COMPARATIVE STUDY OF TENSILE BOND STRENGTH BETWEEN RESIN CEMENTS AND HYBRID MATERIALS

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การศึกษาเปรียบเทียบกำลังแรงยึดดึงระหว่างเรซินซีเมนต์และวัสดุชนิดไฮบริด

น.ส.ชาคริยา ดอนปิ่นไพร

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Thesis Advisor	Professor Morakot Piemjai, D.D.S., M.D.Sc., Ph.D

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วัตถุประสงค์: เพื่อศึกษาเปรียบเทียบค่าความแข็งแรงยึดดึงระหว่างวัสดุไฮบริด (VITA ENAMIC®, SHOFU Block HC, Katana AVENCIA และวัสดุทดลอง) กับเรซินซีเมนต์ 2 ชนิด คือ ซุปเปอร์บอนด์และรีไลย์เอ็กซ์ยูสองร้อย โดยใช้ร่วมกับยูนิเวอร์แซลเซรามิกไพรม์เมอร์และรีไลย์เอ็กซ์เซรามิกไพรม์เมอร์ตามลำดับ *วิธีการศึกษา:* เตรียมชิ้นตัวอย่างวัสดุไฮบริดทั้งหมดตัดขนาด 4x4x1 มิลลิเมตร^3 จำนวน 20 ชิ้นของวัสดุแต่ละชนิด นำชิ้นตัวอย่างไปขัดผิวด้วยกระดาษทรายซิลิกอนคาร์ไบด์ ความละเอียด 400 และ 600 และนำไปปรับสภาพผิวโดยวัสดุ VITA ENAMIC® ใช้ด้วยกรดไฮโดรฟลูออริกเข้มข้นร้อยละ 5 ร่วมกับเซรามิกไพรม์เมอร์ วัสดุ SHOFU Block HC และ Katana AVENCIA ใช้การเป่าทรายด้วยผงอะลูมินาขนาด 50 ไมครอน ร่วมกับเซรามิกไพรม์เมอร์ตามที่บริษัทแนะนำ ส่วนวัสดุทดลองใช้กรดฟอสฟอริกความเข้มข้นร้อยละ 65 หลังจากนั้นทำการสุ่มชิ้นตัวอย่างมายึดกับเคลือบฟันวัวที่ถูกลงบล็อคด้วยเดนทัลสโตน ด้วยเรซินซีเมนต์ 2 กลุ่ม กลุ่มละ 10 ตัวอย่าง หลังจากนั้นนำชิ้นตัวอย่างไปแช่น้ำที่อุณหภูมิ 37 องศาเซลเซียส 24 ชั่วโมง ก่อนนำมายึดกับแท่งพีเอ็มเอ็มเอเพื่อทดสอบแรงยึดดึงด้วยเครื่องทดสอบสากล ตรวจสอบตำแหน่งแตกหักบนผิววัสดุด้วยกล้องจุลทรรศน์ชนิดสเตอริโอและนำค่าความแข็งแรงยึดดึงมาทดสอบทางสถิติ *ผลการศึกษา:* การใช้วัสดุทดลองให้ค่าความแข็งแรงยึดดึงสูงที่สุด ตามด้วยวัสดุ VITA ENAMIC®, Katana AVENCIA และ SHOFU Block HC ตามลำดับ โดยไม่พบความแตกต่างกันระหว่างการใช้เรซินซีเมนต์ 2 ชนิด ($p>0.05$) และพบความล้มเหลวเกิดขึ้นที่บริเวณรอยต่อระหว่างเรซินซีเมนต์และวัสดุไฮบริดทั้งหมดในกลุ่มวัสดุ SHOFU Block HC *บทสรุป:* ชนิดของส่วนอนินทรีย์และการเตรียมพื้นผิวของวัสดุไฮบริดมีผลต่อค่าความแข็งแรงยึดดึง

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KEYWORD: hybrid material, filler, resin cement, tensile bond strength

Chakriya Donpinprai : COMPARATIVE STUDY OF TENSILE BOND STRENGTH BETWEEN RESIN CEMENTS AND HYBRID MATERIALS. Advisor: Prof. Morakot Piemjai, D.D.S., M.D.Sc., Ph.D

Purpose: To compare tensile bond strength among the hybrid materials (VITA ENAMIC®, SHOFU Block HC, Katana AVENCIA and Experimental material) using two primers (Universal ceramic primer and RelyX ceramic primer) and two resin cements (Super-Bond C&B and RelyX^{TM} U200). *Methods:* Twenty blocks of 4×4×1 mm^3 were prepared from each material type. In VITA ENAMIC® group, specimens were conditioned with 5% Hydrofluoric acid and primer. In SHOFU Block HC and Katana AVENCIA groups, specimens were treated with 50µm alumina and primer. In experimental material, specimens were etched with 65% Phosphoric acid. Ten specimens were randomly assigned to each group for different resin cement then cemented on bovine enamel. All cemented specimens were stored in water at 37°C for 24 hours. TBS was tested with a universal testing machine. The fractured interface was examined with a stereomicroscope and TBS data were statistically analyzed. *Result:* The experimental material showed the highest TBS values, followed by VITA ENAMIC®, Katana AVENCIA, and SHOFU Block HC, respectively. There was no significant difference between different resin cement ($p>0.05$). In SHOFU Block HC group, all fractured specimens showed adhesive failure. *Conclusion:* The types of inorganic components and surface treatment of the hybrid materials have an effect on TBS values.

Field of Study: Prosthodontics		Student's Signature ....
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CHAPTER I Introduction

Background and rationale

Ceramics and resin composite restorations are restorative materials that have been widely used because of their aesthetic properties. However, dental ceramic has some problems due to its hardness and brittleness. Their extreme hardness could potentially cause the opposing tooth to wear and their brittleness makes restorations susceptible to fracture.(1) Fracture of veneering porcelain is a complication that can occur in every dental ceramic material. Several studies(2-4) have suggested many methods to repair the ceramic restoration intraorally. Hydrofluoric acid etching appears to be a successful method for silicate-based material. However, using Hydrofluoric acid etching intraorally can cause an injury of the oral soft tissue.

Ease of repair or replacement is an advantage for resin composite restoration. This is because it is less invasive and is able to prolong tooth-retention in the long-term.(5, 6) Resin composite has less brittleness and hardness thus it does not wear the opposing tooth. On the other hand, the wear of itself is higher and it has lower color stability which limits its use.(7)

Nowadays, CAD/CAM (Computer-aided design/Computer-aided manufacturing) technology has gained importance and popularity in dentistry. It has been developed to possess many advantages such as speed, quality control, and repeatability.(8) The recently introduced CAD/CAM hybrid ceramic presents the advantages of ceramic and polymer properties including improved flexural properties, low abrasiveness, and more color stability and durability.(9) Many commercial brands of hybrid ceramics are launched to the market such as polymer-infiltrated ceramic network (PICN) material (VITA ENAMIC), Zirconia-silica ceramic (SHOFU Block HC), and resin nanoceramic (Lava Ultimate).

Minimally invasive treatment options have become increasingly favored in restorative dentistry. Adhesive bonding is an essential part in the placement of indirect restorations, which rely on adhesive cementation for retention, durability, and clinical effectiveness.(10) As a result, the adhesive bond between different materials must be suited to each material's physical characteristics.(11) A good adhesive bonding between the substrate and the material side could provide successful restorations.

Bond strength plays an important role in representing mechanical properties. Bonding to enamel is easy and durable due to its high mineral content and low water content. The etch-and-rinse technique is the most effective technique for bonding enamel. Etching partially demineralizes hydroxyapatite crystals then followed by polymerization of the resin, as well as creating hybridized enamel by micromechanical interlocking.(12-14) The hybridization zone is fundamental for successful restorations. It is a structure that is resistant to chemical attacks and impermeable to oral fluids, bacterial substances, and demineralizing agents.(14, 15)

Developing materials to have such a good adhesion property and ease of preparation is considered necessary for restorations. The hypothesis of this research is that the experimental material containing hydroxyapatite structure can provide a higher bond strength compared to commercial hybrid ceramic materials.

Research Question

Would the microstructures and compositions of hybrid materials have an effect on tensile bond strength value?

Research Objective

To compare tensile bond strength of experimental material and commercial hybrid ceramic materials when using various types of resin cement.

Research Hypothesis

H_0: There is no difference of tensile bond strength among hybrid material prostheses with various types of resin cement.

H_1: There is difference of tensile bond strength among hybrid material prostheses with various types of resin cement.

Proposed Benefits

To develop materials with good adhesion properties, ease of preparation, and improved clinical efficacy.

Keywords Hybrid ceramics, Tensile bond strength, Surface treatment, Resin cement, Filler

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 Hybrid Ceramics

Dental ceramics have recently been classified into three main categories: glass-ceramics; polycrystalline ceramics; and resin matrix ceramics, also known as hybrid ceramics (HCs). Hybrid ceramics are composed of organic matrix highly filled with ceramic particles. The goal was to (1) achieve a modulus of elasticity that was closer to dentin than typical ceramics, (2) design a material that was easier to mill and modify, and (3) repair easily with composite resin.(16) Thus, this material is combined the physical and mechanical advantages of ceramic and polymer such as improved flexural properties, low abrasiveness, and more color stability and durability.(9)

Hybrid ceramics can be divided by their inorganic composition, as follows:

1. Resin nanoceramic (Lava Ultimate, 3M ESPE)

It consists of a highly cured resin matrix reinforced with approximately 80% by weight nanoceramic particles.

2. Glass-ceramic in a resin interpenetrating matrix (eg, Enamic, Vita)

This is composed of both feldspathic ceramic network (86% by weight/75% by volume) and polymer network (14% by weight/25% by volume). The polymer network is composed of urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA).

3. Zirconia-silica ceramic in a resin interpenetrating matrix (Shofu Block HC, Shofu)

This is composed of zirconia-silica particles and different organic matrix in various weight percentage of ceramics.

The properties of these materials are depended on amount, size and type of crystalline phase.(8) Increasing in the inorganic composition leads to mechanical reinforcement. In contrast, the resilient property is reduced. Several inorganic fillers are used in hybrid ceramic such as glass particles, alumina, and zirconium.

2.2 Bonding to hybrid ceramics

To create a sufficient bond to hybrid ceramics, mechanical or chemical pre-treatments are recommended. Using various surface treatment techniques is depending on the composition of materials, such as silanization, etching with hydrofluoric acid (HF), and sandblasting.(17-20)

Peumans et al. reported that pre-treatments using 5% Hydrofluoric acid (HF) and silane enhanced bond strength in both Lava Ultimate and VITA ENAMIC. HF acid treatment is effective because it can partially dissolve the polymer and glassy phases which possibly increased micromechanical retention surface. After that, silane application increases the surface wetting of bonding area to improve the bond strength. However, sandblasting is not proper for glass ceramics surface treatment. It creates microcracks in the ceramic surface and leads to failures of prosthesis.(11)

Sandblasting with silanization can improve the bond strength of the CAD/CAM resin materials. Many studies reveal that using sandblasting with aluminum oxide 50 micron and then treating surface materials with a resin primer, which has MMA, can improve bond strength in SHOFU Block HC group. Sandblasting increases bond strength by exposing a clean, contaminant-free surface and enabling micromechanical retention for the cement at the roughened surface.(19, 21) The bond strength of CAD/CAM resin block (Shofu Block HC) using 4-META/MMA-TBB resin cement (Super Bond C&B, SB, Sun Medical, Moriyama, Japan) with sandblasting and silane coupling agent containing MMA has been reported a range between 19-24 MPa.(19) Nevertheless, there is no agreement on the most favorable surface treatment between resin cement and hybrid ceramics.

2.3 Resin cement

Resin cement is a luting agent that used to join a tooth to a prosthesis. It must show a low viscosity to flow along with the interfaces between the tooth surface and prosthesis. The variation of resin cement products is depended on composition, concentration and filler contents.(22)

O'Brien classified resin cement into 2 categories based on compositions(23)

1. Acrylic resin cement – The powder consists of polymethylmethacrylate and benzoyl peroxide as initiators. The liquid consists of methyl methacrylate monomer and amine as an accelerator. The polymerization occurs after the polymer is dissolved by monomer through peroxide-amine interaction. The adhesive promoter, 4-META (4-methacryloyloxyethyl trimellitate anhydride), is added to methacrylate monomer for assisting dentin bonding, initiated by tri-n-butyl borane (TBB).
2. Dimethacrylate cement – Resin cement in this type is similar to composite restorative material. It consists of an aromatic dimethacrylate, usually Bis-GMA, with monomer and fillers. Adhesive promoter can be added to resin cement such as phosphate or carboxyl groups such as MDP (10-Methacryloyloxydecyl dihydrogen phosphate). It is classified into 3 groups as chemical-cured, light-cured, and dual-cured resin cement.

2.4 Hybridized enamel

Hybrid layer is created by the demineralization of the dental hard tissue surface and subsurface, followed by the infiltration of monomers and polymerization.(14) The structure is composed of hydroxyapatite crystals and resin bonded efficiently. This zone is stable and impermeable to oral fluids or bacterial substances, and thus it could resist chemical attack and provide stable adhesion.(15,

24) Therefore, the completed hybrid layer is necessary for the restored tooth to provide a longer function.

Bonding to enamel was first described by Buonocore in 1955. Due to the larger mineral content and lower water content of enamel compared to dentin, it is easier, stronger, and more durable than bonding to dentin. The etch-and-rinse technique, with Phosphoric acid, is the most effective technique for bonding enamel. Thirty-five to thirty-seven percent of phosphoric acid etching for 30 seconds showed resin penetration in the range of approximately 15 µm in depth.(25-27) Etching partially demineralizes hydroxyapatite crystals creating numerous pits on enamel. This is followed by resin polymerization, which is absorbed by capillary attraction within enamel and is referred to as resin tags. There are two types of resin tags available. “Macro”-tags fill the space surrounding the enamel prisms while “micro”-tags fill the space within the tiny etch-pits at the cores of the etched enamel prisms. The latter is gained more retention to the enamel.(12, 13) Thus, the diffusivity of resin copolymers is key to create a resin-enamel hybrid layer.

4-methacryloxyethyl trimellitate anhydride (4-META) in methyl methacrylate (MMA) initiated by tri-n-butyl borane (TBB) (4-META/MMA-TBB) is MMA-based adhesive resin cement. It is a durable resin cement that bonds to the tooth structure.(28) 4-META has been shown to help monomers penetrate tooth structures.(14) Moreover, The molecular weight of MMA (mw=100) is the smallest among the dental polymerizable methacrylates and therefore it has a higher rate of diffusivity than the others.(15)

2.5 Bond strength testing

Bond strength plays an important role to represent mechanical properties. One way for evaluating the success of surface treatment is to measure the bond strength of adhesive systems to a prosthetic substrate. The popular methods to measure bond strength are shear bond strength test and tensile bond strength test. It can be divided into 2 types depending upon the size of the bonded area: micro-, smaller than 3 mm^2, and macro-test set-up.(29) In contrast to the shear bond strength test, more stress distribution and more uniform stress are associated with tensile bond strength methods.(29, 30)

Microtensile bond testing (μTBS) was first introduced by Sano et al in 1994.(31) This technique was created to test the bond strength of adhesive materials to a small area of tooth substrates. The technique's benefits include a better stress distribution at the bonded interface of small specimens under loading and the ability to partition one material into several specimen pieces. However, this testing is technical demanding, time-consuming and the fast dehydration of small samples.(32) Conversely, macrotensile bond testing is easy to prepare specimens and less time-consuming than microtensile bond strength testing.(33)

Several specimen types have been reported as hourglass, slap(rectangular), stick(square), and dumbbell shape.(30) The dumbbell shape specimen is efficient to identify defects in specimens when measuring tensile bond strength. Especially in dentin, remaining demineralized dentin is easy to detect in dumbbell shape specimens.(34) However, a higher incidence of pre-test failures occurs during trimming or sawing specimens by interfacial stress. Due to lack of agreement on specimen design, some authors have recommended the non-trimming specimen for reducing the stress before testing.(30, 35)

CHAPTER III Research Methodology

3.1 Materials and equipment

<u>Equipment</u>

1. Carbimet paper disc (Buehler; Lake Bluff, IL)
2. Polishing Machine (PRESI MINITECH 233, USA)
3. A low-speed saw (Isomet, Buehler, Lake Bluff, IL)
4. A micrometer (Mitutoyo Co., Kawasaki, Japan)
5. Scotch Magic Invisible Tape; 3M
6. Ultrasonic bath (VGT-1990, QTD, China)
7. Sandblasting machine (Vario Basic® Renfert, Germany)
8. Incubator (Contherm 160M, Contherm Scientific Ltd, Korokoro, Lower Hutt, New Zealand)
9. Universal testing machine (Shimadzu, Kyoto, Japan)
10. Light curing unit (EliparTrilightTM S10, 3M-ESPE St. Paul, MN, USA)
11. PVC mold ½ “

Table 1 *Trade names, manufacturers, and compositions.*

Material	Type	Manufacturer	Composition	Lot number
VITA ENAMIC® (EN)	Hybrid ceramic	Vita Zahnfabrik, Germany	86% feldspar ceramic Polymer: UDMA, TEGDMA	78560
SHOFU Block HC (SH)	Hybrid ceramic	Shofu Inc., Kyoto, Japan	61% zirconium silicate, silicon dioxide Polymer: UDMA, TEGDMA	77671
Katana AVENCIA (KA)	Hybrid ceramic	Kuraray Noritake Dental, Niigata, Japan	62% alumina filler (20 nm), silica filler (40 nm), Polymer: UDMA, TEGDMA	000957
Experimental material (EX)			90% Hydroxyapatite Polymer: PMMA	
Super-Bond	MMA-	Sun Medical,	Monomer: MMA, 4-META	TK1

C&B (SB)	based resin cement	Moriyama, Japan	Catalyst: TBB Polymer: PMMA	
RelyX^{TM} U200 (RX)	Adhesive resin cement	3M-ESPE St. Paul, MN, USA	Base paste: Methacrylate monomers containing phosphoric acid group, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: Methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigment, rheological additives	5289014
Universal ceramic primer	Priming agent	Sun Medical, Moriyama, Japan	Liquid A: Methacrylic monomer, others Liquid B: Methacrylic monomer, Silane coupling agent	TX1
RelyX Ceramic Primer	Priming agent	3M-ESPE St. Paul, MN, USA	Ethyl, alcohol, water, Methacryloxypropyltrimethoxysilane	N988623

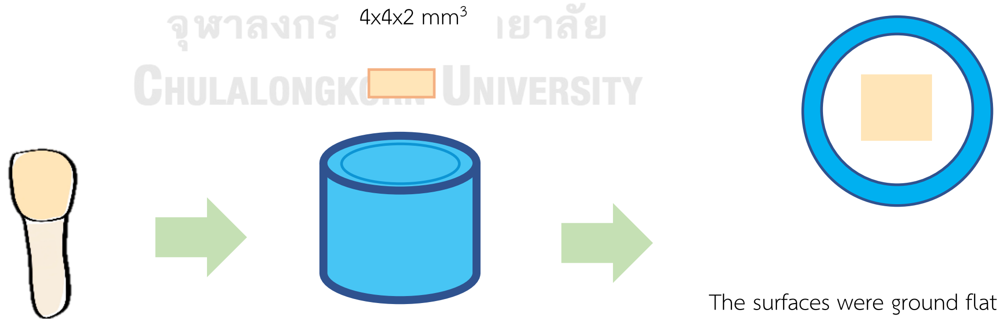
3.2 Experimental procedures

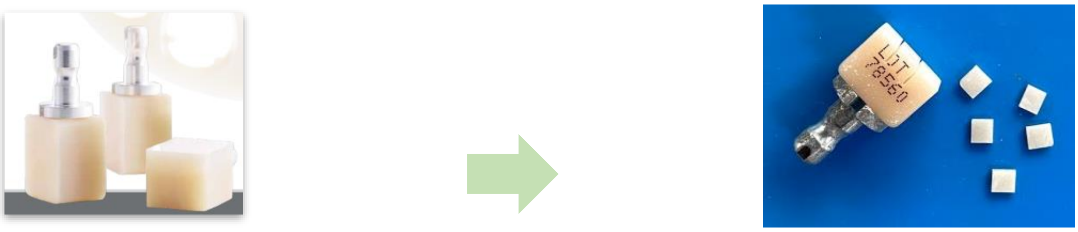
Part I Specimen preparation

The bovine tooth (enamel side) was sectioned into 4x4x2 mm^3 with a diamond disk(Slow speed cutting machine, Model Isomet, Buehler, IL, USA) then embedded in polyvinyl chloride (PVC) size ½” with dental stone in total 80 blocks. The surfaces were ground flat for surface standardization with 400-grit and 600-grit silicon carbide (Si-C) abrasive paper in a polishing machine (Minitech 233, Presi, Le Locle, Switzerland) under water cooling. After the polishing procedures, the substrate surfaces were ultrasonically cleaned in distilled water for 15 minutes and then air-dried.

The G power program was used to calculate the sample size and power for this research. The sample size was calculated using data from a previous pilot study (Table 4), which generated a sample size of 10 for each group.

The hybrid ceramic blocks and experimental material were sectioned into 4x4x1 mm^3 20 specimens per group (4 groups). The surfaces of all ceramic sections were ground flat for surface standardization with 400-grit and 600-grit silicon carbide (Si-C) abrasive paper in a polishing machine under water cooling. After the polishing procedures, the substrate surfaces were ultrasonically cleaned in distilled water for 15 minutes and then air-dried.





Shofu Block HC, VITA ENAMIC, Katana AVENCIA and Experimental material were cut into 4x4x1 mm^3

The surfaces were ground with 400-grit and 600-grit silicon carbide abrasive paper in a polishing machine and then cleaned in distilled water for 15 minutes.

Figure 1 *shows the steps of specimen preparation.*

The hybrid ceramic specimens and experimental material were randomly divided into 2 subgroups (n=10) for a different type of resin cement. The enamel blocks, experimental material, and hybrid ceramic specimens were treated on their surface depending on their type of material as shown in Table 2. To control the bonding area, a hole was punched in polyethylene tape 3 mm. diameter and placed on the center of the enamel surfaces.

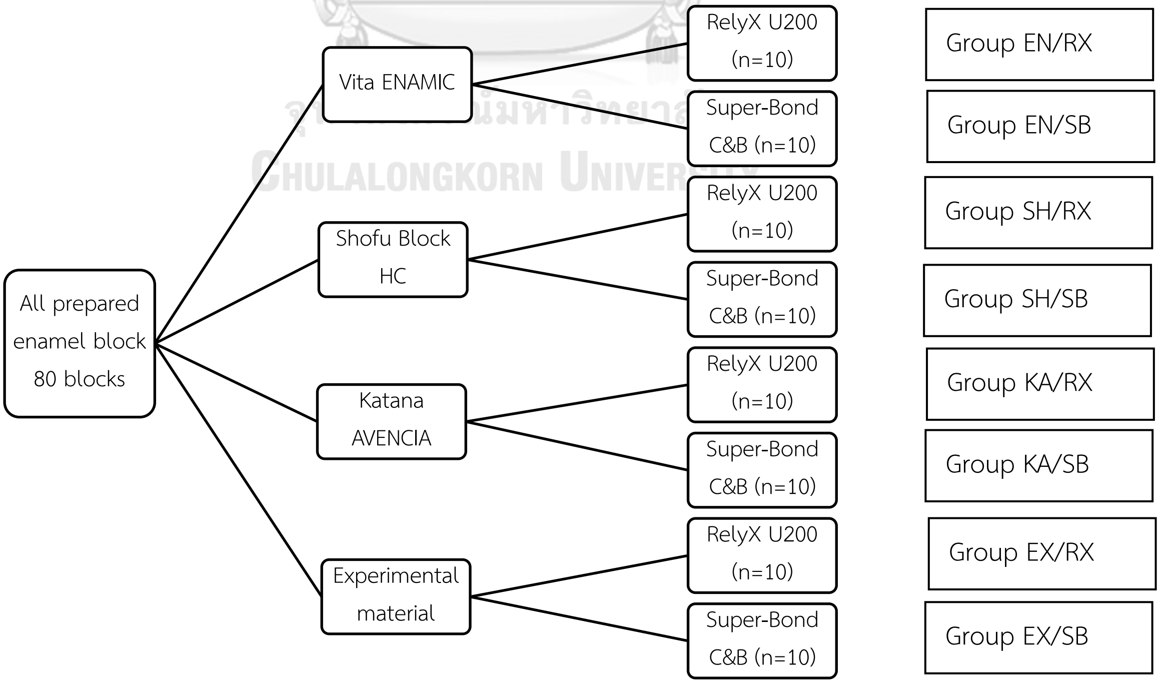


Figure 2 *shows a schematic diagram of the experimental procedure.*

***Table 2** Surface pretreatment methods.*

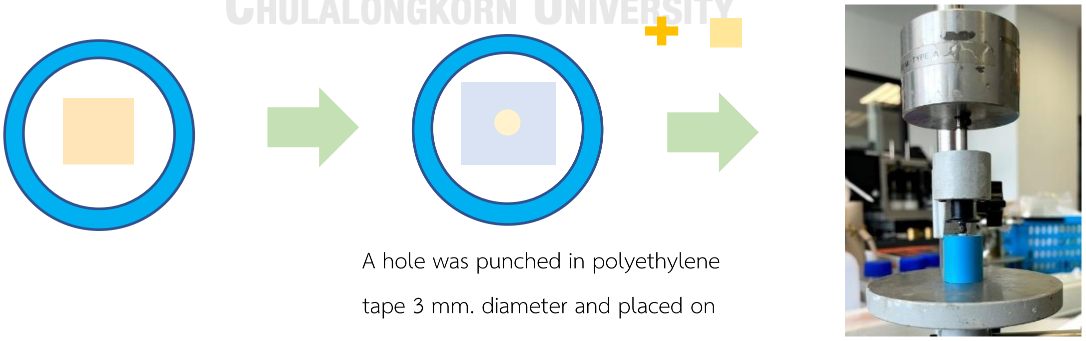
Surface treatment	**Material**	**Super-Bond C&B**	**RelyX$^{\text{TM}}$ U200**
	Enamel block	65% Phosphoric acid (Red activator) 30s, rinse 10s, air-dried 10s	37% Phosphoric acid 15s, rinse 10s, air-dried 10s
	Vita Enamic	5% Hydrofluoric acid (HF) for 60s rinse 10s, air-dried 10s and Universal Ceramic Primer	5% Hydrofluoric acid (HF) for 60s rinse 10s, air-dried 10s and RelyX Ceramic primer
	Shofu Block HC	Sandblasting with 50-μm aluminum oxide particles perpendicular to the surface from a distance of 10 mm. for 10s at a pressure 2 bar and Universal Ceramic Primer was applied	Sandblasting with 50-μm aluminum oxide particles perpendicular to the surface from a distance of 10 mm. for 10s at a pressure 2 bar and RelyX Ceramic Primer was applied
	Katana Avencia Block	Sandblasting with 50-μm aluminum oxide particles perpendicular to the surface from a distance of 10 mm. for 10s at a pressure 2 bar and followed by Universal Ceramic Primer	Sandblasting with 50-μm aluminum oxide particles perpendicular to the surface from a distance of 10 mm. for 10s at a pressure 2 bar and followed by RelyX Ceramic Primer
	Experimental material	65% Phosphoric acid (Red activator) 30s, rinse 10s, air-dried 10s	65% Phosphoric acid (Red activator) 30s, rinse 10s, air-dried 10s

Adhesion		- Mixing monomer 4 drops and catalyst 1 drop (4-META/MMA-TBB) -PMMA powder was applied on a specimen with brush dip technique	- Dosing of the two paste in 1:1 ratio then mixing the paste - Light-polymerized at the 4 proximal sides and the top surface, each for 20 seconds

Part II Cementation

Resin cement was applied in equal amount on each enamel block then all specimens were bonded. Luting was performed under 10N perpendicular pressure for 10 seconds at room temperature and light-polymerized at the 4 proximal sides and the top surface, each for 20 seconds in RelyX^{TM} U200 group. The blocks were stored in distilled water at 37°C for 24 hours in an incubator (Contherm 160M, Contherm Scientific Ltd, Korokoro, Lower Hutt, New Zealand) according to ISO/TS 11405

PMMA rods (cylindrical shape 5 mm. diameter and height 30 mm. with a hole) were ultrasonically cleaned for 15 minutes and bonded to the specimen then tensile bond strength was tested.



A hole was punched in polyethylene tape 3 mm. diameter and placed on the center of the enamel surfaces.

Luting was performed under 10N perpendicular pressure for 10 seconds.

Figure 3 Luting procedure

Part III Tensile bond strength

PVC tube was fixed to the holder and PMMA rod was held with a 2-mm diameter metal bar. Bending forces were avoided during specimen mounting. The specimens were aligned in a universal testing machine (SHIMADZU, EZ-S 500N model, Japan) and loaded in tension at a cross-headed speed of 1.0 mm/min until failure occurred. The force at failure was recorded in newtons. The cross-sectional area of the fractured specimen was remeasured with a micrometer (Mitutoyo Co., Kawasaki, Japan) and the surface area was calculated. Tensile strength was calculated in megapascal (MPa) by dividing the maximum load at failure (N) with the bonding area (mm^2). The debonded specimens were examined under a stereomicroscope (Olympus Stereo Microscopes, SZ61, Japan) at 40x magnification to determine the failure patterns and following by scanning electron microscope analysis (FEI Quanta 250) at ×5000 magnification.

The tensile bond strength was tested by using a universal testing machine at a crosshead speed of 1.0 mm/min until failure occurs.

PMMA rods were bonded with specimens.

***Figure 4** shows the universal testing machine with notched-edge shear bond strength testing.*

3.3 Statistics analysis

Statistical analysis was performed using SPSS version 22.0 (SPSS Inc, Chicago, Illinois) as follows:

If the population was normally distributed and homogeneity of variance, two-way analysis of variance (two-way ANOVA) was used to compare mean of tensile strength in each group at a 95% confidence level followed by Tukey’s HSD Post-hoc test to compare between groups at a 95% confidence level. But if the population was not normally distributed, the Kruskal Wallis test was used at a 95% confidence level.

CHAPTER IV Results

4.1 Tensile bond strength

The tensile bond strengths of all groups were analyzed (Table 3). Two-way ANOVA showed no statistically significant interaction between materials and resin cements ($p \geq 0.05$). No statistically significant differences in tensile bond strengths were found among types of resin cement.

One-way ANOVA and Games-Howell post hoc test were used for statistical analysis on material types. The highest bond strength was observed for experimental material groups while the lowest bond strength was observed for SHOFU Block HC groups, regardless of resin cement.

In Super-Bond C&B group showed Experimental material (12.09±2.08 MPa) had the highest value followed by VITA ENAMIC® (9.01±3.04 MPa), Katana AVENCIA (8.19±3.59 MPa), and SHOFU Block HC (5.34±1.33 MPa) respectively, but there was no significant difference between Experimental material and VITA ENAMIC®.

In RelyX^{TM} U200 group showed Experimental material (11.06±2.40 MPa) had significant difference and the highest value followed by VITA ENAMIC® (7.14±2.86 MPa), Katana AVENCIA (7.02±2.90 MPa), and SHOFU Block HC (6.00±1.98 MPa) respectively.

Table 3 *Mean values and standard deviations (MPa) of tensile bond strength for all groups.*

	VITA ENAMIC (EN)		SHOFU Block HC (SH)		KATANA AVENCIA (KA)		Experimental Material (EX)	
	Mean TBS ± SD	Failure mode A/C/M	Mean TBS ± SD	Failure mode A/C/M	Mean TBS ± SD	Failure mode A/C/M	Mean TBS ± SD	Failure mode A/C/M
Super-Bond C&B (SB)	9.01 ± 3.04^{AC}	70/0/30	5.34 ± 1.33^{B}	100/0/0	8.19 ± 3.59^{BC}	60/0/40	12.09 ± 2.08^{A}	0/0/100
RelyX™ U200 (RX)	7.14 ± 2.86^{B}	70/0/30	6.00 ± 1.98^{B}	100/0/0	7.02 ± 2.90^{B}	70/0/30	11.06 ± 2.40^{A}	0/0/100

Mean values represented with the different superscript uppercase letters (row) were significantly different at $p>0.05$

Percentage of failure mode [A: adhesive failure at the cement–materials interface/ C: cohesive failure within the luting cement/ M: mixed failure].

4.2 Failure mode

The analysis of failure modes indicated that adhesive failure pattern was the most common failure mode found in SHOFU Block HC group. Mixed failure was also the most common failure mode showed in an experimental group.

4.3 Stereomicroscope and SEM Analysis

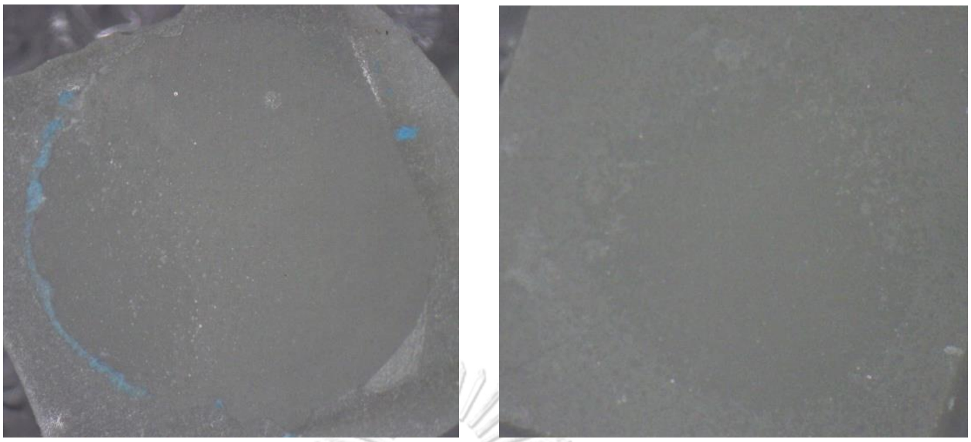


Figure 5 *Stereomicroscope images of the fractured surface in SH/RX group (left) and SH/SB group (right) at magnification 30x. The specimens showed adhesive failure between resin cement and SHOFU Block HC interface.*

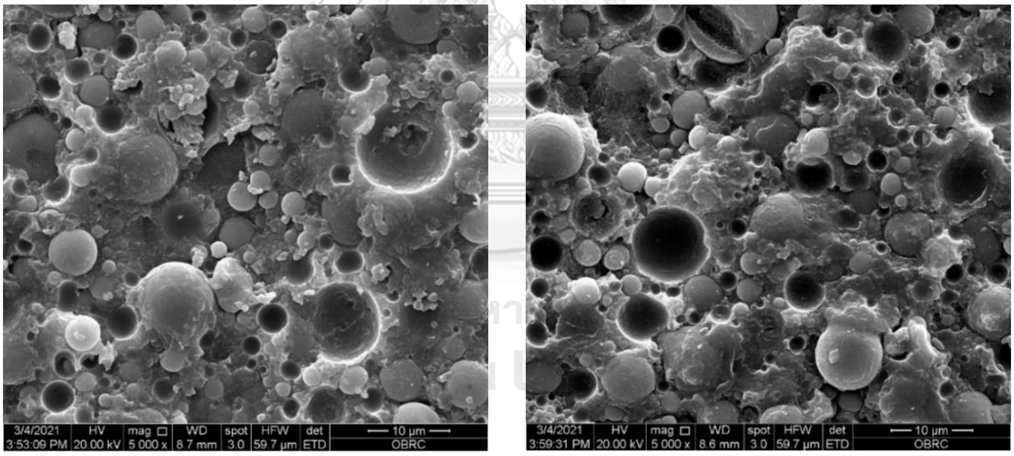


Figure 6 *SEM micrographs of the fractured surface in SH/RX (Left) group and SH/SB (Right) group at magnification 5000x showed the dislodgement of filler particles from SHOFU Block HC due to sandblasting.*

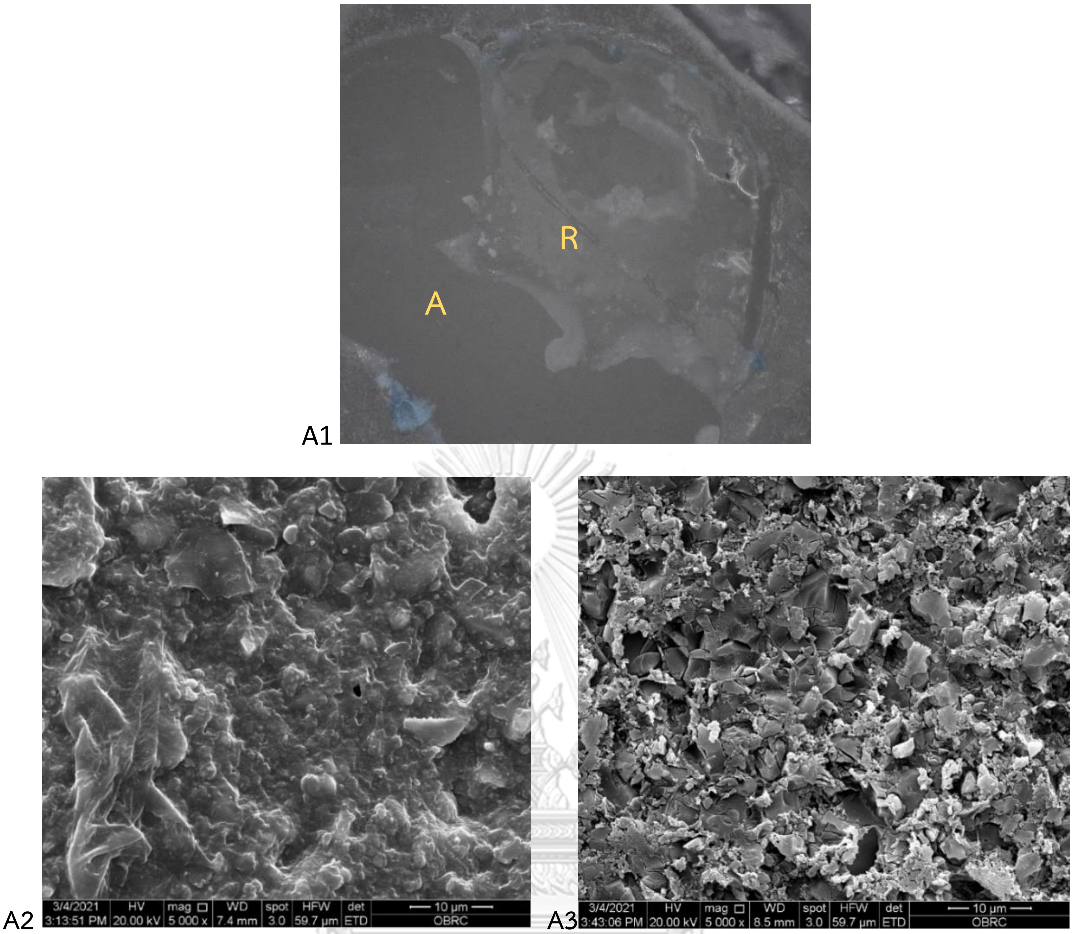


Figure 7 *A1) Stereomicroscope image of the fractured surface in EN/RX group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

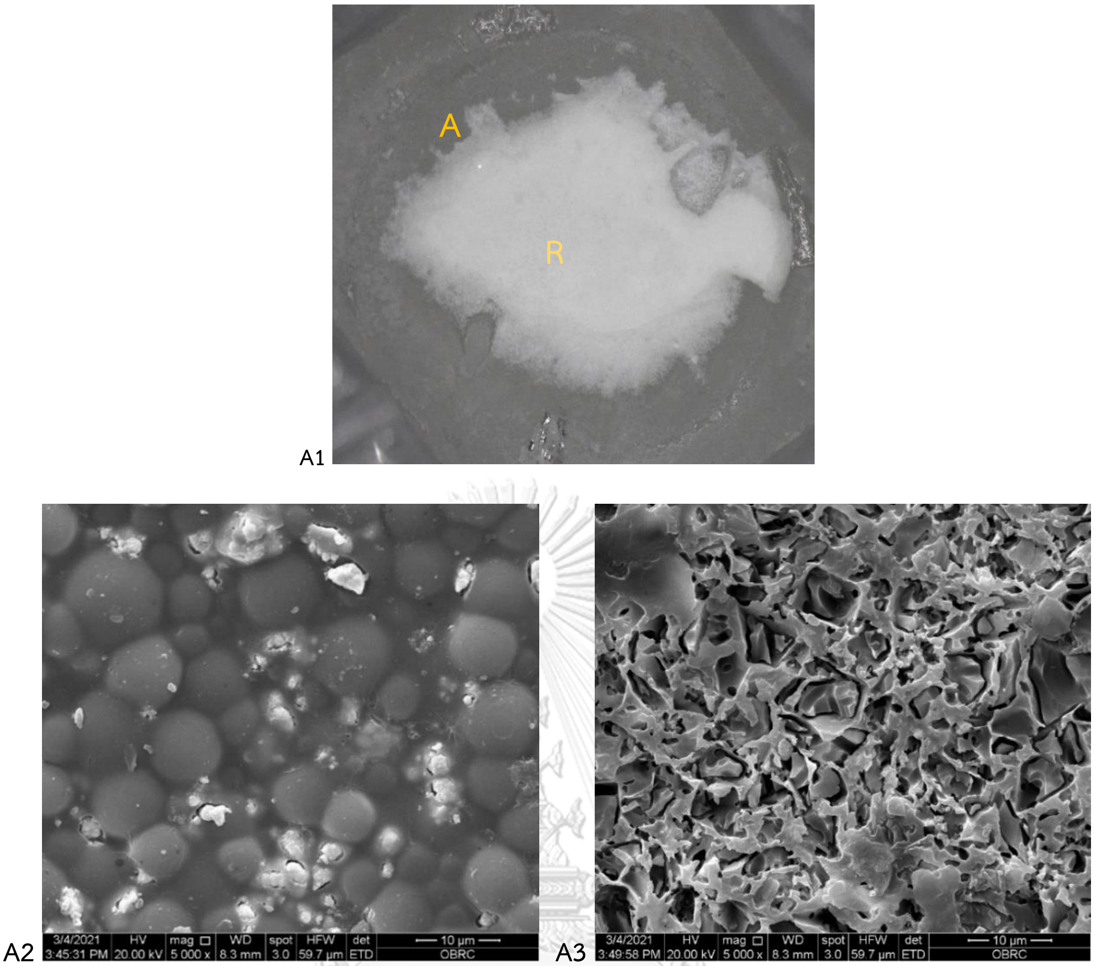


Figure 8 *A1) Stereomicroscope image of the fractured surface in EN/SB group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

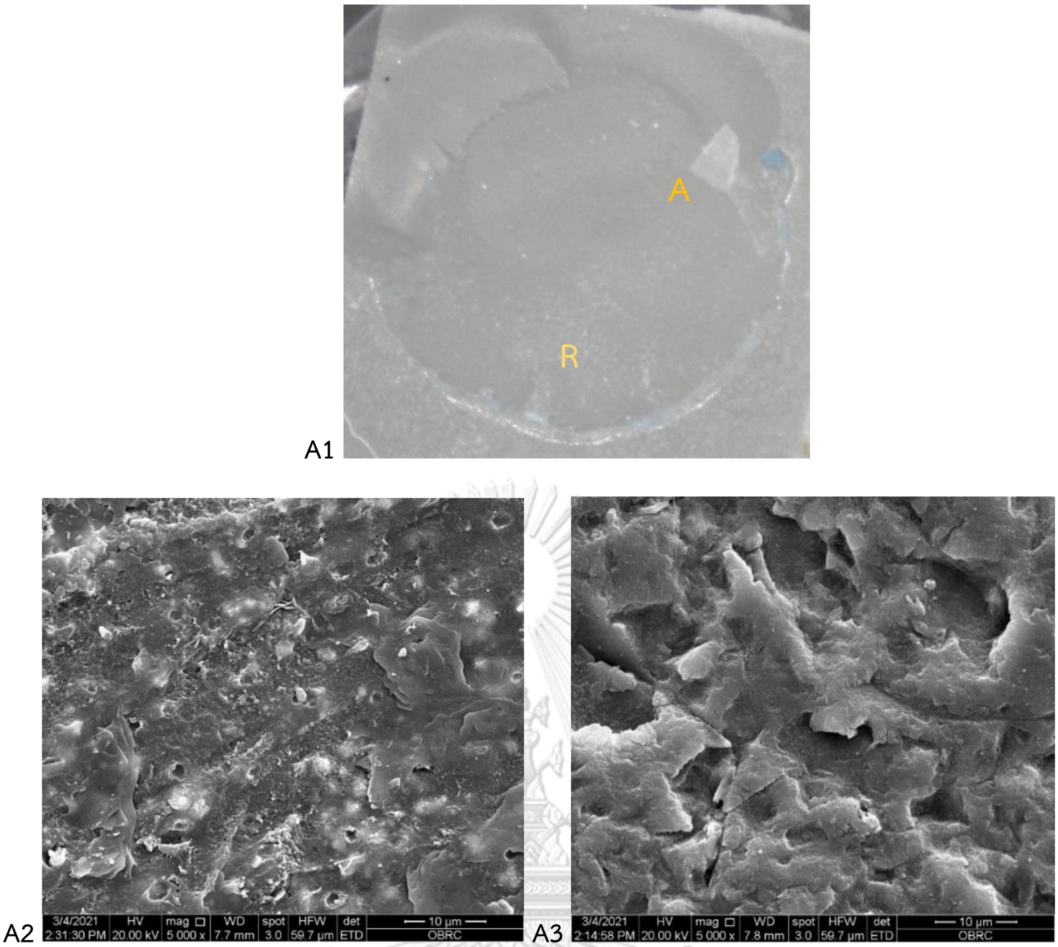


Figure 9 *A1) Stereomicroscope image of the fractured surface in KA/RX group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

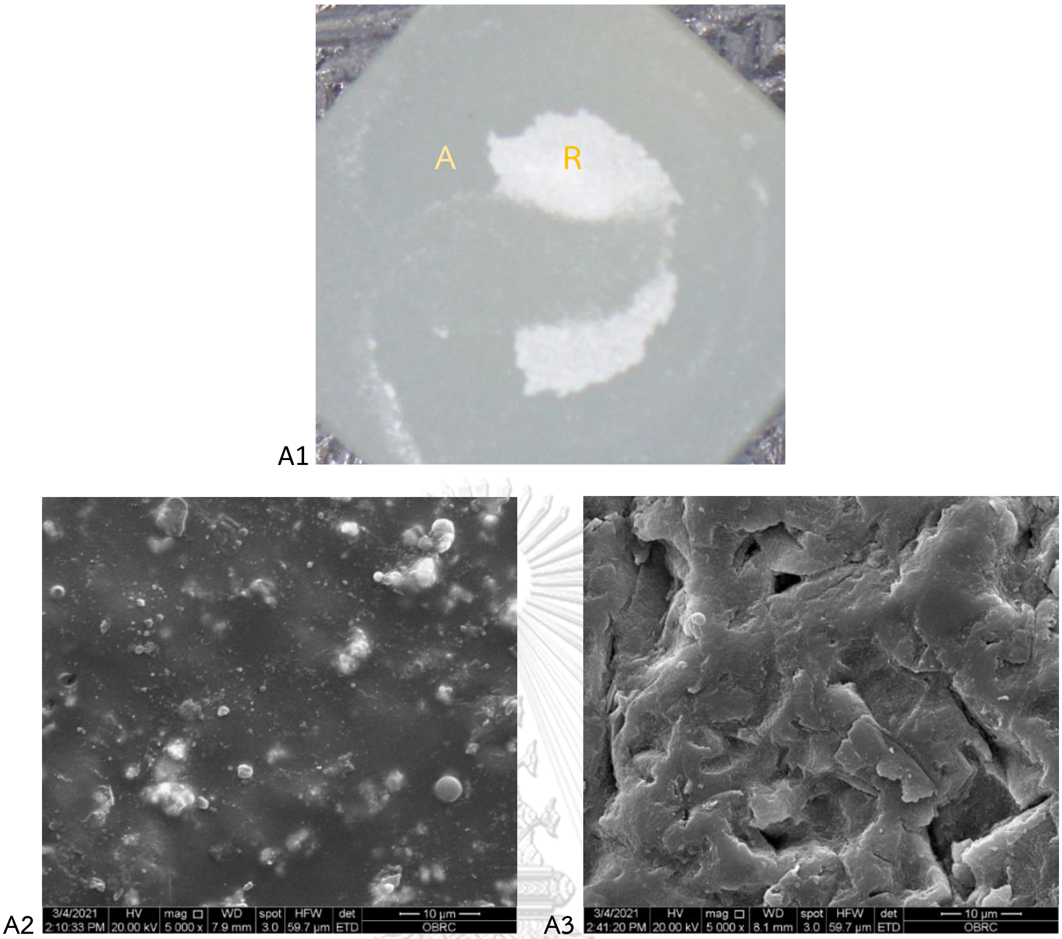


Figure 10 *A1) Stereomicroscope image of the fractured surface in KA/SB group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

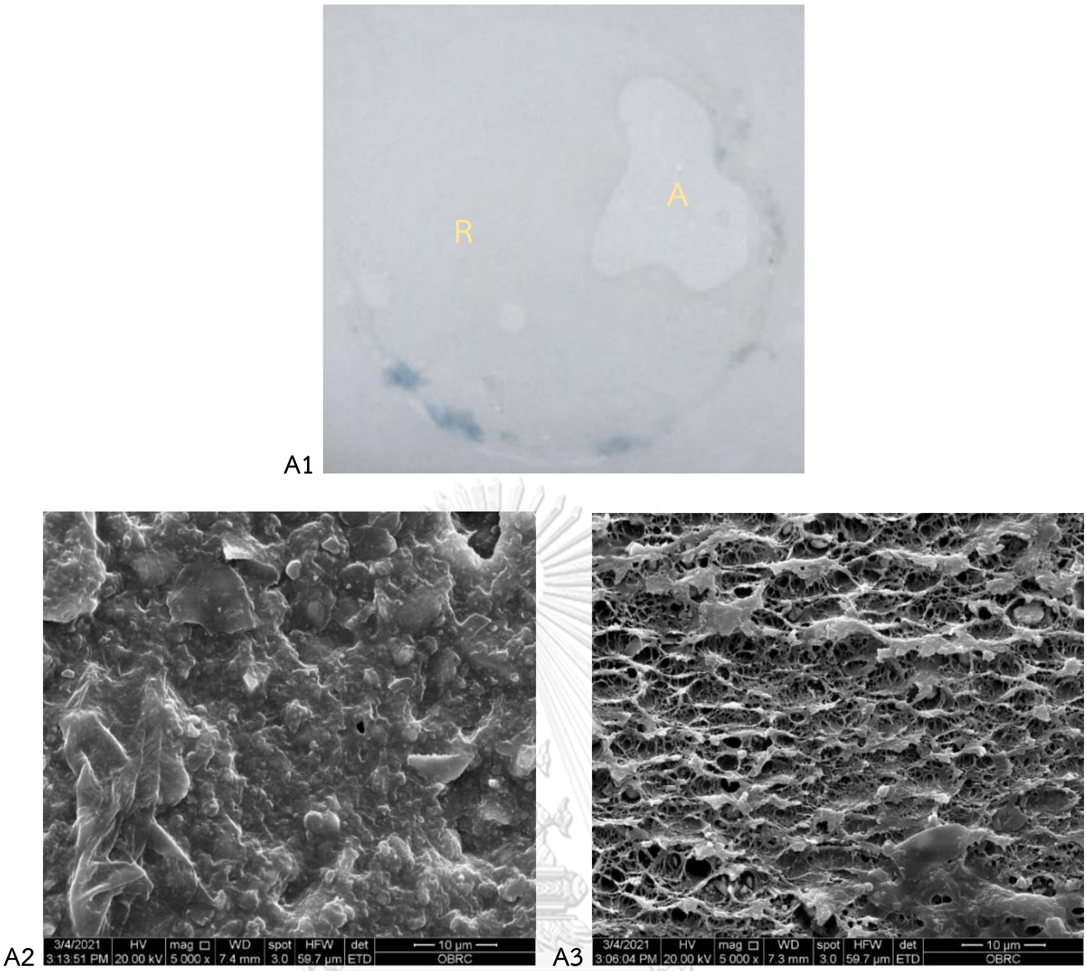


Figure 11 *A1) Stereomicroscope image of the fractured surface in EX/RX group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

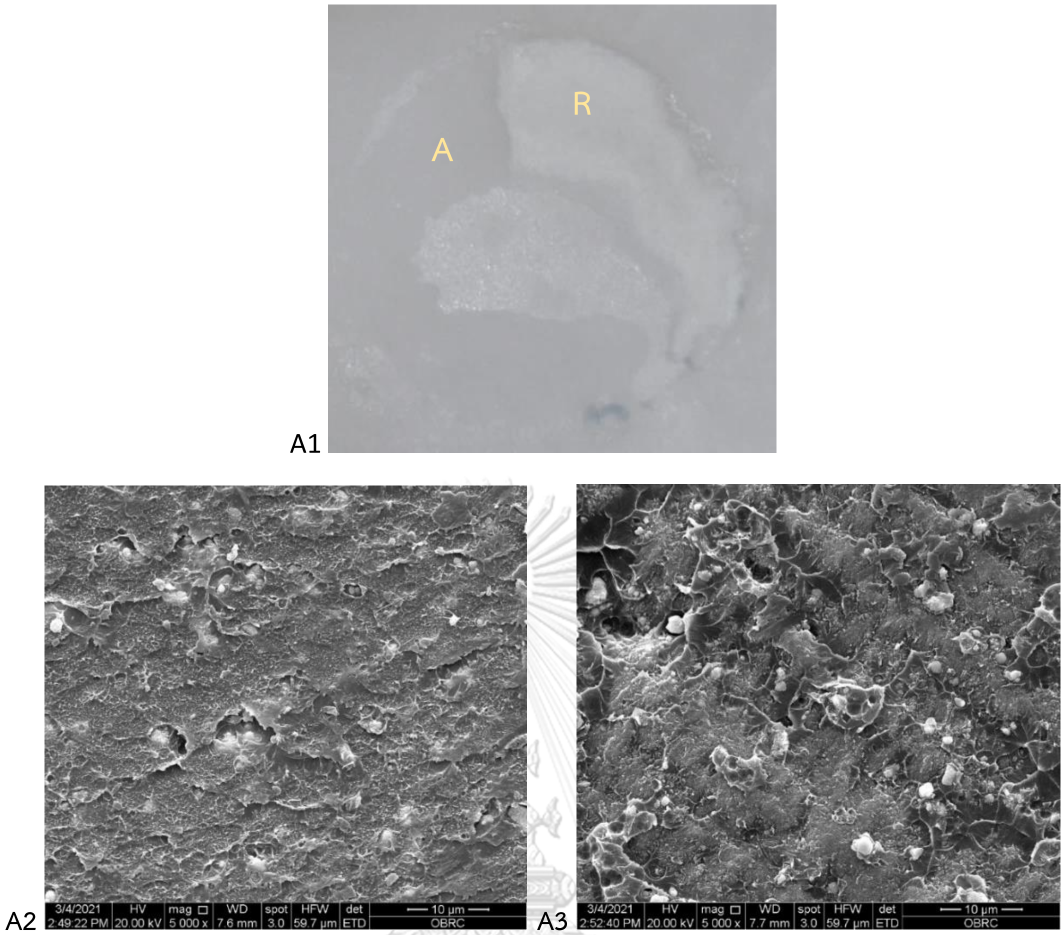


Figure 12 *A1) Stereomicroscope image of the fractured surface in EX/SB group at magnification 30x. The specimen showed mixed failure. A2) SEM micrographs at magnification 5000x showed cohesive failure in resin cement at R area. A3) adhesive failure between resin cement and ceramic at A area.*

CHAPTER V Discussion

5.1 Discussion

The purpose of this study was to compare tensile bond strength of the experimental material and commercial hybrid ceramic materials when using Super-Bond C&B or RelyX^{TM} U200 as a resin cement. As opposed to the shear bond strength test, the tensile bond strength test offers a more precise estimate of bond strength, more uniform and homogeneous stress distribution.(30, 36) The results showed that the lowest bond strength was found in SHOFU Block HC group while the highest bond strength was found in an experimental group. Mixed failure is associated with higher bond strength, while adhesive failure is associated with lower bond strength, which is consistent with the findings that mixed failure was the most common form of failure in an experimental group in both types of resin cement from all models while 100% adhesive failure was found in SHOFU Block HC group. (Figure 5)

The four hybrid materials in this study contained 61-90wt% inorganic components and varied in types. Except for the experimental material group, the others consisted of UDMA and TEGDMA as a polymer. SHOFU Block HC contained 61% nano-filler zirconium metallic glass as an inorganic part in the form of large spherical particles 1-10 µm. Katana AVENCIA contained 62% SiO_2 and Al_2O_3 ranging between size 20 and 50 nm.(37, 38) VITA ENAMIC® contained 86% feldspathic ceramic network and an infiltrated resin while SHOFU Block HC and Katana AVENCIA structure are composed of fillers dispersed in a resin matrix.(9, 39) Finally, the other is the experimental material that contained a 90% hydroxyapatite scaffold and infiltrated PMMA resin, which is the highest percentage of an inorganic component among the hybrid materials in this study. Therefore, the inorganic-matrix compositions and microstructures of the four hybrid materials studied in this study varied.

Physical methods including sandblasting with alumina or etching with hydrofluoric acid were used for increasing bond strength by improving mechanical interlocking, increasing wettability and increasing surface area.(11) Following the surface pretreatment protocols recommended by manufacturers, SHOFU Block HC and Katana AVENCIA were recommended using air abrasion while VITA ENAMIC® was recommended using hydrofluoric acid followed by a silane coupling agent.(17-19) Hydrofluoric acid can be used for etchable filler such as feldspathic ceramic, which is a filler in VITA ENAMIC®. Whereas fillers like zirconia and aluminum can use the air abrasion method, which is not proper for glass ceramics surface treatment. This technique may create microcracks in the ceramic surface and lead to mechanical failures of prosthesis.(11, 40)

A silane coupling agent recommended by manufacturers is effective for improving bonding ability by chemically bonding between silica-based inorganic fillers and resin cements as was shown in several studies.(8, 40, 41) However, the silanization effect was depended on the microstructure and the amount of inorganic content.(11, 20, 42) For the VITA ENAMIC® structure, the glass-ceramic network, which is exposed on the material surface due to physical methods, can cause the process of silanization. This process resulted in the tight bonding of the material with the resin cement. Whereas the chemical bond with silane does not occur in SHOFU Block HC due to the inorganic part which composes of zirconium silicate filler.(4) Corresponding to the previous study,(21) pre-test failure could be found in SHOFU Block HC more than the other groups because of the amount and its type of inorganic content. It can be concluded that the differences between inorganic-matrix compositions and microstructures seem to be responsible for the bonding ability of these hybrid materials regardless of resin cement.

For the SHOFU Block HC and Katana AVENCIA structures, sandblasting was recommended as the surface pretreatment method. This method can improve bond strength by exposing filler particles and enhancing micro-mechanical retention at the roughened surface.(43) These exposed filler particles can be reachable for silanization. However, SEM micrograph (Figure 6) showed the dislodgement of filler particles from SHOFU Block HC due to sandblasting, which corresponded to previous studies.(37, 44)

When sandblasting is at 2 bar blasting pressure, the material surface was destroyed and creating the 1-10 micron gap between the filler particles and resin matrix. Due to the manufacturing process, inorganic filler particles are often treated with silane.(45) The incorporation of silanized filler particles in the resin matrix will improve the mechanical strength and optimize the hydrolytic stability of composite resin. For bonding to non-silica-based filler particles like zirconia, a silane coupling agent alone may be insufficient. As a consequence of lacking polar bonds, zirconia-filler surfaces are incapable of forming chemical bonds with silane hydroxyl groups. As a result, after mechanical roughening, inorganic components may be detached due to the weakened bond to the matrix. Consequently, the filler particles were detached from the resin matrix and using the silane coupling agent after the surface pretreatment method was less effective. This had a negative impact on SHOFU Block HC's bonding capacity and tensile bond strength value.

Mixed failure is associated with higher bond strength, whereas adhesive failure is associated with lower bond strength, which is consistent with the findings in Figure 7-12, which shows that mixed failure was the most common kind of failure in the other groups. In the SHOFU Block HC group, only adhesive failure was observed. There is a correlation with 100% adhesive failure found in this group which means the weakest point was at the connection between the material and the resin cement. As a result of unnecessary damage to the material surface, the surface pretreatment protocols of SHOFU Block HC should be revised by manufacturers and effective surface treatment is needed to improve zirconia bonding to the resin matrix.

The highest bond strength was found in the experimental material group which the composition consisted of 90% hydroxyapatite in a scaffold pattern. The microhardness and tensile bond strength test of an experimental material were examined in the previous study and the results were promising.(46) Acid etching with phosphoric acid was recommended as the surface treatment method for this group.

There is an easy process for the preparation of surface materials without inducing tissue irritation as hydrofluoric acid and destroying the surface of material as sandblasting.(47) As previously mentioned, creating a sufficient bond to hybrid materials was depended on several factors including materials selection and surface pretreatment methods. In this present study, the inorganic-matrix compositions and microstructure directly affected the bonding ability regardless of resin cement. According to the promising results of this material in tensile bond strength and convenience of surface treatment, other properties should be further investigated for developing this material.

5.2 Limitation

The test was carried out 24 hours after cementation, further research could be carried out to see whether longer storage times, thermocycling, or different resin cement systems can be used.

5.3 Conclusion

Within the limits of the study, it can be concluded that the bond strength of hybrid ceramic materials is influenced by surface treatment, compositions, and microstructure of materials. In the SHOFU Block HC group, the surface pretreatment protocols should be revised by manufacturers. The experimental material should be further investigated.

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APPENDIX

Table 4 G Power Calculation

Statistical test ANCOVA: Fixed effects, main effects and interactions

Input parameter		**Output parameter**	
Effect size f	0.4	Noncentrality parameter λ	11.68
α err prob	0.05	Critical F	2.75
Power (1-β err prob)	0.8	Denominator df	65
Numerator df	3	Total sample size	73
Number of groups	8	Actual power	0.80
Number of covariates	0		

Table 5 Tensile bond strength values (MPa) and mode of failure

Groups	Number	Tensile bond strength values (MPa)	Mode of failure
VITA ENAMIC/Super-Bond C&B	1	5.97	A
	2	8.27	A
	3	6.19	A
	4	7.96	A
	5	15.53	M
	6	11.52	M
	7	4.912	A
	8	9.15	A
	9	7.44	A
	10	13.19	M
VITA ENAMIC/ RelyX$^{\text{TM}}$ U200	1	6.18	A
	2	10.99	M
	3	6.17	A
	4	11.65	M

	5	10.96	M
	6	4.46	A
	7	4.78	A
	8	4.88	A
	9	5.90	A
	10	5.46	A
SHOFU Block HC/Super-Bond C&B	1	3.74	A
	2	3.56	A
	3	5.57	A
	4	5.02	A
	5	4.92	A
	6	4.53	A
	7	4.92	A
	8	6.44	A
	9	7.26	A
	10	7.39	A
SHOFU Block HC/RelyX^{TM} U200	1	3.72	A
	2	3.46	A
	3	5.60	A
	4	8.11	A
	5	6.63	A
	6	4.06	A
	7	5.88	A
	8	7.66	A
	9	9.44	A
	10	5.49	A
Katana AVENCIA/Super-Bond C&B	1	4.77	A
	2	11.09	M
	3	4.77	A
	4	6.70	A
	5	4.69	A
	6	14.48	M

	7	5.33	A
	8	7.65	A
	9	12.49	M
	10	9.96	M
Katana AVENCIA/RelyX^{TM} U200	1	8.27	A
	2	3.40	A
	3	9.18	M
	4	7.80	A
	5	12.60	M
	6	8.58	M
	7	3.11	A
	8	6.89	A
	9	5.02	A
	10	5.31	A
Experimental material/Super-Bond C&B	1	14.44	M
	2	9.59	M
	3	10.73	M
	4	11.57	M
	5	13.89	M
	6	10.02	M
	7	12.16	M
	8	15.29	M
	9	9.76	M
	10	13.45	M
Experimental material/RelyX^{TM} U200	1	7.97	M
	2	8.32	M
	3	9.67	M
	4	11.15	M
	5	14.50	M
	6	10.04	M
	7	12.88	M
	8	14.40	M

	9	9.21	M
	10	12.45	M

[A: adhesive failure at the cement–materials interface/ C: cohesive failure within the luting cement/ M: mixed failure].

Table 6 Descriptive Statistics

Dependent Variable: TBS

Material type	Resin Cement	Mean	Std. Deviation	N
SH	SB	5.3350	1.33297	10
	RX	6.0050	1.98007	10
	Total	5.6700	1.67837	20
EN	SB	9.0130	3.40623	10
	RX	7.1410	2.86291	10
	Total	8.0770	3.20944	20
KA	SB	8.1930	3.58904	10
	RX	7.0160	2.90095	10
	Total	7.6045	3.23303	20
EX	SB	12.0900	2.08009	10
	RX	11.0580	2.39744	10
	Total	11.5740	2.24775	20
Total	SB	8.6578	3.60525	40
	RX	7.8050	3.14348	40
	Total	8.2314	3.38806	80

Table 7 Levene's Test of Equality of Error Variances[a]

Dependent Variable: TBS

F	df1	df2	Sig.
2.262	7	72	.039

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

a. Design: Intercept + Material_type + ResinCement + Material_type * ResinCement

Table 8 Tests of Between-Subjects Effects

Dependent Variable: TBS

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	395.030[a]	7	56.433	7.939	.000	.436
Intercept	5420.443	1	5420.443	762.538	.000	.914
Material_type	363.012	3	121.004	17.023	.000	.415
ResinCement	14.544	1	14.544	2.046	.157	.028
Material_type * ResinCement	17.475	3	5.825	.819	.487	.033
Error	511.806	72	7.108			
Total	6327.279	80				
Corrected Total	906.836	79				

a. R Squared = .436 (Adjusted R Squared = .381)

Table 9 Test of Homogeneity of Variances

TBS

ResinCement	Levene Statistic	df1	df2	Sig.
SB	4.195	3	36	.012
RX	.853	3	36	.474

Table 10 ANOVA

TBS

Resin Cement		Sum of Squares	df	Mean Square	F	Sig.
SB	Between Groups	231.632	3	77.211	10.097	.000
	Within Groups	275.284	36	7.647		
	Total	506.916	39			
RX	Between Groups	148.854	3	49.618	7.552	.000
	Within Groups	236.522	36	6.570		
	Total	385.376	39			

Table 11 Robust Tests of Equality of Means

TBS

ResinCement		Statistic[a]	df1	df2	Sig.
SB	Welch	24.078	3	18.609	.000
RX	Welch	8.707	3	19.762	.001

a. Asymptotically F distributed.

Table 12 Multiple Comparisons

Dependent Variable: TBS

Resin	Cement	(I) Material_type	(J) Material_type	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
							Lower Bound	Upper Bound
SB	Games-Howell	SH	EN	-3.67800*	1.15668	.036	-7.1256	-.2304
			KA	-2.85800	1.21070	.141	-6.4790	.7630
			EX	-6.75500*	.78125	.000	-9.0011	-4.5089
		EN	SH	3.67800*	1.15668	.036	.2304	7.1256
			KA	.82000	1.56472	.952	-3.6035	5.2435
			EX	-3.07700	1.26211	.112	-6.7177	.5637
		KA	SH	2.85800	1.21070	.141	-.7630	6.4790
			EN	-.82000	1.56472	.952	-5.2435	3.6035
			EX	-3.89700*	1.31179	.043	-7.6953	-.0987
		EX	SH	6.75500*	.78125	.000	4.5089	9.0011
			EN	3.07700	1.26211	.112	-.5637	6.7177
			KA	3.89700*	1.31179	.043	.0987	7.6953
RX	Games-Howell	SH	EN	-1.13600	1.10077	.734	-4.2852	2.0132
			KA	-1.01100	1.11068	.800	-4.1911	2.1691
			EX	-5.05300*	.98328	.000	-7.8417	-2.2643
		EN	SH	1.13600	1.10077	.734	-2.0132	4.2852
			KA	.12500	1.28887	1.000	-3.5178	3.7678
			EX	-3.91700*	1.18085	.019	-7.2645	-.5695
		KA	SH	1.01100	1.11068	.800	-2.1691	4.1911
			EN	-.12500	1.28887	1.000	-3.7678	3.5178
			EX	-4.04200*	1.19010	.016	-7.4172	-.6668
		EX	SH	5.05300*	.98328	.000	2.2643	7.8417
			EN	3.91700*	1.18085	.019	.5695	7.2645
			KA	4.04200*	1.19010	.016	.6668	7.4172

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