Effect of different surface treatments on the shear bond strength between lithium disilicate glass-ceramic and resin cement

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A Thesis Submitted in Partial Fulfillment of the Requirements
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ผลของการเตรียมพื้นผิวที่แตกต่างกันต่อค่ากำลังแรงยึดเฉือนของลิเทียมไดซิลิเกตกลาสเซรามิกกับเรซินซีเมนต์

ร.อ.หญิงสิริพรรณ สิมะเศรษฐ์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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สิริพรรณ สิมะเศรษฐ์ : ผลของการเตรียมพื้นผิวที่แตกต่างกันต่อค่ากำลังแรงยึดเฉือนของลิเทียมไดซิลิเกตกลาสเซรามิกกับเรซินซีเมนต์. (Effect of different surface treatments on the shear bond strength between lithium disilicate glass-ceramic and resin cement) อ.ที่ปรึกษาหลัก : รศ. ทพ.ดร.นิยม ธำรงค์อนันต์สกุลD.D.S., M.Sc., Ph.D.

วัตถุประสงค์: การศึกษานี้มีวัตถุประสงค์เพื่อประเมินค่ากำลังแรงยึดเฉือนของลิเทียมไดซิลิเกตกลาสเซรามิกกับเรซินซีเมนต์ ด้วยการเตรียมพื้นผิวที่แตกต่างกัน *อุปกรณ์และวิธีดำเนินการวิจัย:* ชิ้นงานลิเทียมไดซิลิเกตกลาสเซรามิก (Vintage LD Press®) ได้นำลงบล็อคและเตรียมพื้นผิวด้วยกรดไฮโดรฟลูออริก ความเข้มข้น 4.5 % และแบ่งออกเป็น 7 กลุ่มแบบสุ่ม (n=10) ตามการเตรียมพื้นผิวดังนี้ 1) ไม่มีการทาสาร (กลุ่มควบคุม) 2) Silane Primer (KS) 3) Signum® Ceramic Bond I (SGI) 4) Signum® Ceramic Bond II (SGI/SGII) 5) experimental silane (EXP) 6) experimental silane/Signum® Ceramic Bond II (EXP/SGII) และ 7) Experimental/Adper™ Scotchbond Multi-purpose Adhesive (EXP/ADP) จากนั้นชิ้นงานทั้งหมดนำไปผ่านกระบวนการเทอร์โมไซคลิง (Thermal cycling test) โดยแช่น้ำสลับที่อุณหภูมิ 5 และ 55 องศาเซลเซียส และนำมาวัดค่ากำลังแรงยึดเฉือน ค่ากำลังแรงยึดเฉือนที่ได้ นำมาคำนวณสถิติวิเคราะห์ความแปรปรวนแบบเวลช์อโนวา (Welch ANOVA) และเปรียบเทียบความแตกต่างระหว่างกลุ่มชนิดเกมส์โฮเวลล์ (Games-Howell post-hoc test) ที่ระดับความเชื่อมั่นร้อยละ 95 และนำชิ้นงานที่ผ่านการวัดค่ากำลังแรงยึดเฉือนมาสังเกตรูปแบบความล้มเหลวด้วยกล้องจุลทรรศน์ชนิดสเตอริโอที่กำลังขยาย 40 เท่า *ผลการศึกษา:* กลุ่ม EXP/ADP มีค่าเฉลี่ยกำลังแรงยึดเฉือนสูงที่สุด (45.49±3.37 MPa) แต่ไม่มีความแตกต่างจากกลุ่ม EXP/SGII (41.38±2.17 MPa) อย่างมีนัยสำคัญทางสถิติ ($p \geq 0.05$) กลุ่มที่ไม่มีการทาสาร มีค่าเฉลี่ยกำลังแรงยึดเฉือนที่ต่ำที่สุด (18.36±0.69 MPa) แต่ไม่มีความแตกต่างจากกลุ่ม KS (20.17±1.10 MPa) อย่างมีนัยสำคัญทางสถิติ ($p \geq 0.05$) *สรุปผลการศึกษา:* การเตรียมพื้นผิวที่แตกต่างกัน มีผลต่อกำลังแรงยึดเฉือนของลิเทียมไดซิลิเกตกลาสเซรามิกกับเรซินซีเมนต์อย่างมีนัยสำคัญ และการเตรียมพื้นผิวด้วยไซเลนที่ไม่มีสารเจือปน ร่วมกับการใช้และไม่ใช้สารยึดติด ช่วยเพิ่มค่ากำลังแรงยึดเฉือนและเพิ่มคุณภาพในการยึด

สาขาวิชา ทันตกรรมประดิษฐ์ ลายมือชื่อนิสิต ....
ปีการศึกษา 2563 ลายมือชื่อ อ.ที่ปรึกษาหลัก ....

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KEYWORD: Lithium disilicate, Shear bond strength, Silane

Siripan Simasetha : Effect of different surface treatments on the shear bond strength between lithium disilicate glass-ceramic and resin cement . Advisor: Assoc. Prof. NIYOM THAMRONGANANSKUL, D.D.S., M.Sc., Ph.D.

Objective: The study aimed to evaluate the shear bond strength (SBS) of lithium disilicate glass-ceramic and resin cement using different surface treatments. *Methods:* Lithium disilicate blocks (Vintage LD Press ®) were prepared, etched with 4.5 % hydrofluoric acid, and randomly divided into seven groups (n=10), depending on the surface treatments: 1) no surface treatment (control), 2) Silane Primer (KS), 3) Signum® Ceramic Bond I (SGI), 4) Signum® Ceramic Bond II (SGI/SGII), 5) experimental silane (EXP), 6) experimental-silane/Signum® Ceramic Bond II (EXP/SGII), and 7) Experimental/Adper™ Scotchbond Multi-purpose Adhesive (EXP/ADP). The specimens were cemented to resin composite blocks with resin cement and stored in water at 37°C for 24 hours. The specimens underwent 5,000 thermal cycles and were subjected to the SBS test. Data were analyzed with Welch ANOVA and Games-Howell post-hoc tests (α=0.05). Mode of failure was evaluated under the Stereo Microscope. *Results:* The highest mean SBS showed in group EXP/ADP (45.49±3.37 MPa), however this was not significantly different from group EXP/SGII (41.38±2.17 MPa) ($p \geq 0.05$). The lowest SBS was shown in the control group (18.36±0.69 MPa). This was not significantly different from group KS (20.17±1.10 MPa) ($p \geq 0.05$). *Conclusion:* The different surface treatments significantly affected the SBS value between lithium disilicate and resin cement. The application of pure silane coupling agent with or without the application of an adhesive improved the shear bond strength value and bond quality.

Field of Study:	Prosthodontics	Student's Signature ....
Academic Year:	2020	Advisor's Signature ....

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Siripan Simasetha

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INTRODUCTION

Background and Rationale

Since dental materials have improved, patients tend to demand for esthetics and metal-free restorations. Therefore, the clinical application of all-ceramic restoration is widely use nowadays. (1) All-ceramic material can display esthetic ability to mimic the tooth color. The successful clinical application of all-ceramic material depends on the clinicians' ability to make the appropriate treatment plans and carefully choose the appropriate material and cementation protocol that fulfill the patients' needs and expectations. (2)

Lithium disilicate restorations are usually monolithic in which the full contour of the prosthesis is fabricated from a homogeneous single material. It has translucency, opalescence and light diffusion that can be stained, glazed or cut back to layer veneering porcelain to enhance incisal characterization. Because of its high strength 360 to 460 MPa, lithium disilicate can be used for inlays, onlays, three-quarter crowns, full-coverage crowns, and short-span bridges in the anterior region. (3)

The clinical service outcome of lithium disilicate restoration largely depends on the resin-ceramic bond. The strong and substantial resin bonding increases retention, (4, 5) improves marginal adaptation, (6, 7) reduces bacterial microleakage, (7, 8) and improves fracture resistance. (9) The resin-ceramic bond can be generated through micromechanical retention, chemical bonding to a silica-based ceramic surface and surface wettability. (10) To gain micromechanical retention, the surface can be prepared by airborne particle abrasion and/or etching with hydrofluoric acid. But airborne particle abrasion is not suggested as a result of a significant reduction in the flexural strength of lithium disilicate. (11) Hydrofluoric acid etching dissolves the glass phase from the matrix and creates microporosity and increases the surface areas. (12) Chemical bonding between the resin-ceramic surfaces can be accomplished by using a silane coupling agent. The silane coupling agent is a bifunctional molecule that encourages adhesion through the covalent bonds with hydroxyl (OH) groups on the ceramic surface. One functional group can react with the inorganic ceramic surface and the other methacrylate group is capable of reacting with an organic resin matrix.

Silane bonds to Si-OH on a ceramic surface by condensation reaction and the methyl methacrylate double bonds provide bonding to the adhesive. When the adequate Si-OH sites on the ceramic surface, satisfy bonding will be achieved. (12) A thin silane application on the ceramic surface should be made and followed by the protocol of the silane manufactures. The application of unfilled resin/adhesive before cementation with resin cement will enhance infiltration to the superficial irregularities of etched lithium disilicate glass-ceramic resulting in increased bond strength. (13) Furthermore, the resin cement plays an important role on the bond to high crystalline content ceramics and the adhesion between dental ceramics and resin cement is the result of a physicochemical interaction across the interface between resin and ceramic.

Therefore, the purpose of this study was to evaluate the shear bond strength of lithium disilicate glass-ceramic and resin cement using different surface treatments.

Research Question

Would the different surface treatments have an effect on shear bond strength of lithium disilicate glass-ceramic and resin cement?

Research Objective

To evaluate the shear bond strength of lithium disilicate glass-ceramic and resin cement using different surface treatments.

Research Hypotheses

$H1_0$: There would be no difference to be found in shear bond strength (SBS) between lithium disilicate glass-ceramic and resin cement using different silane surface treatments.

$H1_1$: There would be a difference to be found in shear bond strength (SBS) between lithium disilicate glass-ceramic and resin cement using different silane surface treatments.

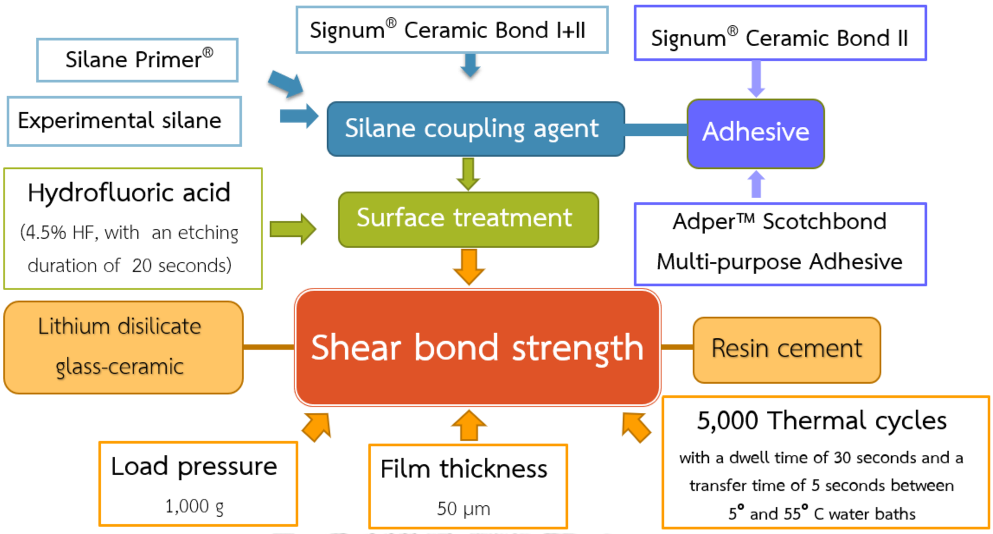
$H2_0$: There would be no difference to be found in shear bond strength (SBS) between lithium disilicate glass-ceramic and resin cement using different silanes followed by different adhesives.

$H2_1$: There would be a difference to be found in shear bond strength (SBS) between lithium disilicate glass-ceramic and resin cement using different silanes followed by different adhesives.

Proposed Benefits

To provide recommendations to the clinicians for achieving optimal shear bond strength between lithium disilicate glass-ceramic and resin cement using silane and adhesive.

Conceptual framework



Chapter II

LITERATURE REVIEW

Presently, all ceramic restorations are widely used because of the patients' demand of esthetics and biocompatibility. All-ceramic restorations have improved translucency, esthetics, wear resistance, color stability and structural durability to fabricate inlay, onlay, anterior to posterior crowns and bridges. (1, 3, 14) There are several ceramic classification based on its composition, fabrication techniques, clinical applications, microstructures, temperature of fusion, and translucency. (15) The clinical success of all-ceramic material depends on the clinicians' ability to select the appropriate material and cementation technique to fulfill the patients' expectations.

1. Classification of all-ceramic restorations

All-ceramic restorations can be classified by their compositions into 3 main types as follows

1.1 Silica-based ceramic

Silica-based ceramic that can be subdivided into 3 types

1.1.1 Feldspathic porcelain

It is produced from the mixture of potassium feldspar, quartz, and kaolin. It has high translucency, brittleness, low fracture resistance that is used as veneering porcelain in metal ceramic and all-ceramic restorations. (16, 17)

1.1.2 Leucite-based ceramic

It incorporated leucite crystal in the material to increase flexural strength and fracture resistance by preventing crack propagation. (18) The available leucite reinforced glass-ceramic is IPS Empress® (Ivoclar Vivadent, Schaan, Liechtenstein), which consists of silicon dioxide, aluminum oxide, and leucite crystals. It is recommended for inlays, onlays, veneers and crowns. (19) The flexural strength is approximately 160 MPa.

1.1.3 Lithium disilicate-based ceramic

It consisted of lithium disilicate crystals ($Li_2Si_2O_5$), which was first developed as IPS Empress II® (Ivoclar Vivadent, Schaan, Liechtenstein). IPS Empress II® composed of 65 vol% lithium disilicate crystal needle-shaped crystals (3–6 μm × 0.8 μm) embedded in a glass matrix, with a 1 vol% porosity, (20) using the lost-wax technique. Once a wax pattern is fabricated, it will be invested in the special investment material and burned out to leave a mold for melted ingot to be pressed and flow in. In order to gain the optical characteristics of natural teeth, the cores are lately veneered with a very translucent fluorapatite ceramic. It contained 19–23% of fluorapatite crystals ($Ca_5(PO_4)_3F$) embedded in a glassy matrix. The flexural strength is 360 MPa which is more than IPS Empress®. Later in 2005, Ivoclar Vivadent introduced IPS e.max® Press using the lost-wax/heat-pressing technique. Its flexural strength is 360–460 MPa and its fracture toughness (KIC) is 2.8–3.5 $MPam^2$. The high mechanical performance of this material as a result of a layered, tightly interlocked distribution of the elongated disilicate crystals, impeded crack propagation across the planes and incompatibility between the thermal expansion coefficients of lithium disilicate crystals and the glassy matrix, induces a tangential, compressive stress around the crystals. (21) IPS e.max® Press composed of a refined size of lithium disilicate crystals to improve physical properties and translucency acquired through a different firing process. (22) Because it has acceptable translucency and esthetics, it is recommended to use as inlays, onlays, three-quarter crowns, and full-coverage crowns in anterior and posterior regions. (23) Lithium disilicate-based ceramic has higher flexural strength, abrasive resistance, and chemical durability than feldspathic ceramic. It is indicated for onlays, three-quarter crowns, anterior fixed partial denture prosthesis. (3) Because the high strength and toughness of IPS e.max® Press, it can be used as monolithic restorations that have higher fatigue resistance than bilayered restorations. The Ivoclar Vivadent has developed the CAD/CAM technology, IPS e.max® CAD which is suitable for chairside fabrication. IPS e.max® CAD is different from IPS e.max® Press because it exhibits 2 phases of crystallization. At the firing phase it forms into 60%, by weight, lithium metasilicate crystals and a 40%, by weight, lithium disilicate, which appears in blue color. At this phase, the flexural strength is in between 130-150 MPa which promotes higher cutting efficiency, easier and faster working time and lower wear on the milling tools. (14, 20, 21) The final crystallization phase temperature is at 850°C. The flexural strength can be raised up to 417 MPa. (24) Due to its satisfaction strength it can be used as posterior monolithic crown.(25) However, because it has more crystalline phase which make IPS Empress®, It has lower opacity when compared to IPS e.max® CAD. (26) The available lithium disilicate glass-ceramics apart from IPS Empress II® and IPS e.max® Press/CAD are as follows

- Vintage LD Press® (Shofu Inc., Kyoto, Japan) in form of ingot fabricated with heat press technique which comes along with Vintage LD® in form of powder used for layering and cut-back technique and in case that the clinician would like to modify internal and external characterizations, Vintage ART LF® will be utilized.
- GC Initial® LiSi Press (GC America Inc., Alsip, Illinois, U.S.A.) comes in form of the ingot for the heat-pressing technique and has GC Initial® LiSi as a veneering feldspathic porcelain.
- Rosetta SP® (HASS Corp., Gyeonggi-do, Korea) comes in form of ingot for the heat-pressing technique and Rosetta SM® for CAD/CAM system.

Microstructure of Lithium disilicate-based ceramics

IPS e.max® Press

IPS e.max® Press has 70% of lithium disilicate crystal ($Li_2Si_2O_5$) which appears as needle-like crystals that incorporated in glassy matrix. The length of lithium disilicate crystal is approximately 3-6 μm.

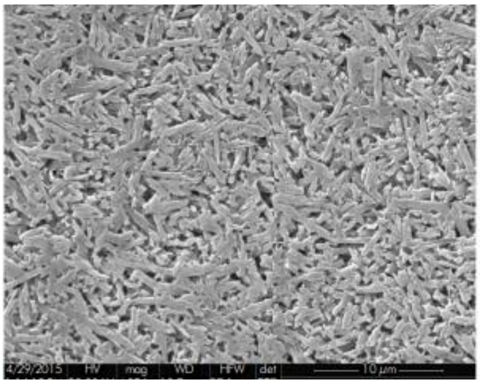


Figure 1 SEM of Etched IPS e.max® Press with 5% Hydrofluoric acid
for 20 seconds at 4026x magnification
Modified picture from Sundfeld et al (2018) (27)

Vintage LD Press®

Ohashi et al (2017) (28) reported density of Vintage LD® Press compared with IPS e.max® Press and GC Initial® LiSi Press that Vintage LD Press® lithium disilicate crystal were thinly distributed than that of GC Intial® LiSi Press and IPS e.max® Press. The Vintage LD Press® lithium silicate crystal size is approximately 1-3 μm. This study reported that IPS e.max® Press lithium disilicate crystal length is approximately
1-4 μm.

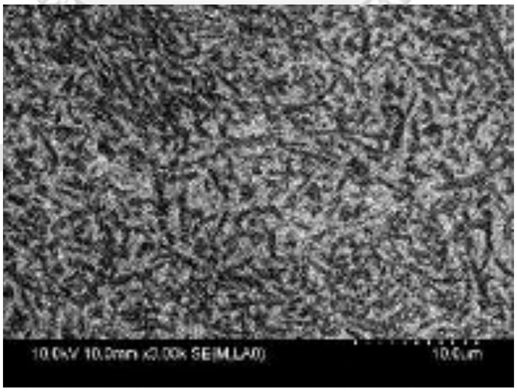


Figure 2 SEM of Vintage LD Press® at 3000X magnification
Modified picture from Ohashi et al (2017) (28)

GC Initial® LiSi Press

Due to several lithium disilicate glass-ceramic being new, the availability of studies in markets is limited, according to Ohashi et al (2017) (28), the lithium disilicate crystal of GC Initial® LiSi Press is approximately 1-1.5 µm but the density of lithium disilicate crystal is not much different from that of IPS e.max® Press.

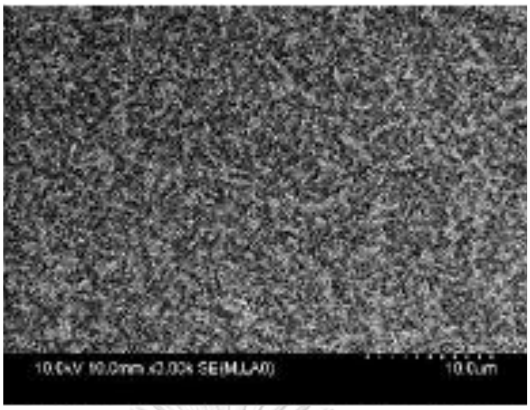


Figure 3 SEM of GC Initial® LiSi Press at 3000x magnification
Modified picture from Ohashi et al (2017) (28)

1.2 Alumina-based ceramic

Alimina-based ceramic consisted of dense particle of Aluminium oxide (Al_2O_3) or Zirconium oxide (ZrO_2) and a few of silica. It is indicated for crowns and bridge. (29, 30) The examples of alumina-based ceramic are Procera® AllCeram alumina (Nobel Biocare AB, Goteborg, Sweden), In-Ceram Alumina, and In-Ceram Zirconia (VITA, Zahnfabrik, Germany).

1.3 Zirconia-based ceramic

Zirconia-based ceramic has zirconia as the main component and a few silica. Due to its structural durability and high flexural strength, it can be used as posterior bridges. (29) The examples of zirconia-based ceramic are IPS e.max® ZirCAD (Ivoclar Vivadent, Schaan, Liechtenstein) and Cercon® (Dentsply Ceramo, York, Pennsylvania, U.S.A.).

2. Surface treatment of lithium disilicate glass-ceramic

There are several materials for lithium disilicate surface treatment as follows

Hydrofluoric acid

Hydrofluoric acid removed glassy matrix and exposed lithium disilicate crystals. (13, 31) The chemical process of hydrofluoric acid and lithium disilicate occurs when lithium disilicate is treated with hydrofluoric acid, tetrafluorosilane (SiF_4) is formed and then reacts with hydrofluoric acid and forms hydrofluorosilicic acid (H_2SiF_6). The glassy matrix is dissolved and rinsed away. This creates microporosity and a high free energy surface state resulting in increased ceramic surface area with enhanced micromechanical retention between resin cement and ceramic interface. (12) It also elevates bond strength and bond durability of ceramic and resin cement. (13, 32-34) The etched ceramic surface has a low contact angle and high wettability. Additionally, acid etching exposes hydroxyl groups on the ceramic surface resulting in increased chemical adhesion to silane coupling agent. Hydrofluoric acid is hazardous liquid. Therefore, a clinician should select an appropriate hydrofluoric acid concentration and duration of etching for optimum bonding and carefully use with the patients. The recommended concentration of hydrofluoric acid is 5 % with 20 seconds of etching duration. Higher concentrations and etching durations are not necessary. (35) Etching with hydrofluoric acid and applying silane showed the best bond strength compared to the other methods, such as sandblasting, lasers, or roughening with diamond bur. (36-40) The application of unfilled resin before cementation with resin cement will enhance infiltration to the superficial irregularities of etched lithium disilicate glass-ceramic resulting in increased bond strength. (13)

Air-particle abrasion or sandblasting

Air-particle abrasion or sandblasting with particles of aluminum oxide are inferior when compared to etching, with or without the use of silane coupling agent. (37, 39, 41) Menees et al (2014) (11) reported that using aluminum oxide particle on IPS e.max® CAD at pressure higher than 55 kPa shown significant lower flexural resistance. The previous study stated that when there is no hydrofluoric acid, sandblasting can be considered as an alternative surface treatment. (42)

Silane coupling agent

The silane coupling agent is silicon-based synthetic chemical that contains inorganic and organic molecules. A typical general structure is $(RO)_3SiCX\text{-}Y$, where RO is hydrolyzed group, such as methoxy, ethoxy, or acetoxy. X is the carbon backbone and Y is an organofunctional group such as amino, methacryloxy and epoxy. The silane coupling agent acts at the interface between inorganic and organic materials. The silane coupling agent bonds to resin cement, which is considered as an organic material with the covalent bond. It also bonds to silica-based ceramic, which is considered as an inorganic material with a hydrogen bond. The silane coupling agent can be applied on silica-based restorative materials, metallic restorations, (43, 44) ceramic restoration, E-glass fiber reinforced resin composites, etc. (45) Because silane coupling agents create a water resistant bond at the interface between inorganic fillers and organic materials resulting in a good adhesion and provide bonding stability. Therefore, application of silane in glass ceramic is a standard procedure. (34) The most commonly used silane in dentistry is ϒ-methacryloxypropyl trimethoxysilane (ϒ-MPTS or 3-MPS), which is producedin pre-hydrolyzed single-bottle system and later developed into a two-component silane system to prolong the service life. The pre-hydrolyzed single-bottle system or pre-activated system consists of ethanol, pre-hydrolyzed silane, and water. The pre-hydrolyzed single-bottle system has higher hydrolysis rate than the two-component silane system. (12) The two-component silane system consists of unhydrolyzed silane monomer dissolved in ethanol and aqueous acetic acid solution. The hydrolysis reaction occurs when unhydrolyzed silane and aqueous acetic acid solution are combined. The aqueous acetic acid solution hydrolyzed ester which is on one end of silane to silanol group (Si-OH). The silanol group reacts with hydroxyl group in silicon dioxide of ceramic as a condensation polymerization to form siloxane bond and on the other end of silane reacts with double carbon bond of resin cement as additional polymerization that forms monomers and dimers that later on will have siloxane bonds from condensation polymerization forming a molecule of oligomer and polymer compounds. The example of the two-component silane system is BIS-SILANE (Bisco Dental Products, Schaumburg, Illinois, U.S.A.). The molecules size will be determined by the types of solvent, temperature and acid-base condition. (12) The small molecules of monomer are well adsorbed on ceramic surface resulting in increased in bonding ability of resin cement and ceramic. The small molecules of monomer will be formed during the freshly mixed process. Using of heated air blowing can remove excess silane coupling agent and oligomer compound of the pre-hydrolyzed single-bottom system, (46) and does not give much different bond strength from the two-component silane system. Adhesion mechanism of resin and silane can be explained by 2 processes. Primarily, acid activates silane and substrate to form silanol group that reacts with hydroxyl group on substrate surface by a condensation reaction. The resin and silane adhesion formation are activated by light-curing unit. The adhesion capability depends on the materials. The materials like silica, glass, and quartz can form strong siloxane (Si-O-Si), which create strong adhesion. Pure metal and metal alloys create mild adhesion. Amaral et al (2006) (47) suggested usage of appropriate silane protocol to assure the success of bond strength and longevity of dental restoration. As per Anusavice (2013), (48) the silane coupling agent increases the surface energy of ceramic substrates and improves wettability of adhesive and cement. When silane is applied on the ceramic surface and dried, the interphase layer is generated. The interface layer of silane coupling agent is needless for the bonding process. Thus taking away of the outermost layer of the silane film and preserving the most stable and chemisorbed layer on the ceramic surface will improve the bond strength. (49) Aboudzadeh et al (2007) (50) prepared hydrolyzed silane by adding 2% by volume of 3-MPS to a mixture 70% ethanol and 30% distilled water by weight. The pH of the solution was adjusted to 4.5-5.5 by adding a few drops of acetic acid. For this technique, the Si-OR groups of silanes were transformed to activate Si-OH groups. In this study, the author followed Aboudzadeh et al (2007) (50) hydrolyzed silane preparation method.

Universal adhesives

Universal adhesives are simplified adhesives in a single bottle, suitable for different substrates such as dentin, enamel, resins, alloys, and ceramics. (51) Universal adhesives contain several ingredients such as phosphate monomers called 10-methacryloxydecyl dihydrogen phosphate (10-MDP), bisphenol A glycidyl methacrylate (Bis-GMA), hydroxyethyl methacrylate (HEMA), silane coupling agent, etc. These monomers are responsible for the adhesive capacity of these adhesives between the ceramic, polymeric and dental substrates. (40) Adding of 10-MDP in silane coupling agent decreases bond strength (52) and has high susceptibility to hydrolytic degradation (12) comparing to application of silane followed by this type of adhesive. (33) Its high acidity causes the silane coupling agent to have low stability and have a condensation reaction in between its silanol group. A large molecule of Bis-GMA in the universal adhesive may inhibit condensation polymerization of hydroxyl group and silanol group. (53)

Tribochemical silica coating

Tribochemical silica coating uses the CoJet™ system for the chair-side technique and the Rocatec® system (3M-ESPE, St. Paul, MN, U.S.A.) for the laboratory base system, which are considered as a cold silicatisation method. The silicatisation process transferred the object material as kinetic energy that generates local frictional heat at the impact area with no changes of temperature. The CoJet™ system is based on airborne micro-blasting sand, which is especially silica-modified aluminum trioxide size 30 µm and provides the ceramics with a reactive silica-rich outer surface inclined to silanization. The indication of CoJet™ is to repair ceramic, composite, and metal restoration with composite. The Rocatec® system, the surface to be coated is cleaned and roughened by blasting with high-purity aluminium oxide sand 110 µm; Rocatec® Pre and followed by tribochemical coating of the microblasted surface with silica-modified aluminium oxide. The aluminium oxide sand is coated with a thin layer of SiO_2 which the sand particle for Rocatec® Plus is 110 µm and for Rocatec® Soft is 30 µm. Neis et al (2015) (54) reported that in feldspathic porcelain a tribochemical process was the only tested surface treatment that show lower values of tensile bond strength.

Lasers

The Nd:YAG, Er:YAG, and a femtosecond laser consisted of a titanium system. The systems composed of neodymium and erbium were used for increasing the roughness of the ceramic material. Gökçe et al (2007) (55) reported that low power Er: YAG laser can be used as an alternative surface treatment for ceramic but the chairside use of a laser may not be as practical as etching with hydrofluoric acid.

3. Resin cement

Resin cement is widely used in the present because resin-ceramic interface affects the survival outcome of the restoration. The required properties of luting material is that, it should have high bonding ability to the tooth and restoration, high mechanical properties, marginal sealability, biocompatibility to the oral tissues, appropriate dissolution resistance in an intraoral condition, adequate compressive and tensile strengths under masticatory function, (56) radiopacity, and color stability under the oral fluids. And the removal of excess resin cement should be easy. (57) The strong and substantial resin bonding increases retention, (4, 5) improves marginal adaptation, (6, 7) reduces bacterial microleakage, (7, 8) and improves fracture resistance. (9) The resin-ceramic bond can be generated through micromechanical retention, chemical bonding to a silica-based ceramic surface, and surface wettability. (10, 58) As per Sundfeld et al (2018) (27) resin cement enhances mechanical and chemical bonding of silica-based ceramic to the tooth. Resin cement composed of a resin matrix and filler which are combined together with organo-silane coupling agent. Resin matrix is methacrylate-based materials which can be Bis-GMA (Bisphenol-A glycidyl dimethacrylate), TEGDMA (Triethyleneglycoldimethacrylate), and UDMA (Urethane dimethacrylate). The resin matrix consists of a high molecular weight of diacrylate monomer which make it has a high viscosity. Therefore, adding a low molecular weight dimethacrylate will decrease resin cement viscosity. The filler contents such as silica and glass particles are added to increase mechanical properties and wear resistance. The amount of filler will affect viscosity, flowability and thickness of resin cement.(57) Resin cement can be used as a luting material in metal, porcelain fused to metal, ceramic and resin composite restorations which can be inlays, onlays, veneers, crowns and fixed partial denture prostheses. Resin cement can be classified by polymerization mechanism, adhesive system, etc. It can be classified by polymerization mechanism into 3 types, chemical-cure resin cement which has tertiary amine and benzoyl peroxide compounds mix together. The tertiary amine compound activates a peroxide compound, which will form free radicals to break the carbon double bond (C=C) at the terminal of dimethacrylate monomer, forming a crosslink of the carbon single bond (C-C).The advantage of this resin cement is, it is suitable for the area that light could not be transmitted. (59) But it has low color stability and short working time. Light-cure resin cement, it has photo-initiator to activate polymerization reaction. The photo-initiator such as camphorquinone which will be activated to the excited stage and then reacted with tertiary amine compound to form free radicals to break carbon double bond (C=C) at the terminal of dimethacrylate monomer to form a short polymerization chain of single carbon bond (C-C) which later will form a long polymerization chain. This type of resin cement has long working time and color stability. Dual-cure resin cement, which composes of self-cure initiator and photo-initiator. The chemical reaction occurs between benzoyl peroxide and tertiary amine compounds. Phenolic compound uses as an inhibitor to slow the polymerization reaction. When it is exposed to the light, camphorquinone will initiate the polymerization reaction. (60) This type of resin cement can be applied to metal, porcelain fused to metal and all-ceramic restoration. (61) It allows sufficient degree of monomer conversion underneath the restoration and promote high mechanical properties than chemical-cure resin cement. (62) According to resin cement adhesive system classification, total-etch adhesive resin cement procedure is complicated and if the moisture control and dentin wettability are not properly manipulated, the bond strength will be decreased. (63) Later on self-etch adhesive resin cement was introduced to prohibit collagen collapse in the dentin during acid etching. Nevertheless, when hardening is retarded, moisture solubility along adhesive can cause decreasing bond strength. (64) Thus, self-adhesive resin cement that do not require surface pretreatment was developed. The self-adhesive resin cement is composed of resin matrix, filler, and acidic monomer will dissolve inorganic matrix and form chemical bond to the tooth. Acidity of functional monomer should not be too high to make it have hydrophilicity after polymerization. (65)

Chapter III

RESEARCH METHODOLOGY

Materials

1. Lithium disilicate glass-ceramic (Vintage LD Press® ingot, Shofu Inc., Kyoto, Japan)
2. 4.5% Hydrofluoric acid (IPS-Ceramic Etching Gel®, Ivoclar Vivadent, Schaan, Liechtenstein)
3. Silane coupling agent (Silane Primer, Kerr Corporation, Orange, CA, U.S.A.)
4. Silane coupling agent (Signum® Ceramic Bond I+II, Kulzer, Hanau, Germany)
5. Silane coupling agent (Sigma-Aldrich, St. Louis, MO, U.S.A.)
6. Adhesive (Adper™ Scotchbond Multi-purpose Adhesive, 3M-ESPE St. Paul, MN, U.S.A.)
7. Resin cement (RelyX™ U200 Self-Adhesive Resin cement, 3M-ESPE St. Paul, MN, U.S.A.)
8. Resin composite (Filtek™ Z350 XT Universal Restorative System, 3M-ESPE St. Paul, MN, U.S.A.)
9. Self-curing acrylic resin (Ortho-Jet™, Lang, Illinois, U.S.A.)

Equipment

1. Silicon carbide paper 320, 600 and 800 grit (3M Wetordry abrasive sheet, 3M-ESPE St. Paul, MN, U.S.A.)
2. Polishing Machine (Nano 2000 grinder-polisher with FEMTO 1000 polishing head, Pace Technologies, Arizona, U.S.A.)
3. PVC tube 20 mm diameter
4. Ultrasonic cleaner (VGT-1990 QTD, Guangzhou, Guangdong, China)
5. Adhesive tape (Scotch 3M Tape, 3M-ESPE St. Paul, MN, U.S.A.)
6. Polyvinyl siloxane impression material (Elite HD, Zhermack, Badia Polesine, Italy)
7. Light curing unit (Bluephase N®, Ivoclar Vivadent, Schaan, Liechtenstein)
8. Incubator (Contherm 160 M, Contherm Scientific Ltd., Wellington, New Zealand)
9. Thermo Cycling Unit (King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand)
10. Universal Testing Machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan)
11. Stereo Microscope (SZ61, Olympus Corporation, Tokyo, Japan)
12. ImageJ Image Processing and Analysis in Java (The National Institutes of Health and The Laboratory for Optical and Computational Instrumentation, WI, U.S.A.)

Table 1 Trade names and compositions of experimental materials used in this study

Trade names	Manufacturers	Compositions	Lot number
Vintage LD Press® (Lithium disilicate)	Shofu Inc, Kyoto, Japan	Lithium disilicate-based ceramic	021601
IPS-Ceramic Etching Gel® (4.5% Hydrofluoric acid)	Ivoclar Vivadent, Schaan, Liechtenstein.	Aqueous solution of hydrofluoric acid	Y34242
Silane Primer (Silane)	Kerr Corporation, Orange, CA, USA.	3-MPS, BisEMA, TEGDMA, and ethanol	6825763
Signum® Ceramic Bond I+II (Silane)	Kulzer,Hanau, Germany.	Signum® Ceramic Bond I: Isopropanol, acetone, silane, acids, monomer, initiators, and stabilizers Signum® Ceramic Bond II: Silane, initiators, stabilizers, monomers, and silicic acid	K010112 K010711
Experimental silane	- Sigma-Aldrich, St. Louis, MO, USA	Alcohol, distilled water, and acetic acid 3-MPS	SHBJ3136
Adper™ Scotchbond Multi-purpose Adhesive (Adhesive)	3M ESPE, St. Paul, MN, USA.	Bis-GMA, HEMA, and initiators	N979519
RelyX™ U200 (Self-adhesive and dual-cure resin cement)	3M ESPE, St. Paul, MN, USA.	Base: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, and rheological additives Catalyst: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, rheological additives, and pigments	4819681
Filtek^{TM} Z350 XT Universal Restorative (Resin composite)	3M ESPE, St. Paul, MN, USA.	**Organic matrix:** BisGMA, UDMA, BisEMA, and TEGDMA **Inorganic particle:** Non-agglomerated nanoparticles of silica and nanoagglomerates formed of zirconium/silica particles	N912324

Abbreviations: BisEMA: Ethoxylated bisphenol-A dimethacrylate; TEGDMA: Triethyleneglycoldimethacrylate; 3-MPS: 3-(Trimethoxysilyl)propyl methacrylate; BisGMA: Bisphenol-A glycidyl dimethacrylate; HEMA: hydroxyethyl methacrylate; UDMA: Urethane dimethacrylate. All information supplied by manufacturers

Methods

Sample size calculation

The pilot study, the shear bond strength test was measured from lithium disilicate glass-ceramic and resin cement using different surface treatments. The G*Power 3.1.9.2. was used to calculate the sample sizes, with a significance level (α=0.05), power (0.95), means and standard deviations of each of the groups are, group 1 (8.64±2.27 MPa), group 2 (7.51±3.09 MPa), group 3 (22.04±3.16 MPa), group 4 (21.45±3.43 MPa), group 5 (8.38±2.69 MPa), and group 6 (24.79±4.96 MPa). The calculated results showed 2 samples for each experimental group. Therefore, the author preferred to set 10 samples in each experimental group for a better evaluation (n=10).

Specimen Preparation

Lithium disilicate specimen preparation

Lithium disilicate glass-ceramic (Vintage LD Press® ceramic ingots, Shofu Inc., Kyoto, Japan) were pressed into the circular molds with 5mm diameter and 3 mm length in an automatic press furnace (PANAMAT® 640/620, DEKEMA Dental-Keramikofen GmBH, Freilassing, Germany). The specimens were observed under Stereo Microscope (SZ61, Olympus Corporation, Tokyo, Japan) at 40x magnification. The specimens were randomly divided into 7 groups (n=10).

Each specimen was embedded in a PVC block size 20 mm diameter and 25 mm length using self-curing acrylic resin (Ortho-Jet™, Lang, Illinois, U.S.A.). The upper surface was polished to a flat surface with wet polishing using 320, 600, and 800 grit silicon carbide abrasive paper (3M Wetordry abrasive sheet, 3M-ESPE St. Paul, MN, U.S.A.) with polishing machine (Nano 2000 grinder-polisher with FEMTO 1000 polishing head, Pace Technologies, Arizona, U.S.A.) with load of 2 kg/cm^2. The silicon carbide paper was rotated with speed of 100 rpm. in anti-rotation movement and the specimen were rotated in rotation movement. The polishing cycle is 2 minutes. The silicon carbide paper was changed when the polish cycle ended.

All the specimens were immersed in an ultrasonic cleaner (VGT-1990 QTD, China) for 10 minutes to remove debris, and again the specimens were observed under Stereo Microscope (SZ61, Olympus Corporation, Tokyo, Japan) at 40x magnification, then subsequently etched with 4.5% hydrofluoric acid (IPS Ceramic Etching-gel®, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds, rinsed with water and dried with air blow and again immersed in the ultrasonic cleaner for 10 minutes. The surface treatment of each experimental group is described in Table 2.

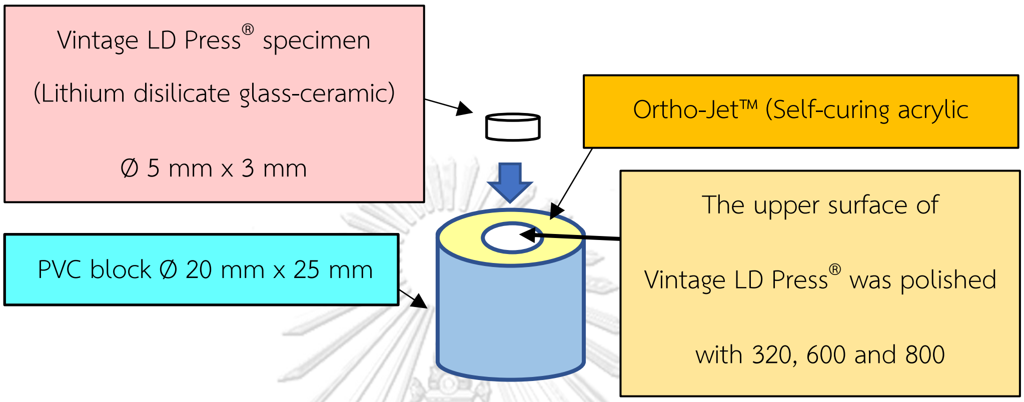


Figure 4 Lithium disilicate glass-ceramic placement in PVC block

Figure 5 An adhesive tape application to identify the bonded area

The adhesive tape (Scotch 3M Tape,3M-ESPE St. Paul, MN, U.S.A.) with a 2.38 mm diameter (ISO 29022:2013) (66) and 50 μm thickness (ISO 4049:2009) (67) was firmly attached on the upper surface of Vintage LD Press® specimen to define the area of bonding and control the film thickness of the resin cement.

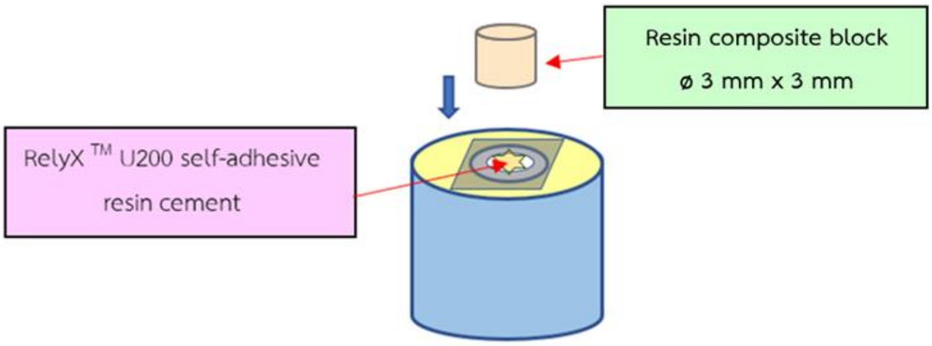


Figure 6 RelyX™ U200 resin cement and resin composite block cementation to the bonded area

Resin composite block preparation

The resin composite blocks (Filtek Z350XT,3M-ESPE, St. Paul, MN, U.S.A.) were fabricated from putty silicone mold with 3 mm diameter and 3 mm length using the light-curing unit (Bluephase N®, Ivoclar Vivadent, Schaan, Liechtenstein) with intensity of 1200 mW/cm.² as per the manufacturer's instruction.

Resin composite block cementation

The self-adhesive resin cement (RelyX™ U200, 3M-ESPE, St. Paul, MN, U.S.A.) was cemented with the Vintage LD Press® as per manufacturer's instruction by mixing of base paste and catalyst paste on the mixing pad, and placing the composite block on the treated Vintage LD Press® surface under a constant weight of 1000 g. Removed the excess cement with a new disposable micro applicator (Cotisen micro applicator dispenser, Huanghua premise dental, Huanghua, Hebei, China). The light-curing unit was used to apply on 4 joining surfaces for 40 seconds per joining surface.

Preparation of experimental silane

The experimental silane used in this study was prepared as per a previous study, (50) through mixing of a ratio of 70% ethanol and 30% distilled water in a glass container. The pH of the solution was changed to the range of 4.5-5.5 using acetic acid and measured for accuracy using a digital pH meter (Orion 420a pH, Thermo Electron Corp, Massachusetts, U.S.A.). The solution was then moved to a new plastic container where it was then mixed with a silane coupling agent: 3-Trimethoxysilyl propyl methacrylate (3-MPS) (Sigma-Aldrich, Missouri, U.S.A.). The mixing process entailed slowly adding the agent using a stirring procedure to produce a 2% concentrated solution. It was then left for 60 minutes with no actions to allow hydrolysis to take place and form the final silane mixture. At that point, a magnetic stirrer and bar (Hotplate stirrer UC152, Stuart Scientific, Staffordshire, U.K.) were used to gently mix the solution for 10 minutes.

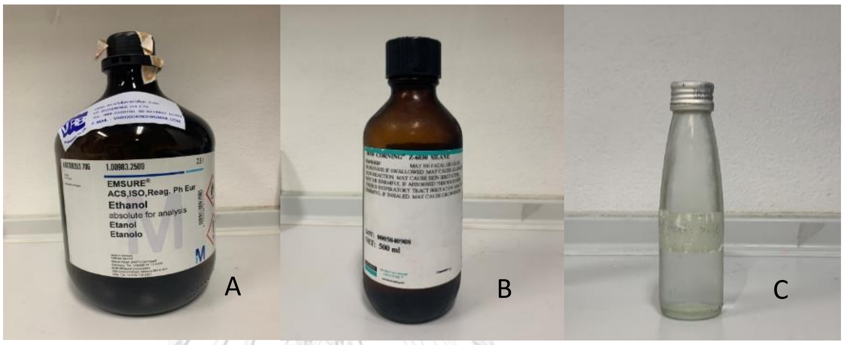


Figure 7 Materials used in experimental silane:
A) Ethanol, B) 3-MPS, and C) Acetic acid

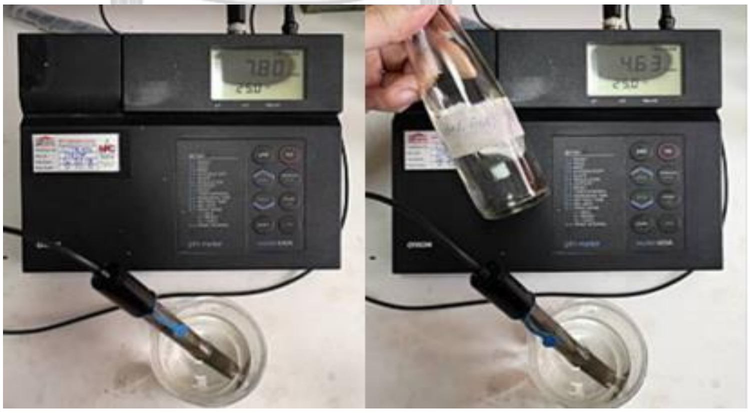


Figure 8 The pH of the solution was changed
to the range of 4.5-5.5 using acetic acid

Table 2 Demonstration of surface treatment of each experimental group

Group	Abbreviation	Surface treatments
1	HF/RXU200 (Control)	Step 1: Etching of lithium disilicate (LS) prepared block with 4.5% HF for 20 seconds, rinsed with water for 60 secs, and air-dried for 20 seconds. Step 2: No silane treatment (Control) Step 3: Application of RelyX™ U200 resin cement by mixing of base paste and catalyst paste on the mixing pad, and placing the composite block on the treated LS surface under a constant weight of 1000 g for 10 seconds. Removed the excess cement with a new disposable micro applicator. **All groups 1 - 7 follow the same procedure in step 1 and step 3.** **Step 2 was altered depending on the silane used**
2	HF/KS/ RXU200	Step 2: Surface treatment with Kerr Silane Primer. The micropipette was used to take 3 microliters of Kerr Silane Primer and placed on the etched LS surface then applied with a disposable micro applicator as a single film and left untouched for 60 seconds.
3	HF/SGI/ RXU200	Step 2: Surface treatment with Signum® Ceramic Bond I. The micropipette was used to take 3 microliters of Signum® Ceramic Bond I and placed on the etched LS surface then applied with a disposable micro applicator as a single film, left untouched for 10 seconds, and allowed it to dry.
4	HF/SGI/SGII/ RXU200	Step 2: Surface treatment with Signum® Ceramic Bond I as previously described in group 3 and followed by application of Signum® Ceramic Bond II. The micropipette was used to take 3 microliters of Signum® Ceramic Bond II on the silanized LS surface as a thin layer and rubbed it for 30 seconds. Any excess will be removed with a new disposable micro applicator.

Abbreviations: HF; 4.5% Hydrofluoric acid, LS; Lithium disilicate glass-ceramic, RXU 200; RelyX™ U 200, KS; Silane Primer, SGI; Signum® Ceramic Bond I, SGII; Signum® Ceramic Bond II, EXP; Experimental silane, ADP; Adper™ Scotchbond™ Multi-purpose Adhesive.

Group	Abbreviation	Surface treatments
5	HF/EXP/ RXU200	Step 2: Surface treatment with experimental silane. The micropipette was used to take 3 microliters of experimental silane and placed on the etched LS surface then applied with a disposable micro applicator as a single film, left untouched for 60 seconds, and allowed it to dry.
6	HF/EXP/SGII/ RXU200	Step 2: Surface treatment with experimental silane as previously described in group 5, followed by Signum® Ceramic Bond II as previously described in group 4.
7	HF/ EXP/ADP/ RXU200	Step 2: Surface treatment with experimental silane as previously described in group 5, followed by Adper™ Scotchbond Multi-purpose Adhesive. The micropipette was used to take 3 microliters of Adper™ Scotchbond Multi-purpose Adhesive and applied on the silanized LS surface then rubbed with a disposable micro applicator as a single film, left untouched for 60 seconds and allowed it to dry. Any excess will be removed with a new disposable micro applicator

Abbreviations: HF; 4.5% Hydrofluoric acid, LS; Lithium disilicate glass-ceramic, RXU 200; RelyX™ U 200, KS; Silane Primer, SGI; Signum® Ceramic Bond I, SGII; Signum® Ceramic Bond II, EXP; Experimental silane, ADP; Adper™ Scotchbond™ Multi-purpose Adhesive.

The specimens were stored in distilled water and stored in the Incubator (Contherm 160 M, Contherm Scientific Ltd., Wellington, New Zealand) under 37°C for 24 hours. The specimens underwent 5,000 thermal cycles (Thermo Cycling Unit, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand) with a 30 second dwell time and a 5 second transfer time between 5 and 55°C and subjected to the shear bond strength test.

Testing of shear bond strength

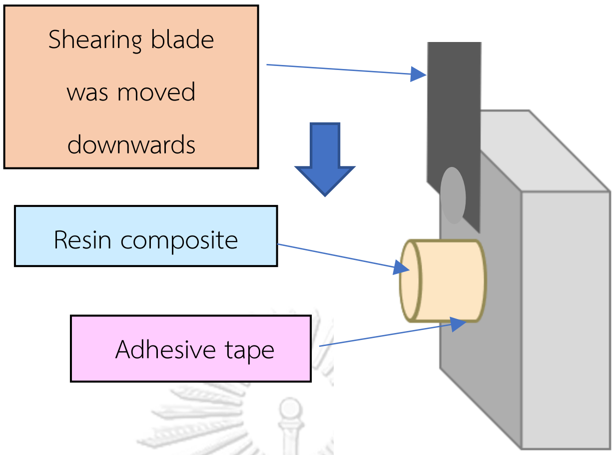


Figure 9 Notched-edge shear bond strength

All of the specimens were mounted in the universal testing machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan). The shear bond strength was tested using the notched-edge shearing blade. The notched-edge shearing blade was placed parallel to the bonding site. The crosshead speed is 1.0 mm per minute (ISO 29022:2013). (66) The shear bond strength in megapascal (MPa) was determined from the maximum force prior to the bond failure (N) divided by the bonding area (mm^2) between resin cement and lithium disilicate glass ceramic as the following formula:

$$\sigma=F/A_b$$

where σ is stress (MPa), F is force (N), and A is bonding area (mm^2)

Mode of failure evaluation

The fractured parts were evaluated with Stereo Microscope (SZ 61, Olympus, Tokyo, Japan) at 40x magnification. The mode of failure was adapted from Matinlinna et al (2011) (68) and categorized into 3 types.

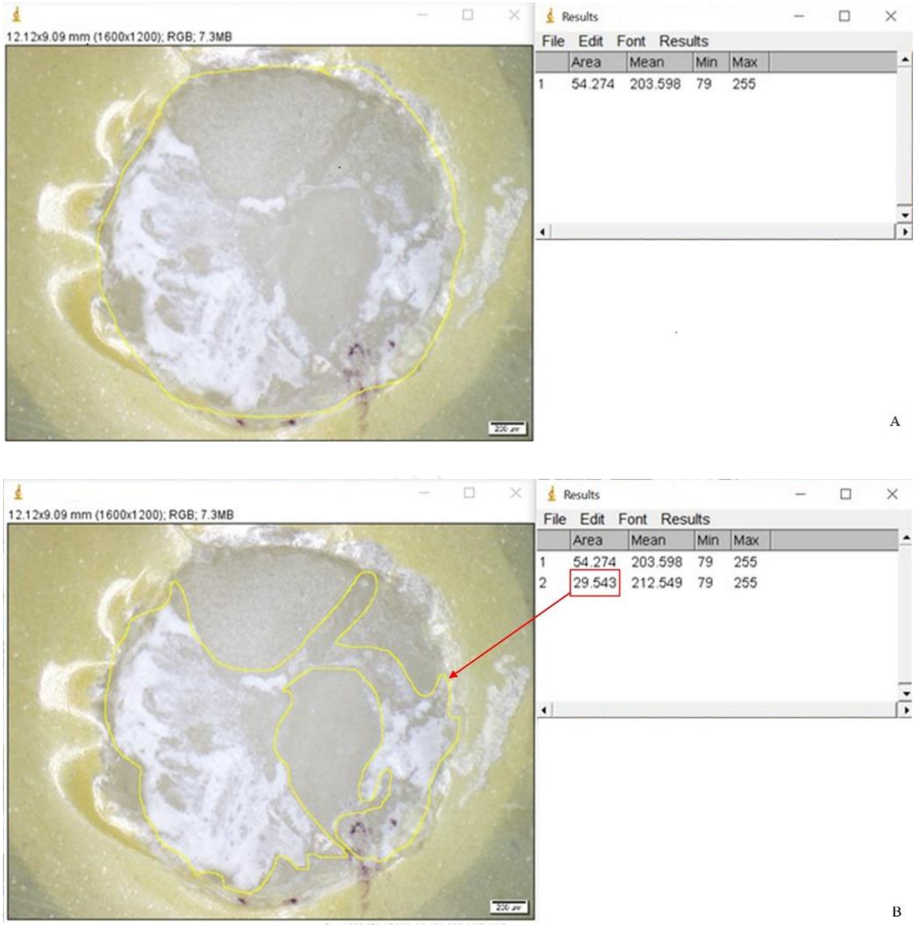
- When less than 40% of the resin cement could be observed on the surface of lithium disilicate glass-ceramic, it was categorized as an adhesive failure (AF) which means that there is no bond between lithium disilicate glass-ceramic and resin cement.

- When at least 60% of the resin cement could be observed on the surface of lithium disilicate glass-ceramic, it was categorized as a cohesive failure (CF) which means that failure occurs in the resin cement.

- When more than 40% but less than 60% of the resin cement could be observed on the surface of lithium disilicate glass-ceramic, it was categorized as a mixed failure (MF) which means that it has both adhesive and cohesive failures.

The percentage of area in mode of failure was measured using ImageJ software then calculated using the marked area of resin cement on the bonded ceramic surface divided by the total bonded area and multiplying by 100 as in the following example.

The percentage of area of the fractured specimen was measured using ImageJ software. The total bonded area measured was 54.27 (Figure 10A) and the area of resin cement was 29.54 (Figure 10B). Therefore, the percentage of area in mode of failure was calculated from the area of resin cement divided by the total bonded area and multiplying by 100, which was 54.41%. Therefore, it was categorized as a mixed failure.



A

	Area	Mean	Min	Max
1	54.274	203.598	79	255

B

	Area	Mean	Min	Max
1	54.274	203.598	79	255
2	29.543	212.549	79	255

Figure 10 The percentage of area in mixed failure was determined using ImageJ software

Statistics analysis

The data was analyzed by IBM SPSS Statistic for windows version 22.0. Welch ANOVA and Games-Howell post-hoc multiple comparisons test were used to analyze the difference among groups. All *p*-value < 0.05 were considered statistically significant.

Chapter IV

RESULTS

Shear bond strength

According to the statistical analysis results using Welch ANOVA, the shear bond strength was significantly affected by the different silane surface treatments application. The mean shear bond strength obtained from silane surface-treated groups is shown in MPa and mode of failure after 5,000 thermocycling are shown in Table 3 and Figure 12.

The lowest shear bond strength was shown in a control group (18.36±0.69 MPa), which was not significantly different from group 2 (20.17±1.10 MPa) ($p \geq 0.05$). However, the shear bond strength of groups 1 and 2 were significantly different from groups 3 (25.16±1.35 MPa), 4 (30.03±2.80 MPa), 5 (32.52±1.32 MPa), 6 (41.38±2.17 MPa), and 7 (45.49±3.37 MPa) ($p < 0.05$).

The shear bond strength of group 4 (30.03±2.80 MPa) was significantly higher than group 3 (25.16±1.35 MPa) ($p < 0.05$). The shear bond strength of group 4 (30.03±2.80 MPa) and group 5 (32.52±1.32 MPa) were not significantly different ($p \geq 0.05$).

Meanwhile, the shear bond strength of group 4 (30.03±2.80 MPa) was significantly different from group 6 (41.38±2.17 MPa) and group 7 (45.49±3.37 MPa) ($p < 0.05$). Group 7 exhibited the highest shear bond strength, but it was not significantly different from group 6 ($p \geq 0.05$).

Table 3 Means and standard deviations (SD) of shear bond strength obtained from each respective silane surface treatment groups in MPa and mode of failure

Group	Surface treatments	Mean±SD (MPa)	Mode of failure (AF/CF/MF)
1	HF/RXU200 (Control)	18.36±0.69 ^{a}	10/0/0
2	HF/KS/RXU200	20.17±1.10 ^{a}	9/0/1
3	HF/SGI/RXU200	25.16±1.35 ^{d}	3/4/3
4	HF/SGI/SGII/RXU200	30.03±2.80 ^{b}	1/5/4
5	HF/EXP/RXU200	32.52±1.32 ^{b}	1/6/3
6	HF/EXP/SGII/RXU200	41.38±2.17 ^{c}	0/6/4
7	HF/EXP/ADP/RXU200	45.49±3.37 ^{c}	0/7/3

The same superscript indicates no significant difference (p≥0.05).

Abbreviations: HF; 4.5% Hydrofluoric acid, RXU 200; RelyX™ U 200, KS; Kerr Silane Primer, SG I; Signum® Ceramic Bond I, SG II; Signum® Ceramic Bond II, EXP; Experimental silane, ADP; Adper™ Scotchbond™ Multi-purpose Adhesive. AF; Adhesive failure, CF; Cohesive failure, MF; Mixed failure.

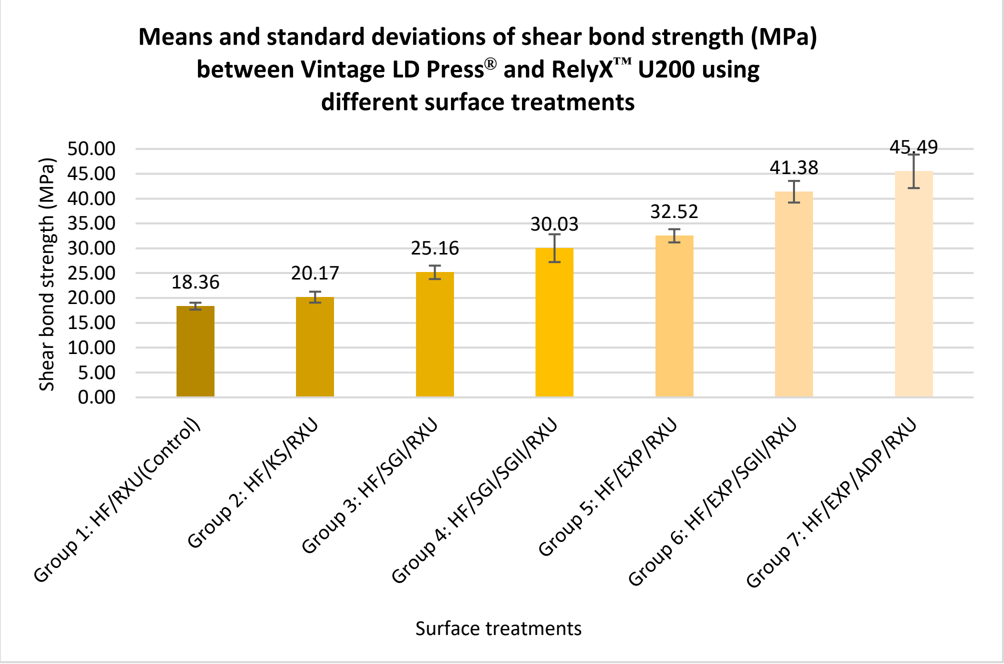


Figure 11 Means and standard deviations of shear bond strength (MPa) between Vintage LD Press® and RelyX™ U200 using different surface treatments

Mode of failure evaluation

The results of the mode of failure evaluation under Stereo Microscope at the magnification of 40x has shown that adhesive failure was highly exhibited in group 1 (100%) and group 2 (90%). The adhesive failure was not exhibited in group 6 and group 7. Cohesive failure was highly exhibited in group 7 (70%), group 6 (60%) and group 5 (60%). Mixed failure was exhibited approximately 30-40% in groups 3, 4, 5, 6, and 7.

Microscopic images of debonded Vintage LD Press® under Stereo Microscope at 40x magnification with modes of failure were shown in figure 11-13. Adhesive failure was highly exhibited in group 1 (Figure 11a) and group 2 (Figure 11b). Mixed failure was exhibited in group 4 (Figure 12a) and group 6 (Figure 12b). Cohesive failure was highly exhibited in group 7 (Figure 13a), group 6 (Figure 13b), group 5 (Figure 13c).

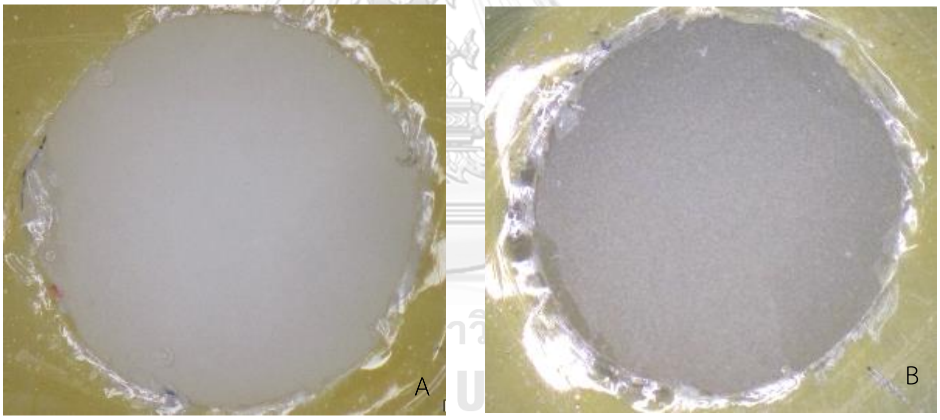


Figure 12 Stereo Microscopic images of adhesive failure;
group 1 (A) and group 2 (B)

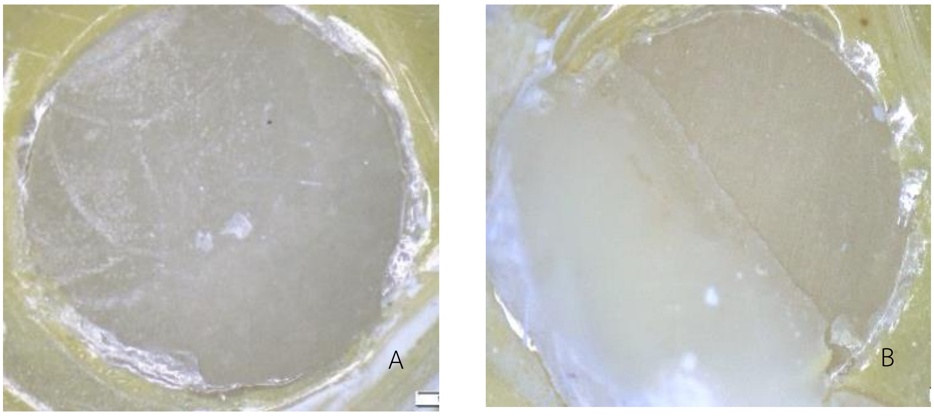


Figure 13 Stereo Microscopic images of mixed failure;
group 4 (A) and group 6 (B)

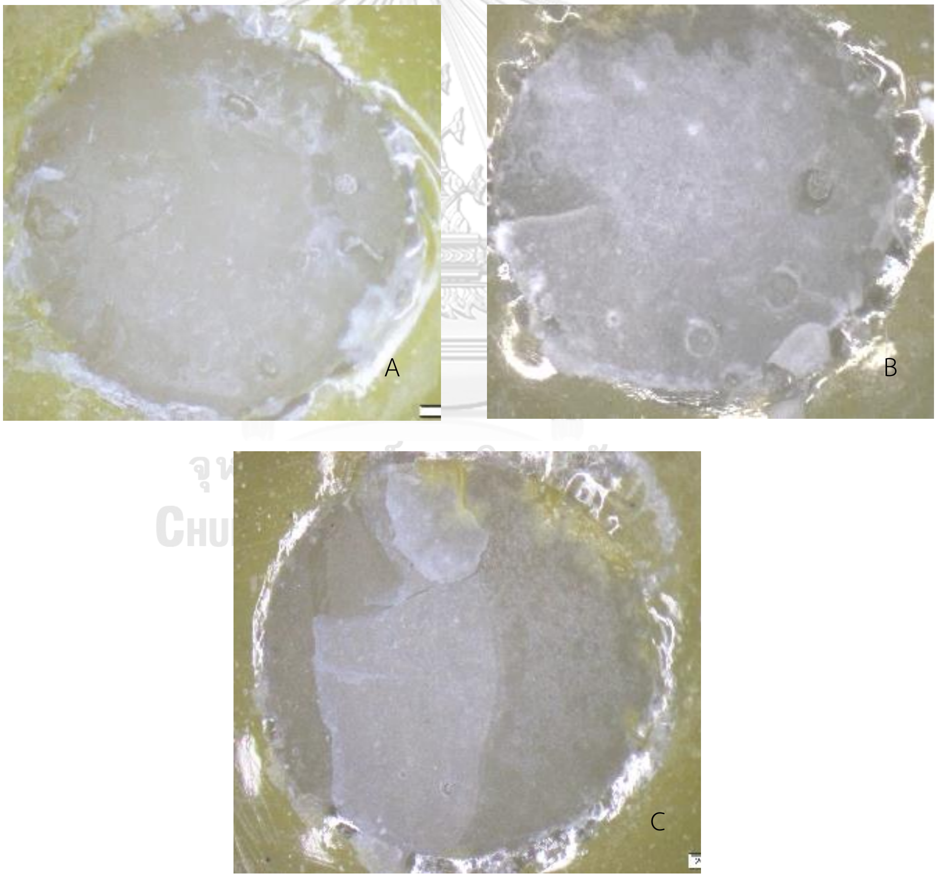


Figure 14 Stereo Microscopic images of cohesive failure;
group 7 (A), group 6 (B), and group 5 (C)

Chapter V

DISCUSSION AND CONCLUSION

Discussion

This study evaluated the SBS of lithium disilicate glass-ceramic and resin cement using different surface treatments. Results of this study found there were significant differences of SBS between lithium disilicate glass-ceramic and resin cement using different silanes surface treatments. The study also found there were significant differences in SBS between lithium disilicate glass-ceramic and resin cement using different silanes followed by different adhesives. Therefore, the null hypotheses were rejected.

At the present time ceramic restoration is in high demand. The bond ability and bond durability have become a major concern for restoration. There are several lithium disilicate glass-ceramics available in the market such as IPS e.max® Press, Initial® LiSi Press, Rosetta SP®, and Vintage LD Press®. Ohashi et al 2017 (28) has shown the microstructure under SEM of various types of lithium disilicate glass-ceramic available in the market. Vintage LD Press® has a wider distribution of lithium disilicate crystal and a much greater glassy matrix than that of GC Initial® LiSi Press and IPS e.max® Press. (28) There are limited studies of bond strength of Vintage LD Press®. Thus, Vintage LD Press® was chosen in this study.

In this study, all of the specimens were etched with 4.5% hydrofluoric acid. It is recommended the silica-based ceramic have a proper surface treatment. The recommended treatment is etching the ceramic internal surface with hydrofluoric acid and application of a silane coupling agent (34, 69) to create micromechanical retention. Micromechanical retention occurs when lithium disilicate glass-ceramic is treated with hydrofluoric acid. Tetrafluorosilane is formed and then reacts with hydrofluoric acid and forms hydrofluorosilicic acid (H_2SiF_6). The glassy matrix will be dissolved and rinsed away. Acid-etching created microporosity, increases the surface area and a high free energy surface state. (12) This will decrease contact angle between the lithium disilicate glass-ceramic surface and resin cement. Acid-etching increases surface wettability for silane coupling agent resulting in bond strength and bond durability improvement. In this study, all of the specimens underwent the thermal cycling test for 5,000 cycles. Thermocycling is used to predict clinical service when there is an initial seal between materials under pressure so that the test will be clinically representative. (70)

The shear bond strength of group 2 was not significantly different from the control group. The group 2; Silane Primer is a resin-containing silane coupling agent with pH 7.3 which was developed to reduce clinical surface pretreatment steps and expected to bond with resin in the resin luting cement. (53) Tarateeraseth et al (2020) (71) studied the contact angles of unetched and etched lithium disilicate glass ceramic after silane application. The study after Silane Primer application on unetched and etched lithium disilicate surface contact angles were not significantly different which correlated with the previous study. (53) Moreover, acid etching on lithium disilicate in resin-containing silane does not improve surface wettability. (71) A previous study (53) implied that the similar contact angle of unetched and etched lithium disilicate glass-ceramic suggested that the chemical bond between lithium disilicate glass-ceramic surface and silane did not occur or slightly occurred. Furthermore, Dimitriadi et al (2019) (72) stated that Silane Primer showed slight silanol activity which was confirmed by the similar siloxane (Si-O-Si) peak between the etched ceramic and polished ceramic surfaces. There are many factors affecting bond quality, surface wettability and surface energy are some of the factors. The additive like resin may impede condensation reaction of silane coupling agent causing low bond strength, low contact angle, (53, 71) and does not enhance bond strength between resin and lithium disilicate glass-ceramic. (73) The condensation reaction formed stabilized siloxane and released water. The addition of resin in silane coupling agent may delay water vaporization. (53) Moreover, the additive in silane might affect ceramic surface polarity and surface energy causing a rising of the contact angle. (71) Additionally, the pH value of Silane Primer is at 7.3, which has lower acidity than an appropriate hydrolysis pH which occurs at 4. (74) This might be the reasons that Silane Primer has lower bond strength than the other groups. Meanwhile, group 1; control gained some shear bond strength from the hydrofluoric acid etching on lithium disilicate glass-ceramic creating microporosity, hence increasing in surface area and surface energy. (13, 32-34)

Signum® Ceramic Bond I (SGI) was considered as a silane coupling agent. (75, 76) Meanwhile, Signum® Ceramic Bond II (SGII) was considered as an adhesive. (76) It could be implied group 4 (SGI/SGII) had higher shear bond strength than group 3 (SGI) because of the applied adhesive. Some studies recommended the use of an unfilled resin as an optional procedure to enhance surface wettability, reduce etched surface irregularities, and reinforce etched lithium disilicate surface. This resulted in gaining a high bond strength. (77, 78) The bond quality was improved in group 4, a higher mixed failure was observed than in group 3. Exhibition of mixed failure and reduction of adhesive failure in group 4 referred to the bond quality improvement. (79)

Group 5; application of an experimental silane (EXP) showed significantly higher bond strength than group 3 (SGI). This may be due to the different types of solvent used, the concentration of the silane, and silane molecular structure in the manufacturing process. The hydrophilicity of solvent affects the hydrolysis rate. (80) EXP solvent was ethanol which has lower hydrophilicity than propanol. Isopropanol and acetone were used as solvents in SGI. Furthermore, the variation of liquid solvent affects the surface tension. The lower surface tension solvents tend to have a favorable wettability. However, EXP was mixed at a pH of 4.6 and the pH value of SGI was 4.5 which both correspond to the optimum hydrolysis rate of silanol occurring at a pH value of 4. (74) Thus, the hydrolysis pH factor was not considered among these groups. EXP and SGI were considered as conventional pre-hydrolyzed silanes, which have a more favorable silanol activity than the universal adhesive. (72) Therefore, it could be implied group 2 (KS) has lower bond strength than group 5 (EXP) and group 3 (SGI).

Meanwhile, group 4 (SGI/SGII) and group 5 (EXP) were not different in shear bond strength. This may be due to variations of solvents used in each silane group. Ethanol was mainly used as solvent and silane dissolves better in ethanol than water. Moreover, silane molecular structure, silane concentration, temperature, and humidity may affect silane hydrolysis. (80)

The shear bond strength of Group 6 (EXP/SGII) was not different from the application of EXP followed by Adper™ Scotchbond Multi-purpose Adhesive (ADP). ADP contained no silane coupling agent, composed of Bis-GMA and hydroxyethyl methacrylate (HEMA), tertiary amine, and initiator. This adhesive was used in a three-step total-etch adhesive system. Meanwhile, the SGII composition showed that there was a presence of silane coupling agent, but the amount of silane coupling was not shown. It could be implied the amount and concentration of silane coupling agent in the bonding agent might be insufficient causing an adverse effect on the bonding agent. However, group 6 and group 7 exhibited more cohesive failure than the other groups and no adhesive failure was observed. This implied better bond quality when using an adhesive as an optional surface treatment.

When considering the commercial silanes and experimental silane with or without an adhesive application, high SBS values were found in the groups which used the experimental silane. This was because it reached an optimum pH silanol hydrolysis rate which occurred at a pH value of 4. The pH value is a main factor affecting silane hydrolysis. The experimental silane used ethanol as a solvent because silane is more easily dissolved in ethanol. It also has a higher hydrophilicity than propanol which makes it tend to have a higher hydrolysis rate.

The results of this study corresponded with the previous study. (77, 78) The results of the study showed the bond strength value correlated with the mode of failure. The high shear bond strength value groups tended to have more mixed and cohesive failures than the ones that had a lower SBS value which tended to have more adhesive failure.

Limitation

There are some limitations/possible limitations in this study; This study was an in vitro study. It could not simulate a cyclic load in the oral condition completely.

The further studies may require using various types of additives in silane coupling agents with different types of resin cements.

Conclusion

Within the limitations of this study, it can be concluded that:

1. The different types of surface treatments significantly affected the shear bond strength value between Vintage LD Press® ceramic and RelyX™ U200 resin cement.
2. Application of pure silane coupling agent with or without the application of an adhesive improves the shear bond strength value and bond quality. The presence of mixed and cohesive failures indicate bond quality improvement.
3. Application of pure silane coupling agent followed by an adhesive improved the shear bond strength value and bond quality.

REFERENCES

1. Kelly JR, Benetti P. Ceramic materials in dentistry: historical evolution and current practice. Aust Dent J 2011;56:84-96.

2. McLaren EA, Whiteman YY. Ceramics: rationale for material selection. Compend Contin Educ Dent 2010;31(9):666-8, 70, 72 ; quiz 80, 700.

3. Conrad HJ, Seong Wj Fau - Pesun IJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. J Prosthet Dent 2007;98(5):389-404.

4. Gorodovsky S, Zidan O. Retentive strength, disintegration, and marginal quality of luting cements. J Prosthet Dent 1992;68(2):269-74.

5. el-Mowafy O. The use of resin cements in restorative dentistry to overcome retention problems. J Can Dent Assoc 2001;67(2):97-102.

6. Rosentritt M, Behr M, Lang R, Handel G. Influence of cement type on the marginal adaptation of all-ceramic MOD inlays. Dent Mater 2004;20(5):463-9.

7. Albert FE, El-Mowafy OM. Marginal adaptation and microleakage of Procera AllCeram crowns with four cements. Int J Prosthodont 2004;17(5):529-35.

8. Sorensen JA, Kang SK, Avera SP. Porcelain-composite interface microleakage with various porcelain surface treatments. Dent Mater 1991;7(2):118-23.

9. Jensen ME, Sheth JJ, Tolliver D. Etched-porcelain resin-bonded full-veneer crowns: in vitro fracture resistance. Compendium 1989;10(6):336-8, 40-1, 44-7.

10. Filho AM, Vieira LC, Araujo E, Monteiro Junior S. Effect of different ceramic surface treatments on resin microtensile bond strength. J Prosthodont 2004;13(1):28-35.

11. Menees TS, Lawson NC, Beck PR, Burgess JO. Influence of particle abrasion or hydrofluoric acid etching on lithium disilicate flexural strength. J Prosthet Dent 2014;112(5):1164-70.

12. Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. Dent Mater 2012;28(5):467-77.

13. Sundfeld D, Naves LZ, Costa AR, Correr AB, Consani S, Borges GA, et al. The Effect of Hydrofluoric Acid Concentration on the Bond Strength and Morphology of the

Surface and Interface of Glass Ceramics to a Resin Cement. Oper Dent 2015;40(5):470-9.

14. Guess PC, Schultheis S, Bonfante EA, Coelho PG, Ferencz JL, Silva NR. All-ceramic systems: laboratory and clinical performance. Dent Clin North Am 2011 55:333-52.

15. McLaren EA, Cao PT. Ceramics in dentistry—part I: classes of materials. Inside Dentistry 2009;5(9):94-105.

16. Anusavice K. Phillip’s science of dental materials. 11, editor: WB Saunders 2003.

17. Kimmich M, Stappert CF. Intraoral treatment of veneering porcelain chipping of fixed dental restorations: a review and clinical application. J Am Dent Assoc 2013;144(1):31-44.

18. Dong JK, Luthy HF, Wohlwend AF, Scharer P. Heat-pressed ceramics: technology and strength. Int J Prosthodont 1992;5(1):9-16.

19. El-Mowafy O, Brochu JF. Longevity and clinical performance of IPS-Empress ceramic restorations-a literature review. J Can Dent Assoc 2002;68(4):233-7.

20. Zarone F, Ferrari M, Mangano F, Leone R, Sorrentino R. Digitally Oriented Materials:Focus on Lithium Disilicate Ceramics. Int J Dent 2016;1:1-10.

21. Denry I, Holloway JA. Ceramics for Dental Applications: A Review. Materials 2010;3(1):351-68.

22. Santos MJ, Costa MD, Rubo JH, Pegoraro LF, Santos GC, Jr. Current all-ceramic systems in dentistry: a review. Compend Contin Educ Dent 2015;36(1):31-7.

23. Giordano R, McLaren EA. Ceramics overview: classification by microstructure and processing methods. Compend Contin Educ Dent 2010;31(9):682-700.

24. Zogheib LV, Bona AD, Kimpara ET, McCabe JF. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. Braz Dent J 2011;22:45-50.

25. Guess PC, Zavanelli RA, Silva NR, Bonfante EA, Coelho PG, Thompson VP. Monolithic CAD/CAM lithium disilicate versus veneered Y-TZP crowns: comparison of failure modes and reliability after fatigue. Int J Prosthodont 2010;23:434-42.

26. Bagis B, Turgut S. Optical properties of current ceramics systems for laminate veneers. J Dent 2013;41:24-30.

27. Sundfeld D, Palialol ARM, Fugolin APP, Ambrosano GMB, Correr-Sobrinho L,

Martins LRM, et al. The effect of hydrofluoric acid and resin cement formulation on the bond strength to lithium disilicate ceramic. Braz Oral Res 2018;32:32-43.

28. Ohashi K, Kameyama Y, Wada Y, Midono T, Miyake K, Kunzelmann KH, et al. Evaluation and comparison of the characteristics of three pressable lithium disilicate glass ceramic materials. Int J Adv Res 2017;7(11):16711-16.

29. Raigrodski AJ, Chiche GF, Potiket NF, Hochstedler JL, Mohamed SE, Billiot SF, et al. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. J Prosthet Dent 2006;96(4):237-44.

30. Haselton DR, Diaz-Arnold AM, Hillis SL. Clinical assessment of high-strength all-ceramic crowns. J Prosthet Dent 2000;83(4):396-401.

31. Vidotti HA, Garcia RP, Conti PC, Pereira JR, Valle AL. Influence of low concentration acid treatment on lithium disilicate core/veneer ceramic bond strength. J Clin Exp Dent 2013;5(4):157-62.

32. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89(3):268-74.

33. Kalavacharla VK, Lawson NC, Ramp LC, Burgess JO. Influence of Etching Protocol and Silane Treatment with a Universal Adhesive on Lithium Disilicate Bond Strength. Oper Dent 2015;40(4):372-8.

34. Tian T, Tsoi JK, Matinlinna JP, Burrow MF. Aspects of bonding between resin luting cements and glass ceramic materials. Dent Mater 2014;30(7):147-62.

35. Puppin-Rontani J, Sundfeld D, Costa AR, Correr AB, Puppin-rontani RM, Borges GA, et al. Effect of Hydrofluoric Acid Concentration and Etching Time on Bond Strength to Lithium Disilicate Glass Ceramic. Oper Dent 2017;42(6):606-15.

36. Matinlinna JP, Vallittu PK. Bonding of resin composites to etchable ceramic surfaces - an insight review of the chemical aspects on surface conditioning. J Oral Rehabil 2007;34(8):622-30.

37. Yucel MT, Aykent F, Akman S, Yondem I. Effect of surface treatment methods on the shear bond strength between resin cement and all-ceramic core materials. J Non Cryst Solids 2012;358(5):925–30.

38. Colares RCR, Neri JR, Souza AMB, Pontes KMF, Mendonca JS, Santiago SL. Effect of Surface Pretreatments on the Microtensile Bond Strength of Lithium-Disilicate

Ceramic Repaired with Composite Resin. Braz Dent J 2013;24(4):349-52.

39. Aboushelib MF, Sleem D. Microtensile bond strength of lithium disilicate ceramics to resin adhesives. J Adhes Dent 2014;16(6):547-52.

40. Erdemir U, Sancakli HS, Sancakli E, Eren MM, Ozel S, Yucel T, et al. Shear bond strength of a new self-adhering flowable composite resin for lithium disilicate-reinforced CAD/CAM ceramic material. J Adv Prosthodont 2014;6(6):434-43.

41. Yavuz T, Ozyilmaz OY, Dilber E, Tobi ES, Kilic HS. Effect of Different Surface Treatments on Porcelain-Resin Bond Strength. J Prosthodont 2017;26(5):446-54.

42. Alberto CG, Edgar DM. Alternatives of surface treatments for adhesion of lithium disilicate ceramics Alternativas de tratamientos de superficie para adhesión de cerámica de disilicado de litio. Revista Cubana de Estomatología 2018;55(1):59-72.

43. Al Moaleem M. Repairing of Fractured Metal Ceramic Restorations: Techniques Review. Int J Contemp Dent 2013;4:21–30 2013;4:21-30.

44. Kocaagaoglu H, Manav T, Albayrak H. In Vitro Comparison of the Bond Strength between Ceramic Repair Systems and Ceramic Materials and Evaluation of the Wettability. J Prosthodont 2017;3:238-43.

45. Zhang M, Matinlinna JP. E-Glass Fiber Reinforced Composites in Dental Applications. Silicon 2012;4(1):73-78.

46. Hooshmand T, Noortb van R K, Keshvad A. Storage effect of a pre-activated silane on the resin to ceramic bond. Dent Mater 2004;20(7):635-42.

47. Amaral R, Ozcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: the effect of surface conditioning. Dent Mater 2006;22(3):283-90.

48. Anusavice, Kenneth J, Phillips, W. R, Shen C, Rawls HR. Phillips' science of dental materials. 12, editor. Rio de Janeiro: Elsevier; 2013.

49. Ishida H. Molecular characterization of composite interfaces. 1 ed. G. IHaK, editor. New York: Plenum Press; 1985.

50. Aboudzadeh MA, Mirabedini SM, Atai M. Effect of silane-based treatment on the adhesion strength of acrylic lacquers on the PP surfaces. Int j Adhes 2007;27(7):519-26.

51. Garboza CS, Berger SB, Guiraldo RD, Fugolin AP, Gonini-Junior A, Moura SK, et al. Influence of Surface Treatments and Adhesive Systems on Lithium Disilicate Microshear

Bond Strength. Braz Dent J 2016;27(4):458-62.

52. Kim RJ, Woo JS, Lee IB, Yi YA, Hwang JY, Seo DG. Performance of universal adhesives on bonding to leucite-reinforced ceramic. Biomater Res 2015;19:11.

53. Chen L, Shen H, Suh BI. Effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers. J Prosthet Dent 2013;110(5):402-7.

54. Neis CA, Albuquerque NL, Albuquerque Ide S, Gomes EA, Souza-Filho CB, Feitosa VP, et al. Surface treatments for repair of feldspathic, leucite - and lithium disilicate-reinforced glass ceramics using composite resin. Braz Dent J 2015;26:152-55.

55. Gokce B, Ozpinar B, Dundar M, Comlekoglu E, Sen BH, Gungor MA. Bond strengths of all-ceramics: acid vs laser etching. Oper Dent 2007 Mar-Apr;32(2):173-8 2007;32(2):173-8.

56. Blatz MB. Long-term clinical success of all-ceramic posterior restorations. Quintessence Int 2002;33(6):415-26.

57. Simon JF, Darnell LA. Considerations for proper selection of dental cements. Compend Contin Educ Dent 2012 Jan;33(1):28-30 32 34-5; quiz 36, 38 2012;33(1):28-30,32,34-5; quiz 36, 38.

58. Nagai T, Kawamoto Y, Kakehashi Y, Matsumura H. Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents. J Oral Rehabil 2005;32(8):598-605.

59. Stamatacos C, Simon JF. Cementation of indirect restorations: an overview of resin cements. Compend Contin Educ Dent 2013;34(1):42-4, 46.

60. Stansbury JW. Curing dental resins and composites by photopolymerization. J Esthet Dent 2000;12(6):300-8.

61. Pegoraro TA, da Silva NR, Carvalho RM. Cements for use in esthetic dentistry. Dent Clin North Am 2007;51(2):453-71.

62. el-Badrawy WA, el-Mowafy OM. Chemical versus dual curing of resin inlay cements. J Prosthet Dent 1995;73(6):515-24.

63. Mak YF, Lai SC, Cheung GS, Chan AW, Tay FR, Pashley DH. Micro-tensile bond testing of resin cements to dentin and an indirect resin composite. Dent Mater 2002;18(8):609-21.

64. Knobloch LA, Gailey D, Azer S, Johnston WM, Clelland N, Kerby RE. Bond

strengths of one- and two-step self-etch adhesive systems. J Prosthet Dent 2007;97(4):216-22.

65. Ferracane JL, Stansbury JW, Burke FJ. Self-adhesive resin cements - chemistry, properties and clinical considerations. J Oral Rehabil 2011;38(4):295-314.

66. International, Organization, for, & Standardization. (2013). Dentistry — Adhesion — Notched-edge shear bond strength test, ISO 29022. In. Switzerland.

67. International, Organization, for, & Standardization. (2009). Dentistry — Polymer-based restorative materials, ISO 4049. In. Switzerland.

68. Matinlinna JP, Lassila LV. Enhanced resin-composite bonding to zirconia framework after pretreatment with selected silane monomers. Dental Materials 2011;27(3):273-80.

69. Lee H-Y, Han G-J, Chang J, Son H-H. Bonding of the silane containing multi-mode universal adhesive for lithium disilicate ceramics. Restor Dent Endod 2017;42(2):95-104.

70. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent 1999;27(2):89-99.

71. Tarateeraseth T, Sriamporn T, Thamronganankul N. The effect of various types of silane coupling agents on the wettability of hydrofluoric acid-etched/unetched lithium disiicate surfaces. J Dent Assoc Thai 2020;70(2):150-7.

72. Dimitriadi M, Zafiropoulou M, Zinelis S, Silikas N, Eliades G. Silane reactivity and resin bond strength to lithium disilicate ceramic surfaces. Dental Materials 2019;35(8):1082-94.

73. Lanza MDS, Vasconcellos WA, Miranda GLPd, Peixoto RTRdC, Lanza LD. Different bonding agents effect on adhesive bond strength: lithium disilicate glass ceramic. Rev Odontol UNESP 2020;49:1-9.

74. Matinlinna JP, Lassila LV, Ozcan M, Yli-Urpo A, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. Int J Prosthodont 2004;17(2):155-64.

75. Altinci P, Mutluay M, Tezvergil-Mutluay A. Repair bond strength of nanohybrid composite resins with a universal adhesive. Acta Biomater Odontol Scand 2018;4(1):10-19.

76. Bergoli CD, de Carvalho RF, Luz JN, Luz MS, Meincke DK, Saavedra Gde S. Ceramic Repair Without Hydrofluoric Acid. J Adhes Dent 2016;18(4):283-7.

77. Hooshmand T, van Noort R, Keshvad A. Bond durability of the resin-bonded and silane treated ceramic surface. Dent Mater 2002;18(2):179-88.

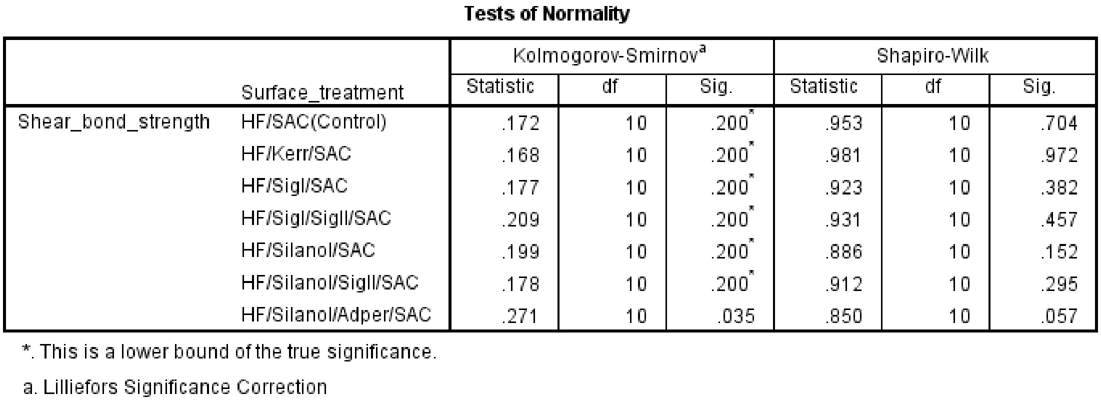
78. Naves LZ, Soares CJ, Moraes RR, Gonçalves LS, Sinhoreti MA, Correr-Sobrinho L. Surface/interface morphology and bond strength to glass ceramic etched for different periods. Oper Dent 2010;35(4):420-7.

79. Abduljabbar T, AlQahtani MA, Jeaidi ZA, Vohra F. Influence of silane and heated silane on the bond strength of lithium disilicate ceramics - An in vitro study. Pakistan journal of medical sciences 2016;32(3):550-54.

80. Matinlinna JP, Lung CYK, Tsoi JKH. Silane adhesion mechanism in dental applications and surface treatments: A review. Dent Mater 2018;34(1):13-28.

APPENDIX

Table 4 Test of normality



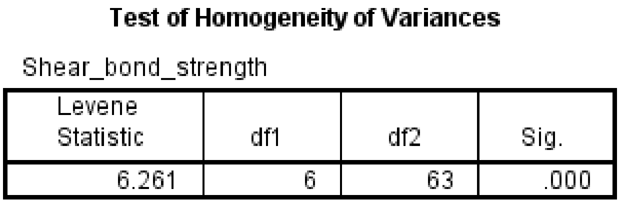
Tests of Normality

	Surface_treatment	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
Shear_bond_strength	HF/SAC(Control)	.172	10	.200*	.953	10	.704
	HF/Kerr/SAC	.168	10	.200*	.981	10	.972
	HF/SigI/SAC	.177	10	.200*	.923	10	.382
	HF/SigI/SigII/SAC	.209	10	.200*	.931	10	.457
	HF/Silanol/SAC	.199	10	.200*	.886	10	.152
	HF/Silanol/SigII/SAC	.178	10	.200*	.912	10	.295
	HF/Silanol/Adper/SAC	.271	10	.035	.850	10	.057

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

a. Lilliefors Significance Correction

Table 5 Test of homogeneity of variances

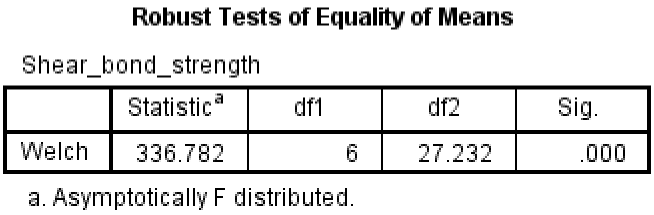


Test of Homogeneity of Variances

Shear_bond_strength

Levene Statistic	df1	df2	Sig.
6.261	6	63	.000

Table 6 Test of equality of means



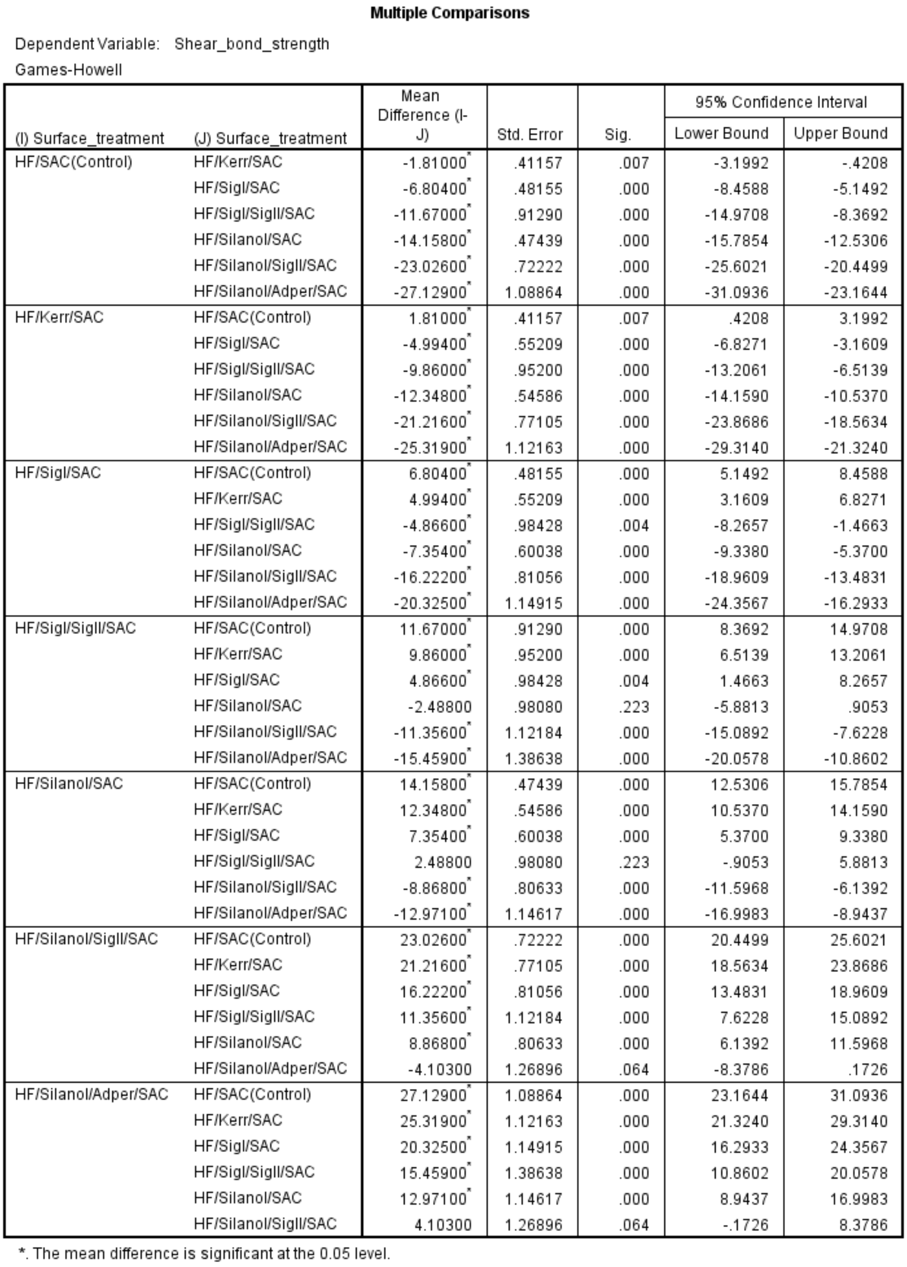
Robust Tests of Equality of Means

Shear_bond_strength

	Statistic[a]	df1	df2	Sig.
Welch	336.782	6	27.232	.000

a. Asymptotically F distributed.

Table 7 Multiple comparisons test



Multiple Comparisons

Dependent Variable: Shear_bond_strength

Games-Howell

(I) Surface_treatment	(J) Surface_treatment	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
HF/SAC(Control)	HF/Kerr/SAC	-1.81000*	.41157	.007	-3.1992	-.4208
	HF/SigI/SAC	-6.80400*	.48155	.000	-8.4588	-5.1492
	HF/SigI/SigII/SAC	-11.67000*	.91290	.000	-14.9708	-8.3692
	HF/Silanol/SAC	-14.15800*	.47439	.000	-15.7854	-12.5306
	HF/Silanol/SigII/SAC	-23.02600*	.72222	.000	-25.6021	-20.4499
	HF/Silanol/Adper/SAC	-27.12900*	1.08864	.000	-31.0936	-23.1644
HF/Kerr/SAC	HF/SAC(Control)	1.81000*	.41157	.007	.4208	3.1992
	HF/SigI/SAC	-4.99400*	.55209	.000	-6.8271	-3.1609
	HF/SigI/SigII/SAC	-9.86000*	.95200	.000	-13.2061	-6.5139
	HF/Silanol/SAC	-12.34800*	.54586	.000	-14.1590	-10.5370
	HF/Silanol/SigII/SAC	-21.21600*	.77105	.000	-23.8686	-18.5634
	HF/Silanol/Adper/SAC	-25.31900*	1.12163	.000	-29.3140	-21.3240
HF/SigI/SAC	HF/SAC(Control)	6.80400*	.48155	.000	5.1492	8.4588
	HF/Kerr/SAC	4.99400*	.55209	.000	3.1609	6.8271
	HF/SigI/SigII/SAC	-4.86600*	.98428	.004	-8.2657	-1.4663
	HF/Silanol/SAC	-7.35400*	.60038	.000	-9.3380	-5.3700
	HF/Silanol/SigII/SAC	-16.22200*	.81056	.000	-18.9609	-13.4831
	HF/Silanol/Adper/SAC	-20.32500*	1.14915	.000	-24.3567	-16.2933
HF/SigI/SigII/SAC	HF/SAC(Control)	11.67000*	.91290	.000	8.3692	14.9708
	HF/Kerr/SAC	9.86000*	.95200	.000	6.5139	13.2061
	HF/SigI/SAC	4.86600*	.98428	.004	1.4663	8.2657
	HF/Silanol/SAC	-2.48800	.98080	.223	-5.8813	.9053
	HF/Silanol/SigII/SAC	-11.35600*	1.12184	.000	-15.0892	-7.6228
	HF/Silanol/Adper/SAC	-15.45900*	1.38638	.000	-20.0578	-10.8602
HF/Silanol/SAC	HF/SAC(Control)	14.15800*	.47439	.000	12.5306	15.7854
	HF/Kerr/SAC	12.34800*	.54586	.000	10.5370	14.1590
	HF/SigI/SAC	7.35400*	.60038	.000	5.3700	9.3380
	HF/SigI/SigII/SAC	2.48800	.98080	.223	-.9053	5.8813
	HF/Silanol/SigII/SAC	-8.86800*	.80633	.000	-11.5968	-6.1392
	HF/Silanol/Adper/SAC	-12.97100*	1.14617	.000	-16.9983	-8.9437
HF/Silanol/SigII/SAC	HF/SAC(Control)	23.02600*	.72222	.000	20.4499	25.6021
	HF/Kerr/SAC	21.21600*	.77105	.000	18.5634	23.8686
	HF/SigI/SAC	16.22200*	.81056	.000	13.4831	18.9609
	HF/SigI/SigII/SAC	11.35600*	1.12184	.000	7.6228	15.0892
	HF/Silanol/SAC	8.86800*	.80633	.000	6.1392	11.5968
	HF/Silanol/Adper/SAC	-4.10300	1.26896	.064	-8.3786	.1726
HF/Silanol/Adper/SAC	HF/SAC(Control)	27.12900*	1.08864	.000	23.1644	31.0936
	HF/Kerr/SAC	25.31900*	1.12163	.000	21.3240	29.3140
	HF/SigI/SAC	20.32500*	1.14915	.000	16.2933	24.3567
	HF/SigI/SigII/SAC	15.45900*	1.38638	.000	10.8602	20.0578
	HF/Silanol/SAC	12.97100*	1.14617	.000	8.9437	16.9983
	HF/Silanol/SigII/SAC	4.10300	1.26896	.064	-.1726	8.3786

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

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