THE EFFECT OF SURFACE TREATMENT ON SHEAR BOND STRENGTH OF RESIN-MATRIX CERAMICS AND DUAL-CURED RESIN CEMENT

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A Thesis Submitted in Partial Fulfillment of the Requirements
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ผลของการปรับสภาพพื้นผิวต่อความแข็งแรงเฉือนของเรซินเมทริกซ์เซรามิกและเรซินซีเมนต์ชนิดบ่มตัวสองแบบ

นายณัฐพงศ์ บุญชวนสกุล

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Thesis Title	THE EFFECT OF SURFACE TREATMENT ON SHEAR BOND STRENGTH OF RESIN-MATRIX CERAMICS AND DUAL-CURED RESIN CEMENT
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Field of Study	Prosthodontics
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ณัฐพงศ์ บุญชวนสกุล : ผลของการปรับสภาพพื้นผิวต่อความแข็งแรงเฉือนของเรซินเมทริกซ์เซรามิกและเรซินซีเมนต์ ชนิดบ่มตัวสองแบบ. (THE EFFECT OF SURFACE TREATMENT ON SHEAR BOND STRENGTH OF RESIN-MATRIX CERAMICS AND DUAL-CURED RESIN CEMENT) อ.ที่ปรึกษาหลัก : รศ.ทพ. ดร.นิยม ธำรงค์อนันต์สกุล

วัตถุประสงค์: การศึกษานี้มีวัตถุประสงค์เพื่อประเมินผลของการใช้สารปรับสภาพพื้นผิวด้วยเททระไฮโดรฟูราน ร่วมกับไซเลน และสารยึดติด ต่อค่าแรงยึดของวัสดุเรซินเมทริกซ์เซรามิกและเรซินซีเมนต์ *วิธีดำเนินการวิจัย* :นำวัสดุเรซินเมทริกซ์เซรามิกได้แก่ Enamic, Cerasmart, Shofu block HC มาตัดเป็นสี่เหลี่ยมจัตุรัสขนาด 6x6x2 มิลลิเมตร^3 และจัดเข้ากลุ่มทั้ง 10 กลุ่มแบบสุ่มโดยแบ่งตามการปรับสภาพพื้นผิว ดังนี้ กลุ่มควบคุม(C), สารยึดติด (Ad),เททระไฮโดรฟูราน 1นาที (T1),ไซเลน/สารยึดติด (Si/Ad), เททระไฮโดรฟูราน1นาที/สารยึดติด(T1/Ad),เททระไฮโดรฟูราน1นาที/ไซเลน/สารยึดติด(T1/Si/Ad),เททระไฮโดรฟูราน2นาที/ไซเลน/สารยึดติด(T2/Si/Ad),เททระไฮโดรฟูราน3นาที/ไซเลน/สารยึดติด(T3/Si/Ad),เททระไฮโดรฟูราน4นาที/ไซเลน/สารยึดติด(T4/Si/Ad),เททระไฮโดรฟูราน5นาที/ไซเลน/สารยึดติด(T5/Si/Ad) จากนั้นนำชิ้นงานไปแช่น้ำที่ 37องศาเซลเซียส เป็นเวลา 24 ชั่วโมง จากนั้นนำไปวัดค่าแรงยึดเฉือนแล้วนำผลของค่าแรงยึดของการทดสอบในแต่ละกลุ่มมาหาค่าเฉลี่ยและส่วนเบี่ยงเบนมาตรฐาน และทำการวิเคราะห์สถิติด้วยการวิเคราะห์ความแปรปรวนแบบสองทาง (Two-way ANOVA) ร่วมกับทดสอบความแตกต่างระหว่างค่าเฉลี่ยรายคู่ ของ Bonferroniที่ระดับความเชื่อมั่นร้อยละ 95 *ผลการศึกษา*:พบว่าในกลุ่มของเททระไฮโดรฟูราน 3นาที/ไซเลน/สารยึดติด(T3/Si/Ad) ให้ค่าแรงยึดเฉลี่ยสูงที่สุด(25.37 ± 4.73 MPa)อย่างมีนัยสำคัญเกือบทุกกลุ่ม ยกเว้นกลุ่มT4/Si/AdและT5/Si/Ad โดยที่ Enamic ให้ค่าแรงยึดเฉือนมากที่สุด 28.12 ± 5.45 MPa CerasmartและShofu block HC ตามลำดับ ความล้มเหลวแบบผสมพบเป็นส่วนใหญ่ในกลุ่มปรับผิวด้วยเททระไฮโดรฟูราน ร่วมกับไซเลน และสารยึดติด *สรุปผลการศึกษา*:การใช้สารปรับสภาพพื้นผิวด้วยเททระไฮโดรฟูราน ร่วมกับไซเลน และสารยึดติด มีผลต่อค่าแรงยึดของวัสดุเรซินเมทริกซ์เซรามิกและเรซินซีเมนต์

สาขาวิชา	ทันตกรรมประดิษฐ์	ลายมือชื่อนิสิต
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ปี	2562	ลายมือชื่อ อ.ที่ปรึกษาหลัก
การศึกษา		....

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KEYWORD: Resin matrix ceramics, Tetrahydrofuran, Shear bond strength

Nuttapong Bunchuansakul : THE EFFECT OF SURFACE TREATMENT ON SHEAR BOND STRENGTH OF RESIN-MATRIX CERAMICS AND DUAL-CURED RESIN CEMENT. Advisor: Assoc. Prof. Dr. NIYOM THAMRONGANANSKUL

Objective:The purpose of this study was to evaluate the effect of Tetrahydrofuran (THF) on shear bond strength (SBS) between resin matrix ceramics (RMC) materials and resin cements.*Methods*:RMC materials(Enamic,Cerasmart,Shofu block HC) were cut into square piece of approximately 6x6x2 mm^3 and randomly divided into 10 groups following the surface treatment:no treatment(C),adhesive agent(Ad),THF1min(T1),silane/adhesive(Si/Ad),THF1min/adhesive(T1/Ad),THF1min/silane/adhesive(T1/Si/Ad),THF2mins/silane/adhesive(T2/Si/Ad),THF3mins/silane/adhesive(T3/Si/Ad), THF4mins/silane/adhesive(T4/Si/Ad),THF5mins/silane/adhesive(T5/Si/Ad).Specimens were cemented to composite resin rod with resin cement and kept them in water at 37°C for 24 hours.The SBS measurements were tested with universal testing machine.Failure modes were examined by stereomicroscope.The SBS values were analyzed with two-way ANOVA and Bonferroni's post hoc tests (α = 0.05).*Results*: The highest mean SBS for all RMC materials was found in group T3/Si/Ad (25.37 ± 4.73 MPa) significantly greater than almost all groups ($p < 0.05$), except for T4/Si/Ad and T5/Si/Ad.In addition, Enamic showed the highest SBS value (28.12 ± 5.45 MPa) followed by Cerasmart and Shofu block HC,respectively. Mixed failure was the most common found in THF with silane and adhesive agent groups.*Conclusion*:Tetrahydrofuran with silane and adhesive agent affected to bond strength of RMC materials and resin cements.

Field of Study:	Prosthodontics	Student's Signature ....
Academic Year:	2019	Advisor's Signature ....

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Nuttapong Bunchuansakul

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Chapter I Introduction

Background and rationale

One of the main purposes of restorative dentistry is to create functional and esthetic restorations. Ceramics are extensively used as indirect restorations due to their esthetic appearance, good fracture resistance and low wear rate.(1, 2) However, ceramics have limitations on success rates because of their toughness, brittleness, and potential to wear opposing teeth.(3, 4) Nowadays, not only esthetic expectations, but also chairside fabrication of restorations are necessary. As the results, computer-aided design/computer-aided manufacturing (CAD/CAM) technology is wildly used and different types of machinable materials such as ceramics, acrylic resins, and composite resins developed to complete the requirements.(3, 5, 6)Resin matrix ceramics (RMC) have been recently developed for CAD/CAM technology. RMC combines the benefits of composite resins, improved flexural properties and low abrasiveness, as well as color stability and durability of ceramics.(7, 8) Available commercial products of resin matrix ceramic materials include a polymer-infiltrated ceramic network (PICN) material (VITA ENAMIC), nanohybrid composite resin materials such as resin nanoceramic (Shofu Block HC ,Lava Ultimate) and a nanoparticle-filled resin (Cerasmart). In addition, RMC have the ability to distribute stress due to modulus of elasticity near to dentine and the capacity of milling-adjusting which is more convenient and safes compared to glass matrix or polycrystalline ceramics.(3, 9,10)

The bond strength between cement and resin or ceramic CAD/CAM materials has a major role in providing the improvement of fracture resistance and keeping the marginal integrity of the restorations.(11, 12) To create a sufficient bond, mechanical or chemical pre-treatments are essential.(13, 14)Depending on the composition of the material, various surface treatment techniques such as silanization, silica coating (Co-jet), etching with hydrofluoric acid (HF) and sandblasting could be applied.(15-17) Many studies attempted to improve the bond strength of RMC materials to different resin cements by using different surface treatments. However, some methods of surface treatments are still inconclusive.(18-22)Tetrahydrofuran (THF) is an organic solvent and it could be used as solvent in dental adhesive systems to form bond strength stability.(23) THF could also be used with silane for improving the shear bond strength of glass fiber post.(24) There is still not enough information on THF to be used as surface treatment for enhancing the bond strength between resin cement and RMC. Therefore, the purpose of this study was to evaluate the effect of THF on bond strength between RMC and dual cure resin cements. The null hypothesis is that surface treatment by using THF with silane and adhesive agent would not affect the shear bond strength of RMC to dual cure resin cement.

Research Objective

To evaluate the effect of THF on bond strength between RMC and dual cure resin cements.

Research Question

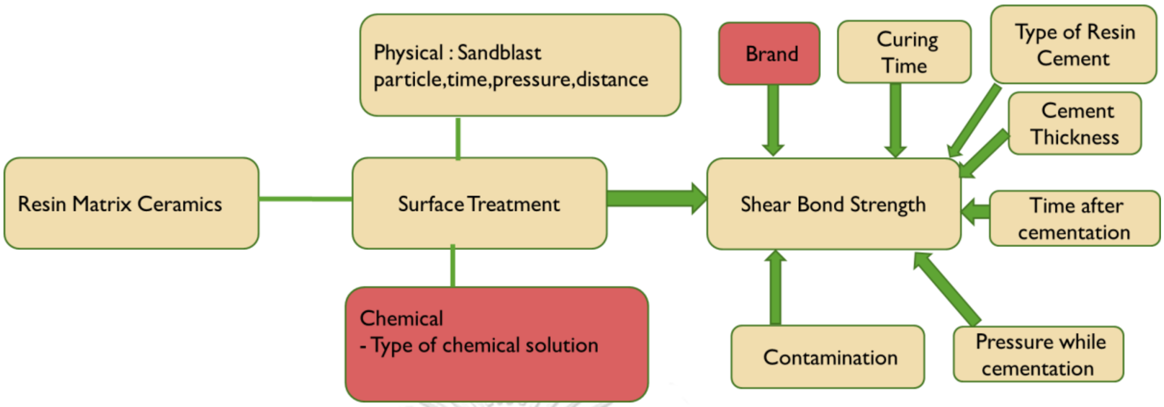
Would surface treatment by THF with silane and adhesive on Resin matrix ceramics using dual cure resin cement have an effect to shear bond strength?

Research Hypothesis

H 0: There is no difference of shear bond strength among Resin matrix ceramics with dual cure resin cement in surface treatment by using THF with silane and adhesive agent

H 1: There is difference of shear bond strength among Resin matrix ceramics with dual cure resin cement in surface treatment by using THF with silane and adhesive agent

Conceptual framework



Proposed Benefits

To provide an appropriate surface treatment method for enhancing bond strength among Resin matrix ceramics with dual cure resin cement and better result in clinical performance.

Keywords

Resin matrix ceramics,Tetrahydrofuran,shear bond strength

Research design

Laboratory and experimental research

Location of the Experimental Database

Dental Material R&D Center, Faculty of Dentistry, Chulalongkorn University

Chapter II Literature Review

2.1 Resin Matrix ceramics

Resin-matrix ceramic materials can be divided into several groups from compositions such as PICN (polymer-infiltrated ceramic network e.g. Enamic, Vita) which have a dual network of ceramic and a polymer, zirconia-silica ceramic in a resin interpenetrating matrix (e.g. Shofu Block HC) that contain silica powder and zirconium silicate in ceramic contents and resin nanoceramic (e.g. Lava Ultimate).(9) However, the materials can be classified into 2 subclasses which are PICN materials and dispersed fillers which included Lava Ultimate, Shofu Block HC and Cerasmart into the same group is shown in Table 1.(25)

Table 1 shows the classification of resin-matrix ceramic materials, manufacturers and their composition.

Material	Type	Manufacturer	Composition
Vita Enamic	Polymer infiltrated Ceramic Network	Vita Zahnfabrik, , Germany	86 wt% feldspar ceramic, 14 wt% polymer,UDMA,TEGDMA
Cerasmart	Dispersed Fillers	GC Corp., Tokyo, Japan	Nanoparticle-filled resin containing 71 wt% silica and barium glass filler,UDMA
Shofu Block HC	Dispersed Fillers	Shofu Inc., Kyoto, Japan	Filler composition: 61%, incl. zirconium silicate, silicon dioxide, UDMA, TEGDMA
Lava Ultimate	Dispersed Fillers	3M ESPE	UDMA Silica (20 nm) +zirconia (4-11 nm) + zirconia-silica clusters (0.6-10 μm) (79 wt%)

Many studies have researched the bond strength between resin-matrix ceramic materials and different resin cements by different surface treatment. However, there is some method of surface treatments still have disagreement.

Elsaka et al.2014 found that using either HF acid etching or sandblasting with a silane for Enamic can increased bond strength significantly but Lava Ultimate, there was no different significantly value in any type of surface treatment(20). On the other hand, Peumans et al. revealed that both Lava Ultimate and Enamic were improved bond strength by pre-treatments with HF acid and silane. HF acid treatment in

Enamic may cause by partial dissolved the polymer and glassy phases which possibly increased micromechanical retention surface then silane application can increase the surface wetting of bonding area consequent to better bond strength.(21) However, Cekic-Nagas et al. showed that treated with 10% HF acid gel did not have effect on bond strength value between resin cement and resin-matrix material which were Enamic, Lava Ultimate and Cerasmart.(18)

According to Frankenberger et al.(12) found that only sandblasting increased highest bond strength for Lava Ultimate whether use silane or not and HF had deleterious to strength value but for Enamic when using hydrofluoric acid etching followed by silane treatment showed the best strength value. Sandblasting is the method expected to increase bond strength by improving micromechanical interlocking, and increasing wettability and surface area. (16, 20) On the contrary, sandblasting to ceramics, does not seem to proper process for surface treatment. Because it may create microcracks in the ceramic surface and lead to premature failures also it effects internal and marginal adaptation.(16, 20, 26)

From the study of Yoshihara et al. revealed that sandblasting with silanization can improved the bond strength of the materials, but sandblasting created surface damage of Shofu Block HC and silane treatment cannot improve the bond strength for these material(22). Reymus M et al. (2019) found that using sandblasting and then treated surface materials with resin primer which have MMA produced more bond strength than use of silane primer in Cerasmart and Shofu Block HC.(27)

In actually, many studies try to improve the bond strength by different surface treatment but there is still have argument in some method of surface treatments for the resin-matrix ceramic materials. This aim of this study is finding that THF with bonding agent for use as surface treatment for enhancing the bond strength between Resin matrix ceramics and dual cure resin (RelyX U200).

2.2 Tetrahydrofuran

Tetrahydrofuran (THF) $(CH_2)_3CH_2O$ is an organic compound that is classified as heterocyclic compound, specifically a cyclic ether. It is a clear colorless liquid with an ethereal odor and low viscosity. It was use as solvent for many polymers such as polyvinyl chloride, unvulcanized rubber, vinyls, polymer coating, cellophane, protective coatings(30).

According to Fontes et al. (2009), used THF as solvent by mixing with HEMA and Phosphate for primer in etch-and-rinse system. The study revealed that after 6-month aging acetone, THF, and THF/water-based primer can maintained bond strength on dentin (23). Further study from Fontes et al. (2013), found that the THF, acetone, or THF/water primer showed high and stable bond strength after 1-year aging. In addition, THF-based primer without water is the only group that having similar bond value between the times 24 h and 1 year. For toxicity, THF showed an intermediate cytotoxicity same as HEMA(28). In addition there is study showed that THF can be used as cleaning agent to enhance bond strength to glass-fiber post when compare to control.(24)

Table 2 shows physical properties of Tetrahydrofuran (**THF**)

Molecular Weight	72.11 g/mol
Density	0.89 g/cm3 (20 °C)
Boiling point	65 - 66 °C (1013 hPa)
Melting Point	-108.5 °C
pH value	7 - 8 (200 g/l, H_2O, 20 °C)
Vapor pressure	173 hPa (20 °C)
LD50 Rat oral	**2.3 mL/kg**

2.3 Shear Bond Strength

According to Phillips' Science of Dental Materials(29), the shear strength is the maximum stress between interfaces of two materials can withstand before failure by sliding or applied force parallel to interface. The shear strength reports value in MPa can calculated by the failure load (in Newtons) divided by the total bonded area (in mm^2). From the following formula:

$$\text{SBS (in MPa)} = \frac{F}{A}$$

F = is the maximum load (Newtons)

A = total bonded area (mm^2)

$A = \pi r^2$ when r is the radius of bonded area (in mm)

Chapter III Research Methodology

3.1 Materials and equipments

<u>Equipments</u>

1.Universal testing machine (SHIMADZU, EZ-S 500N model, Japan)

2.Additional silicone (putty type) Elite HD+ putty soft Zhermack, Italy

3.Glass slab

4.Paper hole puncher

5.Vernier caliper

6.Microbrush (Citisen Micro Applicator, Huanghua Promisee Dental, Hebei, China)

7.Gloves

8.Tissue paper

9.Cement spatula

10.Epoxy resin

11.Silicon carbide paper 300,600 grit

12.Polishing Machine (Minitech 233, Presi, Le Locle, Switzerland)

13.Low speed saw (Isomet 1000: Buehler,USA)

14. PVC mold ½ “

15.Adhesive tape(Scotch blue Painter’s tape, 3M, St. Paul, MN, USA)

16.Light curing unit (Elipar Freelight 2 LED curing light, 3M ESPE, St. Paul, MN, USA)

17.Ultrasonic bath (VGT-1990, QTD, China)

Table 3 shows materials were used in this study, manufacturers and composition, manufacturers, their composition and lot number.

Material	Composition	Manufacturer	Lot No.
Vita Enamic (VE)	86 wt% feldspar ceramic, 14 wt% polymer,UDMA,TEGDMA	Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany	071601
Cerasmart (CS)	Nanoparticle-filled resin containing 71 wt% silica and barium glass filler,UDMA	GC Corp., Tokyo, Japan	1706151
Shofu Block HC (HC)	Filler composition: 61%, incl. zirconium silicate, silicon dioxide, UDMA, TEGDMA	Shofu Inc., Kyoto, Japan	77671
Filtek Z350	Bis-GMA, UDMA, TEGDMA, and Bis-EMA resins and filler	3M ESPE, St. Paul, MN, USA	42424
One Coat Bond SL	HEMA,UDMA,GDMA amorphous silicic	Coltene/Whaledent GmbH, Langenau, Germany	179850
Monobond N primer	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent, Schaan, Liechtenstein	43243
Tetrahydrofuran	Tetrahydrofuran 99.5 %	Loba Chemie Pvt Ltd., Mumbai, Maharashtra, India	LM04671706
RelyX U200	Multifunctional phosphoric acid methacrylates, dimethacrylates, acetate, initiator/stabilizer, powdered glass, silica, substituted pyrimidine, calcium hydroxide, peroxide compound, pigments	3M Deutschland GmbH, Neuss, Germany	4819681

3.2 Experimental procedures

Part I RMC Specimen preparation

Three different resin matrix ceramics materials were used in this study. Manufacturers and compositions of the materials are presented in Table 1. The RMC materials were cut with a diamond disk (Slow speed cutting machine, Model Isomet, Buehler, IL, USA) under cooling water to a square piece (6 × 6 × 2 mm^3). The specimens were embedded in polyvinyl chloride pipe (PVC) diameter 0.5 inch with epoxy resin. After the epoxy resin reached its final setting time, the mounted specimens were polished using a polishing machine (Minitech 233, Presi, Le Locle, Switzerland) with 300 and 600-grit silicon carbide abrasive paper, respectively. The specimens were then ultrasonically cleaned in distilled water for 5 minutes, and air-dried for 15 sec are shown in Figure 1.

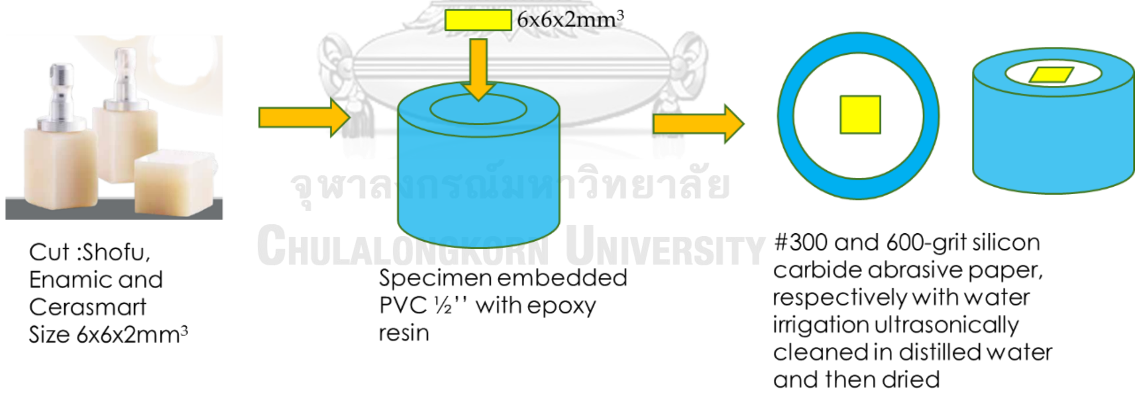


Figure 1 shows the steps of RMC specimen preparation.

Using G power program (following formula) calculated to estimate the sample size and power of this study. The data that calculated from the previously pilot study which calculate sample size (n) is 10 for each group.

Compare two means (use mean and standard deviation

$$n \geq \frac{\left(Z_{1-\alpha/2} + Z_{1-\beta}\right)^2 \left(\sigma_1^2 + \sigma_2^2/r\right)}{\left(\mu_1 - \mu_2\right)^2}$$

The specimens of each RMC were randomly divided into 10 groups and each group was subdivided into 3 subgroups (n=10) according to surface modification methods are shown in Figure 2 schematic diagram. The surface modification details are shown in Table 4 and the flowchart steps of applying chemical agents are shown in Fig 3-5 For the THF groups, lead sheets had been punched as a square shape size 5x5 mm^2, to limit the area of applying agent, then removed after finishing this treatment steps are shown in Figure 4.

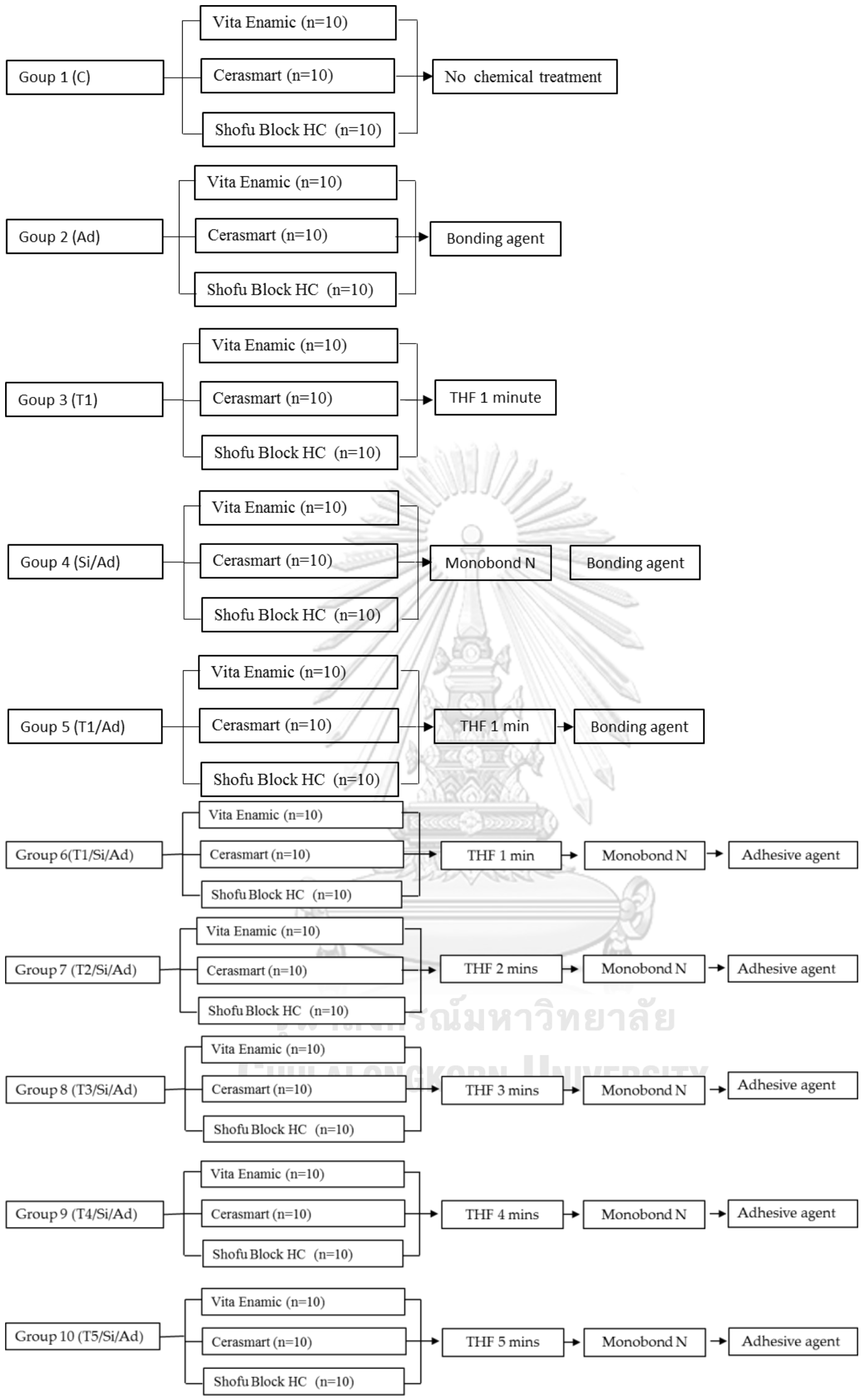


Figure 2 shows schematic diagram of experimental procedure.

Table 4 shows group, code and surface treatment details.

Group	Code	Surface Treatment details
1	C	No Surface treatment (Control)
2	Ad	Two microliters of bonding agent (One Coat Bond SL, Coltene/Whaledent GmbH, Langenau, Germany) were applied to the specimen surface using micropipette (Eppendorf AG, Hamburg, Germany) and rubbed with a disposable microbrush (Citisen Micro Applicator, Huanghua Promisee Dental, Hebei, China) for 10 sec The excess bonding agent was removed with a new disposable microbrush, gently air-dried for 20 sec and light activated for 40 sec.
3	T1	Two drops of THF were applied to the specimen surface and left undisturbed for 1 min, gently air-dried 10 sec.
4	Si/Ad	Two microliters of monobond N were applied to the specimen surface and rubbed with a disposable microbrush for 10 sec and left undisturbed for 1 min, gently air-dried for 20 sec. The bonding was applied as described in group 2.
5	T1/Ad	THF was applied as described in group 4 and the bonding agent was the applied as described in group 2.
6	T1/Si/Ad	THF was applied as described in group 4 and the monobond N and the bonding agent was the applied as described in group 4, respectively.
7	T2/Si/Ad	Two drops of THF were applied to the specimen surface for 2 times. Each round was left undisturbed for 1 min. After the THF application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
8	T3/Si/Ad	Two drops of THF were applied to the specimen surface for 3 times. Each round was left undisturbed for 1 min. After the THF application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
9	T4/Si/Ad	Two drops of THF were applied to the specimen surface for 4 times. Each round was left undisturbed for 1 min. After the THF application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
10	T5/Si/Ad	Two drops of THF were applied to the specimen surface for 5 times. Each round was left undisturbed for 1 min. After the THF application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.

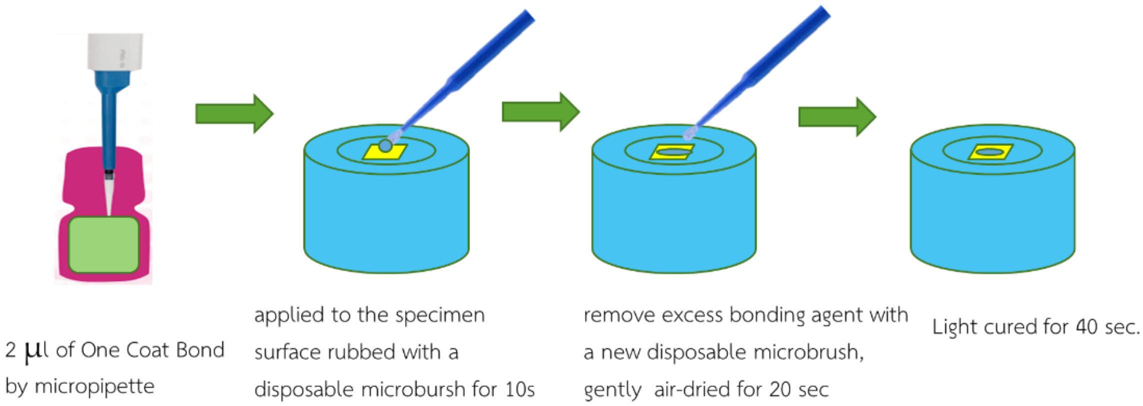


Figure 3 shows the steps of adhesive agent applying.

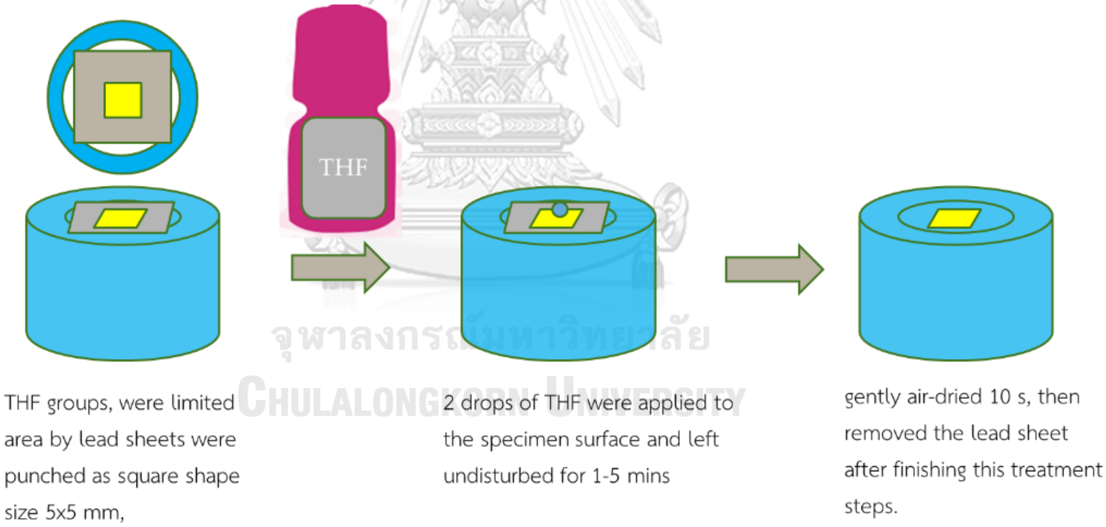


Figure 4 shows the steps of Tetrahydrofuran applying.

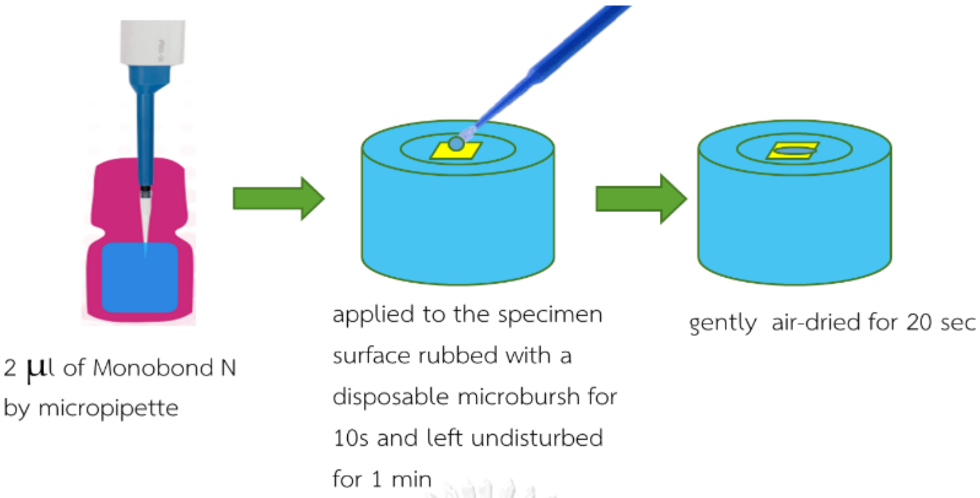


Figure 5 shows the steps of silane applying.

To control the bonding area, an 80-micron thick single-sided adhesive tape (Scotch blue Painter’s tape, 3M, St. Paul, MN, USA) was cut into a square shape with a size of 5x5 mm^2. A 3-mm diameter hole was made in the center of the adhesive tape using a hole- puncher. The adhesive tape was firmly placed and attached to the specimen surfaces, this procedure was performed before cementation.

Part II Composite resin rods

Three hundred composite resin rods were prepared using a custom-made silicone mold (4 mm diameter × 4 mm height). Composite resin Filtek Z 350, 3M ESPE) was condensed with a hand instrument in 2-mm incremental layers and light-polymerized for 40 seconds. (1000 mW/cm2, Elipar Freelight 2 LED curing light, 3M ESPE, St. Paul, MN, USA). The ends of composite resin rods were blasted with 50-micron alumina is shown in Figure 6.

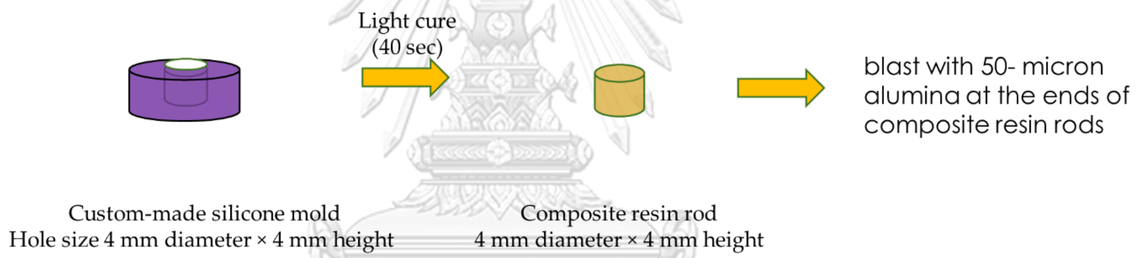


Figure 6 shows composite resin rod preparation.

Part III Cementation

Composite resin rods were bonded to the treated specimens with dual-cure resin cement (RelyX U200,3M Deutschland GmbH, Neuss, Germany) by light-polymerization. Luting was performed under constant weight of 1,000 g applied to the composite rod during the bonding procedure for 10 seconds at room temperature. The cement was activated by a light-curing unit at the 4 proximal sides and the top surface, 20 seconds each. The bonded specimens were kept in 37°C distilled water for 24 hours in an incubator (Contherm 160M, Contherm Scientific Ltd,Korokoro, Lower Hutt, New Zealand) according to ISO/TS 11405 to allow for post-polymerization is shown in Figure 7.

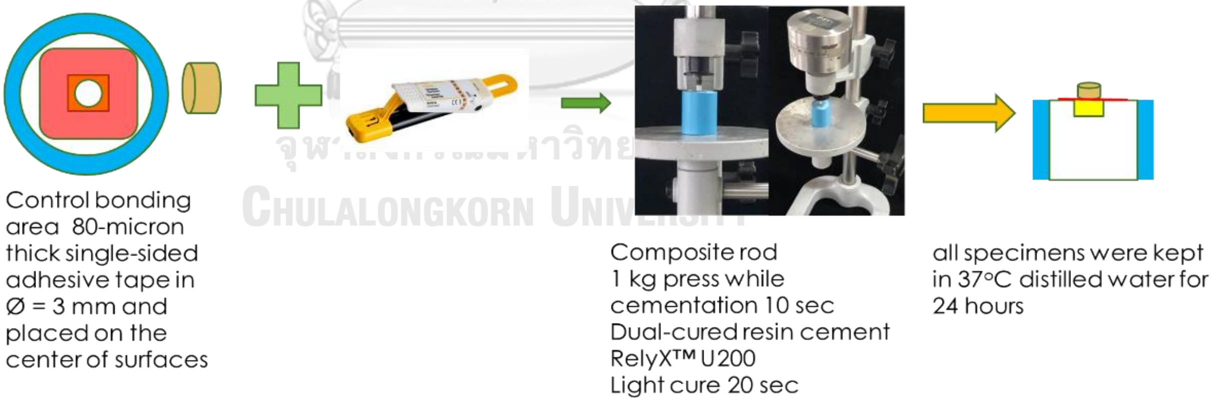


Figure 7 shows the steps of specimen cementation.

Part IV Shear bond strength

The bonded specimens were test with the notched-edge shear bond strength test applied from ISO 29022:2013 using a universal testing machine (Shimadzu, EZ-S 500N model, Japan). The specimen was placed in a metal sample holder, notched-edge shear blade was mounted on the universal testing machine and placed over the composite rod on the aligned specimen as show Figure 8. The blade was positioned precisely over the composite resin rod and force fitted without premature contact to ensure that the load was applied directly to the composite resin rod at a crosshead speed of 1.0 mm/min until failure occurred. Shear bond strength values were calculated in megapascals (MPa) by dividing the maximum load at failure (N) with the bonding area (mm^2). Subsequently, the failure modes were investigated under a stereomicroscope (Olympus Stereo Microscopes, SZ61, Japan) at a magnification of ×40. The failure modes were classified into the following categories: adhesive failure at the cement–materials interface, cohesive failure within the luting cement, cohesive failure in RMC materials and the mixed failure.

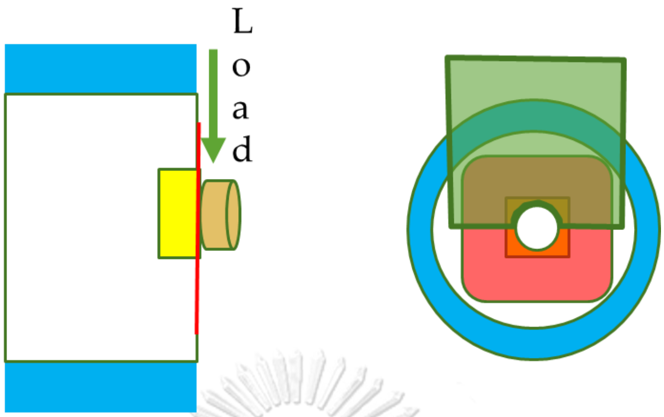


Figure 8 shows the universal testing machine with notched-edge shear bond strength testing.

Part V SEM

The specimens from each RMC material in the control group and the group applying THF for 1 minute, THF for 3 minutes and THF for 5 minutes were evaluated with scanning electron microscope (SEM) analysis (FEI Quanta 250) at ×2000 magnification. For the group with THF application for 3 minutes specimens were analyzed by Energy Dispersive X-ray Spectroscopy (EDX) point-measurements. Specimens in group No.1-10 were not investigated by SEM.

3.3 Statistics analysis

The bond strength values were analyzed by two-way analysis of variance (ANOVA) to determine the significant differences between the surface treatment methods and the different types of RMC at significance level of 0.05 with post hoc comparisons by Bonferroni tests (IBM SPSS Statics for Windows, Version 22.0. Armonk, NY, USA)

Chapter IV Results

4.1 Shear Bond Strength

The SBS are presented in table 5 and Figure 9. Most of the groups showed that the SBS significantly greater than the C group ($p < .05$), except for the T1 group. Also, Si/Ad group presented significantly higher SBS than group C, Ad, T1, T1/Ad ($p< .05$), but still lower than T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad group**.** For T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad showed SBS value between 17.44 -28.12 MPa, which were significantly higher than Ad and Si/Ad groups ($p < .05$).

The mean SBS value were increased when the time of THF application were increased till 3 minutes ,then decreased at 4 and 5 minutes following : T1/Si/Ad (18.58 ± 5.24 MPa), T2/Si/Ad (20.20 ± 5.66 MPa), T3/Si/Ad (25.37 ± 4.73 MPa), T4/Si/Ad (22.78 ± 3.37 MPa) T5/Si/Ad (22.04 ± 6.06 MPa). The highest SBS was found in group T3/Si/Ad but there was no significant difference when compared to T4/Si/Ad and T5/Si/Ad groups.

From the results in table 5, the control group showed Enamic (8.54± 1.56 MPa) had significant difference and the highest value followed by Cerasmart (3.89 ± 2.02 MPa) and Shofu Block HC (2.11 ± 1.22 MPa) respectively.

The adhesive agent group (Ad) revealed Enamic (11.29 ± 1.27 MPa) had the highest value followed by Cerasmart (9.15 ± 1.32 MPa) and Shofu Block HC (7.79 ± 2.12 MPa) respectively, but there was no significant difference.

The THF 1-minute applied group (T1) presented Enamic (6.22± 1.38 MPa) had the highest value followed by Cerasmart (2.44 ± 1.52MPa) and Shofu Block HC (1.89 ± 1.42 MPa) respectively, there was significant difference between Enamic and Shofu Block HC.

The silane with adhesive agent (Si/Ad) group showed Enamic (18.48 ± 4.21 MPa) had significant difference and the highest value followed by Cerasmart (14.1± 2.70 MPa) and Shofu Block HC (12.58 ± 2.56 MPa) respectively.

The THF 1-minute with adhesive agent group (T1/Ad) presented Cerasmart (10.21 ± 2.21 MPa) had the highest value followed by Enamic (9.69 ± 1.95 MPa) and Shofu Block HC (9.57± 3.59MPa) respectively, but there was no significant difference.

The THF 1-minute with adhesive agent and silane group (T1/Si/Ad) group revealed Enamic (20.36 ± 5.40 MPa) had the highest value followed by Cerasmart (17.94 ± 5.81 MPa) and Shofu Block HC (17.44 ± 4.50 MPa) respectively, but there was no significant difference.

The THF 2-minute with adhesive agent and silane group (T2/Si/Ad) group present Shofu Block HC (21.90 ±7.91MPa) had the highest value followed by Enamic (20.67 ± 3.36MPa) and Cerasmart (18.04 ± 4.54 MPa) respectively, but there was no significant difference.

The THF 3-minute with adhesive agent and silane group (T3/Si/Ad) group which had the highest mean SBS value indicated Enamic (28.12 ± 5.45 MPa) had the highest value followed by Cerasmart (24.69 ± 3.87MPa) and Shofu Block HC (23.31 ± 3.68 MPa) respectively, there was significant difference between Enamic and Shofu Block HC.

The THF 4-minute with adhesive agent and silane group (T4/Si/Ad) group presented Enamic (23.98 ± 3.84MPa) had the highest value followed by Cerasmart (22.65 ± 4.00 MPa) and Shofu Block HC (21.63 ± 1.68 MPa) respectively, but there was no significant difference.

The THF 5-minute with adhesive agent and silane group (T5/Si/Ad) group showed Enamic (22.62 ± 5.70 MPa) had the highest value followed by Cerasmart (22.13± 5.86 MPa) and Shofu Block HC (21.38 ± 7.12 MPa) respectively, but there was no significant difference.

The comparison of mean SBS among brands of RMC from table 6 demonstrated that VE showed the highest SBS (16.99 ± 8.01 MPa) followed by CS (14.52± 8.31 MPa) and HC (13.96 ± 8.88 MPa), respectively. The significant difference was founded in VE with CS and HC groups but there was no significant difference between CS and HC groups.

The two-way ANOVA analysis revealed that the shear bond strength values were significantly influenced by The RMC materials, the surface treatment methods, and the interaction between the RMC materials and surface treatment methods. (p = .001, .001, .222, F = 118.96, 16.91, 1.25, η_p^2 = .80, .11, .08, respectively) Moreover, RMC had a moderate effect size while surface treatment methods and interaction between the RMC materials and the surface treatment methods had a small effect size,(Cohen, 1992)(30).

4.2 Failure Mode

The frequencies of the failure modes observed are presented table 5 and Figure 10. Adhesive failure pattern was the most common failure mode found in C, Ad, T1, T1/Ad group. Mixed failure was also the most common failure mode showed in Si/Ad, T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad.

4.3 SEM Analysis

The SEM image at 2000x magnification in Figure.11-22 showed the different surface morphology of three RMC brands between the control and the THF groups. The specimen's surface treated with THF (Figure 14-22) presented more irregularities and white spot than the control group (Figure 11-13). The surface of the THF for 3-minute group presented more inorganic particle surface when compare with other groups. From the results of SEM/EDX in Figure 17-19 THF for 3 minutes (yellow circle) groups presented the majority of the inorganic particle was silicon element.

From the SEM/EDX image results, showed the different surface of the THF for 3-minute group (Figure 17-19) had more moderate irregularities and inorganic particle which was silicon element, corresponded to shear bond strength value in table 5 that T3/Si/Ad group showed the highest value when compare to other groups.

For the SEM in control group (Figure 11-13) presented low irregularities and white spot when compare to THF group which related to lower shear bond strength value in table 5.THF 1 minute (Figure 14-16) and THF 5-minute (Figure 20-22) groups presented mild irregularities and white spot which related to shear bond strength value in table 5,the T1/Si/Ad and T5/Si/Ad had higher shear bond strength than control group but still lower than T3/Si/Ad.

Chapter V Discussion

5.1 Discussion

The purpose of the present study was to determine the effect of surface treatment by using THF with silane and adhesive agent to three different RMC using dual-cured resin cement in term of shear bond strength. From previous studies, THF could be used as solvent in dental adhesive systems. THF not only showed increased bond strength stability and had an intermediate cytotoxicity close to HEMA but also increased bond strength value to glass fiber post by applied with silane.(11, 23, 28) In the present study, THF with silane and adhesive agent could be used as pre-treatment for improvement of shear bond strength to different RMC. Thus, the null hypothesis was rejected.

THF is an organic compound that is classified as heterocyclic compound, specifically a cyclic ether. THF is used as solvent for many polymers such as polyvinyl chloride, unvulcanized rubber , vinyls, polymer coating, cellophane, protective coatings.(31) According to Inoue and Hayashi study THF was used as the solvent to find that the residual Bis-GMA in resin composite(32). Väkiparta M et al found residual monomers, Bis-GMA and TEGDMA, in fiber-reinforced composites by using THF.(33) RMC materials combined two phases of materials; polymer matrix and condensed filled ceramics particles.(7, 9) Thus, the increased bond strength between RMC and the resin cement of present study could be explained by the fact that THF dissolves partial polymer part at the surface of material. Consequently, the surface of material shows more inorganic part (silica as shows in SEM/EDX results in Figure 17-19) which reacts and promotes adhesion by applying silane. In addition, the THF-3 minute groups were analyzed by EDX point-measurements and revealed moderate irregularities and inorganic particle related to the highest shear bond strength was found in group T3/Si/Ad (Table5).

The silane-coupling-agent acts as bifunctional monomer and adhesion promoter in silica-containing materials. There have been studies describing that silane-coupling-agent reacts to inorganic fillers exposed on surface of material. The other functional monomeric ends molecules of silane can react with the methacrylate groups of the adhesive resin and the integrated polymer parts of RMC materials.(34, 35) The primer in bonding also increased efficiency to bond the CAD/CAM composite blocks.(11, 36, 37) In addition, the use of methacrylate-containing primer combine with a silane-coupling agent increased the bond strength. Another explanation of adhesion mechanism is due to methacrylate monomers of the adhesive agent penetrating to the resin matrix of materials and polymerize to form the interpenetrating network.(27, 36) All explanations correspond to the results of this present study that using THF with silane and adhesive agent shows better improvement of the shear bond strength of RMC In addition, Enamic has the highest bond strength value of RMC in the present study (Table 6). This could result from the difference in the percentage of inorganic component and microstructure, correspond to previous study(38) found that silanization effect to Enamic more than other CAD/CAM composite blocks. In addition, inorganic part and microstructure of RMC affected to silanization.(38)

Mixed failure is correlated with the improved bond strength but adhesive failure means lower bond strength(34), which corresponds with the results in Fig.8 that the mixed failure was predominant type found in THF with silane and adhesive agent. Adhesive failure was commonly found in other groups. But the Si/Ad group mostly found mixed failure due to chemical reaction from silane.

Many surface treatment methods for the RMC materials were observed from previous studies(18-22), chemical and mechanical methods were often used to increased bond strength value. The chemical pre-treatment method which used HF and silane as chemical agents can improve bond strength. HF acid treatment in Enamic caused by partial dissolved the glassy phases and polymer which created microporosities and micromechanical retention surface. Silane application can increase the surface wetting of bonding area and improve a chemical bond to the resin cement and better bond strength as a consequence.(21) However, Cekic-Nagas et al.(18) showed that RMC treated with 10% HF acid gel did not have an effect on bond strength value between resin cement and RMC. In addition, HF acid causes irritation to tissue and considerable health hazard because of toxicity and volatility.(39) Due to, the controversial effects of HF acid to the bond strength of RMC, the surface treatment protocol by HF was not used in this study.

Sandblasting is the mechanical method which use to increase bond strength by improving micromechanical interlocking, and increasing wettability and surface area. (16, 20) However, there is study found that sandblasting to ceramics should be avoid because the materials occurred huge volume loss.(40) Also, Yoshihara et al. revealed that sandblasting created surface damage of Shofu Block HC and silane treatment cannot improve the bond strength for these material. Thus, Tekçe et al. stated that surface sandblasting for 60 seconds showed lower micro-tensile bond strength value when compared with shorter time of duration for Enamic.(41) However, sandblasting to RMC seem still have controversy for surface treatment because it may create microcracks in the surface and lead to premature failures also it effects internal and marginal adaptation.(16, 20, 26) As the results, there are no definite conclusion whether chemical or mechanical surface pre-treatment method is more appropriate for RMC materials.

Self-adhesive resin cement was chosen in this study because self-adhesive resin cement such as RelyX U200 is dual-cured resin cement, easy to use and has the improved mechanical and bonding properties in one step. Moreover, etching, priming and bonding are not necessary for this cement type and self-adhesive resin cement is mostly used in the dental practice.(42)

5.2 Limitation

Due to the limitation of this study shear bond strength was used in the present study because shear test is convenient to prepare specimen and a simple test process. However, shear test could not interpret interface failure as good as mini-dumbbell test.(43) In addition, the test was performed 24 hours after cementation which should have further investigation for increasing time storage, thermo-cycling or vary other resin cement systems.

5.3 Conclusion

THF could be as pre-treatment with silane and adhesive agent. This study showed the improvement of shear bond strength of RMC. Mixed failure pattern was most common failure mode in group of THF with silane and adhesive agent. Among RMC groups, Enamic showed the highest value of bond strength when compared with other materials.

Table 5 shows mean shear bond strength values (MPa ± SD) and number (%) of specimens according to failure mode.

Group	Vita Enamic		Cerasmart		Shofu Block HC		Total
	Mean SBS ± SD	Failure mode AF/CR/CM/MF	Mean SBS ± SD	Failure mode AF/CR/CM/MF	Mean SBS ± SD	Failure mode AF/CR/CM/MF	
C	8.54 ± 1.56	100 / 0 / 0 / 0	3.89 ± 2.02A	100 / 0 / 0 / 0	2.11 ± 1.22A	100 / 0 / 0 / 0	4.84 ± 3.17a
Ad	11.29 ± 1.27A	80 / 0 / 0 / 20	9.15 ± 1.32A	80 / 0 / 10 / 10	7.79 ± 2.12A	100 / 0 / 0 / 0	9.41 ± 2.14b
T1	6.22 ± 1.38A	100 / 0 / 0 / 0	2.44 ± 1.52AB	100 / 0 / 0 / 0	1.89 ± 1.42B	100 / 0 / 0 / 0	3.52 ± 2.40a
Si/Ad	18.48 ± 4.21	0 / 20 / 50 /30	14.10 ± 2.70A	40 / 0/ 10 / 50	12.58 ± 2.56A	40 / 0 / 0 / 60	15.05 ± 4.03
T1/Ad	9.69 ± 1.95A	80 / 10 / 0 / 10	10.21 ± 2.21A	90 / 0 / 0 / 10	9.57 ± 3.59A	80 / 10 / 0 / 10	9.82 ± 2.60b
T1/Si/Ad	20.36 ± 5.40A	0 / 10 / 40 / 50	17.94 ± 5.81A	40 / 0 / 30 / 30	17.44 ± 4.50A	30 / 0 / 40 / 30	18.58 ± 5.24c
T2/Si/Ad	20.67 ± 3.36A	10 / 0 / 20 / 70	18.04 ± 4.54A	20 / 0 / 20 / 60	21.90 ± 7.91A	20 / 0 / 10 / 70	20.20 ± 5.66cd
T3/Si/Ad	28.12 ± 5.45A	0 / 0 / 30 / 70	24.69 ± 3.87AB	20 / 0 / 10 / 70	23.31 ± 3.68B	30 / 0 / 20 / 50	25.37 ± 4.73e
T4/Si/Ad	23.98 ± 3.84A	0 / 0 / 40 / 60	22.65 ± 4.00A	10 / 0 / 20 / 70	21.63 ± 1.68A	30 / 0 / 10 / 60	22.78 ± 3.37de
T5/Si/Ad	22.62 ± 5.70A	0 / 0 / 30 / 70	22.13 ± 5.86A	20 / 0 / 10 / 70	21.38 ± 7.12A	10 / 0 / 20 / 70	22.04 ± 6.06de

Mean values represented with same superscript uppercase letters (row) or lowercase letters (column) are not significantly to Bonferroni multiple comparison test ($p>0.05$).Percentage of failure mode [AF : adhesive failure at the cement–materials interface / CR : cohesive failure within the luting cement / CM: cohesive failure in RMC materials / MF : mixed failure].

Table 6 shows mean of shear bond strength value classified by brand.

Brand	Mean ± SD (MPa)
VE	16.99^{A} ± 8.01
HC	13.96^{B} ± 8.88
CS	14.52^{B} ± 8.31

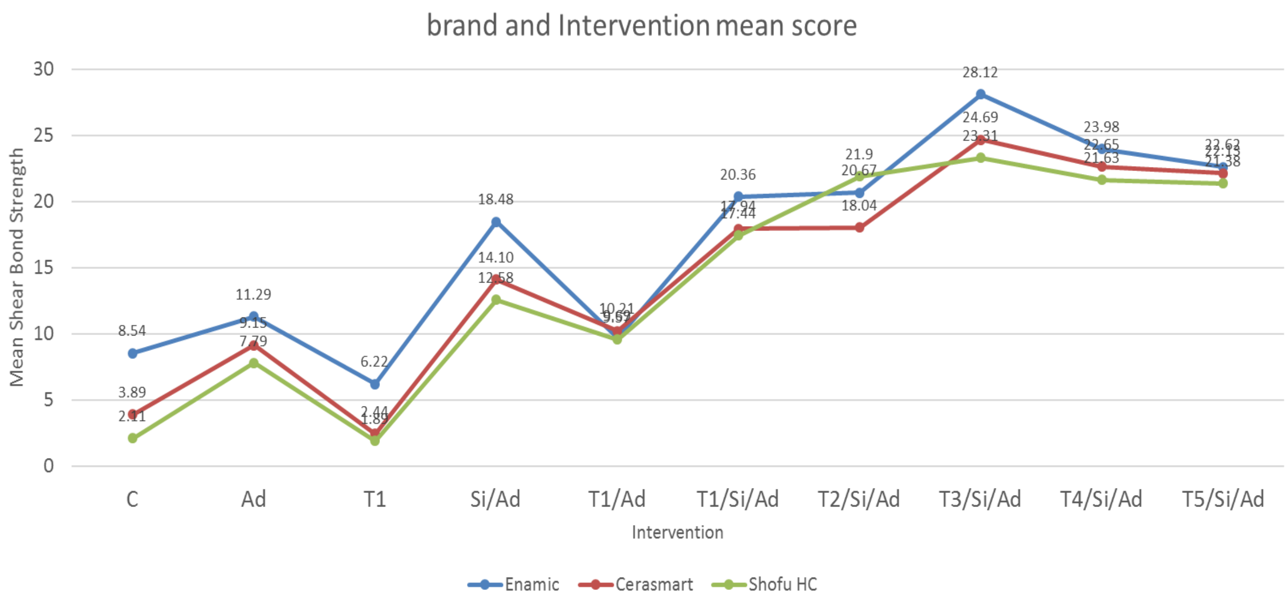


Figure 9 shows the graphs of mean shear bond strength values of control and experimental groups.

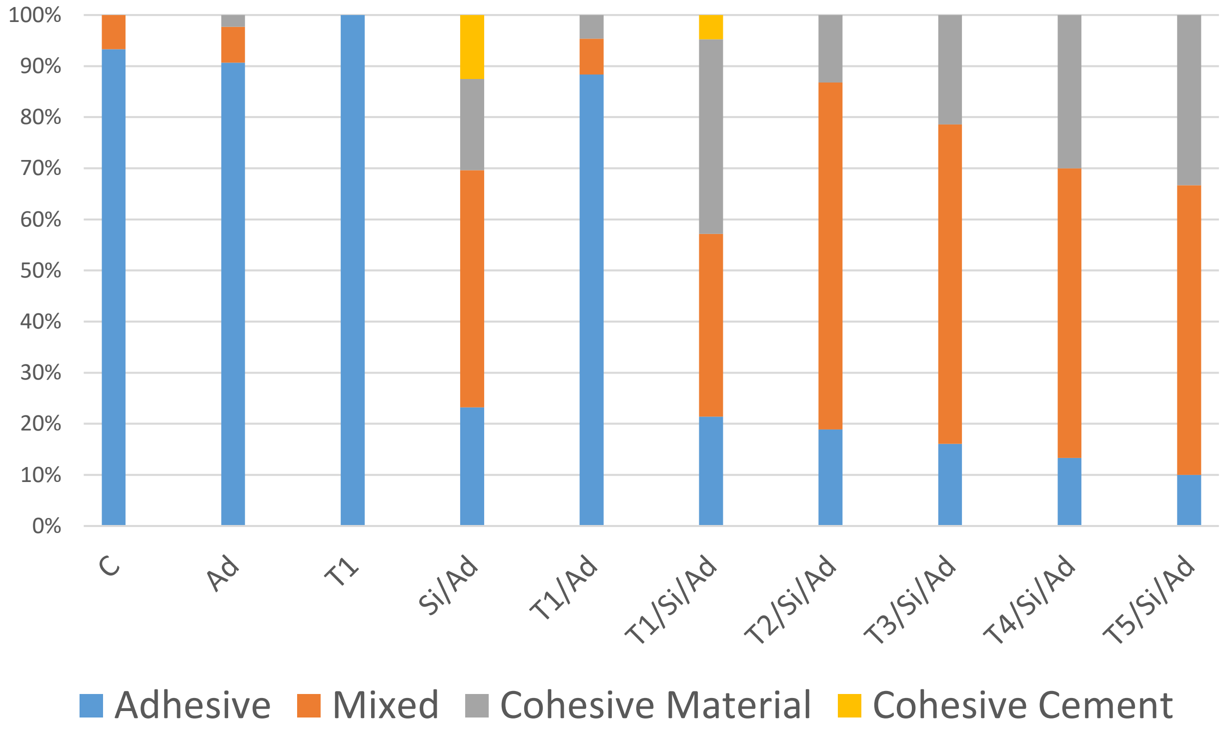


Figure 10 shows the percentages of failure mode of control and experimental groups.

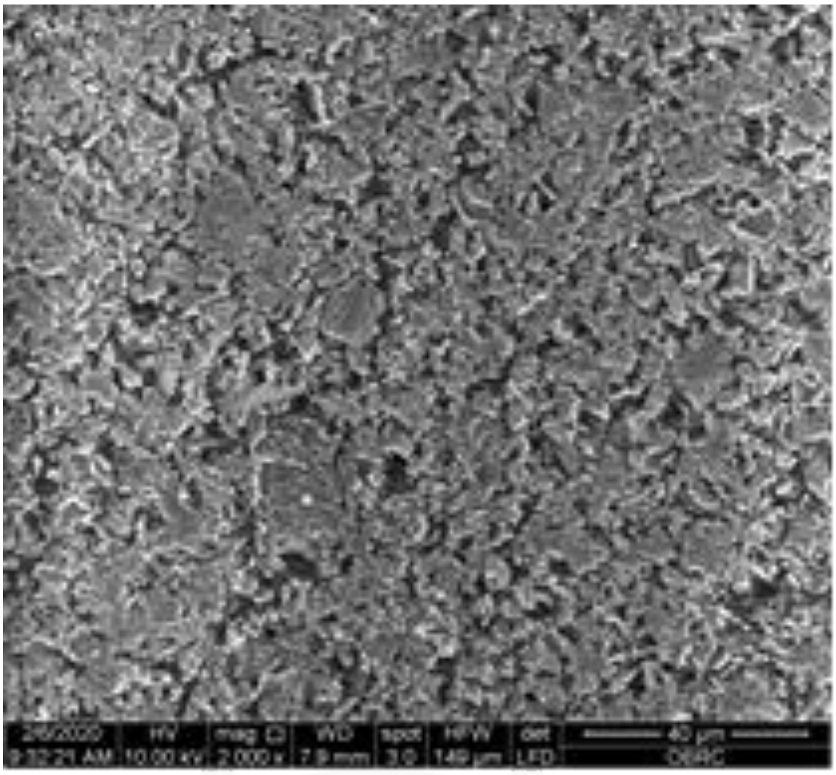


Figure 11 shows the SEM image at 2000x magnification of control group (Enamic).

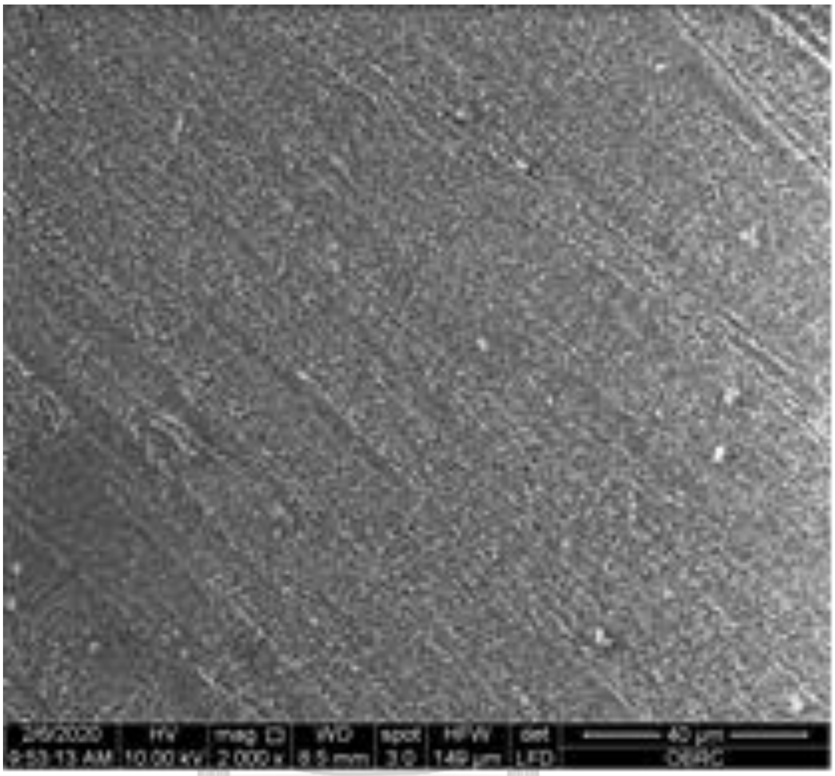


Figure 12 shows the SEM image at 2000x magnification of control group (Cerasmart).

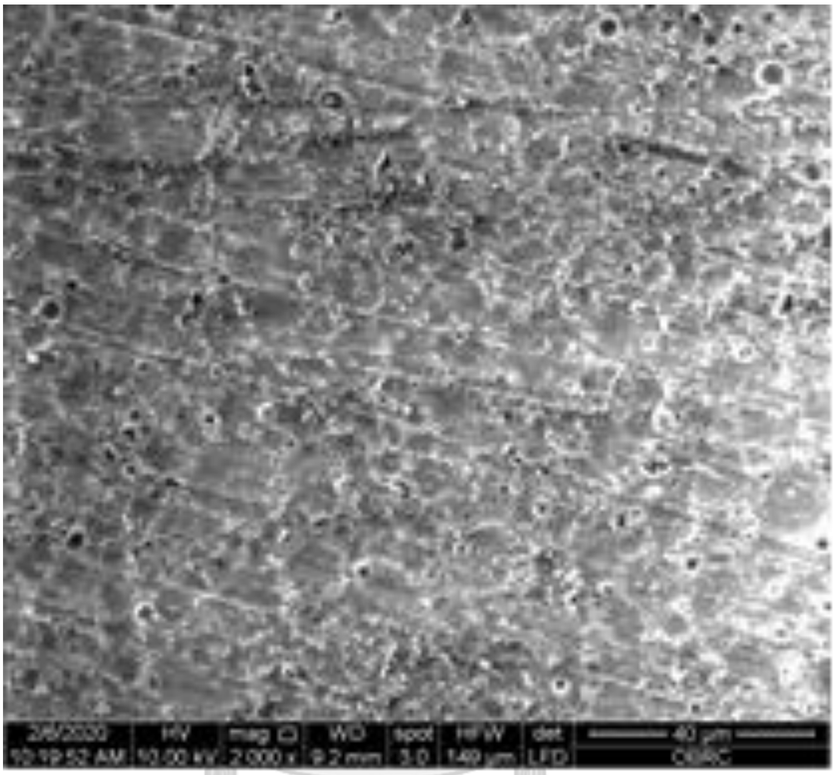


Figure 13 shows the SEM image at 2000x magnification of control group (Shofu Block HC).

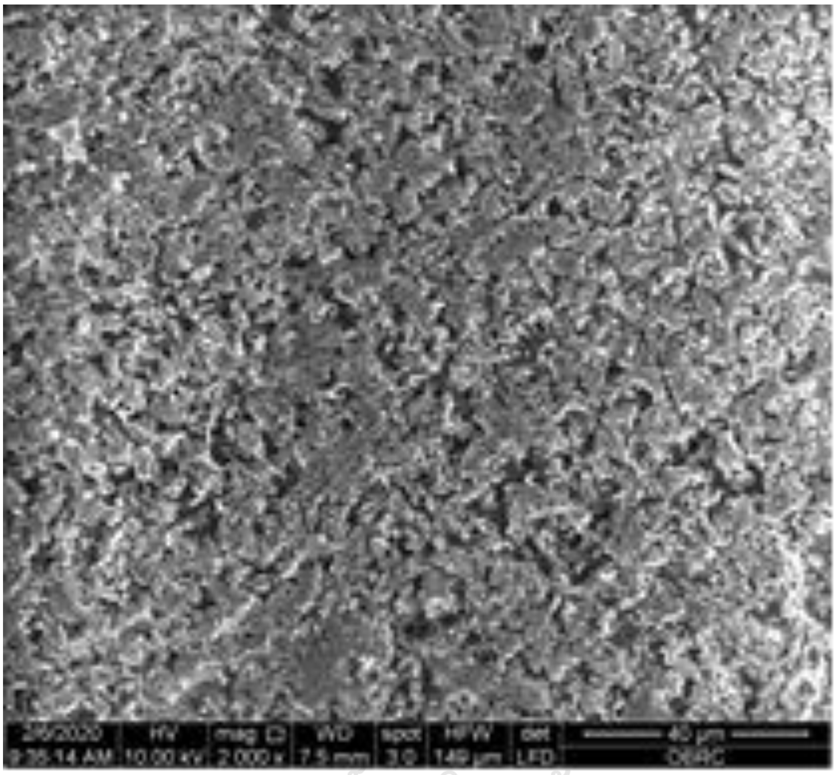


Figure 14 shows the SEM image at 2000x magnification of THF 1-minute group (Enamic).

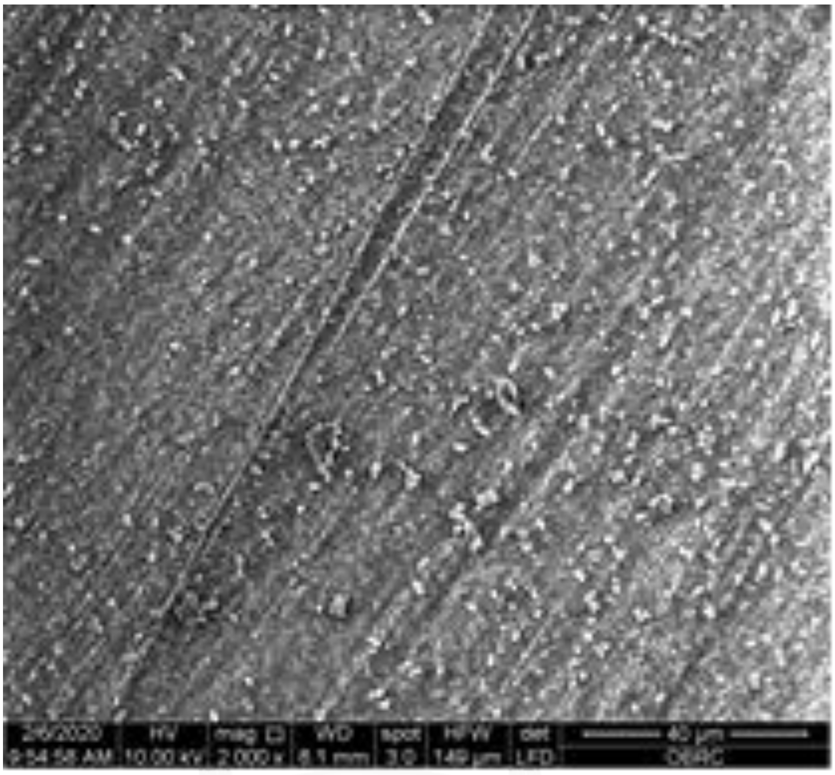


Figure 15 shows the SEM image at 2000x magnification of THF 1-minute group (Cerasmart).

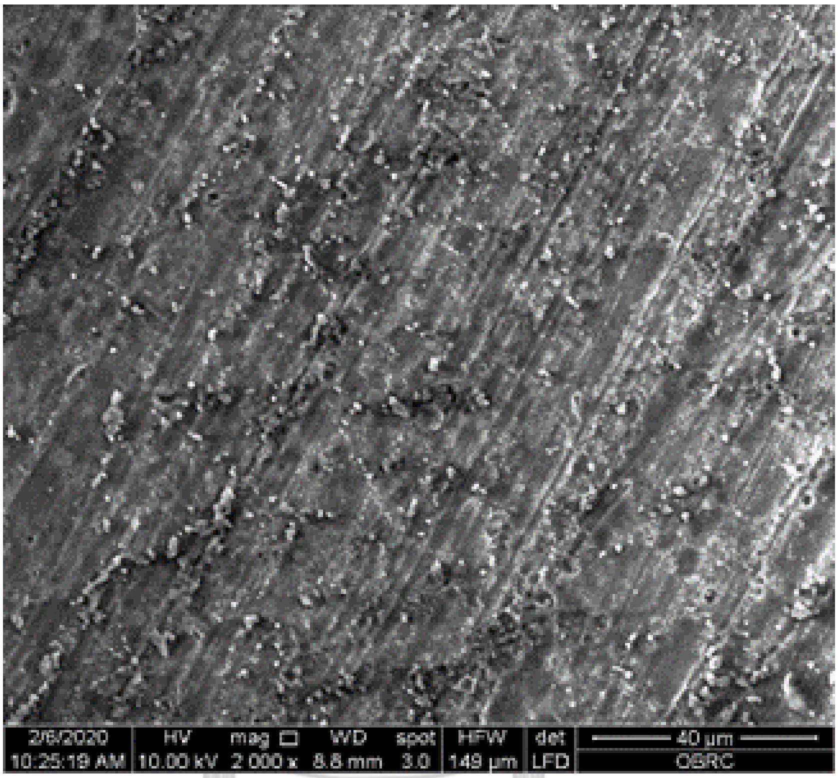


Figure 16 shows the SEM image at 2000x magnification of THF 1-minute group (Shofu Block HC).

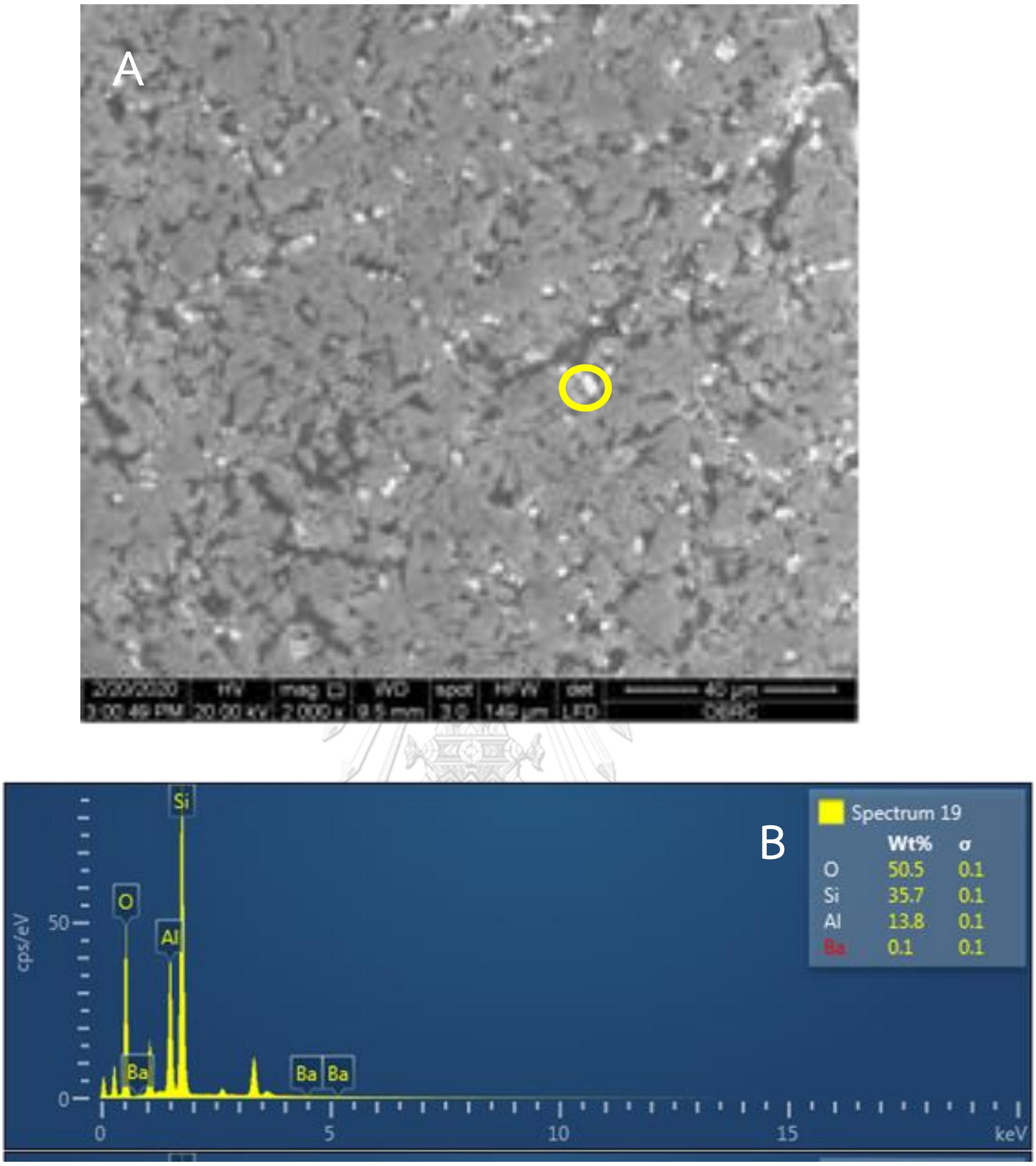


Figure 17 (A) shows the SEM image at 2000x magnification of THF 3-minute group (Enamic) presented more irregularities and moderate white spot, (B) shows the EDX image of silicon element (yellow circle).

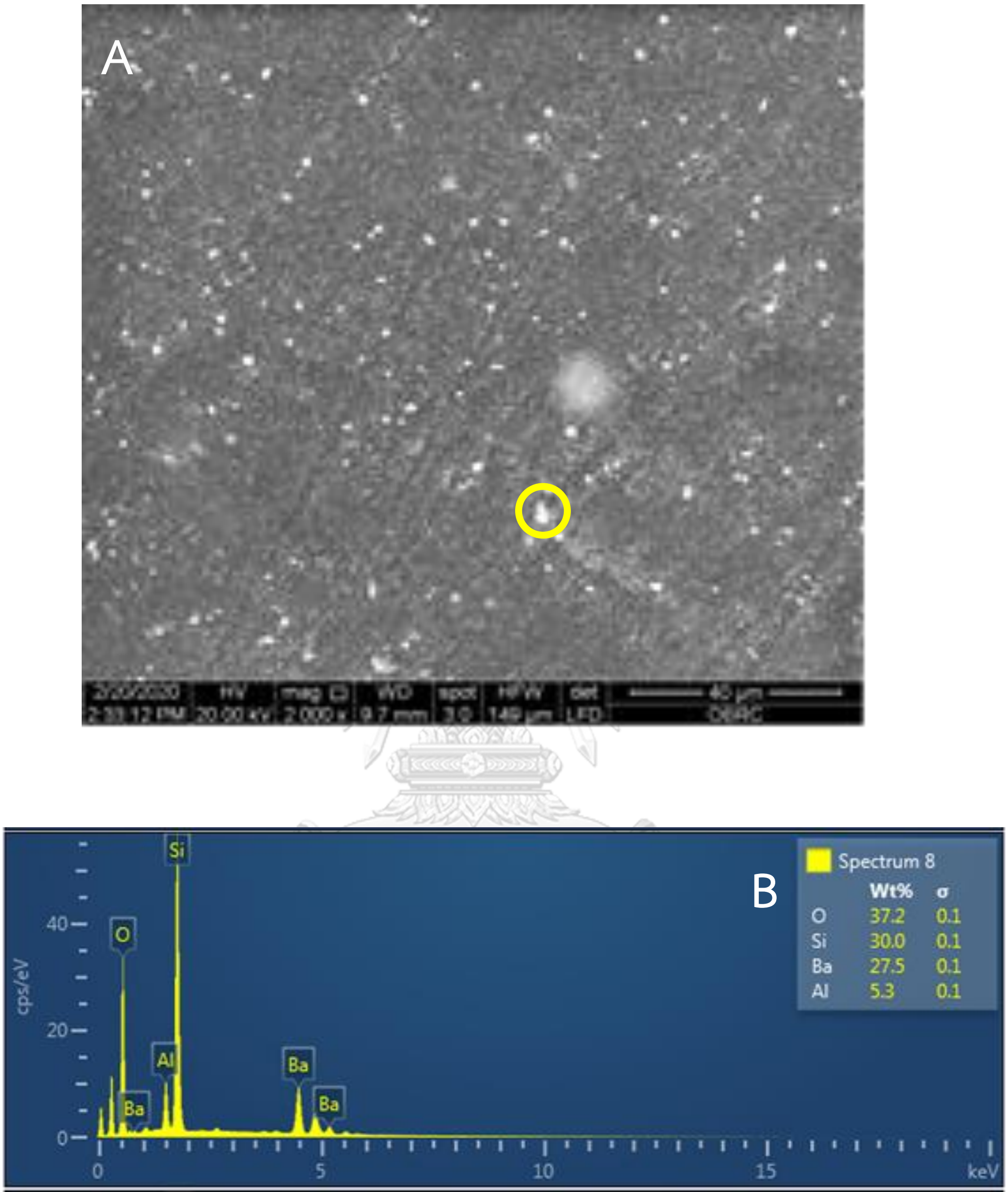


Figure 18 (A) shows the SEM image at 2000x magnification of THF 3-minute group (Cerasmart) presented more irregularities and moderate white spot, (B) shows the EDX image of silicon element (yellow circle).

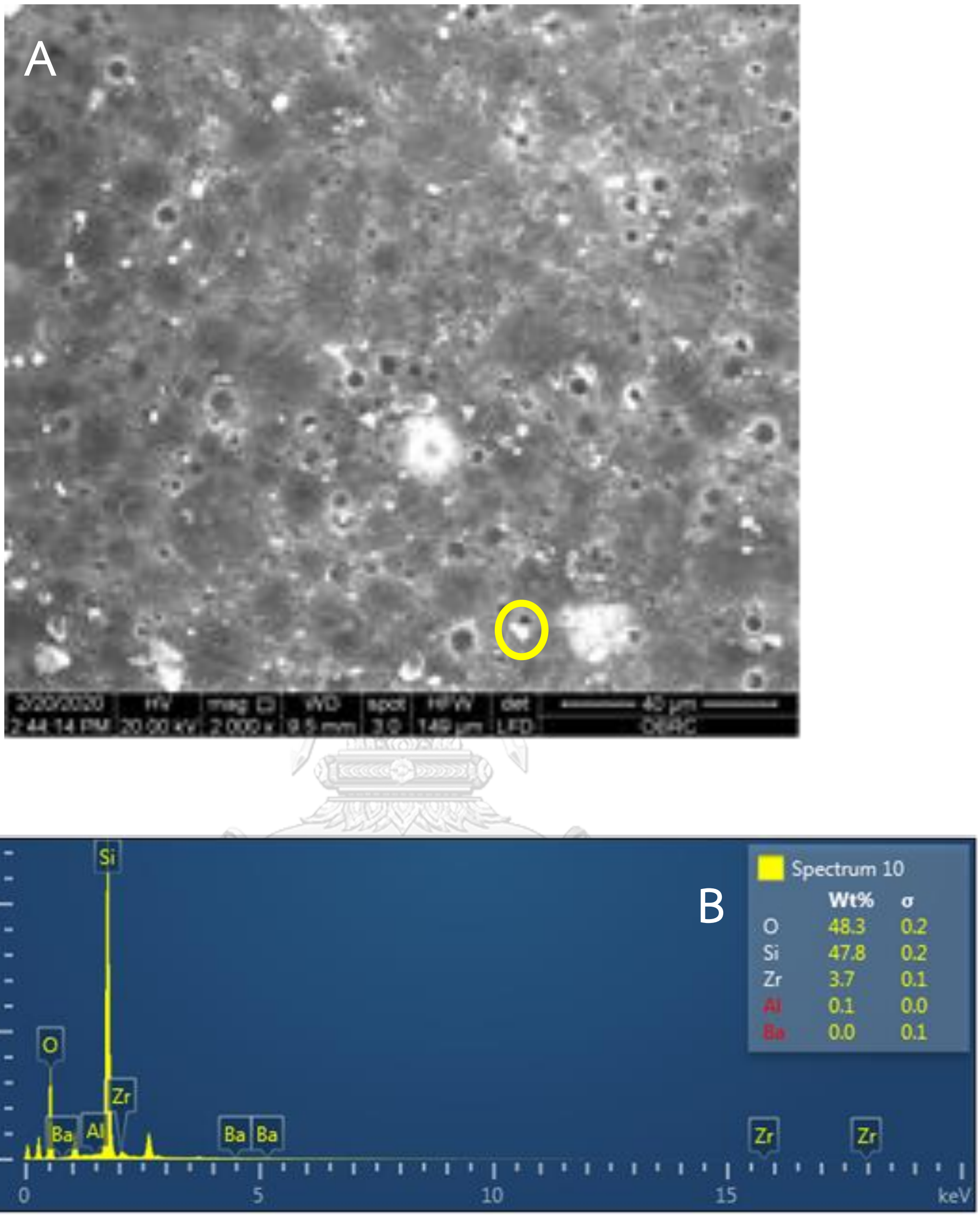


Figure 19 (A) shows the SEM image at 2000x magnification of THF 3-minute group (Shofu Block HC) presented more irregularities and moderate white spot, (B) shows the EDX image of silicon element (yellow circle).

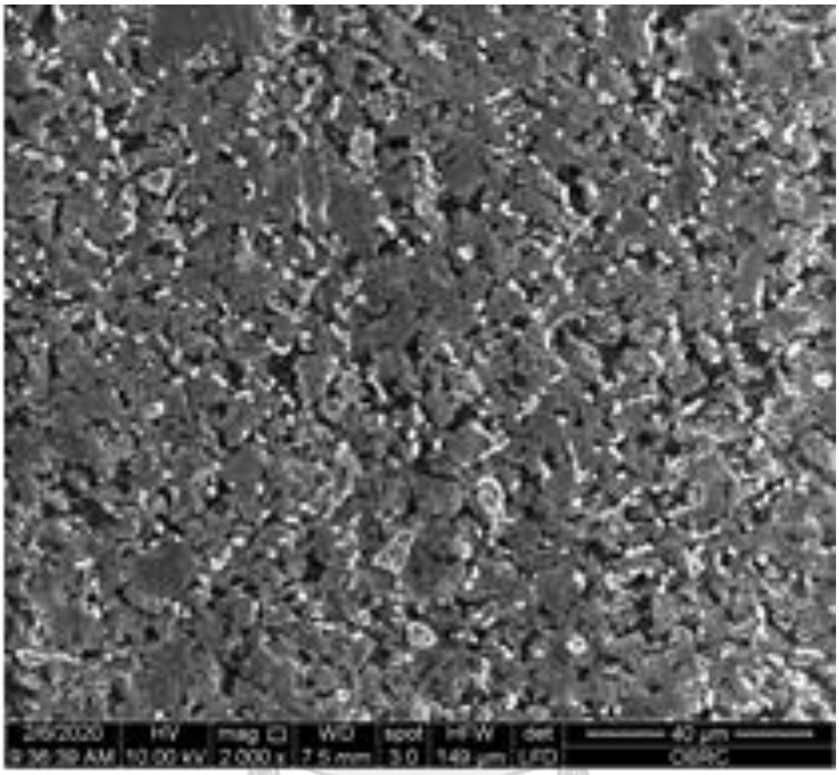


Figure 20 shows the SEM image at 2000x magnification of THF 5-minute group (Enamic).

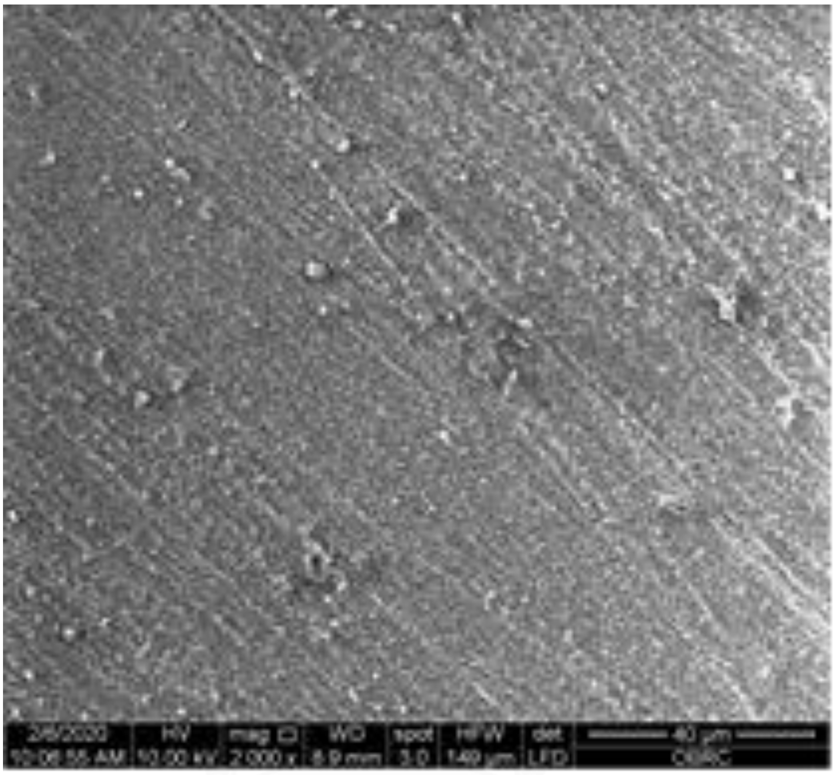


Figure 21 shows SEM image at 2000x magnification of THF 5-minute group (Cerasmart).

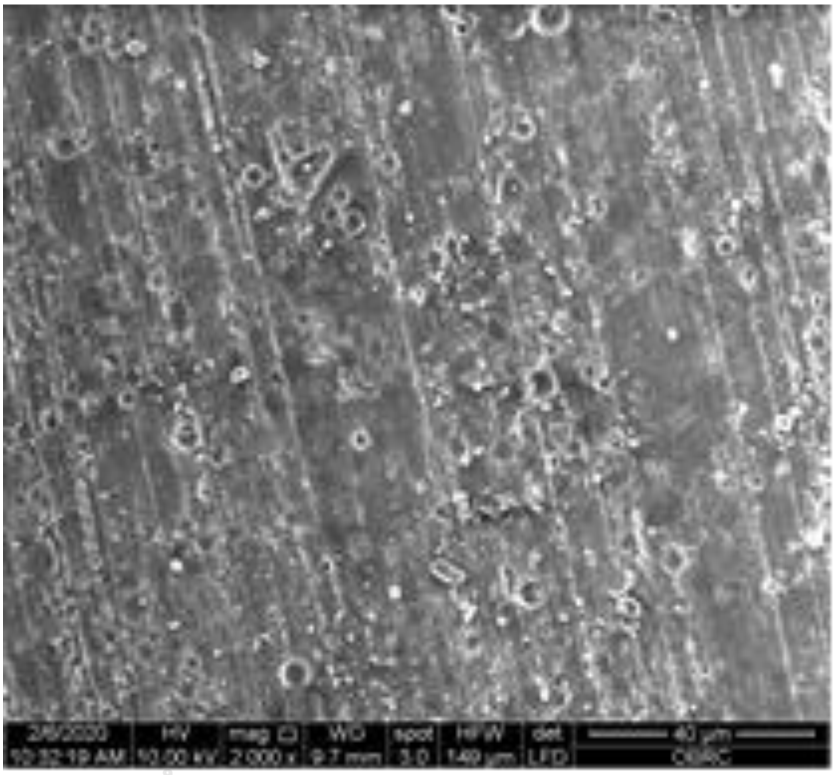


Figure 22 shows the SEM image at 2000x magnification of THF 5-minute group (Shofu Block HC).

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APPENDIX

Table 7 Descriptive Statistics

Dependent Variable: Bond Strength

Intervention	brand	Mean	Std. Deviation	N
Control	Enamic	8.5360	1.56070	10
	Shofu	2.1170	1.21985	10
	Cerasmart	3.8930	2.02016	10
	Total	4.8487	3.17187	30
Adhesive	Enamic	11.2870	1.27059	10
	Shofu	7.7910	2.12049	10
	Cerasmart	9.1480	1.31975	10
	Total	9.4087	2.13996	30
THF1M	Enamic	6.2220	1.37600	10
	Shofu	1.8880	1.42031	10
	Cerasmart	2.4380	1.52420	10
	Total	3.5160	2.40297	30
THF1M+Adhesive	Enamic	9.6860	1.95052	10
	Shofu	9.5710	3.58896	10
	Cerasmart	10.2080	2.20946	10
	Total	9.8217	2.60243	30
Silane+ Adhesive	Enamic	18.4820	4.20703	10
	Shofu	12.5770	2.56727	10

	Cerasmart	14.1040	2.69980	10
	Total	15.0543	4.03477	30
THF1M+Silane+Adhesive	Enamic	20.3560	5.39711	10
	Shofu	17.4400	4.50239	10
	Cerasmart	17.9430	5.81549	10
	Total	18.5797	5.24430	30
THF2M+Silane+Adhesive	Enamic	20.6680	3.36349	10
	Shofu	21.8990	7.91424	10
	Cerasmart	18.0400	4.53693	10
	Total	20.2023	5.65838	30
THF3M+Silane+Adhesive	Enamic	28.1220	5.45276	10
	Shofu	23.3100	3.68422	10
	Cerasmart	24.6920	3.87430	10
	Total	25.3747	4.72562	30
THF4M+Silane+Adhesive	Enamic	23.9790	3.84042	10
	Shofu	21.6360	1.67588	10
	Cerasmart	22.6500	4.00156	10
	Total	22.7550	3.37201	30
THF5M+Silane+Adhesive	Enamic	22.6230	5.69931	10
	Shofu	21.3780	7.12317	10
	Cerasmart	22.1320	5.85509	10
	Total	22.0443	6.06118	30

Total	Enamic	16.9961	8.01036	100
	Shofu	13.9607	8.88468	100
	Cerasmart	14.5248	8.31313	100
	Total	15.1605	8.48573	300

Table 8 Levene's Test of Equality of Error Variances[a]

Dependent Variable: Bond Strength

F	df1	df2	Sig.
5.178	29	270	.000

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + Intervention + brand + Intervention * brand

Table 9 Tests of Between-Subjects Effects

Dependent Variable: Bond Strength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	17368.842[a]	29	598.926	38.859	.000	.807
Intercept	68952.531	1	68952.531	4473.764	.000	.943
Intervention	16500.896	9	1833.433	118.956	.000	.799
brand	521.306	2	260.653	16.912	.000	.111
Intervention * brand	346.640	18	19.258	1.249	.222	.077
Error	4161.414	270	15.413			
Total	90482.788	300				
Corrected Total	21530.256	299				

a. R Squared = .807 (Adjusted R Squared = .786)

Table 10 Estimates : Intervention

Dependent Variable: Bond Strength

Intervention	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
Control	4.849	.717	3.438	6.260
Adhesive	9.409	.717	7.998	10.820
THF1M	3.516	.717	2.105	4.927
THF1M+Adhesive	9.822	.717	8.411	11.233
Silane+ Adhesive	15.054	.717	13.643	16.465
THF1M+Silane+Adhesive	18.580	.717	17.169	19.991
THF2M+Silane+Adhesive	20.202	.717	18.791	21.613
THF3M+Silane+Adhesive	25.375	.717	23.964	26.786
THF4M+Silane+Adhesive	22.755	.717	21.344	24.166
THF5M+Silane+Adhesive	22.044	.717	20.633	23.455

Table 11 Pairwise Comparisons

Dependent Variable: Bond Strength

(I) Intervention	(J) Intervention	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b] Lower Bound	Upper Bound
Control	Adhesive	-4.560*	1.014	.000	-7.901	-1.219
	THF1M	1.333	1.014	1.000	-2.009	4.674
	THF1M+Adhesive	-4.973*	1.014	.000	-8.314	-1.632
	Silane+ Adhesive	-10.206*	1.014	.000	-13.547	-6.864
	THF1M+Silane+Adhesive	-13.731*	1.014	.000	-17.072	-10.390
	THF2M+Silane+Adhesive	-15.354*	1.014	.000	-18.695	-12.012
	THF3M+Silane+Adhesive	-20.526*	1.014	.000	-23.867	-17.185
	THF4M+Silane+Adhesive	-17.906*	1.014	.000	-21.248	-14.565
	THF5M+Silane+Adhesive	-17.196*	1.014	.000	-20.537	-13.854

Adhesive	Control	4.560*	1.014	.000	1.219	7.901
	THF1M	5.893*	1.014	.000	2.551	9.234
	THF1M+Adhesive	-.413	1.014	1.000	-3.754	2.928
	Silane+ Adhesive	-5.646*	1.014	.000	-8.987	-2.304
	THF1M+Silane+Adhesive	-9.171*	1.014	.000	-12.512	-5.830
	THF2M+Silane+Adhesive	-10.794*	1.014	.000	-14.135	-7.452
	THF3M+Silane+Adhesive	-15.966*	1.014	.000	-19.307	-12.625
	THF4M+Silane+Adhesive	-13.346*	1.014	.000	-16.688	-10.005
	THF5M+Silane+Adhesive	-12.636*	1.014	.000	-15.977	-9.294
THF1M	Control	-1.333	1.014	1.000	-4.674	2.009
	Adhesive	-5.893*	1.014	.000	-9.234	-2.551
	THF1M+Adhesive	-6.306*	1.014	.000	-9.647	-2.964
	Silane+ Adhesive	-11.538*	1.014	.000	-14.880	-8.197
	THF1M+Silane+Adhesive	-15.064*	1.014	.000	-18.405	-11.722
	THF2M+Silane+Adhesive	-16.686*	1.014	.000	-20.028	-13.345

	THF3M+Silane+Adhesive	-21.859*	1.014	.000	-25.200	-18.517
	THF4M+Silane+Adhesive	-19.239*	1.014	.000	-22.580	-15.898
	THF5M+Silane+Adhesive	-18.528*	1.014	.000	-21.870	-15.187
THF1M+Adhesive	Control	4.973*	1.014	.000	1.632	8.314
	Adhesive	.413	1.014	1.000	-2.928	3.754
	THF1M	6.306*	1.014	.000	2.964	9.647
	Silane+ Adhesive	-5.233*	1.014	.000	-8.574	-1.891
	THF1M+Silane+Adhesive	-8.758*	1.014	.000	-12.099	-5.417
	THF2M+Silane+Adhesive	-10.381*	1.014	.000	-13.722	-7.039
	THF3M+Silane+Adhesive	-15.553*	1.014	.000	-18.894	-12.212
	THF4M+Silane+Adhesive	-12.933*	1.014	.000	-16.275	-9.592
	THF5M+Silane+Adhesive	-12.223*	1.014	.000	-15.564	-8.881
Silane+ Adhesive	Control	10.206*	1.014	.000	6.864	13.547
	Adhesive	5.646*	1.014	.000	2.304	8.987
	THF1M	11.538*	1.014	.000	8.197	14.880
	THF1M+Adhesive	5.233*	1.014	.000	1.891	8.574

	THF1M+Silane+Adhesive	-3.525*	1.014	.027	-6.867	-.184
	THF2M+Silane+Adhesive	-5.148*	1.014	.000	-8.489	-1.807
	THF3M+Silane+Adhesive	-10.320*	1.014	.000	-13.662	-6.979
	THF4M+Silane+Adhesive	-7.701*	1.014	.000	-11.042	-4.359
	THF5M+Silane+Adhesive	-6.990*	1.014	.000	-10.331	-3.649
THF1M+Silane+Adhesive	Control	13.731*	1.014	.000	10.390	17.072
	Adhesive	9.171*	1.014	.000	5.830	12.512
	THF1M	15.064*	1.014	.000	11.722	18.405
	THF1M+Adhesive	8.758*	1.014	.000	5.417	12.099
	Silane+ Adhesive	3.525*	1.014	.027	.184	6.867
	THF2M+Silane+Adhesive	-1.623	1.014	1.000	-4.964	1.719
	THF3M+Silane+Adhesive	-6.795*	1.014	.000	-10.136	-3.454
	THF4M+Silane+Adhesive	-4.175*	1.014	.002	-7.517	-.834
	THF5M+Silane+Adhesive	-3.465*	1.014	.033	-6.806	-.123
THF2M+Silane+Adhe	Control	15.354*	1.014	.000	12.012	18.695

sive	Adhesive	10.794*	1.014	.000	7.452	14.135
	THF1M	16.686*	1.014	.000	13.345	20.028
	THF1M+Adhesive	10.381*	1.014	.000	7.039	13.722
	Silane+ Adhesive	5.148*	1.014	.000	1.807	8.489
	THF1M+Silane+Adhesive	1.623	1.014	1.000	-1.719	4.964
	THF3M+Silane+Adhesive	-5.172*	1.014	.000	-8.514	-1.831
	THF4M+Silane+Adhesive	-2.553	1.014	.557	-5.894	.789
	THF5M+Silane+Adhesive	-1.842	1.014	1.000	-5.183	1.499
THF3M+Silane+Adhesive	Control	20.526*	1.014	.000	17.185	23.867
	Adhesive	15.966*	1.014	.000	12.625	19.307
	THF1M	21.859*	1.014	.000	18.517	25.200
	THF1M+Adhesive	15.553*	1.014	.000	12.212	18.894
	Silane+ Adhesive	10.320*	1.014	.000	6.979	13.662
	THF1M+Silane+Adhesive	6.795*	1.014	.000	3.454	10.136
	THF2M+Silane+Adhesive	5.172*	1.014	.000	1.831	8.514
	THF4M+Silane+Adhesive	2.620	1.014	.463	-.722	5.961

	THF5M+Silane+Adhesive	3.330	1.014	.052	-.011	6.672
THF4M+Silane+Adhesive	Control	17.906*	1.014	.000	14.565	21.248
	Adhesive	13.346*	1.014	.000	10.005	16.688
	THF1M	19.239*	1.014	.000	15.898	22.580
	THF1M+Adhesive	12.933*	1.014	.000	9.592	16.275
	Silane+ Adhesive	7.701*	1.014	.000	4.359	11.042
	THF1M+Silane+Adhesive	4.175*	1.014	.002	.834	7.517
	THF2M+Silane+Adhesive	2.553	1.014	.557	-.789	5.894
	THF3M+Silane+Adhesive	-2.620	1.014	.463	-5.961	.722
	THF5M+Silane+Adhesive	.711	1.014	1.000	-2.631	4.052
THF5M+Silane+Adhesive	Control	17.196*	1.014	.000	13.854	20.537
	Adhesive	12.636*	1.014	.000	9.294	15.977
	THF1M	18.528*	1.014	.000	15.187	21.870
	THF1M+Adhesive	12.223*	1.014	.000	8.881	15.564
	Silane+ Adhesive	6.990*	1.014	.000	3.649	10.331
	THF1M+Silane+Adhesive	3.465*	1.014	.033	.123	6.806

	THF2M+Silane+Adhesive	1.842	1.014	1.000	-1.499	5.183
	THF3M+Silane+Adhesive	-3.330	1.014	.052	-6.672	.011
	THF4M+Silane+Adhesive	-.711	1.014	1.000	-4.052	2.631

Based on estimated marginal means

*. The mean difference is significant at the

b. Adjustment for multiple comparisons: Bonferroni.

Table 12 Univariate Tests

Dependent Variable: Bond Strength

	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Contrast	16500.896	9	1833.433	118.956	.000	.799
Error	4161.414	270	15.413			

The F tests the effect of Intervention. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

Table 13 Pairwise Comparisons

Dependent Variable: Bond Strength

(I) brand	(J) brand	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
					Lower Bound	Upper Bound
Enamic	Shofu	3.035*	.555	.000	1.698	4.373
	Cerasmart	2.471*	.555	.000	1.134	3.809
Shofu	Enamic	-3.035*	.555	.000	-4.373	-1.698
	Cerasmart	-.564	.555	.932	-1.902	.773
Cerasmart	Enamic	-2.471*	.555	.000	-3.809	-1.134
	Shofu	.564	.555	.932	-.773	1.902

Based on estimated marginal means

*. The mean difference is significant at the

b. Adjustment for multiple comparisons: Bonferroni.

Table 14 Univariate Tests

Dependent Variable: Bond Strength

	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Contrast	521.306	2	260.653	16.912	.000	.111
Error	4161.414	270	15.413			

The F tests the effect of brand. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

Table 15 Estimates :. brand

Dependent Variable: Bond Strength

brand	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
Enamic	16.996	.393	16.223	17.769
Shofu	13.961	.393	13.188	14.734
Cerasmart	14.525	.393	13.752	15.298

Table 16 Pairwise Comparisons

Dependent Variable: Bond Strength

(I) brand	(J) brand	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
					Lower Bound	Upper Bound
Enamic	Shofu	3.035*	.555	.000	1.698	4.373
	Cerasmart	2.471*	.555	.000	1.134	3.809
Shofu	Enamic	-3.035*	.555	.000	-4.373	-1.698
	Cerasmart	-.564	.555	.932	-1.902	.773
Cerasmart	Enamic	-2.471*	.555	.000	-3.809	-1.134
	Shofu	.564	.555	.932	-.773	1.902

Based on estimated marginal means

*. The mean difference is significant at the

b. Adjustment for multiple comparisons: Bonferroni.

Table 17 Estimates Intervention * brand

Dependent Variable: Bond Strength

Intervention	brand	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
Control	Enamic	8.536	1.241	6.092	10.980
	Shofu	2.117	1.241	-.327	4.561
	Cerasmart	3.893	1.241	1.449	6.337
Adhesive	Enamic	11.287	1.241	8.843	13.731
	Shofu	7.791	1.241	5.347	10.235
	Cerasmart	9.148	1.241	6.704	11.592
THF1M	Enamic	6.222	1.241	3.778	8.666
	Shofu	1.888	1.241	-.556	4.332
	Cerasmart	2.438	1.241	-.006	4.882
THF1M+Adhesive	Enamic	9.686	1.241	7.242	12.130
	Shofu	9.571	1.241	7.127	12.015
	Cerasmart	10.208	1.241	7.764	12.652
Silane+ Adhesive	Enamic	18.482	1.241	16.038	20.926
	Shofu	12.577	1.241	10.133	15.021
	Cerasmart	14.104	1.241	11.660	16.548
THF1M+Silane+Ad	Enamic	20.356	1.241	17.912	22.800

hesive	Shofu	17.440	1.241	14.996	19.884
	Cerasmart	17.943	1.241	15.499	20.387
THF2M+Silane+Adhesive	Enamic	20.668	1.241	18.224	23.112
	Shofu	21.899	1.241	19.455	24.343
	Cerasmart	18.040	1.241	15.596	20.484
THF3M+Silane+Adhesive	Enamic	28.122	1.241	25.678	30.566
	Shofu	23.310	1.241	20.866	25.754
	Cerasmart	24.692	1.241	22.248	27.136
THF4M+Silane+Adhesive	Enamic	23.979	1.241	21.535	26.423
	Shofu	21.636	1.241	19.192	24.080
	Cerasmart	22.650	1.241	20.206	25.094
THF5M+Silane+Adhesive	Enamic	22.623	1.241	20.179	25.067
	Shofu	21.378	1.241	18.934	23.822
	Cerasmart	22.132	1.241	19.688	24.576

Table 18 Univariate Tests

Dependent Variable: Bond Strength

	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Contrast	521.306	2	260.653	16.912	.000	.111
Error	4161.414	270	15.413			

The F tests the effect of brand. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

Table 19 Pairwise Comparisons

Dependent Variable: Bond Strength

Intervention	(I) brand	(J) brand	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b] Lower Bound	Upper Bound
Control	Enamic	Shofu	6.419*	1.756	.001	2.190	10.648
		Cerasmart	4.643*	1.756	.026	.414	8.872
	Shofu	Enamic	-6.4.1219*	1.756	.001	-10.648	-2.190
		Cerasmart	-1.776	1.756	.938	-6.005	2.453
	Cerasmart	Enamic	-4.643*	1.756	.026	-8.872	-.414
		Shofu	1.776	1.756	.938	-2.453	6.005

Adhesive	Enamic	Shofu	3.496	1.756	.142	-.733	7.725
		Cerasmart	2.139	1.756	.673	-2.090	6.368
	Shofu	Enamic	-3.496	1.756	.142	-7.725	.733
		Cerasmart	-1.357	1.756	1.000	-5.586	2.872
	Cerasmart	Enamic	-2.139	1.756	.673	-6.368	2.090
		Shofu	1.357	1.756	1.000	-2.872	5.586
THF1M	Enamic	Shofu	4.334*	1.756	.043	.105	8.563
		Cerasmart	3.784	1.756	.096	-.445	8.013
	Shofu	Enamic	-4.334*	1.756	.043	-8.563	-.105
		Cerasmart	-.550	1.756	1.000	-4.779	3.679
	Cerasmart	Enamic	-3.784	1.756	.096	-8.013	.445
		Shofu	.550	1.756	1.000	-3.679	4.779
THF1M+Adhesive	Enamic	Shofu	.115	1.756	1.000	-4.114	4.344
		Cerasmart	-.522	1.756	1.000	-4.751	3.707
	Shofu	Enamic	-.115	1.756	1.000	-4.344	4.114
		Cerasmart	-.637	1.756	1.000	-4.866	3.592
	Cerasmart	Enamic	.522	1.756	1.000	-3.707	4.751
		Shofu	.637	1.756	1.000	-3.592	4.866
Silane+ Adhesive	Enamic	Shofu	5.905*	1.756	.003	1.676	10.134
		Cerasmart	4.378*	1.756	.040	.149	8.607
	Shofu	Enamic	-5.905*	1.756	.003	-10.134	-1.676
		Cerasmart	-1.527	1.756	1.000	-5.756	2.702
	Cerasmart	Enamic	-4.378*	1.756	.040	-8.607	-.149

		Shofu	1.527	1.756	1.000	-2.702	5.756
THF1M+Silane+Adhesive	Enamic	Shofu	2.916	1.756	.294	-1.313	7.145
		Cerasmart	2.413	1.756	.511	-1.816	6.642
	Shofu	Enamic	-2.916	1.756	.294	-7.145	1.313
		Cerasmart	-.503	1.756	1.000	-4.732	3.726
	Cerasmart	Enamic	-2.413	1.756	.511	-6.642	1.816
		Shofu	.503	1.756	1.000	-3.726	4.732
THF2M+Silane+Adhesive	Enamic	Shofu	-1.231	1.756	1.000	-5.460	2.998
		Cerasmart	2.628	1.756	.407	-1.601	6.857
	Shofu	Enamic	1.231	1.756	1.000	-2.998	5.460
		Cerasmart	3.859	1.756	.086	-.370	8.088
	Cerasmart	Enamic	-2.628	1.756	.407	-6.857	1.601
		Shofu	-3.859	1.756	.086	-8.088	.370
THF3M+Silane+Adhesive	Enamic	Shofu	4.812*	1.756	.020	.583	9.041
		Cerasmart	3.430	1.756	.155	-.799	7.659
	Shofu	Enamic	-4.812*	1.756	.020	-9.041	-.583
		Cerasmart	-1.382	1.756	1.000	-5.611	2.847
	Cerasmart	Enamic	-3.430	1.756	.155	-7.659	.799
		Shofu	1.382	1.756	1.000	-2.847	5.611
THF4M+Silane+Adhesive	Enamic	Shofu	2.343	1.756	.549	-1.886	6.572
		Cerasmart	1.329	1.756	1.000	-2.900	5.558
	Shofu	Enamic	-2.343	1.756	.549	-6.572	1.886
		Cerasmart	-1.014	1.756	1.000	-5.243	3.215
	Cerasmart	Enamic	-1.329	1.756	1.000	-5.558	2.900

		Shofu	1.014	1.756	1.000	-3.215	5.243
THF5M+Silane+Adhesive	Enamic	Shofu	1.245	1.756	1.000	-2.984	5.474
		Cerasmart	.491	1.756	1.000	-3.738	4.720
	Shofu	Enamic	-1.245	1.756	1.000	-5.474	2.984
		Cerasmart	-.754	1.756	1.000	-4.983	3.475
	Cerasmart	Enamic	-.491	1.756	1.000	-4.720	3.738
		Shofu	.754	1.756	1.000	-3.475	4.983

Based on estimated marginal means

*. The mean difference is significant at the

b. Adjustment for multiple comparisons: Bonferroni.

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