

ความแข็งแรงพันธะเลื่อนและความคงทนของระบบการซ่อมพอร์ซเลนที่มีไฟร-เมต้าเป็นองค์ประกอบ
พื้นฐาน



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สาขาวิชาทันตกรรมประดิษฐ์ ภาควิชาทันตกรรมประดิษฐ์
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2558
ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

SHEAR BOND STRENGTH AND DURABILITY OF 4-META BASED
PORCELAIN REPAIR SYSTEMS

Miss Pimpisa Sringamprom



A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Prosthodontics

Department of Prosthodontics

Faculty of Dentistry

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Thesis Title	SHEAR BOND STRENGTH AND DURABILITY OF 4-META BASED PORCELAIN REPAIR SYSTEMS
By	Miss Pimpisa Sringamprom
Field of Study	Prosthodontics
Thesis Advisor	Assistant Professor Orapin Komin, D.D.S.,Ph.D.

Accepted by the Faculty of Dentistry, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree

.....Dean of the Faculty of Dentistry
(Assistant Professor Suchit Poolthong, D.D.S.,Ph.D.)

THESIS COMMITTEE

.....Chairman
(Associate Professor Mansuang Arksornnukit, D.D.S.,Ph.D.)

.....Thesis Advisor
(Assistant Professor Orapin Komin, D.D.S.,Ph.D.)

.....Examiner
(Assistant Professor Niyom Thamrongananskul, D.D.S.,Ph.D.)

.....External Examiner
(Associate Professor Pattapon Asvanund, D.D.S.,Ph.D.)

พิมพ์พิศา ศรีงามพร้อม : ความแข็งแรงพันธะเนื้อและความคงทนของระบบการซ่อมพอร์ซเลนที่มีไฟร-เมต้าเป็นองค์ประกอบพื้นฐาน (SHEAR BOND STRENGTH AND DURABILITY OF 4-META BASED PORCELAIN REPAIR SYSTEMS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ผศ. ทญ. ดร.อรพินท์ โคมิน, หน้า.

วัตถุประสงค์: เพื่อเปรียบเทียบค่าความแข็งแรงพันธะเนื้อและความคงทนของเรซินและพอร์ซเลนของระบบการซ่อมพอร์ซเลนชนิดปรับปรุงที่มี 4-เมต้าเป็นองค์ประกอบพื้นฐานกับระบบการซ่อมพอร์ซเลนในปัจจุบันชนิดอื่นๆ

วัสดุอุปกรณ์และวิธีการทดลอง: เฟลด์สปาทิกพอร์ซเลนจำนวน 100 ชิ้น ถูกยึดกับเรซินด้วยระบบการซ่อมพอร์ซเลน 5 ชนิด (กลุ่มละ 20 ชิ้น) ได้แก่ ซูเปอร์บอนด์ ซีแอนด์บี (SB) บอนด์ฟิล เอส บี (BF) ซิงเกิลบอนด์ ยูนิเวอร์เซล (SU) เฮลิโอบอนด์ (HB) และเวอร์ทีส โพล (VF) ชิ้นงาน 10 ชิ้นในแต่ละกลุ่มถูกวัดค่าความแข็งแรงพันธะเนื้อหลังจากแช่น้ำเป็นเวลา 24 ชั่วโมง และอีก 10 ชิ้นถูกวัดหลังการทำเทอมโซคลิง 5,000 รอบ นำค่าที่ได้มาวิเคราะห์ข้อมูลทางสถิติด้วยการวิเคราะห์ความแปรปรวนแบบสองทางและสถิติทดสอบที-เทส ที่ระดับนัยสำคัญ 0.05 จากนั้นตรวจดูชนิดการแตกหักของพื้นผิวทั้งหมดด้วยกล้องจุลทรรศน์ชนิดใช้แสงแบบสเตอริโอ โดยชนิดการแตกหักแบ่งออกเป็นแบบแอคซิซิฟ โคอิซิฟ และชนิดมิกซ์

ผลการทดลอง: ค่าความแข็งแรงพันธะเนื้อเฉลี่ยหลังแช่น้ำเป็นเวลา 24 ชั่วโมงของทั้ง 5 กลุ่มมีค่าระหว่าง 12.3 ถึง 28.8 เมกะปาสคาล และหลังการทำเทอมโซคลิงค่าความแข็งแรงพันธะเนื้อเฉลี่ยมีค่าระหว่าง 4.14 ถึง 25.1 เมกะปาสคาล โดยไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติของค่าความแข็งแรงพันธะเนื้อก่อนและหลังการทำเทอมโซคลิงในกลุ่ม HB, SB และ BF ค่าความแข็งแรงพันธะเนื้อลดลงอย่างมีนัยสำคัญทางสถิติหลังการทำเทอมโซคลิงในกลุ่ม SU และ VF ค่าความแข็งแรงพันธะเนื้อเฉลี่ยได้รับอิทธิพลทั้งจากระบบการซ่อมพอร์ซเลนและการทำเทอมโซคลิง ในทุกกลุ่มพบการแตกของชิ้นงานแบบมิกซ์และแอคซิซิฟเพิ่มขึ้นหลังการทำเทอมโซคลิงยกเว้นกลุ่ม HB

สรุปผลการทดลอง: ระบบการซ่อมพอร์ซเลนชนิดปรับปรุงที่มีไฟร-เมต้าเป็นองค์ประกอบพื้นฐานเป็นอีกทางเลือกในการซ่อมพอร์ซเลนได้

ภาควิชา ทันตกรรมประดิษฐ์

ลายมือชื่อนิสิต

สาขาวิชา ทันตกรรมประดิษฐ์

ลายมือชื่อ อ.ที่ปรึกษาหลัก

ปีการศึกษา 2558

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PIMPISA SRINGAMPROM: SHEAR BOND STRENGTH AND DURABILITY OF 4-META BASED PORCELAIN REPAIR SYSTEMS. ADVISOR: ASST. PROF. ORAPIN KOMIN, D.D.S.,Ph.D., pp.

Objective: To compare the resin-porcelain shear bond strengths and their durability using a modified 4-META based porcelain repair system and other current systems.

Material and method: One hundred specimens were prepared from feldspathic porcelain. The specimens were bonded to resin with five porcelain repair systems (20 pieces each): Superbond C&B (SB), Bondfill SB (BF), Single bond universal (SU), Heliobonds (HB) and Vertise flow (VF). In each group, shear bond strength of 10 specimens were tested after 24-h water storage, and the other 10 specimens were tested after 5,000 thermocycles. All data were statistically analyzed by two-way ANOVA and T-test ($p < 0.05$). All substrate surfaces were investigated their fracture modes by a stereomicroscope. Fracture surfaces were categorized as adhesive, cohesive or mixed failure.

Results: The mean shear bond strength among 5 groups at 24-h water storage varied from 12.3 MPa to 28.8 MPa and they varied from 4.14 MPa to 25.1 MPa after thermocycling. There was no statistically significant different in bond strength before and after thermocycling in HB, SB and BF. The significant lower bond strength after thermocycling was statistically significant in SU and VF. The mean shear bond strength was influenced by the type of porcelain repair system and thermocycling. All group exhibited more mixed and adhesive failure, except HB group.

Conclusions: A modified 4-META based porcelain repair system is an alternative choice for repairing porcelain.

Department: Prosthodontics Student's Signature

Field of Study: Prosthodontics Advisor's Signature

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CHAPTER I

INTRODUCTION

Background and Rationale

Porcelain is widely used in crowns and fixed partial dentures because of its esthetics, long-term color stability, translucency, biocompatibility, and wear-resistance. Over time, the fatigue and trauma can create stresses that lead to esthetic veneering porcelain fracture. Fracture can occur in every dental porcelain system, including all-ceramic and porcelain fused to metal (PFM) restorations. Fracture may result from multifactorial problems such as frequently repeated stresses and strains during chewing function or trauma, parafunctional habit, excessive porcelain thickness with inadequate core support, technical mistake during the preparation, crack propagation in the porcelain, improper design of restoration and the chemistry of the oral environment.^{1,2)}

These failure may be classified as simple (fracture in porcelain only), mixed (fracture with both porcelain and core material exposed), and complex (fracture with substantial core material exposure).^{3,4)} Failures occur most frequently in regions that are quite visible, compromising esthetics.²⁾ In one study, the frequencies of veneering porcelain fractures that could be treated by polishing or repairing were considerably higher than those which required replacement of the restoration.⁵⁾ The best method to solve this problem is to identify and correct the reason for the fracture. Many patients do not need to remove the fracture crown especially in long spans fixed partial prostheses. They cannot afford replacement of the restoration, and then seek maintenance and repair. Repairing the fracture areas at the chairside is usually the choice of the patients to correct the esthetics and function immediately. A repair can minimize trauma and preserves both tooth structure and restoration. It may repair by re-cementation with the fracture porcelain, make a new porcelain veneer to cementation and repair with the resin composite.⁶⁻⁸⁾

The requirements of the intraoral repair are to achieve a permanent bonding despite the bite forces and the humidity in the mouth, and to protect the soft

tissues as well as to avoid any health risks for the patient due to the ingredients of the repair system.⁹⁾ The most popular chairside method is repairing with a resin composite, because it can save time, cost and is easy to use. Currently, there are several products available for the repair of chipped or fractured porcelain. The success of the repair depends on the method of surface pretreatment and the adhesive resin systems. Various surface pretreatments have been suggested to increase bond strength and durability, such as abrasion with diamond bur, sandblasting, chemical etching and chemical retention using silane coupling agent.^{5,10)} Recently, material with the capability to repair fractured porcelain without resin composite and resin cement was introduced to the dental market.¹¹⁾ It is a modified 4- Methacryloxyethyl trimellitate anhydride (4-META)-based self-cured, self-adhesive resin with pre-polymerized filler (Bondfill SB; Sun Medical Co, Moriyama, Shiga, Japan). It was claimed to be the restoration of attrition, cervical caries, pit and fissure sealing and ceramic repair, and studies have evaluate the efficiency and characteristics of Bondfill SB as restorative material.¹¹⁻¹³⁾ However, no studies have been found on the use of this material for porcelain repair.

Objectives

1. To compare the resin-porcelain shear bond strength of a modified 4-META based porcelain repair system and other current systems.
2. To investigate the effect of thermocycling on the shear bond strength of porcelain repair systems.

Hypothesis

1. H₀: The resin-porcelain shear bond strength of a modified 4-META based porcelain repair system *does not significantly* differ from other current systems. ($p < 0.05$)
H₁: The resin-porcelain shear bond strength of a modified 4-META based porcelain repair system *significantly differs* from other current systems. ($p < 0.05$)
2. H₀: There is no *significantly difference* in the effect of thermocycling on the shear bond strength of porcelain repair systems. ($p < 0.05$)

H_1 : There is *significantly difference* in the effect of thermocycling on the shear bond strength of porcelain repair systems. ($p < 0.05$)

Research frame

1. This research is a laboratory experimental research.
2. The porcelain is made in the same manner as conventional porcelain following the manufacturer's instructions.
3. All procedures are proceeding by one person under the same instrument.

Limitation

This thesis focuses on porcelain chipping only (simple failure), so the result cannot represent all type of fixed partial dentures fractured.

Expected benefit

1. The results of this study can be the information and suggestion to the dentist for choosing the material for the repair of chipped porcelain.
2. The results of this study will be useful for further studies.

Keywords

4-META	โฟร-เมต้า
Durability	ความคงทน
Porcelain repair	การซ่อมพอร์ซเลน
Shear bond strength	ความแข็งแรงพันธะเฉือน
TBB resin	ทีบีบี เรซิน

CHAPTER II

LITERATURE REVIEW

Porcelain is one of the most popular materials used in fixed prostheses. It is used because of their biocompatibility, aesthetics, color stability and wear resistance. Porcelain contains a crystal phase and a glass phase based on the quartz silica structure. The other consistency is kaolin and feldspar. Porcelain can be classified in many classifications in order to composition: by their microstructure (i.e., amount and type of crystalline phase and glass composition), by the processing technique (powder-liquid, pressed or machined), and by type (feldspathic porcelain, leucite-reinforced porcelain, aluminous porcelain), etc. Porcelains are brittle, which means that they display a high compressive strength but low tensile strength and may be fractured under very low strain. Feldspathic porcelain presents fracture toughness of approximately $0.9-1.5 \text{ MPa/m}^2$, flexural strength 55-87 MPa whereas alumina and zirconia reinforced high strength ceramic exhibit fracture toughness of $3-9 \text{ MPa/m}^2$ and flexural strength about 300-700 MPa. Therefore, Feldspathic porcelain are known to be susceptible to fracture.^{14,15)}

There are a lot of reports in chipping of the veneering porcelain that made from feldspathic porcelain. A long-term clinical study on porcelain fused to metal crown found that there was 6.25% of porcelain fracture.¹⁶⁾ According to Pjurtursson et al¹⁷⁾ reported a 5-year survival rate of porcelain chipping was 3.7% for all-ceramic single crown and 5.7% for metal-ceramic crown. While, Sailer et al¹⁸⁾ performed a 5-year survival rate and incidence of complications for all ceramic fixed dental prostheses (FDPs) and metal-ceramic FDPs. The frequencies of veneering porcelain fracture in fixed partial prostheses were higher for all ceramic FDPs (13.6%) compared with those of metal-ceramic FDPs (2.9%).

The fracture of veneering porcelain was the most frequent reason for failure of fixed dental prostheses.¹⁸⁾ Failures occur most frequently in regions that are quite visible, compromising esthetics.^{2,19)} The majority (65%) of failures had been observed in the anterior region, whereas 35% were in the posterior region. 60% of the failure

occurred at the labial surfaces, 27% at the buccal, 5% at the incisal, and 8% at the occlusal of the restorations.²⁾ These fractures were mainly in the maxilla. Porcelain is inherently brittle material. Once crack initiates in the surface, it will propagate until the restoration material breaks away from understructure. Fracture may result from multifactorial problem such as trauma, hard substrates with in food bitten on by the patient, parafunctional habits, and mechanical fatigue. Sometimes, the reason may be due to poor and inadequate laboratory techniques. There may be poor substructure design leaving unsupported porcelain over the substructure. In some case, it may due to poor abutment preparation, leading to insufficient porcelain thickness that will be subject to fracture in normal function.^{1,2,15)}

When the fracture happens, it is necessary to access the possible causes of fracture. Since crown and bridge are cemented adhesively, their removal for indirect repair without creating any damage to the tooth structure or the restoration itself is difficult.¹⁴⁾ Many patients cannot afford replacement of the restoration and seek maintenance and repair, especially in long span bridge. Heintze SD²⁰⁾ found that the frequency of veneering porcelain fractures that could be treated by polishing or repairing was considerably higher than those which required replacement of the restoration.

Repair of fractured porcelain aims to reestablish the function and esthetics of restorations and preventing the accumulation of microorganisms on the fractured surface. The easiest way to repair fractured veneering porcelain is to polish the fractured surface thoroughly to minimize surface flaws. However, there are several methods to repair porcelain such as overcasting crown, recementation with the broken piece of porcelain with resin cement, prepare the restoration for new veneer and adhesively to existing restoration and repair with the resin composite.⁵⁻⁷⁾ The most popular chairside method is repairing with resin composite. In 1977 Eames et al^{21,22)} reported the method to repair the porcelain with resin composite and follow up after one year. They found that the bond strength was still good. Many studies^{14,23-28)} have evaluated the appropriate method for repairing the porcelain

surface. The success of repairing depends on a method of pretreatment surface and adhesive resin systems.

A surface pretreatment depends on micromechanical retention and chemical retention to the porcelain surface. For micromechanical retention, the main objective is to make rough surface of fractured porcelain. Common treatment options are grinding with diamond bur, sandblasting, acid etching and combination methods.^{5,10)} Grinding with diamond bur is the simple method to rough the surface. It increases surface area to bond with resin cement but we cannot predict the bond strength in long term.^{6,9)} Sandblasting with aluminum oxide particle will hit the porcelain surface with high energy, they will generate small surface flaw. Cracks usually originate from this flaw. Excessive airborne particle abrasion induces chipping of ceramic material. Kato et al²⁴⁾ found that air abrasion could rough the porcelain surface but post-thermocycling bond strength indicated that air abrasion was not retentive.

Another treatment is acid etching with hydrofluoric acid (HF) that can dissolve bonds in silicate substances such as feldspathic porcelain. The application of 2-10% hydrofluoric acid, it will react silicon dioxide (SiO₂) on porcelain surface to Hexafluorosilicate (H₂SiF₆) that make the surface like a beehive according to the formulation in Figure 1.^{24,29)} Canay S²⁵⁾ found that the application of 9.5% HF etching for 1 minute resulted in the production of small channels, which were approximately 3-4 μm wide. This will allow the formation of a mechanical interlock with the resin.

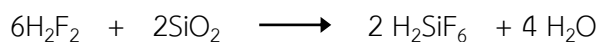


Figure 1 Formulation of hydrofluoric acid on the porcelain surface.

However, intraoral use of hydrofluoric acid is controversial because of its hazardous properties.³⁰⁾ Clinicians should use it with a rubber dam. Acid etchant that are less hazardous than hydrofluoric acid are preferred intraorally such as phosphoric acid (H₃PO₄), acidulated phosphate fluoride (APF gel).¹⁹⁾ Kato et al²⁴⁾ found insufficient retention produced with 60 s conditioning using phosphoric acid and APF gel. Ten

minutes of etching with the APF gel increased roughness. According to many studies^{9,28,31)} reported that SEM finding of etched porcelain surfaced with HF and APF gel showed that etching with APF gel might be adequate and produced morphological changes compared with HF when there was appropriate timing (10 mins). It might be used as an alternative etchant to hydrofluoric acid in a specific clinical situation or used with silane coupling agent. However, it should be noted that etching effect of the APF gel is inferior to the hydrofluoric acid.^{24,25)}

Silane coupling agent creates chemical bond between the porcelain and the hydrophobic resin (such as composite resin). In the silanization process, silanes are bifunctional molecules that bond silicon dioxide with the OH groups on the ceramic surface (Figure 2). They usually contain a silane coupler and a weak acid, which enhances the formation of siloxane bonds. Silanization also increases wettability of the ceramic surface.¹⁰⁾

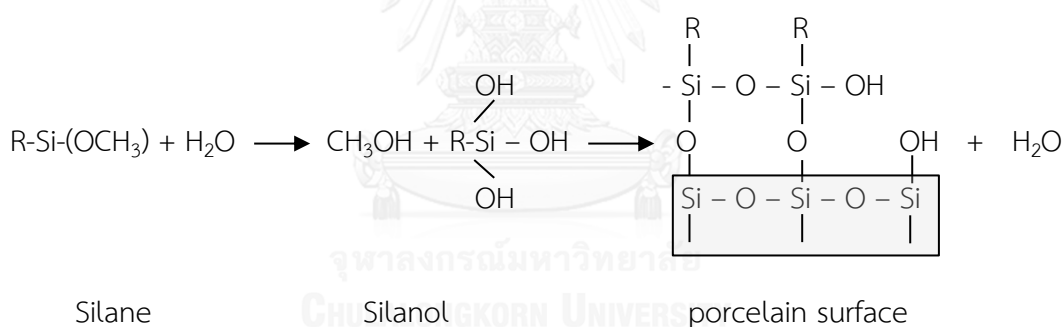


Figure 2 Silanization process

Some silane agents that contained carboxylic acid provided sufficient bond strength even without hydrofluoric acid etching, and other was successful after acid etching with phosphoric acid.³²⁾ Previous study⁴⁾ reported that air-abraded porcelain was not retentive unless a silane agent was used in combination. According to Kussano M³³⁾ reported that acid etching is not necessary for adhesion between composite resin and porcelain if the silane is used. Silane coupling agent can be categorized into 3 main groups: unhydrolyzed single liquid silane primer, prehydrolyzed single liquid silane primer, and 2 or 3 liquid silane primer. Single

bottle has a limited shelf life and are susceptible to rapid solvent evaporation and hydrolyzation. Some manufacturers add a silane coupler to their bonding system.¹⁰⁾

Current porcelain repair system has various surface pretreatment methods. There is no specific protocol for repairing. It is common to use different surface pretreatment in conjunction with an available adhesive systems and resin composite. Previous studies have compared the bond strength of several porcelain repair systems. They have reported bond strength values vary widely from 3 to 60 MPa. For example, Appeldoorn et al³⁴⁾ found that the mean shear bond strength of composite resin bonded to porcelain by the use of eight commercially available porcelain repair systems after 24 hours water storage was 12.0-23.5 MPa and after 3 months of water storage and thermocycling was 4.2-20.7 MPa. While Kato et al³⁵⁾ examined the shear bond strength and durability of porcelain bonding systems joined to feldspathic porcelain. The mean bond strength before and after thermocycling varied from 30.3-65.3 MPa and 5.3-45.2 MPa, respectively. Another study³⁶⁾ evaluate the shear bond strength of currently used ceramic repair systems and the effect of 2 years simulation of thermo-mechanical loading. They found that some ceramic repair systems tested (Z 100 kit®, Monobond S/Tetric® and Porcelain Etch®) exhibited a shear bond strength greater than the control group Rocatec®. Juliana et al¹⁹⁾ evaluated the shear bond strength of 5 metal-ceramic repair systems. The mean bond strength varied from 16.18-21.05 MPa.

Bonding between the composite resin and porcelain was frequently investigated by shear bond strength methodologies.^{3,19,27,34-40)} It has been reported that shear stresses are major stresses that generated in vivo bonding failure of restorations. Shear bond strength test is a test to qualify adhesion of a cement system, although some criticism has been expressed to the shear bond strength measurement mode.¹⁹⁾ Some studies^{14,41)} investigated by tensile bond strength or microshear bond strength or microtensile bond strength. Each method has its own merits. However, there is no studies indicated which method is the better over the counter.

The resin-porcelain bond is susceptible to chemical, thermal and mechanical influences under intraoral conditions.⁹⁾ The simulation of such influences in the

laboratory is to identify the long term durability of a bonding procedure and superior materials and techniques because clinical trials are costly and time consuming.⁴²⁾ Long term water storage and thermocycling of the bonded specimens are accepted methods to stimulate aging process. The evaluation of initial bond strength cannot answer the question of whether a bonding system will show successful long term stability. Most studies reveal significant difference between before and after mean bond strength value.^{34-37,43)} Exposing the specimens to thermocycling speed up the diffusion of water in between the resin and the porcelain. Changing the temperature creates stress at the interface of the two materials because of different coefficients of thermal expansion.⁹⁾ Five thousand thermocycles have been proposed to represented 6 months clinical service.⁴²⁾

According to the previous studies, considering to the many variables and mixed results from different size of specimens, storage condition and test methodologies have been reported in laboratory studies. The comparison of bond strength of currently porcelain repair system with the recently systems is difficult. A modified 4- methacryloxyethyl trimellitate anhydride (4-META) / Methyl methacrylate-Tri-n-butylborane (MMA-TBB)-based resin, Bondfill SB (Sun Medical Co, Ltd., Moriyama, Kyoto, Japan)¹¹⁾ was introduced to the dental market. It was claimed to be for the restoration of attrition, cervical caries, pit and fissure sealing and ceramic repair. It is an acrylic-based adhesive resin containing trimethylolpropane trimethacrylate (TMPT) reactive organic filler which creates a tough restoration. It presents greatly wear resistance compared to the conventional acrylic resins.

The material polymerization is initiated by Tri-n-butylborane (TBB) which has been used in 4-META-based resins (Superbond C&B, Sun Medical, Moriyama, Shiga, Japan) for a long time. This material forms strong bonds to enamel, dentin, and restorative materials without gapping and discoloration when appropriate primers are used.^{12,13)} There are literatures to evaluate efficiency and characteristic of Bondfill SB as a restoration material.^{11-13,44)} However, no studies have been found on the use of this material for porcelain repair. Therefore, the aim of this study is to compare this repair system to other current systems that has several surface pretreatment. Superbond C&B is resin cement and use with Porcelain Liner M for repairing

porcelain. Single Bond Universal (3M ESPE, St. Paul, MN, USA) is a universal adhesive that contain silane coupling agent. Another system (Ivoclar Vivadent AG, Schaan, Liechtenstein) has bonding agent (Heliobond) and separate silane coupling agent (Monobond N) for surface pretreatment. The last system (Vertise Flow, Kerr Dental Product, Orange, CA, USA), there is no conditioning agent.



CHAPTER III

MATERIAL AND METHODS

One hundred specimens, 5-mm diameter and 3-mm thick, were prepared from feldspathic porcelain (Vintage MP, Shofu, Kyoto, Japan) (Figure 3) according to manufacturer's instructions. All specimens were embedded in polyvinyl chloride (PVC) tubing, 22-mm diameter, with clear acrylic resin. (Figure 4)



Figure 3 Feldspathic porcelain

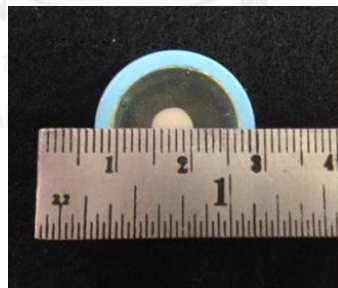


Figure 4 Specimens in PVC tube with clear acrylic resin

The specimens were wet-polished with 220- and 320- grit silicon carbide papers with polishing machine (Nano2000, PACE technologies, St. Tucson, AZ). The surface roughness of all specimens was calculated with profilometer (Talyscan 150, Taylor Hobson LTD, Leicester, UK) (Figure 5). The specimens were cleaned in an ultrasonic cleaner (Branson 5210, Branson, Dietzenbach-Steinberg, Germany)

(Figure 6) with distilled water for 5 min, air dried and stored in closed contamination until used.



a.



b.

Figure 5 The machine used in this study: (a) Polishing machine, (b) Profilometer.



Figure 6 Ultrasonic cleaner.

All specimens were randomly assigned into five groups for the following intra-oral repair systems (n = 20): Superbond C&B (SB, Sun Medical, Moriyama, Shiga, Japan), Bondfill SB (BF, Sun Medical, Moriyama, Shiga, Japan), Single Bond Universal (SU, 3M ESPE, St. Paul, MN, USA), Heliobond (HB, Ivoclar Vivadent AG, Schaan, Liechtenstein) and Vertise Flow (VF, Kerr Dental Product, Orange, CA, USA). The materials used are lists in Table 1.

A polyethylene sheet with a 2.5-mm diameter hold is fixed with adhesive tape on the specimens to define the bond area. The silicone mold was fixed above the sheet to hold a 3-mm diameter and 3-mm high aluminum ring (Figure 7). The repair material was bonded to the porcelain in accordance with the manufacturer's instructions (Figure 8) and constrained by aluminum ring. The resin composite was applied and polymerized in layers of maximum 2-mm thick, using a curing light (Elipar S10; 3M ESPE, St. Paul, MN, USA) for 40 s.

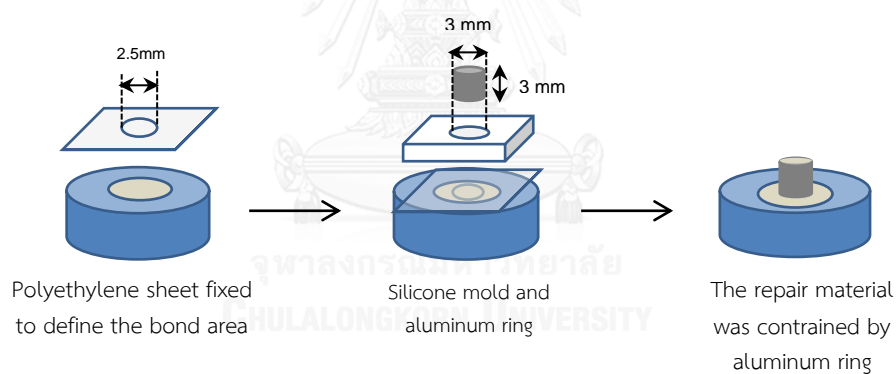


Figure 7 Diagram for specimen's preparation

Table 1 Material used in this study

Type	Material (Lot no.)	Code	Composition*(Lot. no)	Manufacture
Luting agent	Superbond C&B	SB	Base (GM1): 4-META, Methyl methacrylate, Catalyst (GR22): TBB, TBB-O, Hydrocarbon, Powder (GM1): PMMA, metal oxide Red activator (GL1): 65% phosphoric acid	Sun Medical, Moriyama, Shiga, Japan
	Bondfill SB	BF	Base (GR1): 4-META, Methyl methacrylate, Catalyst (EV2 IF): TBB, TBB-O, Hydrocarbon, Powder (GR1): PMMA, TMPT pre-polymerized filler, pigment	Sun Medical, Moriyama, Shiga, Japan
	Single Bond Universal (548708)	SU	MDP, Bis-GMA, HEMA, decamethylene DMA, ethanol, water, silane treated silica, 2-propenoic acid, -methyl-, reaction products with 1,10-decanediol and phosphorous oxide, copolymer of acrylic and itaconic acid, dimethylaminobenzoate, camphorquinone, (dimethylamino)ethyl methacrylate, methyl ethyl ketone	3M EPSE, St. Paul, MN, USA
	Heliobond (S46831)	HB	Bis-GMA, TEGDMA	Ivoclar Vivadent, Schaan, Liechtenstein
	Vertise Flow (5326583)	VF	Uncured methacrylate ester monomers Inert mineral fillers, Ytterbium Fluoride, activators, stabilizers and colorants	Kerr Corporation, Orange, CA, USA
Etchant	Porcelain etchant (140818)	-	9.6% hydrofluoric Acid	Pulpdent Corporation, Watertown, MA, USA
Conditioner	Porcelain Liner M (GK3)	-	Liquid A(FR1): 4-META, Methyl methacrylate Liquid B(FL1S): 3-(trimethoxysilyl) propyl methacrylate	Sun Medical, Moriyama, Shiga, Japan
	Monobond N (S55074)	-	Ethanol, Methacrylated phosphoric acid ester	Ivoclar Vivadent, Schaan, Liechtenstein
Resin composite	Metalfil CX (GS21)	-	UDMA, TEGDMA, PolyTMPT, Silica, amorphous	Sun Medical, Moriyama, Shiga, Japan
	Tetric N-Ceram (T08035)	-	UDMA, Ytterbium Trifluoride, ethoxyylated bisphenol A dimethacrylate, Bis-GMA	Ivoclar Vivadent, Schaan, Liechtenstein
	Filltek Z350 XT (N576840)	-	silane treated ceramic, silane treated silica, UDMA, Bis-GMA, Bis-EMA, silane treated zirconia, PEGDMA, TEGDMA, 2,6-di-tert-butyl-p-cresol	3M EPSE, St. Paul, MN, USA

*According to Material Safety Data Sheet

Abbreviation: 4-META: 4-methacryloxyethyltrimellitic acid anhydride, TBB-O: partially oxidized tri-n-butylborane, PMMA: Poly(methyl methacrylate), TMPT: trimethylolpropane trimethacrylate, MDP: methacryloxydecyl dihydrogen phosphate, Bis-GMA: Bisphenol A-glycidyl methacrylate, HEMA: hydroxyethyl methacrylate, DMA: Dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate, PEGDMA: polyethylene glycol dimethacrylate

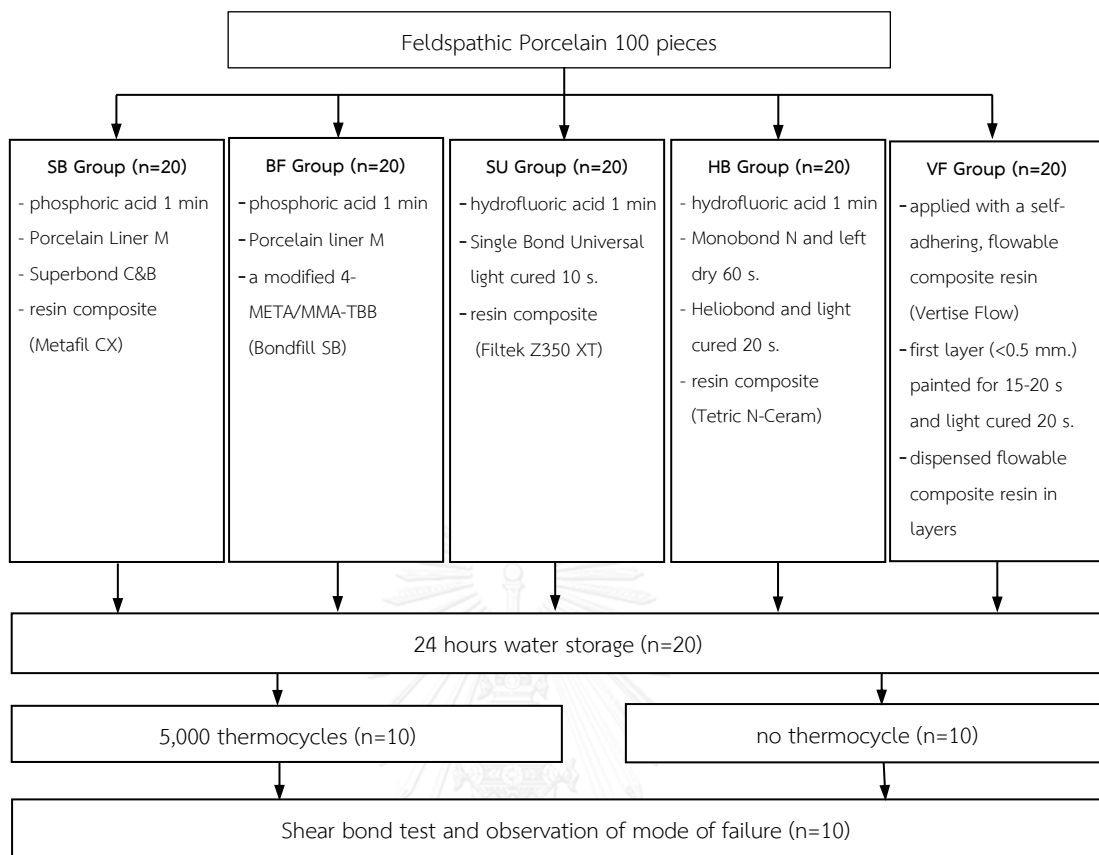


Figure 8 Study design

Thirty minutes after preparation, the specimens were stored in 37°C distilled water for 24 hours. Half of the specimens in each group were tested for 24-h shear bond strength using universal testing machine (EZ-S, Shimadzu, Kyoto, Japan) with a cross head speed of 0.5 mm. per min until fracture. (Figure 9, 10) The remaining specimens were thermocycled for 5,000 cycles in a thermocycling unit (King Mongkut's University of Technology Thonburi, Thailand) between 5°C and 55°C water with a dwell time of 30 s and resting interval of 10 S between baths followed by shear bond strength testing according to ISO 10477⁴⁵⁾.

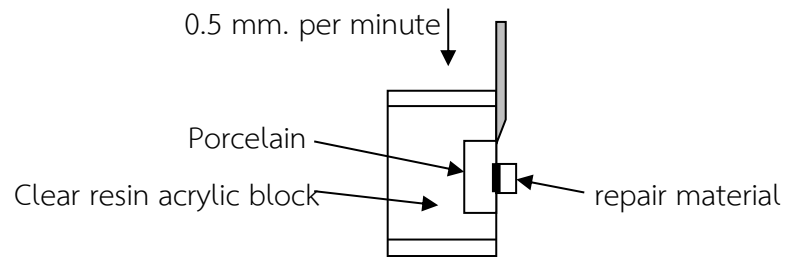


Figure 9 Model for shear bond strength testing



Figure 10 Universal testing machine (EZ-S, Shimadzu, Kyoto, Japan)

After shear bond strength testing, all fractured surfaces were examined by a stereomicroscope (SZ 61, Olympus, Tokyo, Japan) at 30X to determine the mode of failure, and recorded by a single calibrated observer as

Type I: Cohesive failure inside the porcelain (Cohesive)

Type II: Mixed failure of cohesive in the porcelain and the luting agent and adhesive failures at the luting agent-porcelain interfaces (Mixed)

Type III: Adhesive failure at the luting agent-porcelain interfaces (Adhesive)

The statistical analysis

- The data of each group (24-h water storage and thermocycles) were analyzed with One-way ANOVA and Tukey HSD test (equal variance) or Dunnett's T3 test (unequal variance) at significant level of 0.05. Then T-test was analyzed to compare the same material before and after thermocycling.
- Two-way ANOVA were analyzed to examine the influence of two factors using SPSS (SPSS version 22.0; IBM, Chicago, IL, USA).



CHAPTER IV

RESULTS

The mean shear bond strength

The descriptive analysis of mean shear bond strengths and standard deviation of five porcelain repair systems after 24-h water storage and thermocycles are shown in Table 2 and 3. The same letters denote groups that are not significantly different ($p < 0.05$).

Table 2 Mean and standard deviation (SD) of shear bond strength (MPa) of 24-h water storage group.

Group	Shear bond strength
SB	21.8 (4.49) ^{b,c}
BF	26.7 (2.34) ^{a,b}
SU	20.9 (5.12) ^c
HB	28.8 (4.66) ^a
VF	12.3 (3.95) ^d

Values with different lower case superscripts are statistically significantly different ($p < 0.05$).

Table 3 Mean and standard deviation (SD) of shear bond strength (MPa) of thermocycles group.

Group	Shear bond strength
SB	21.4 (4.91) ^e
BF	25.1 (4.52) ^e
SU	13.9 (5.01) ^f
HB	25.1 (5.24) ^e
VF	4.14 (1.04) ^g

Values with different lower case superscripts are statistically significantly different ($p < 0.05$).

When tested after 24-h water storage (Table 2), the data were normal distribution and equal variance, therefore they were analyzed by One-way ANOVA and Tukey HSD test. There was statistically significant difference between mean shear bond strength at least two groups ($p=0.000$). There was no statistically significant difference between SB and BF, SB and SU, BF and HB. HB showed the highest mean bond strength while VF showed the lowest mean bond strength.

When tested after thermocycles (Table 3), the data were normal distribution and unequal variance, therefore they were analyzed by One-way ANOVA and Dunnett's T3 test. There was statistically significant difference between mean shear bond strength at least two groups ($p=0.000$). There was no statistically significant difference between three systems SB, BF and HB. These three systems statistically significantly differ from SU and VF. The bond strength of HB was similar to that of BF (25.1 MPa) although HB (28.8 MPa) had the higher bond strength than BF (26.7 MPa) after 24-h water storage. VF still showed the lowest mean bond strength.

Figure 11 showed the data of 24-h water storage was compared with the data of 5,000 thermocycles, the data were analyzed by independent sample t-test. The lower bond strength after thermocycling was statistically significant only for SU ($p=0.006$) and VF ($p=0.000$). There was no statistically significant before and after thermocycling for HB ($p=0.113$), SB ($p=0.864$) and BF ($p=0.344$). The bond strength of SB was minimal changes while VF was maximum changes.

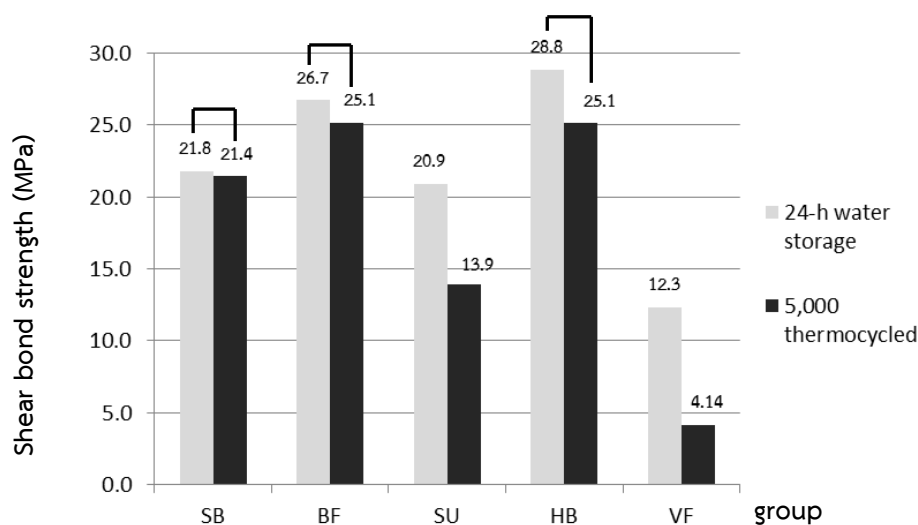


Figure 11 Mean shear bond strength after 24-h water storage and thermocycles

The Two-way ANOVA results are presented in table 4. The analysis showed that the bond strength was effected by the type of porcelain repair system ($p=0.000$), thermocycling ($p=0.000$) and their interaction ($p=0.021$) that means different repair system and number of thermocycling effect on the shear bond strength.

Table 4 Analysis of variance for shear bond strength

Source	Sum of squares	df	Mean Square	F	sig.
Group	4618.645	4	1154.662	61.683	0.000
Storage condition	433.333	1	433.333	23.149	0.000
Group * Storage condition	228.575	4	57.144	3.053	0.021

The failure mode analysis

The failure mode analysis is shown in figure 12. Microscope representing each failure type are presented in figure 13-15. After 24-h water storage, SB and HB showed 50% cohesive failure in the porcelain. HB did not show adhesive failure. SU showed 20% cohesive failure. BF showed only 10% cohesive failure and VF did not show any cohesive failure in the porcelain. After thermocycling, SB exhibited more mixed failure and showed less cohesive failure (30%). BF also showed more mixed failure and less adhesive failure. SU exhibited more adhesive failure but did not show any cohesive failure. HB had the same failure pattern as 24-h water storage (50% cohesive failure). VF showed only adhesive failure in every specimen.

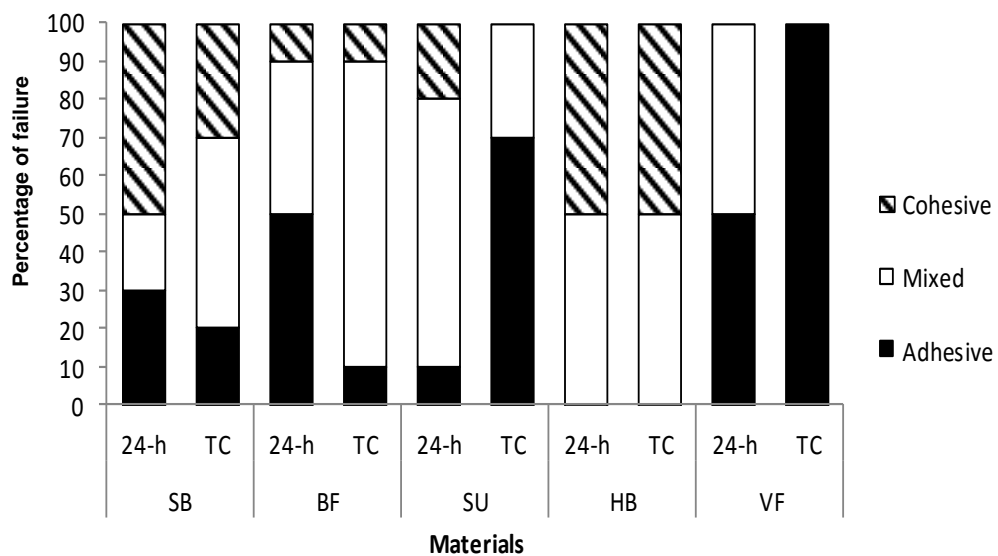


Figure 12 Failure mode analysis after 24-hour water storage (24-h) and thermocycles (TC).

Cohesive: Cohesive failure inside the porcelain;

Mixed: Mixed failure of cohesive in the porcelain and the luting agent and adhesive failures at the luting agent-porcelain interfaces;

Adhesive: Adhesive failure at the luting agent-porcelain interfaces

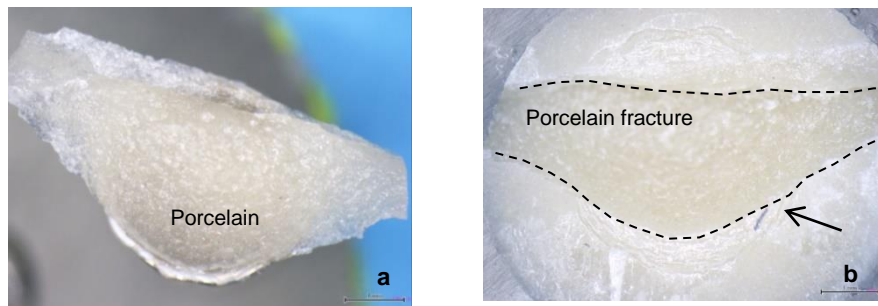


Figure 13 Cohesive failure in porcelain: (a) restorative site, (b) porcelain site

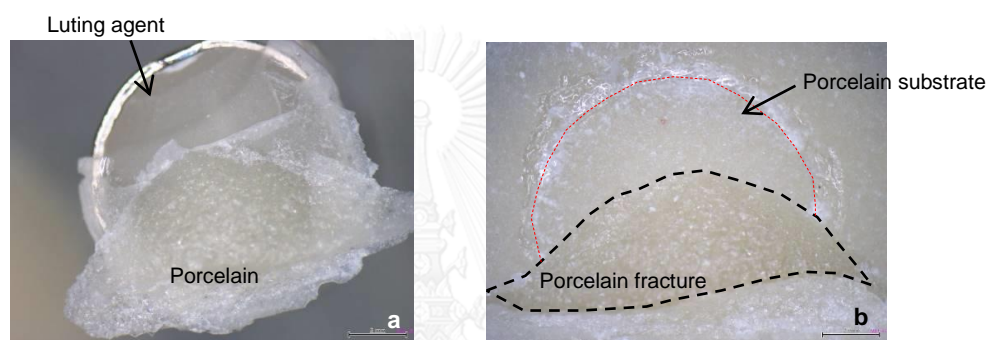


Figure 14 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site

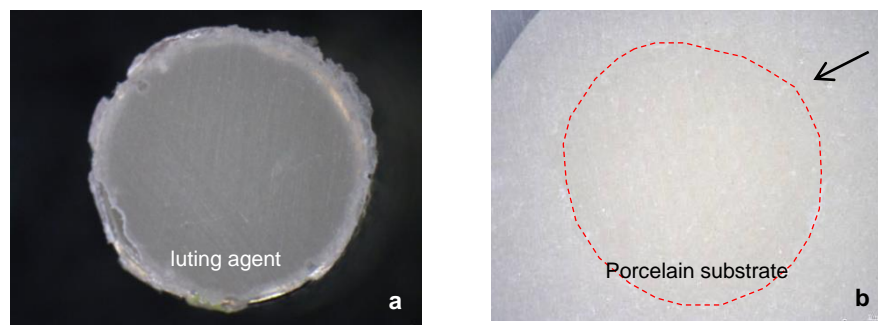


Figure 15 Adhesive failure between luting agent and porcelain: (a) restorative site, (b) porcelain site

CHAPTER V

DISCUSSION

In this study, the result showed that the bond strength was influenced by the type of porcelain repair system and thermocycling. Therefore, the null hypothesis that there is no difference in shear bond strength of Bondfill SB and currently used systems was rejected. The second null hypothesis that there is no difference in the effect of thermocycling on shear bond strength was also rejected.

Since there are several porcelain repair systems available on the dental market that are based on the different surface pretreatment, it is difficult for clinicians to choose the best system. The results of previous studies were not comparable because of different specimen preparation and testing methodologies. In this study, four porcelain repair systems were selected for comparison with Bondfill SB. Adhesion principles usually rely on a combination of mechanical and chemical bonding. For Superbond C&B and Bondfill SB are based on phosphoric acid (H_3PO_4) treatment followed by a silane coupling agent. The other two systems (Heliobond and Single Bond Universal) used hydrofluoric acid (HF) to dissolve the porcelain glass matrix and create more micromechanical retention. Heliobond also uses separate silane and bonding agent, while Single Bond Universal is a universal adhesive bonding that contains the silane. The last system (Vertise Flow), there is no conditioning step. The differences in bond strengths could be related to micromechanical retention and the efficiency of the chemical reactions of silanes in forming bonds between substrate and resin system. There is some evidence that silane with different chemical compositions and concentrations in the solvent result in different adhesion.¹⁰⁾

From table 2, when the results are evaluated at 24-h water storage, Heliobond had the highest mean bond strength followed by Bondfill SB, Superbond C&B, Single Bond Universal and Vertise Flow. HF etching followed by the separate silane (Heliobond) resulted in mean bond strength superior to H_3PO_4 etching

followed by separate silane (Bondfill SB, Superbond C&B) and HF etching only (Single Bond Universal). However, there was no significant difference in mean bond strength between Superbond C&B and Bondfill SB, because of their similar composition of the adhesive components (4-META and TBB). One difference in the composition is related to the trimethylolpropanetriacrylate (TMPT) filler, which is only present in Bondfill SB. This filler is in a flexible acrylic matrix that creates a tough restoration and lead to lower brittleness.^{11,46)} Therefore, Bondfill SB showed higher bond strength than Superbond C&B. Vertise Flow showed the lowest bond strength. This repair system does not require any surface pretreatment according to the manufacturer instruction, leading to poor bond strength, possibly because the viscous characteristics of the flowable composite⁴⁷⁾ preventing flow into the surface of the porcelain to create a strong bond.³⁹⁾ In addition, the acidic functional component of Vertise Flow may not provide enhanced siloxane bonds, resulting in decreased bond strength values.³²⁾ According to a recent study³⁹⁾ also reported the low bond strength of Vertise Flow using leucite-reinforced ceramic for repairing substrate in their study.

From the result of this study (Figure 11), every system showed lower bond strength after 5,000 thermocycles. This reduction in bond strength may be due to the uptake of water and thermal challenge causing changes of the mechanical properties within the silane and resin layers.³⁶⁾ Superbond C&B showed the lowest decrease in bond strength value, while Vertise Flow showed the highest decrease. The lower decrease in bond strength shows more durability in resin system. The bond strength after thermocycling of Bondfill SB and Heliobond was similar. Their bond strength was superior to the other systems, although the initial bond strengths were different. Three of the five systems in this study (Superbond C&B, Bondfill SB and Heliobond) did not show a significant decrease in bond strength when 24-h water storage was compared with 5,000 thermocycles. According to Kato H³⁵⁾ found that the bond strength of Superbond C&B and Porcelain Liner M did not decrease significant after thermocycling. Another two systems (Single Bond Universal and Vertise Flow) that did not use a separate silane application showed a significant decrease in bond strength after thermocycling.

According to a previous study³²⁾, silane can improve bond strength and durability with or without using HF etching. Although silane is one of the components in Single Bond Universal, it still showed a low bond strength after thermocycling. This implies that silane in Single Bond Universal failed to produce any significant chemical bonds with the porcelain and that the use of separate silanes may be more favourable.⁴⁸⁾ This can be explained by the mixture of various components within the same bottle, as it has been reported that bis-GMA may inhibit the action of silane by disrupting the condensation reaction with the hydroxyl group of a silica-based ceramic.⁴⁹⁾ Furthermore, the acidic functional monomer 10-MDP (10-methacryloxydecyl dihydrogen phosphate) may impede the ideal chemical interaction between silane and ceramics owing to the tendency for premature hydrolysis in an acidic environment.⁵⁰⁾

Considering about the failure mode analysis of 24-h water storage and thermocycles (Figure 12) was cohesive, mixed and adhesive failure. Cohesive failure in porcelain indicates that the bonding of resin-porcelain is superior to the actual inherent strength of the porcelain substrate.⁵¹⁾ Adhesive failure means the bonding system fail to produce any chemical bonds with the porcelain. Mixed failure is a combination of cohesive failure and adhesive failure. This means that some areas of bond are weak and produce adhesive failure. From this study indicated that Heliobond produced a strong bond of resin to porcelain. Fifty percent of the specimens in HB group failed cohesively in porcelain before and after thermocycles. While Single Bond Universal produced 20% of cohesive failure after 24-h water storage and no cohesive failure after thermocycling. In addition, no cohesive failure was observed in the system that yielded the lowest bond strength (Vertise Flow). It had only 50% mixed failure that produced a small area of cohesive porcelain fracture with an adhesive failure. For Superbond C&B and Bondfill SB was similar, there was more mixed failure after thermocycling. Although these two system recorded high bond strength after thermocycling, but only 30% (Superbond C&B) and 10% (Bondfill SB) of the specimens exhibited cohesive failure. From the result, the specimens appeared to fail combination in the resin layer of Superbond C&B (Figure 21) and in Bondfill SB (Figure 25). Superbond C&B does not contain filler¹²⁾

and mechanical failure in the resin layer of Superbond C&B is shown by its cohesive failure, consistent with a previous study.³⁴⁾ Bondfill SB exhibited the most mixed failure after thermocycling because some specimens showed failure in the Bondfill SB and adhesive failure between Bondfill SB and porcelain. The flexural strength of Bondfill SB⁵²⁾ is 66 MPa, similar to feldspathic porcelain, therefore it failed in both Bondfill SB (restorative material) and porcelain.

No agreement exists on minimal bond strength for retention of a resin bond to porcelain in the oral cavity. Some authors^{40,53)} have recommended a shear bond strength of 20 MPa for success of the repaired restorations (the adhesion between a conventional etched enamel).¹⁵⁾ The result of three systems (Superbond C&B, Bondfill SB, Heliobond) exceeded shear bond strength of 20 MPa. For Single bond Universal, the bond strength was higher than 20 MPa only in 24-h water storage (20.9 MPa) and Vertise Flow did not reach the level of 20 MPa in 24-h water storage (12.3 MPa) and after thermocycling (4.14 MPa). Whereas, some author⁴⁰⁾ suggested a strength of 5 MPa is given in ISO 10477 for polymer-based crown and bridge veneering material. All tested repair systems was higher than the ISO requirement of 5 MPa, except Vertise Flow after thermocycling. In addition, some authors^{23,34)} proposed that the minimum resin-porcelain bond strength must not less than 10-13 MPa. The failure mode will change from cohesive failure in the porcelain to adhesive failure when the bond strength is lower than 10-13 MPa^{23,34)} After 24-h water storage, the bond strengths of the Superbond C&B, Bondfill SB, Single Bond Universal and Heliobond were higher than the proposed minimum bond strength (10-13 MPa), while Vertise Flow was within the range. Therefore, Vertise Flow showed more adhesive failure than the other systems. After thermocycling, Vertise Flow had a bond strength less than the 10-13 MPa suggested, which is why all specimens showed adhesive failure.

Shear bond strength test method was preferred for this study because it has been reported that shear stresses are major stresses that generated in vivo bonding failure of restorations.¹⁾ In the previous studies,^{3,19,27,34-40)} bonding between the composite resin and porcelain was frequently investigated by shear bond strength methodologies but these test method may cause cohesive failure in the porcelain.¹⁰⁾

While feldspathic porcelain used in this study was mechanically weaker than the machinable ceramics block used in the other studies.³⁸⁾ Therefore, it might show cohesive failure in the porcelain, consistent with the previous study.^{13,19,34,35,37,54,55)}

However, in the present study the systems that appeared to be best for repairing porcelain are Bondfill SB and Heliobond, which showed superior mean bond strength after thermocycling. When comparing these two systems, other factors such as manipulation, setting time and number of steps need to be considered. Heliobond has more steps than Bondfill SB, as it uses HF etching and silane for surface pretreatment, a bonding agent and light curing for polymerization of the restoration. It may take longer to place but it can be polished immediately. Bondfill SB can be used without bonding or light curing because it is a self-cured material, but it is quite difficult to manipulate when the restoration is not cured and if the cavity is quite big, and it needs to mature for approximately 8-10 mins before finishing and polishing. Thus, there are advantages and disadvantages of both systems. The important issue is that the bond strength of Bondfill SB is close to Heliobond after thermocycling, but without using HF etching. Due to Hazardous surface effect of HF etching for both patient and operator, it must be used very carefully during the repair of porcelain surface. The elimination of the hazardous HF etchant step can be considered as an advantage in the dental surgery, especially in elderly patient.^{30,32,36)}

The result of this study is limited in this experimental study. The data cannot predict the clinical performance and the performance of ceramic repairs in vivo.⁹⁾ The further clinical observation is recommended.

CHAPTER VI

CONCLUSION

With the limitations of this study, the following conclusions were reached: the bond strength of a modified 4-META based porcelain repair system was not significantly different from Superbond C&B and Heliobond, but greater than Single Bond Universal and Vertise Flow. Systems with a separate silane were more durable after thermocycling. Therefore, based on this laboratory study, a modified 4-META based porcelain repair system appears to be the material of choice for repairing porcelain.



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Raw data of all groups

Table 5 Raw data of all groups

No.	Bond strength									
	24-h water storage					Thermocycles				
	SBW	BFW	SUW	HBW	VFW	SBT	BFT	SUT	HBT	VFT
1	18.59	25.42	26.62	26.92	13.25	30.04	23.13	6.44	29.53	2.19
2	19.93	29.92	12.99	36.44	10.55	15.67	22.22	8.79	27.15	4.00
3	20.64	23.35	16.06	27.66	8.91	17.44	29.22	13.48	22.02	3.53
4	28.86	29.01	20.61	20.52	11.19	21.59	17.80	10.18	35.90	4.88
5	20.73	25.82	18.09	24.69	11.21	24.62	30.87	15.89	24.18	4.09
6	15.85	25.19	18.94	32.51	18.40	24.54	24.43	20.95	21.77	2.77
7	27.26	29.60	20.15	27.23	18.21	18.04	25.26	14.51	20.72	5.18
8	24.44	25.29	19.57	27.12	15.77	15.34	27.53	16.78	21.26	5.15
9	16.24	24.79	28.20	31.66	7.66	20.49	30.98	10.59	29.82	4.68
10	25.27	28.59	27.92	33.19	8.15	26.39	19.92	21.42	19.26	4.96
Mean	21.8	26.7	20.9	28.8	12.3	21.4	25.1	13.9	25.1	4.14
SD	4.49	2.34	5.12	4.66	3.95	4.91	4.52	5.01	5.24	1.04

Normality test for bond strength groups
One-Sample Kolmogorov-Smirnov Test

24-h water storage

SB (24-h water storage)

One-Sample Kolmogorov-Smirnov Test ^a		
		bond
N		10
Normal Parameters ^{b,c}	Mean	21.7811
	Std. Deviation	4.48580
Most Extreme Differences	Absolute	.192
	Positive	.192
	Negative	-.123
Test Statistic		.192
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = SB

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

BF (24-h water storage)

One-Sample Kolmogorov-Smirnov Test ^a		
		bond
N		10
Normal Parameters ^{b,c}	Mean	26.6978
	Std. Deviation	2.33771
Most Extreme Differences	Absolute	.246
	Positive	.246
	Negative	-.190
Test Statistic		.246
Asymp. Sig. (2-tailed)		.087 ^d

a. group = BF

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

SU (24-h water storage)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	20.9146
	Std. Deviation	5.11595
Most Extreme Differences	Absolute	.224
	Positive	.224
	Negative	-.168
Test Statistic		.224
Asymp. Sig. (2-tailed)		.168 ^d

a. group = SU

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

HB (24-h water storage)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	28.7935
	Std. Deviation	4.65526
Most Extreme Differences	Absolute	.196
	Positive	.196
	Negative	-.143
Test Statistic		.196
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = HB

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

VF (24-h water storage)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	12.3295
	Std. Deviation	3.95459
Most Extreme Differences	Absolute	.211
	Positive	.211
	Negative	-.131
Test Statistic		.211
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = VF

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

Thermocycles

SB (Thermocycles)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	21.4162
	Std. Deviation	4.91275
Most Extreme Differences	Absolute	.154
	Positive	.154
	Negative	-.137
Test Statistic		.154
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = SB

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

BF (Thermocycles)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	25.1354
	Std. Deviation	4.51644
Most Extreme Differences	Absolute	.117
	Positive	.098
	Negative	-.117
Test Statistic		.117
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = BF

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

SU (Thermocycles)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	13.9026
	Std. Deviation	5.00784
Most Extreme Differences	Absolute	.146
	Positive	.146
	Negative	-.120
Test Statistic		.146
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = SU

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

HB (Thermocycles)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	25.1024
	Std. Deviation	5.23755
Most Extreme Differences	Absolute	.222
	Positive	.222
	Negative	-.132
Test Statistic		.222
Asymp. Sig. (2-tailed)		.178 ^d

a. group = HB

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

VF (Thermocycles)

One-Sample Kolmogorov-Smirnov Test^a

		bond
N		10
Normal Parameters ^{b,c}	Mean	4.1432
	Std. Deviation	1.03789
Most Extreme Differences	Absolute	.196
	Positive	.158
	Negative	-.196
Test Statistic		.196
Asymp. Sig. (2-tailed)		.200 ^{d,e}

a. group = VF

b. Test distribution is Normal.

c. Calculated from data.

d. Lilliefors Significance Correction.

e. This is a lower bound of the true significance.

One-way ANOVA

24-h water storage

ANOVA

bond

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1629.115	4	407.279	22.863	.000
Within Groups	801.634	45	17.814		
Total	2430.748	49			

Test of Homogeneity of Variances

bond

Levene Statistic	df1	df2	Sig.
1.249	4	45	.304

Post Hoc Tests

Multiple Comparisons

Dependent Variable: bond

	(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	SB	BF	-4.91670	1.88754	.086	-10.2801	.4467
		SU	.86646	1.88754	.991	-4.4969	6.2298
		HB	-7.01235*	1.88754	.005	-12.3757	-1.6490
		VF	9.45160*	1.88754	.000	4.0882	14.8150
	BF	SB	4.91670	1.88754	.086	-.4467	10.2801
		SU	5.78316*	1.88754	.029	.4198	11.1465
		HB	-2.09565	1.88754	.800	-7.4590	3.2677
		VF	14.36830*	1.88754	.000	9.0049	19.7317
	SU	SB	-.86646	1.88754	.991	-6.2298	4.4969
		BF	-5.78316*	1.88754	.029	-11.1465	-.4198
		HB	-7.87881*	1.88754	.001	-13.2422	-2.5155
		VF	8.58514*	1.88754	.000	3.2218	13.9485

Multiple Comparisons

Dependent Variable: bond

	(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
						HB	SB
		BF	2.09565	1.88754	.800	-3.2677	7.4590
		SU	7.87881*	1.88754	.001	2.5155	13.2422
		VF	16.46395*	1.88754	.000	11.1006	21.8273
VF	SB	-9.45160*	1.88754	.000	-14.8150	-4.0882	
		BF	-14.36830*	1.88754	.000	-19.7317	-9.0049
		SU	-8.58514*	1.88754	.000	-13.9485	-3.2218
		HB	-16.46395*	1.88754	.000	-21.8273	-11.1006

*. The mean difference is significant at the 0.05 level.

Homogeneous Subsets

		bond					
		group	N	Subset for alpha = 0.05			
				1	2	3	4
Tukey HSD ^a	VF	10	12.3295				
	SU	10		20.9146			
	SB	10		21.7811	21.7811		
	BF	10			26.6978	26.6978	
	HB	10				28.7935	
	Sig.			1.000	.991	.086	.800

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

Thermocycles

ANOVA

bond

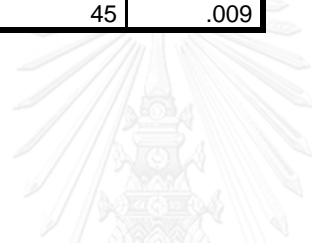
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3218.105	4	804.526	40.997	.000
Within Groups	883.089	45	19.624		
Total	4101.195	49			

Test of Homogeneity of Variances

bond

Levene Statistic	df1	df2	Sig.
3.862	4	45	.009

Post Hoc Tests



Multiple Comparisons

Dependent Variable: bond

	(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Dunnett T3	SB	BF	-3.71928	2.11029	.575	-10.3749	2.9364
		SU	7.51354*	2.21841	.030	.5224	14.5046
		HB	-3.68630	2.27084	.671	-10.8458	3.4732
		VF	17.27294*	1.58784	.000	11.7432	22.8027
	BF	SB	3.71928	2.11029	.575	-2.9364	10.3749
		SU	11.23282*	2.13253	.001	4.5042	17.9614
		HB	.03298	2.18701	1.000	-6.8766	6.9426
		VF	20.99222*	1.46545	.000	15.9052	26.0793
	SU	SB	-7.51354*	2.21841	.030	-14.5046	-.5224
		BF	-11.23282*	2.13253	.001	-17.9614	-4.5042
		HB	-11.19984*	2.29151	.001	-18.4227	-3.9770
		VF	9.75940*	1.61727	.001	4.1233	15.3955

Multiple Comparisons

Dependent Variable: bond

	(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
HB	SB	SB	3.68630	2.27084	.671	-3.4732	10.8458
		BF	-.03298	2.18701	1.000	-6.9426	6.8766
		SU	11.19984*	2.29151	.001	3.9770	18.4227
		VF	20.95924*	1.68847	.000	15.0662	26.8523
VF	SB	SB	-17.27294*	1.58784	.000	-22.8027	-11.7432
		BF	-20.99222*	1.46545	.000	-26.0793	-15.9052
		SU	-9.75940*	1.61727	.001	-15.3955	-4.1233
		HB	-20.95924*	1.68847	.000	-26.8523	-15.0662

*. The mean difference is significant at the 0.05 level.

Homogeneous Subsets

		bond			
		N	Subset for alpha = 0.05		
group			1	2	3
Tukey HSD ^a	VF	10	4.1432		
	SU	10		13.9026	
	SB	10			21.4162
	HB	10			25.1024
	BF	10			25.1354
	Sig.			1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

T-Test

SB

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2- tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
bond	Equal variances assumed	.073	.790	.173	18	.864	.36495	2.10375	-4.05486	4.78476
	Equal variances not assumed			.173	17.853	.864	.36495	2.10375	-4.05746	4.78736

BF

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2- tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
bond	Equal variances assumed	3.844	.066	.972	18	.344	1.56237	1.60820	-1.81634	4.94108
	Equal variances not assumed			.972	13.499	.348	1.56237	1.60820	-1.89892	5.02366

SU

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
bond	Equal variances assumed	.000	.996	3.097	18	.006	7.01203	2.26388	2.25580	11.76826
	Equal variances not assumed			3.097	17.992	.006	7.01203	2.26388	2.25565	11.76842



HB

Independent Samples Test

		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
bond	Equal variances assumed	.231	.636	1.666	18	.113	3.69100	2.21593	-.96449	8.34649
	Equal variances not assumed			1.666	17.756	.113	3.69100	2.21593	-.96909	8.35109

VF

Independent Samples Test

	Levene's Test for Equality of Variances		t-test for Equality of Means							
	F	Sig.	t	df	Sig. (2- tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference		
								Lower	Upper	
bond	Equal variances assumed	14.344	.001	6.332	18	.000	8.18629	1.29290	5.47000	10.90258
	Equal variances not assumed			6.332	10.234	.000	8.18629	1.29290	5.31443	11.05815

Stereomicroscope micrograph of interface of fracture surface

SB group

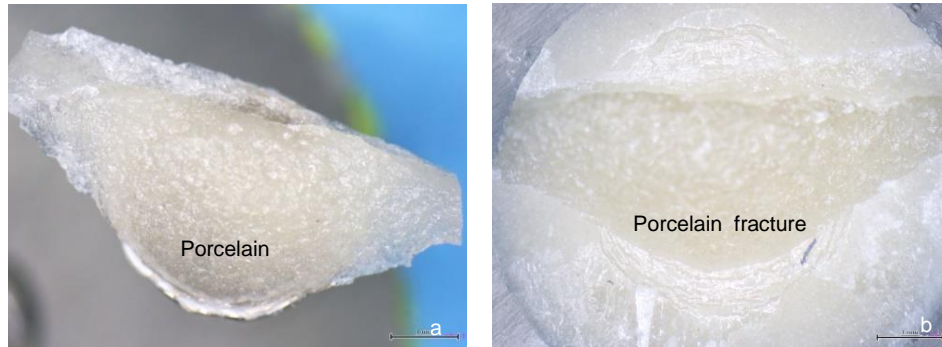


Figure 16 Cohesive failure in porcelain: (a) restorative site, (b) porcelain site (SB group)

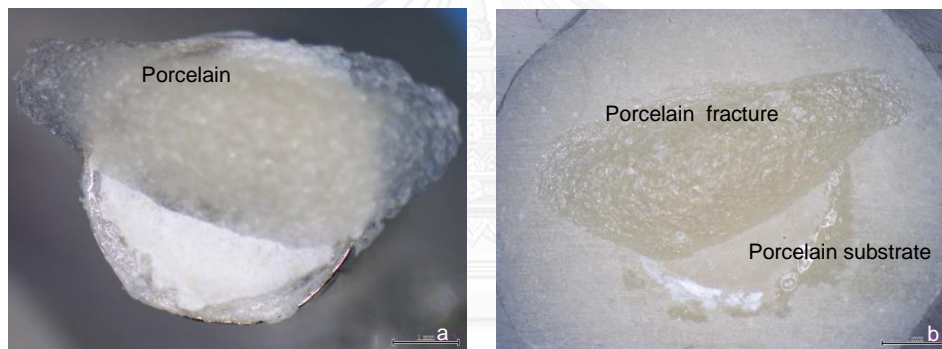


Figure 17 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site (SB group)

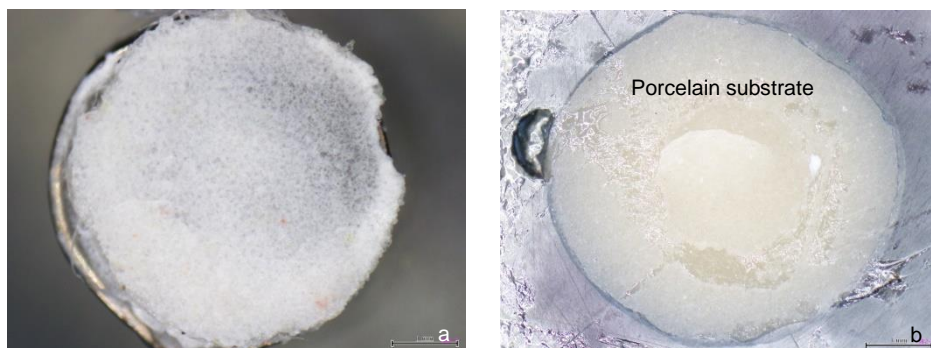


Figure 18 Adhesive failure between luting agent and porcelain: (a) restorative site, (b) porcelain site (SB group)

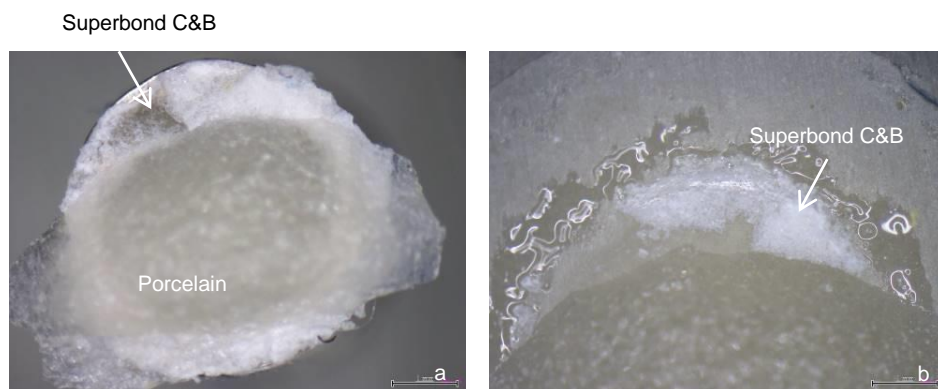


Figure 19 Mixed failure (failure in Superbond C&B and cohesive failure in the porcelain): (a) restorative site, (b) porcelain site (SB group)



BF group



Figure 20 Cohesive failure in porcelain: (a) restorative site, (b) porcelain site (BF group)

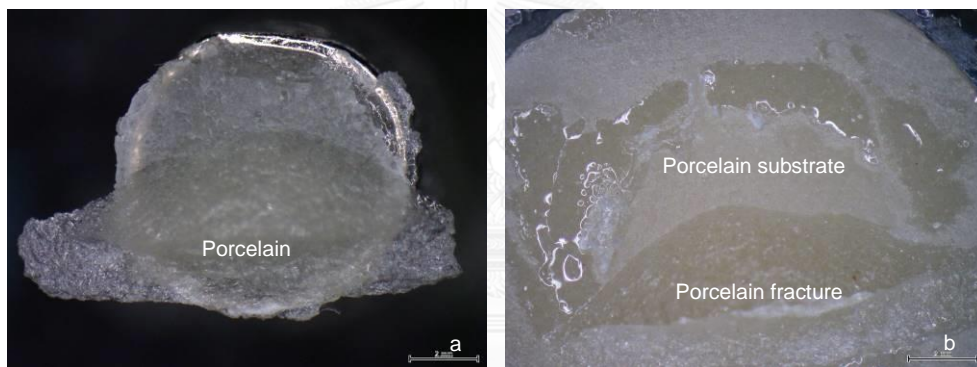


Figure 21 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site (BF group)

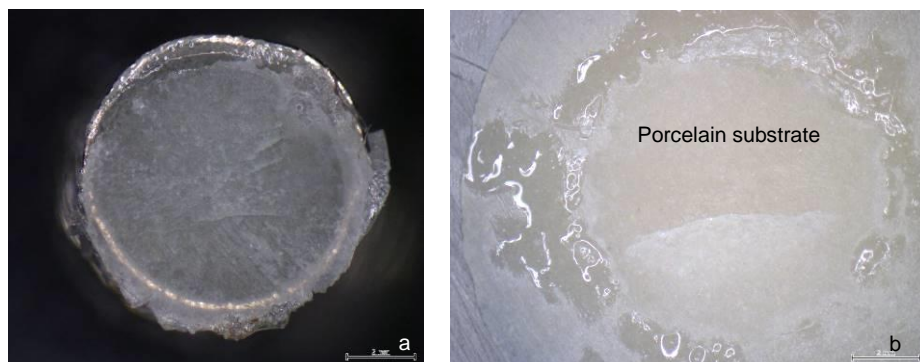


Figure 22 Adhesive failure between luting agent and porcelain: (a) restorative site, (b) porcelain site (BF group)

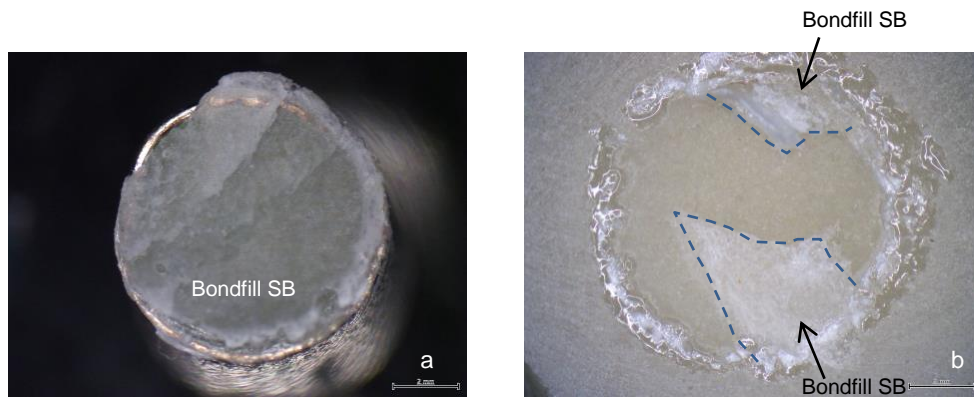


Figure 23 Mixed failure (adhesive failure between Bondfill SB and porcelain and cohesive failure in Bondfill SB): (a) restorative site, (b) porcelain site (BF group)



SU group

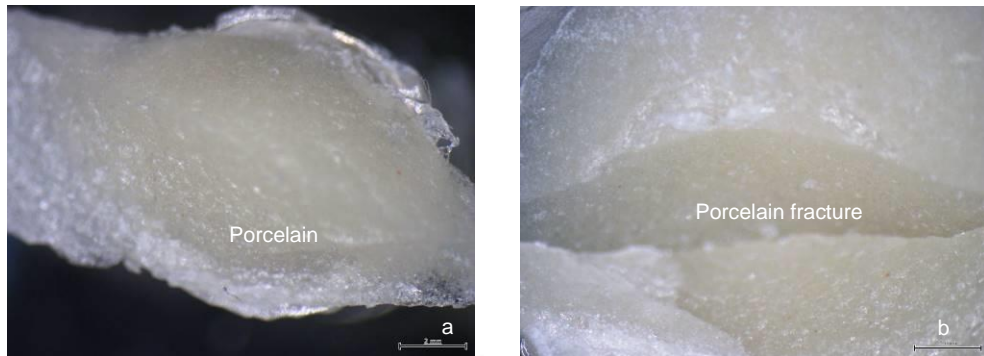


Figure 24 Cohesive failure in porcelain: (a) restorative site, (b) porcelain site (SU group)

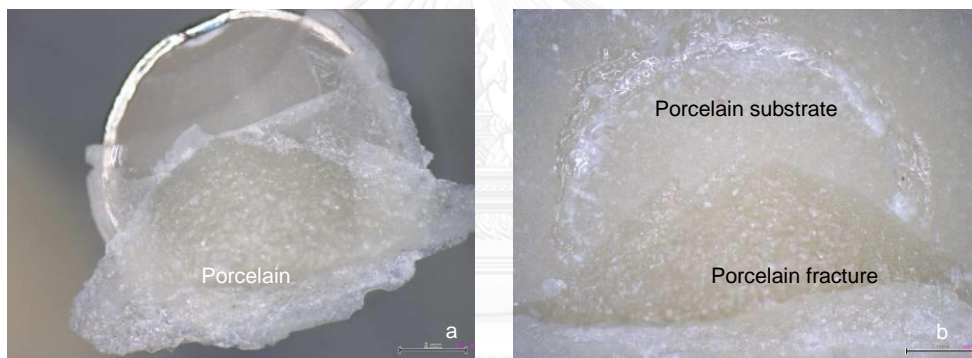


Figure 25 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site (SU group)

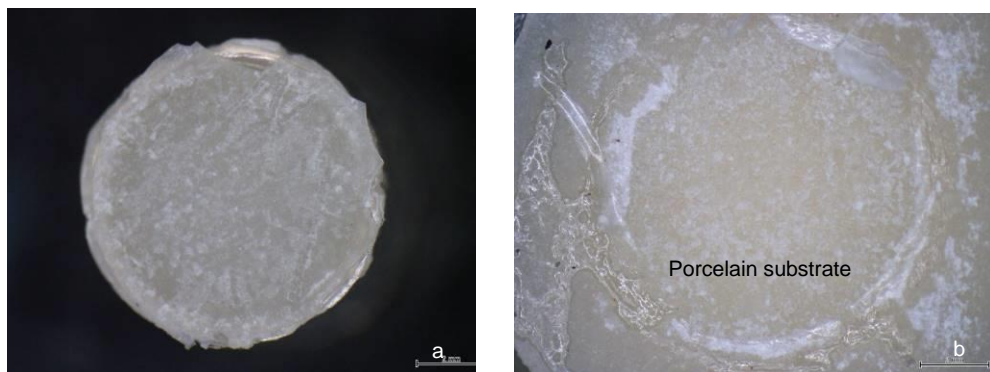


Figure 26 Adhesive failure between luting agent and porcelain: (a) restorative site, (b) porcelain site (SU group)

HB group

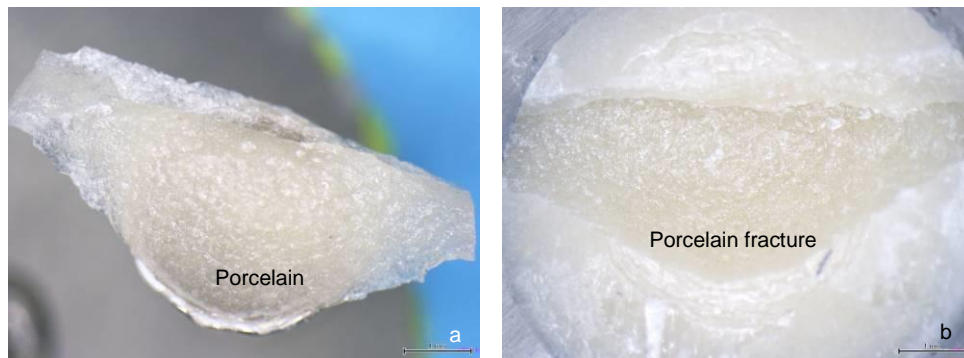


Figure 27 Cohesive failure in porcelain: (a) restorative site, (b) porcelain site (HB group)

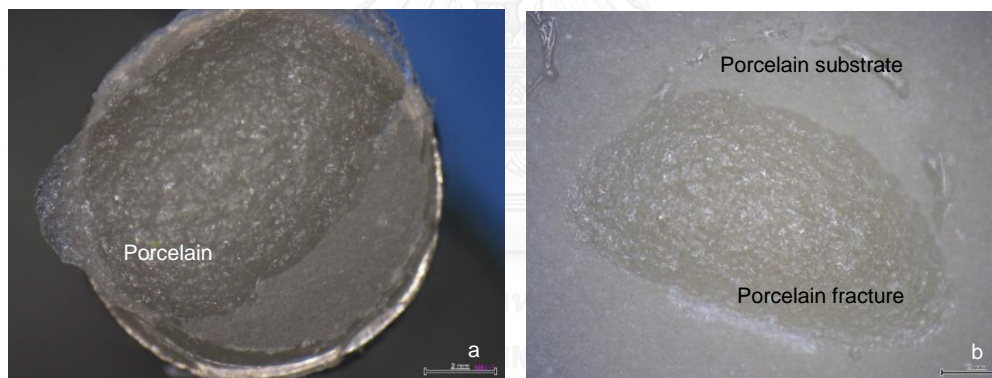


Figure 28 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site (HB group)

VF group

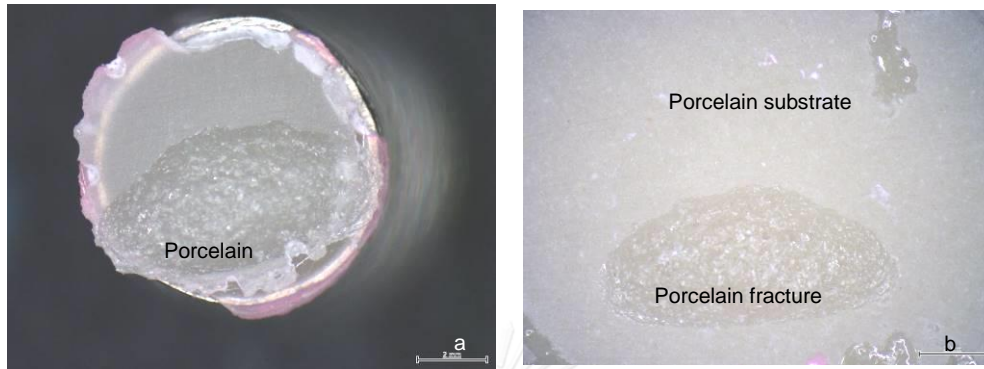


Figure 29 Mixed failure (adhesive failure between luting agent and porcelain and cohesive failure in porcelain): (a) restorative site, (b) porcelain site (VF group)

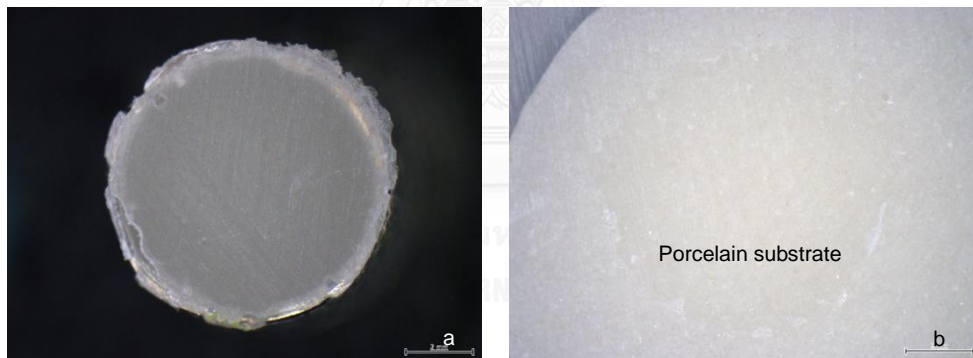


Figure 30 Adhesive failure between luting agent and porcelain: (a) restorative site, (b) porcelain site (VF group)

VITA

Miss Pimpisa Sringamprom was born in Bangkok, Thailand on December 20, 1985. In 2010, she graduated the degree of Doctor of Dental Surgery (D.D.S.) from Faculty of Dentistry, Mahidol University, Bangkok. After graduation, she worked as a dentist in Detudom crown prince hospital, Det Udom, Ubon Ratchathani (2010 - 2011) and Ban Phaeo Hospital (Public Organisation), Ban Phaeo, Samutsakorn (2011 - 2013). After that she started her study for the Master of Science Program in Prosthodontics at Faculty of Dentistry, Chulalongkorn University in 2013.

E-mail: s.pimpisa@outlook.com

