical bonding with silane and adhesive (Single Bond Universal Adhesive), concluding that application of surface treatment to create mechanical bonding on repair of PICN with composite resin provides greater retention on materials.

In this study, The PICN etched by HF attained highest bond strength values. 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. (Yen TW et al., 2008) Consequently, the surface of the ceramic becomes rough and promoting micro-mechanical interlocking with resin composite. (Chaiyabutr Y et al., 2008) The HF change their surface energy and the bonding potential of ceramic to resin, thus enhancing bond between ceramic and composite resin.

Sandblasting caused roughness on surfaces. However, the shear bond strength of HF and sandblast groups are not significantly different. Particle size 50 μm was use in this study, since crack formation in material has been observed with larger-sized particles (120 μm), also the duration of the procedure should not exceed 30 seconds (K. Papadopoulos et al., 2020). According to Tecke et al., reported that over 30 seconds of sandblasting duration, superficial cracks are formed that may expanding up to 3 μm into the material mass. Therefore, considering the size of particles and duration of the sandblast before repaired PICN is recommended.

In this study, sandpaper was used to imitate effect made by diamond bur (Medium coarse) which is used in clinical situation. It created roughness on the surface. Surface preparation with sandpaper creates deep grooves and streaks which form macro- and microretentive areas. (Shaymaa E. Elsaka., 2015) Grinding with sandpaper has the following advantages: accessibility and ease of execution (Duzyol M et al., 2016) and the interpenetration of the adhesive in these retentions to form a siloxane bond between the fillers and the polymer matrix. (Tezvergil A et al., 2003)

In any case, it is stated in the literature that the use of adhesive bonding agents and silanization enhance bonding when repairing hybrid ceramic restorations. (M.M. Wahsh, O.H. Ghallab, 2015) In this study, the surface treatment by making surface roughness was the most efficient surface treatment in our study. The result of this study confirmed the importance of micromechanical preparation in repaired PICN.

After shear bond strength tested, all specimens were evaluated by stereomicroscope, it is possible to affirm that fracture occurred mostly in the adhesive zone, while cohesive failures were less frequent, which benefits the real evaluation and interpretation of bond strength data. (F Campos et al., 2016)

According to the shear bond strength test and failure mode analysis performed in this study, it was revealed that each group with respect to resin composite, surface treatment and ceramic material predominantly showed adhesive failure between ceramic and resin composite. The cohesive failure inside ceramic indicates that the bond between the ceramic and resin composite seemed to exceed the strength of the material itself. (Cekic-Nagas et al., 2016)

Even though surface treatment with HF etching was suggested as the most appropriate surface treatment method for ceramics, the effects of HF on oral tissues are potentially harmful (Filho AM et al., 2014) such as the potential for systemic intoxication, can cause eye lesions and can irritate soft tissues. (Ozcan M et al., 2012) Moreover, HF removes the glass matrix, conserving only the polymer component. On the other hand, the other treatments only create a rough surface, maintaining both the glass matrix and the polymer. Thus, the polymer alone at the interfaces could lead to weaker bond strengths. Probably, this is the main difference between the acid-etched hybrid material and conventional feldspar-based ceramic. (Pollyanna Nogueira Ferreira da SILVAe et al., 2017)

In the dental literature, the shear test is the most widely used tests in measuring bond strength. The shear bond strength test has been found to be the fastest and easiest method for reliable results. But non-homogeneous stress distribution in the test procedure may eventually cause erroneous interpretation of the results. Then, in this study, smaller specimens were chosen to reduce the bonded area and to ensure loading of the direct shear stress on the adhesion site. (Eliane Placido et al., 2017)

Furthermore, The PICN microstructure imaged through SEM showed clearly difference between control group and others. While in sandpaper, sandblast and HF group were slightly different. However, HF group appeared much rougher than the other 2 groups. There was a variation in the surface microstructures of the VITA ENAMIC® which showed distinctive irregularities, creating a microretentive roughness and randomly distributed gaps and micropores.

One of the limitations of this study was the pretest failures, which were dominant in the control group after storage within 24 hours. However, it was evident that although the hybrid ceramic includes resin in composition, it requires surface treatment for bonding longevity at the interface.

In this study used immediate bond which is storage within 24 hours. The relevance of this study was that it stimulated different surface treatments for the PICN. Further studies should be conducted to investigate on effect of other type of ceramic and thermocycling.

CHAPTER 6

Conclusion

The limitations of this study, the following conclusion can be drawn:

The type of different surface treatment significantly affected shear bond strength values which the hydrofluoric acid group is the highest.

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APPENDIX

Table 4: The mean different of surface treatment factor

					95% Confidence interval for Mean			
	N	Mean	Std. Deviation	Std. Error	Lower Bound	Upper Bound	Minimum	Maximum
Control	10	5.80	1.60	.51	4.65	6.94	4.00	8.88
HF	10	13.00	3.09	.98	10.80	15.21	8.86	17.58
Sandblast	10	10.85	2.14	.68	9.32	12.38	8.50	14.74
Sandpaper	10	10.18	2.10	.67	8.69	11.67	7.58	14.22
Total	40	9.96	3.45	.55	8.85	11.06	4.00	17.58

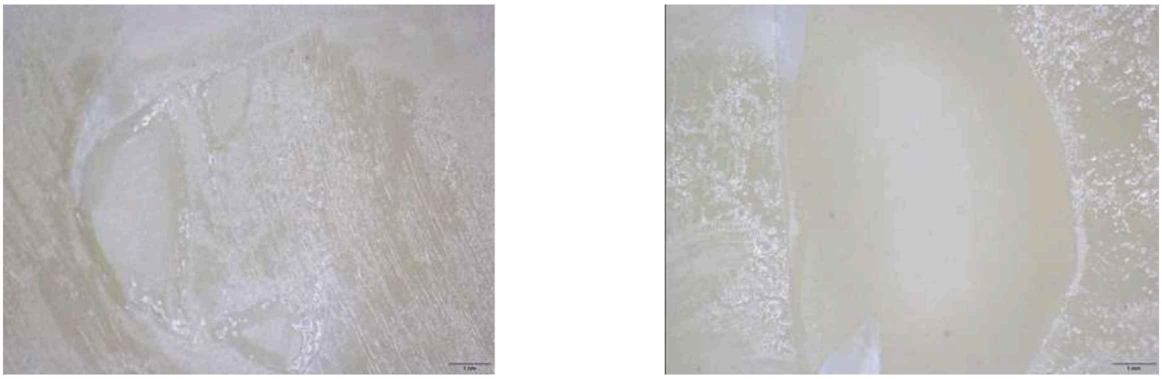
Table 5: Statistic test between mean shear bond strength of each factor

					95% Confidence interval	
Type (I)	Type (J)	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
Control	HF	-7.21*	1.03	.00	-9.97	-4.45
	Sandblast	-5.06*	1.03	.00	-7.82	-2.30
	Sandpaper	-4.39*	1.03	.00	-7.15	-1.62
HF	Control	7.21*	1.03	.00	4.45	9.97
	Sandblast	2.16	1.03	.17	-.61	4.92
	Sandpaper	2.82*	1.03	.04	.06	5.59
Sandblast	Control	5.06*	1.03	.00	2.30	7.82
	HF	-2.16	1.03	.17	-4.92	.61
	Sandpaper	.67	1.03	.91	-2.10	3.43
Sandpaper	Control	4.39*	1.03	.00	1.62	7.15
	HF	-2.82*	1.03	.04	-5.59	-.06
	Sandblast	-.67	1.03	.91	-3.43	2.10

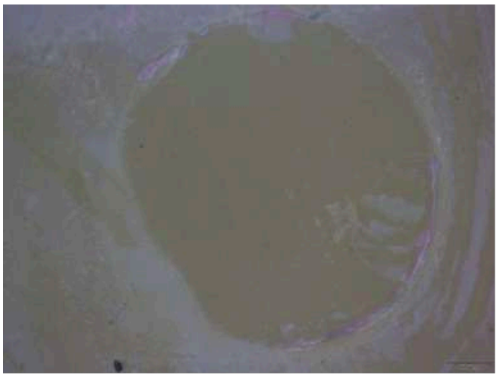
Table 6: Failure modes of each surface treatment after 24 hours storage in temperature 37^{o} C.

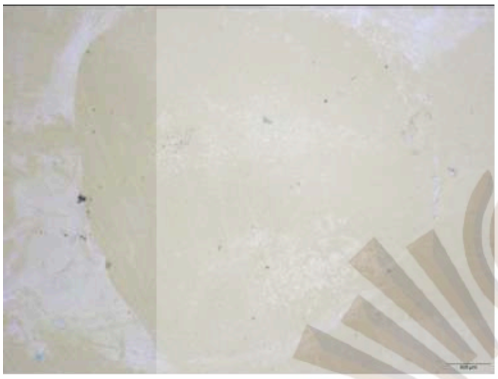
	Control	HF	Sandblast	Sandpaper
Adhesive	100%	40%	20%	40%
Cohesive	0%	20%	10%	20%
Mixed	0%	40%	70%	40%

(Fig.12) The stereomicroscope photographs in HF group.

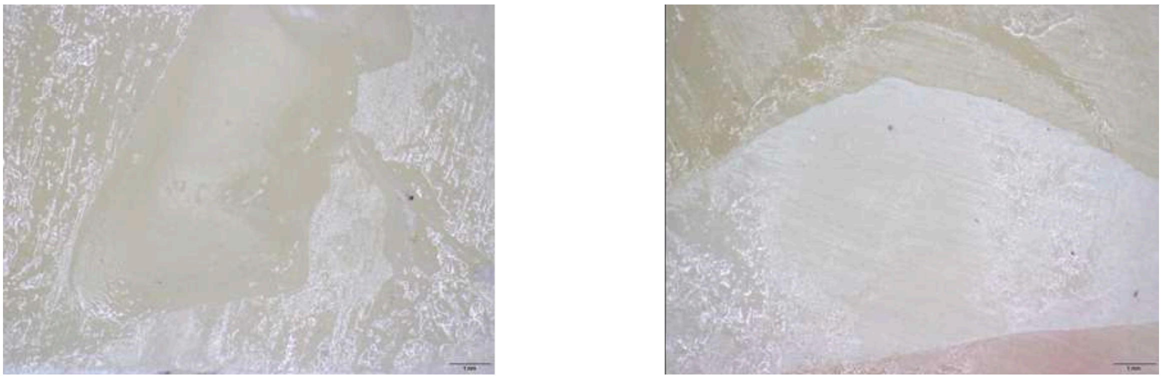


(Fig.13) The stereomicroscope photographs in sandblast group.

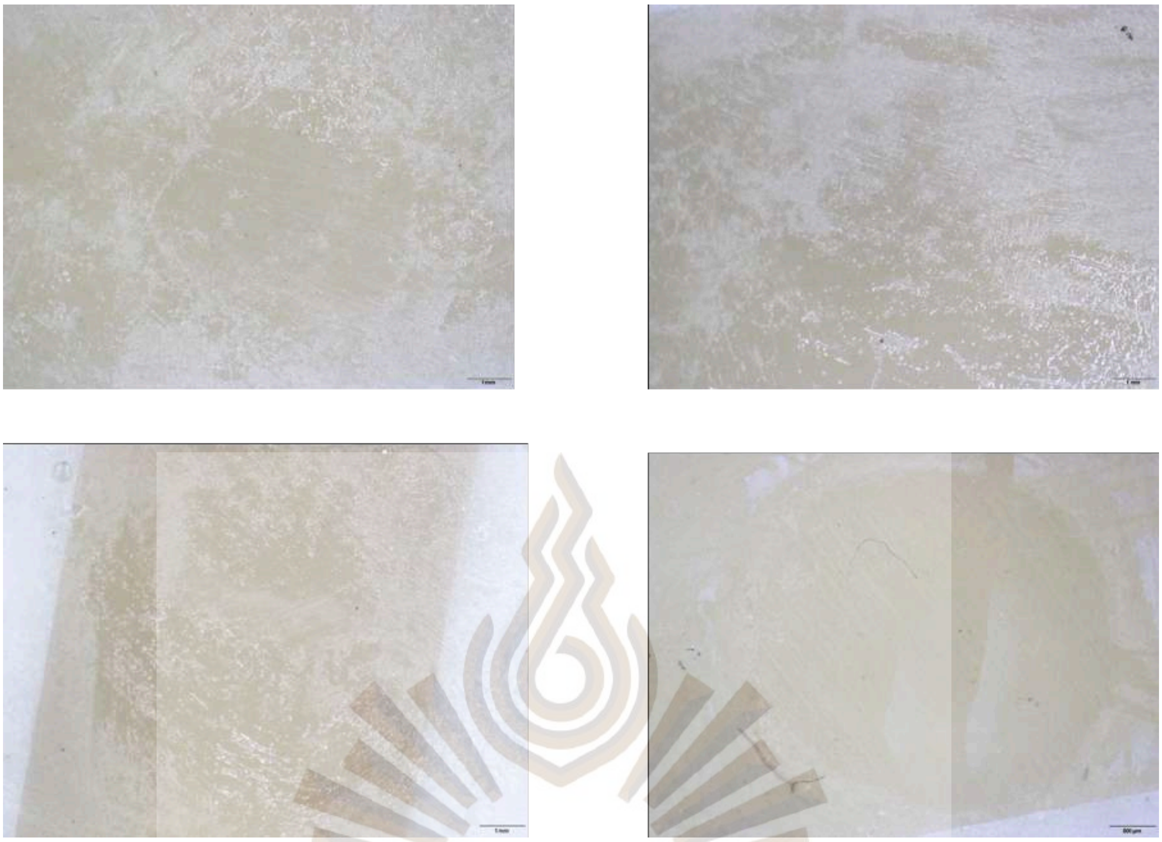




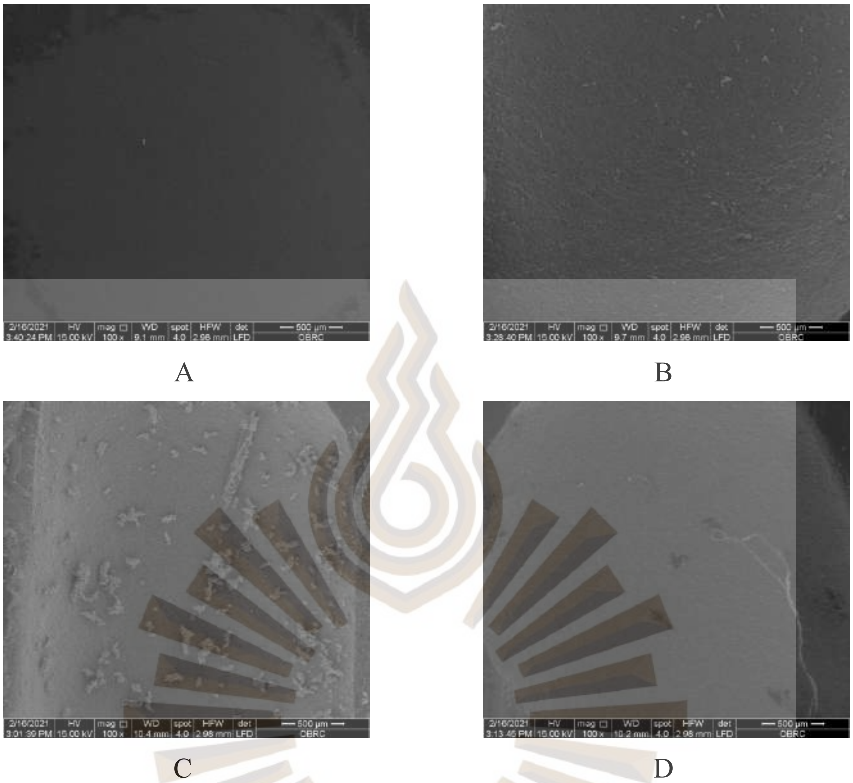
(Fig.14) The stereomicroscope photographs in sandpaper group.



(Fig.15) The stereomicroscope photographs in control group.



(Fig.16) SEM micrographs of ceramic surfaces. Representative images: A: Control, B: HF, C: Sandblast and D: Sandpaper (Original magnification: 100X)



(Fig.17) SEM micrographs of ceramic surfaces. Representative images: A: Control, B: HF, C: Sandblast and D: Sandpaper (Original magnification: 500X)