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EFFECT OF DIFFERENT CLEANING AGENTS ON SHEAR BOND STRENGTH OF RESIN  
CEMENTS TO CONTAMINATED CERAMICS



Mr. Jitti Doungsri

จุฬาลงกรณ์มหาวิทยาลัย

**CHULALONGKORN UNIVERSITY**

A Thesis Submitted in Partial Fulfillment of the Requirements  
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By	Mr. Jitti Doungsri
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จิตติ ดวงศรี : ผลของสารทำความสะอาดชนิดต่างๆต่อกำลังแรงยึดเหนี่ยวของซีเมนต์เรซินต่อเซรามิกที่มีการปนเปื้อน. (EFFECT OF DIFFERENT CLEANING AGENTS ON SHEAR BOND STRENGTH OF RESIN CEMENTS TO CONTAMINATED CERAMICS) อ.ที่ปริกษานิพนธ์หลัก: รศ. ทพ. ดร. แมนสรวง อักษรนุกิจ, 89 หน้า.

การศึกษานี้มีจุดประสงค์เพื่อตรวจสอบผลของสารทำความสะอาดชนิดต่างๆต่อกำลังแรงยึดเหนี่ยวของซีเมนต์เรซินต่อเซรามิกที่มีการปนเปื้อนน้ำลาย การศึกษานี้ใช้ชิ้นตัวอย่างที่เป็น เซรามิก ลิเทียม ไดซิลิเกต และ เซอร์โคเนีย จำนวน 240 ชิ้น โดยผิวของชิ้นตัวอย่างได้มีการเตรียมผิวเซรามิกตามคำแนะนำของผู้ผลิต และได้ถูกแบ่งแบบสุ่มออกเป็น 6 กลุ่ม หนึ่งกลุ่มเป็นกลุ่มควบคุมที่เซรามิกไม่มีการปนเปื้อน ส่วนกลุ่มที่เหลือเซรามิกจะมีการปนเปื้อนน้ำลาย แล้วถูกทำความสะอาดด้วยสารทำความสะอาด 5 ชนิด ได้แก่ น้ำ, กรดฟอสฟอริก 37%, กรดไฮโดรฟลูออริก 5%, ไอโวลีน และสารละลายโซเดียม ซิลิเกต 30% หลังจากนั้นแต่ละชิ้นคอมโพสิตจะถูกยึดบนผิวเซรามิกด้วยซีเมนต์เรซิน รีไลเอกซ์ยูสองร้อย, พานาเวียเอฟสอง และ ซุปเปอร์บอนด์ซีแอนด์บี (RelyX U200, Panavia F2.0, Superbond C&B) หลังจากนั้นชิ้นตัวอย่างจะถูกเก็บในน้ำกลั่น 37 องศาเซลเซียสเป็นเวลา 24 ชั่วโมง หลังจากนั้นทำการทดสอบกำลังแรงยึดเหนี่ยวด้วยเครื่องทดสอบสากลที่ความเร็วหัวตัด 0.5 มิลลิเมตรต่อนาที ผลของการทดสอบพบว่า กลุ่มเซรามิก ลิเทียม ไดซิลิเกตที่ใช้ ซีเมนต์ รีไลเอกซ์ยูสองร้อย ในการยึดกับเรซินคอมโพสิต เมื่อปนเปื้อนน้ำลาย แล้วทำความสะอาดด้วย ไอโวลีน หรือ สารละลายโซเดียม ซิลิเกต 30% มีค่ากำลังแรงยึดเหนี่ยวสูงกว่ากลุ่มอื่นอย่างมีนัยสำคัญ ส่วนกลุ่มเซรามิก ลิเทียม ไดซิลิเกตที่ใช้ ซีเมนต์ พานาเวียเอฟสอง และ ซุปเปอร์บอนด์ซีแอนด์บี พบว่าหลังการปนเปื้อนน้ำลาย เมื่อทำความสะอาดด้วย ไอโวลีน, สารละลายโซเดียม ซิลิเกต 30% หรือ กรดไฮโดรฟลูออริก 5% มีค่ากำลังแรงยึดเหนี่ยวสูงกว่ากลุ่มอื่นอย่างมีนัยสำคัญ ในขณะที่ กลุ่มเซอร์โคเนีย ที่ใช้ซีเมนต์ รีไลเอกซ์ยูสองร้อย และ พานาเวียเอฟสอง เมื่อมีการปนเปื้อนน้ำลายแล้วทำความสะอาดด้วย ไอโวลีน หรือสารละลายโซเดียม ซิลิเกต 30% มีค่ากำลังแรงยึดเหนี่ยวสูงกว่ากลุ่มอื่นอย่างมีนัยสำคัญ และกลุ่มเซอร์โคเนีย ที่ใช้ซีเมนต์ ซุปเปอร์บอนด์ซีแอนด์บี เมื่อมีการปนเปื้อนน้ำลายแล้วทำความสะอาดด้วย ไอโวลีน มีค่ากำลังแรงยึดเหนี่ยวสูงกว่ากลุ่มอื่นอย่างมีนัยสำคัญ จากการศึกษาสรุปได้ว่า ไอโวลีน และสารละลายโซเดียม ซิลิเกต 30% มีประสิทธิภาพในการทำความสะอาดผิว เซรามิก ลิเทียม ไดซิลิเกต ที่มีการปนเปื้อนน้ำลายมากกว่าวิธีอื่น ในขณะที่ไอโวลีน มีประสิทธิภาพที่สูดในการทำความสะอาดผิว เซอร์โคเนีย ที่มีการปนเปื้อนน้ำลาย

จุฬาลงกรณ์มหาวิทยาลัย  
CHULALONGKORN UNIVERSITY

ภาควิชา ทันตกรรมประดิษฐ์

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สาขาวิชา ทันตกรรมประดิษฐ์

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# # 5376107832 : MAJOR PROSTHODONTICS

KEYWORDS: LITHIUM DISILICATE CERAMIC / ZIRCONIA / CONTAMINATION / SALIVA / CLEANING AGENT / SHEAR BOND STRENGTH

JITTI DOUNGSRI: EFFECT OF DIFFERENT CLEANING AGENTS ON SHEAR BOND STRENGTH OF RESIN CEMENTS TO CONTAMINATED CERAMICS. ADVISOR: ASSOC. PROF. MANSUANG ARKSORNNUKIT, Ph.D., 89 pp.

The objective of this study was to investigate the effect of different cleaning agents on shear bond strength of resin cements to saliva contaminated ceramics. Three hundred and sixty slabs of ceramic specimens (lithium disilicate ceramic and zirconia) were prepared. The round specimens of lithium disilicate ceramics with diameter of 15 mm and a thickness of 2 mm and the square specimens of zirconia with a width of 10 mm and a thickness of 1 mm were used in this study. The substrate surfaces were conditioned according to manufacturer's instructions. Specimens of each ceramic were randomly divided into six groups (n=10). The first group was control group. The others were contaminated with saliva and then cleaned using five cleaning conditions. They were deionized water, 37% phosphoric acid, 5% hydrofluoric acid, Ivoclean and 30% sodium silicate solution. Resin composite block (Clearfil DC core automix) was bonded to ceramic using resin cements ( RelyX U200, Panavia F2.0, Superbond C&B ). The specimens were stored in 37oC distilled water for 24 hours. The shear bond strength tested was performed using a universal testing machine with a cross head speed of 0.5 mm/min. For RelyX U200 group, saliva contaminated lithium disilicate ceramics cleaning with Ivoclean or 30% sodium silicate solution provided statistically significant higher shear bond strengths (24.10 MPa / 24.40 MPa) respectively than the other methods ( $p < 0.05$ ). Panavia F2.0 group and Superbond C&B groups, saliva contaminated lithium disilicate ceramics cleaning with Ivoclean, 30% sodium silicate solution or 5% hydrofluoric acid provided statistically significant higher shear bond strengths (11.96MPa/ 10.76MPa/ 10.08MPa and 27.41MPa/ 28.72MPa/ 27.98MPa ) respectively than the other methods ( $p < 0.05$ ). RelyX U200 and Panavia F2.0 groups, saliva contaminated zirconia cleaning with Ivoclean or 30% sodium silicate solution provided statistically significant higher shear bond strengths (10.71 MPa / 9.24 MPa and 10.55 MPa / 10.06 MPa ) respectively than the other methods ( $p < 0.05$ ). Superbond C&B, saliva contaminated zirconia cleaning with Ivoclean provided statistically significant higher shear bond strengths (20.12 MPa ) than the other methods ( $p < 0.05$ ). The results suggest that Ivoclean and 30% sodium silicate solution were more effective in decontaminating the saliva from lithium disilicate ceramic than the other methods for all resin cements. Ivoclean was the most effective in decontaminating the saliva from zirconia than the other methods for all resin cements.

Department: Prosthodontics

Student's Signature .....

Field of Study: Prosthodontics

Advisor's Signature .....

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# CHAPTER I

## INTRODUCTION

### Background and rationale

Recently, all ceramic restorations have gained popularity over other restorations because of their aesthetics, biocompatibility and durability.<sup>[1]</sup> Tooth-resin bonding has been studied extensively and adhesive has been developed. While resin-ceramic bonding still was not as satisfactory and it needs to be developed to ensure their good adhesion. A strong and durable resin-ceramic bonding provides all-ceramic restoration with high retention, improved marginal adaptation and increasing in fracture resistance of the restored teeth and the restoration.<sup>[2]</sup> Many factors can affect good resin-ceramic bonding. One of them is the cleanliness of the bonding substrate.

In clinical situation, the saliva contamination during try-in is virtually unavoidable. This has led to significantly reduced bond strength.<sup>[3]</sup> Saliva contamination is a main reason for bond strength reduction.<sup>[3-7]</sup> Saliva consists of proteins, enzyme, blood cell, bacteria and various forms of phosphate such as phospholipid in water solution.<sup>[8]</sup> It interferes bond of restoration. Saliva may have left thin residual film on the ceramic surface, which inhibits micromechanical retention and stable chemical bond.<sup>[9]</sup> Non-covalent absorption of saliva proteins might occur on ceramic surface after the ceramic came into contact with saliva for 60 seconds.<sup>[10, 11]</sup> Thus, saliva protein can affect bond strength of restoration. Phosphate group in saliva is actively bound with the bonded surface of zirconia restorations. The phosphate shows a strong affinity and establishes a durable bond to zirconium oxide.<sup>[12]</sup> This phosphate is irreversible with the surface and thus makes the cleaning difficult. Therefore, saliva contamination can pose a problem when the saliva contaminated restorations are adhesively cemented and further resulting in adhesive failure.

Many studies found that contamination could inhibit the formation of stable bond.<sup>[4, 10, 11, 13-15]</sup> Hence, it is imperative that the bonded surface of the restoration is decontaminated before cementation. Achieving properly decontaminated bonding surfaces of the restorations after intraoral try-in is an essential step in creating significant bond strength between tooth structure and restorations. The cleaning methods can be mechanical method, chemical methods or combination of both. Mechanical methods include sandblasting and polishing with polishing paste. Chemical methods include the acid treated surface and cleaning solvent for example alcohol, acetone, phosphoric acid and acidulated phosphate fluoride.

From previous studies, each cleaning method has advantages and disadvantages and it does not provide good adequate bond strength.

Hence this study aims to investigate the efficacy of each cleaning agent to the removal of saliva contaminations on ceramic by means of shear bond strength between ceramic and resin composite in different resin cements.

### Research questions

1. Whether or not different cleaning agents affect the shear bond strength of saliva contaminated lithium disilicate ceramic?
2. Whether or not different cleaning agents affect the shear bond strength of saliva contaminated zirconia?

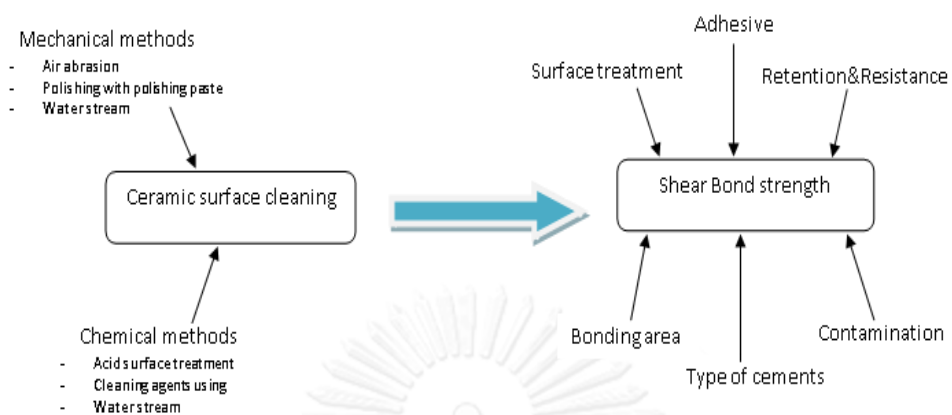
### Objective

1. To study effect of different cleaning agents to shear bond strength of saliva contaminated lithium disilicate ceramic.
2. To study effect of different cleaning agents to shear bond strength of saliva contaminated zirconia.

### Research hypothesis

- $H_0$  : There is no difference in the effect of different cleaning agents to shear bond strength of saliva contaminated lithium disilicate ceramic.
- $H_a$  : There are differences in the effect of different cleaning agents to shear bond strength of saliva contaminated lithium disilicate ceramic.
- $H_0$  : There is no difference in the effect of different cleaning agents to shear bond strength of saliva contaminated zirconia.
- $H_a$  : There are differences in the effect of different cleaning agents to shear bond strength of saliva contaminated zirconia.

## Conceptual framework



## Keywords

Lithium disilicate ceramic

Zirconia

Contamination

Saliva

Cleaning agent

Shear bond strength

## Research design

Laboratory Experimental Research

## Limitations

1. This study is a laboratory experimental research. It does not simulate the same condition it was in the mouth.
2. This research is affected by many variables.
3. The collection of the data of this study was implemented by one researcher using the same apparatus.

### Expected benefits and applications

1. The results could reflect the effect of different cleaning agents on the shear bond strength of resin cements to contaminated ceramics.
2. The results of this study could benefit for dentists to select suitable cleaning agents for clean contaminated ceramic restoration.



## CHAPTER II

### LITERATURE REVIEW

#### Ceramic

Porcelain development in dentistry was established in the late 18th century. Three basic compositions are Kaolin ( $\text{Al}_2\text{O}_3\text{SiO}_2\text{H}_2\text{O}$ ) 50%, Feldspar ( $\text{K}_2\text{OAl}_2\text{O}_3\text{6SiO}_2$ ) 25% and Quartz ( $\text{SiO}_2$ ) 25%. Later, the esthetic was more important. Thus porcelain compositions have changed and developed. The gloss of porcelain surface depends on duration of firing. The porcelain will be translucent when reducing the amount of kaolin and increasing the feldspar. Recently, the basic porcelain's composition is borosilicate glass feldspar. It will be more leucite after firing process.

Duchateau and Chemant developed a method of porcelain artificial teeth and published in the year 1790 to 1824.<sup>[16]</sup> After porcelain in dentistry was further developed, porcelain was used to make a crown. The excellent feature of porcelain fused to metal crown is using two materials together. The crown has strength from metal and esthetic from porcelain.<sup>[17]</sup> Recently, humans are concerned more on cosmetic. The opacity of the opaque porcelain and metal affect the porcelain fused metal crown looks, unlike the natural teeth. Therefore, all-ceramic crowns have been developed.

#### Lithium disilicate ceramic (IPS e.max press)

In the beginning, IPS Empress ceramic was produced with a leucite base. Major composition is silicone dioxide-Alumina-Potassium oxide ( $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O}$ ). The flexural strength increases from 120 megapascal to 200 megapascal after the material is coated and fired. But it is not strong enough for a bridge. The company has developed the composition and properties of materials. It is called IPS Empress II and renamed IPS e.max press later. The strength of IPS e.max press is about 250 megapascal. It is a high fracture resistance and translucent like natural teeth. It can

be used for anterior crown, posterior crown and anterior bridge. Major composition of IPS e.max press is lithium disilicate ( $\text{Li}_2\text{Si}_2\text{O}_5$ ) and the secondary composition is lithium orthophosphate ( $\text{Li}_3\text{PO}_4$ ) different from IPS Empress, which the major composition is leucite. Lithium disilicate is long crystals, which makes it strong and high fracture resistance. Lithium disilicate crystals combined with the glass matrix would inhibit crack propagation by absorbing energy processes such as deviate crack direction or reduce stress on the crack. When large crack grow, the crystal will interfere and absorb the kinetic energy.

### **Zirconia (Zirconium dioxide ceramic)**

Zirconia is the name given to zirconium dioxide ( $\text{ZrO}_2$ ). Zirconia is a polycrystalline material. Zirconium dioxide ( $\text{ZrO}_2$ ) was identified in 1789 by German chemist. In the beginning, zirconium dioxide was used as a ceramic biomaterial in the form of ball heads for Total Hip Replacements (THR). In later years, zirconium dioxide was developed and used as an application in space shuttles, automobiles and dentistry. The type of zirconia used in dentistry is yttria tetragonal zirconia polycrystal (Y-TZP). It is a monophasic ceramic that is formed by directly sintering crystal together. The yttria is added to zirconia to stabilize the structure and maintain the materials properties. Zirconia has good mechanical strength, good toughness, excellent physical properties, white color and biocompatibility. Zirconia was used as endodontic posts, implant abutments, an all-ceramic crowns and fixed partial dentures.



## Resin cement

Resin cements have composition similar to resin composite filling materials such as resin matrix with silane treated inorganic fillers. Resin cements may have high bond strengths both to tooth structure and porcelain, high tensile and compressive strengths, and the lowest solubility of the available cements.<sup>[18]</sup>

### Resin Cement Classifications

Resin cements can be classified according to their adhesive scheme into total-etch, self-etching and self-adhesive

**Total-Etch Resin Cements**—Total-etch resin cements use a 30% to 40% phosphoric acid to etch dentin and enamel. This etching procedure removes the smear layer, and leaves dentinal tubules opened.<sup>[19]</sup> After etching, the adhesive is then applied to the preparation to bond the cement to the tooth. These cements and the adhesives used with them can be light- or dual-cured.<sup>[19]</sup> Total-etch resin cements have increased bond strengths of the resin-based cements and tooth to nearly that of enamel bonding and have significantly reduced microleakage.<sup>[20]</sup> This category provides the highest cement-to-tooth bond but also requires many steps to bond ceramic, composite resin, or metal to the tooth. These cements include RelyX ARC and Superbond C&B.

**Self-Etch Resin Cements**—Self-etch systems apply a self-etching primer to prepare the tooth surface, and mixed cement is applied over the primer. Bonds to tooth structure using this category of cements are almost as high as those of the total-etch cements.<sup>[19]</sup> Self-etching systems are popular among dentists because they are easy to use. Resin cements that incorporate self-etching primers eliminate steps during application with the goal of reducing operator errors and technique sensitivity and ease of use.<sup>[21]</sup> The example of this type of cement is Panavia F2.0.

**Self-Adhesive Resin Cements**—A number of resin cements have been introduced as one-component “universal adhesive cements”. Self-adhesive cements can bond to an untreated tooth surface that has not been micro-abraded or pretreated with an etchant, primer, or bonding agent; thus, cementation is accomplished in a single step. These cements contain phosphoric acid, which is grafted into the resin. Once mixing is initiated, the phosphoric acid reacts with filler particles and dentin in the presence of water, forming a bond. The resin is polymerized into a cross-linked polymer, as is the case with composite resin bonding.<sup>[22]</sup> These cements include RelyX U200 and SpeedCEM.

## Contamination

In clinical try-in procedure, the contamination of restoration luting surface with saliva, blood and silicone disclosing medium are unavoidable.<sup>[3]</sup> Saliva contamination is a main reason for bond strength reduction<sup>[3-7]</sup> Thus, contaminations can affect the bond strength of restoration.

## Saliva

Salivary fluid is an exocrine secretion<sup>[23, 24]</sup> consisting of approximately 99% water, while the other 1% consists of electrolytes (sodium, potassium, calcium, chloride, magnesium, bicarbonate, phosphate) and some polypeptides and oligopeptides of importance to oral health.<sup>[8, 25]</sup> The composition of saliva except water can be divided into two groups. It is organic compounds and inorganic compounds.

### 1) Organic composition of saliva<sup>[26]</sup>

- Proteins comprise the bulk of the organic content of saliva. Most of the proteins in saliva exhibit antimicrobial functions and enzymes
- Free amino acids
- Urea
- Carbohydrates
- Lipids

### 2) Inorganic composition of saliva<sup>[27]</sup>

- Hydrogen ions in saliva has the greatest influence on the chemical reaction in the oral cavity
- Calcium ions is influenced by saliva flow rate

- Phosphate; inorganic phosphate in saliva were found as phosphoric acid ( $\text{H}_3\text{PO}_4$ ) and its conjugates:  $\text{H}_2\text{PO}_4^-$ ,  $\text{HPO}_4^{2-}$  and  $\text{PO}_4^{3-}$ . Its concentration is affected by salivary flow rate as well as pH
- Fluoride

Saliva consists of proteins, enzyme, blood cell, bacteria and various forms of phosphate such as phospholipid in water solution. Saliva may have left thin residual film on the ceramic surface, which inhibits micromechanical retention and stable chemical bond.<sup>[9]</sup> Non-covalent absorption of saliva proteins might occur on ceramic surface after the ceramic came into contact with saliva for 60 seconds.<sup>[10, 11]</sup> Thus, saliva protein can affect bond strength of restoration. Phosphate group in saliva is actively bound with the luting surface of zirconia restorations. The phosphate shows a strong affinity and establishes a durable bond to zirconium oxide.<sup>[12]</sup> This phosphate is irreversible deposited on the surface and thus makes the cleaning difficult. This may be the cause of decrease in bond strength after contamination.

Therefore, cleaning of the bonding substrates of the materials is essential to achieve stable bond strength.

### Cleaning of restorations after try-in procedure

The problems of saliva-protein pollution are discussed in different possible solutions. The cleaning process is necessary to restoration surface. There are many methods to clean the restoration surface. The cleaning methods can be mechanical method, chemical method or combination of both. Mechanical methods include sandblasting and polishing with polishing paste. Chemical methods include the acid treated surface and cleaning solvent for example alcohol, acetone, phosphoric acid and acidulated phosphate fluoride.

**Water steam** : water steam's ability to clean is based primarily on its heat. The steam is applied to cleanable surfaces via a variety of insulated tools and accessories. It provides the energy needed to release contaminations into water suspension, after which they can be removed by wiping. But saliva contaminated dental ceramic restoration cleaned with water is not sufficient. Previous studies showed that saliva contaminated lithium disilicate ceramic<sup>[9]</sup> and zirconia<sup>[14]</sup> cleaning with only water did not increase the bond strength.

**Polishing with polishing paste** : there are many types of polishing paste in dentistry such as pumice, fluoride paste and sodium bicarbonate used with air polishing device. These substances have been used in polishing or to remove saliva contamination. But previous study found that saliva contaminated lithium disilicate ceramic that was cleaned with an air-polishing device with sodium bicarbonate showed lower bond strength than contaminated ceramic. This attributed to the remaining particles of sodium bicarbonate on ceramic surface.<sup>[9]</sup>

**Air abrasion treatment (sandblasting treatment)** : air abrasion treatment is the operation of forcibly propelling a stream of abrasive material against a surface under high pressure to roughen a smooth surface, shape a surface or remove surface contaminants. A typically air is used to propel the blasting material.<sup>[28]</sup> In dentistry, air abrasion treatment with aluminum oxide ( $\text{Al}_2\text{O}_3$ ) particle was popularity. It is commonly used for restoration surface treatment and removing surface contaminants. Previous studies found that air abrasion with 50 micron  $\text{Al}_2\text{O}_3$  at 0.25 MPa at 10 millimeter distance was used on zirconia surface to increase the surface roughness, clean and activate surface.<sup>[29-32]</sup> Airborne-particle abrasion with  $\text{Al}_2\text{O}_3$  was the most effective cleaning method in order to remove saliva contamination from zirconia.<sup>[10, 14]</sup> This method improved resin-zirconia ceramic bond strength with durability.<sup>[2, 15, 30, 31, 33-37]</sup> However, the effect of air abrasion applied to these ceramic is still controversial.<sup>[38, 39]</sup> Air abrasion treatment can induce compressive stresses and phase transformation on the surface, which increase the strength. At the same time, air abrasion treatment also induce flaws and defects which reduce the strength.<sup>[40]</sup> If it was used in the unsuitable conditions, it can also compromise the mechanical strength and damage the ceramic surface by initiating surface defect and creating microcracks,<sup>[38, 41]</sup> decreasing the long-term survival rate of all ceramic crowns.<sup>[38, 39]</sup> Thus, this method may have the negative effect on the bonded surface of the restoration.

**Acid surface treatment** : Acid treated ceramic can improve bond strength of the lithium disilicate ceramic.<sup>[42]</sup> The use of hydrofluoric acid is popular in dentistry. Hydrofluoric acid reacts with the glass matrix of ceramic. The glass matrix is removed and the crystalline structure is exposed.<sup>[43, 44]</sup> The acid treated provide more surface energy in combination with silane.<sup>[45]</sup> The use of hydrofluoric acid for glass ceramic surface cleaning is re-etching after ceramic was etched from laboratory. Previous study showed that 5% hydrofluoric acid provided high bond strength than the other methods when it was used to clean saliva contaminated lithium disilicate ceramic. Second hydrofluoric etching after contamination had no negative influence on ceramic surface.<sup>[9]</sup> On the other hand hydrofluoric acid can reduce the flexural

strength of glass ceramics.<sup>[46]</sup> Re-etching decreases the volume of ceramic and increases flaws in ceramic surface and weakening the surface. Re-etching caused a reduction in the bond strength to resin composite.<sup>[47]</sup> Thus, this method should be more investigated.

**Cleaning agents** : there are many cleaning agents used in cleaning dental restoration surface such as alcohol, acetone and phosphoric acid. Dental textbook recommended the use of organic solution such as alcohol and acetone for the removal of saliva contamination on the luting surface of restoration before cementation.<sup>[48]</sup> In contrast, many previous studies also showed that the use of alcohol and acetone in decontamination ceramic created low bond strength than the other methods.<sup>[4, 10, 11, 15, 49]</sup>

- **Phosphoric acid** : Thirty-seven percent phosphoric acid was also recommended in decontamination of the restoration surface. Previous studies found that saliva contaminated glass ceramic cleaned with phosphoric acid had higher bond strength than the other methods.<sup>[4, 49]</sup> Thus, phosphoric acid gel was recommended for cleaning glass ceramic restorations. Zirconia can form chemical bonds with phosphate group.<sup>[50]</sup> Zirconium oxide can react with phosphoric acid in an acid-base reaction.<sup>[12]</sup> Zirconium phosphate is formed and caused the surface inert to the primer. The phosphoric acid might also decrease the surface energy of the activated zirconia surface.<sup>[10, 15]</sup> Previous study showed saliva contaminated zirconia cleaned with phosphoric acid had lower bond strength and decrease long-term bond strength.<sup>[10]</sup>

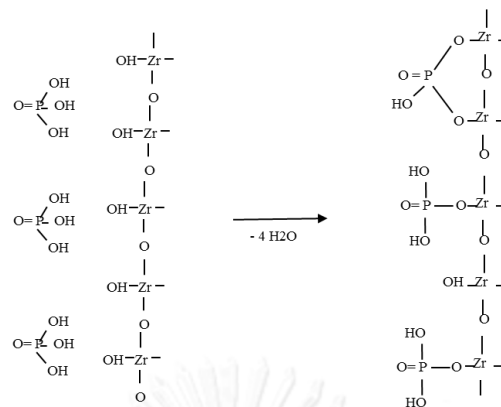


Figure 1 Scheme of the reaction between the zirconium oxide surface and phosphoric acid

Various cleaning agents like cleaning with water, alcohol, acetone, phosphoric acid have been advised and tested. Each cleaning method has advantages and disadvantages but it does not provide adequate bond strength.

- **Ivoclean** : recently, Ivoclean (Ivoclar Vivadent AG, Schaan, Liechtenstein) was introduced as a cleaning agent. The manufacturer claims that it is a non-abrasive cleaning agent. Ivoclean can effectively clean the bonding surface of every kind of prosthetic restorations after intraoral try-in to create optimum prerequisite for the adhesive luting procedure. Ivoclean consists of an alkaline suspension of zirconium oxide particles. Because of the size and concentration of the particles in the medium, phosphate contaminants are much more likely to bond to them than to the surface of the ceramic restoration. Ivoclean absorbs the phosphate contaminants like a sponge and thus leaves behind a clean zirconium oxide surface.<sup>[51]</sup> However, Ivoclean still needs supportive researches.



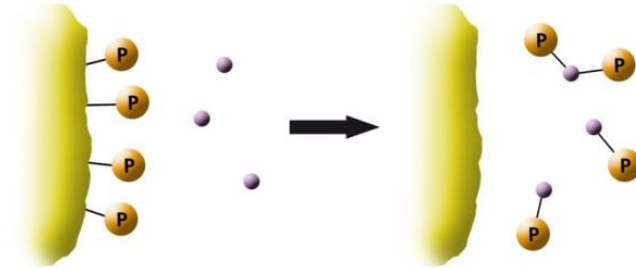


Figure 2 Scheme of cleaning action of Ivoclean on zirconium oxide surface.

Standard compositions	(in wt%)
Zirconium oxide particle	10 - 15
Water	65 - 80
Polyethylene glycol	8 - 10
Sodium hydroxide	≤ 1
Pigments, additives	4 - 5

#### Physical propertie

pH	13 - 13.5
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- **Sodium silicate solution** : Sodium silicate is a colorless compound of oxides of sodium and silica. It has a range of chemical formula varying in sodium oxide ( $\text{Na}_2\text{O}$ ) and silicon dioxide or silica ( $\text{SiO}_2$ ) contents or ratios. The more alkaline silicates including sodium metasilicate ( $\text{Na}_2\text{SiO}_3$ ) are crystalline materials with definite structures and characteristic properties. Sodium silicate is a compound that is commonly used as cleaning agent. These are used chiefly as cleaners and detergents. Sodium silicate is a building agent used in many commercial detergents.<sup>[52]</sup> Builder agent provides water softening and a desirable level of alkalinity (increase pH), which aids in cleaning.<sup>[53]</sup> Sodium silicate also acts as buffer to maintain proper alkalinity in wash water. The purpose of the sodium silicate to prevent mineral deposits on surfaces. The cleaning properties depend on the effect of medium alkalinity with pH 11-12.5. The alkali agents dissolve grease, oils, fats and protein based deposits. Sodium silicate exhibits good detergency, good saponifier. Silicates soften water by the formation of precipitates that can be easily rinsed away, excellent buffering action against acidic compounds, neutralize acid soils.<sup>[52]</sup> Thus, the sodium silicate may be effective in decontamination on the restoration surface and promotes good bond strength.

There are many cleaning agents for decontaminating restoration surface. Hence, this study aims to investigate the efficacy of each cleaning agent in removing saliva contamination on lithium disilicate ceramic and zirconia by means of shear bond strength of resin cements to two kinds of ceramic; lithium disilicate and zirconia.

## CHAPTER III

### METHODOLOGY

#### 1. Specimen Preparation

One hundred and eighty round specimens with diameter of 15 mm. and a thickness of 2 mm. were prepared from lithium disilicate ceramics (IPS e.max press; Ivoclar Vivadent AG, Schaan, Liechtenstein). Another one hundred and eighty square specimens (10×10 mm.) with a thickness of 1 mm were prepared from zirconia (Cercon; Degudent GmbH, Hanue-Wolfgang, Germany). All specimens were wet-polished with 200, 400, 600 and 800 grit silicon carbide papers respectively. The surface roughness of all specimens was calculated with a contact profilometry (Talyscan 150; Leicester, England). The individual specimen surfaces were conditioned according to the manufacturer's instructions as follows.

Lithium disilicate ceramics were ultrasonically cleaned for 10 minutes with distilled water and 5% hydrofluoric acid (IPS Ceramic Etching gel; Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied over the entire surface for 20 seconds, rinsed with distilled water for 15 seconds, air dried and stored in the closed contamination until used.

Zirconia surface was roughened with 50 micron aluminum oxide abrasive at 2.5 bar for 15 seconds at a distance of 10 mm<sup>[10]</sup> (Blast Master; Bangkok, Thailand) and ultrasonically cleaned with distilled water for 10 minutes, air dried and stored in the closed contamination until used.

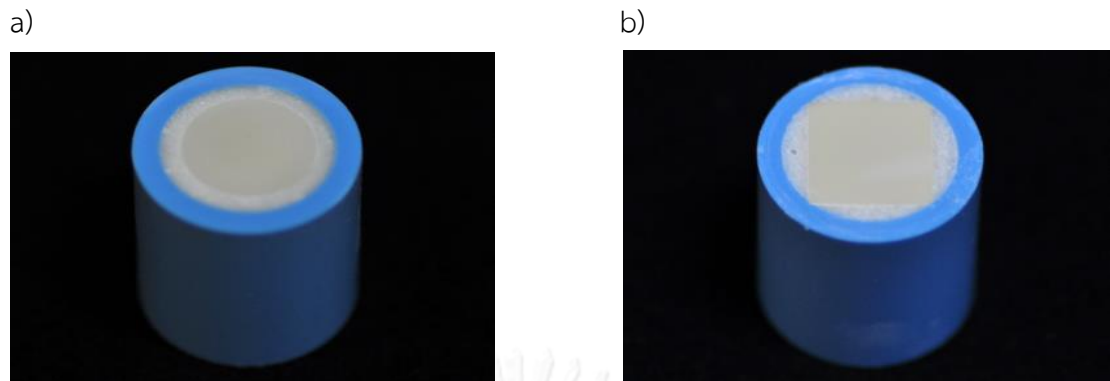


Figure 3 a) illustration of lithium disilicate ceramic specimen b) zirconia specimen

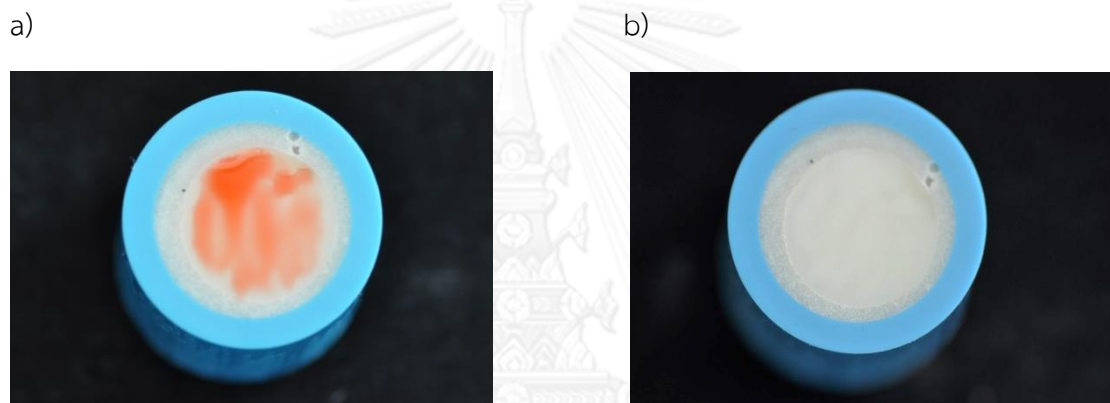


Figure 4 Lithium disilicate ceramic was conditioned with 5% hydrofluoric acid a) hydrofluoric acid applied on ceramic surface b) ceramic surface after rinsed with water

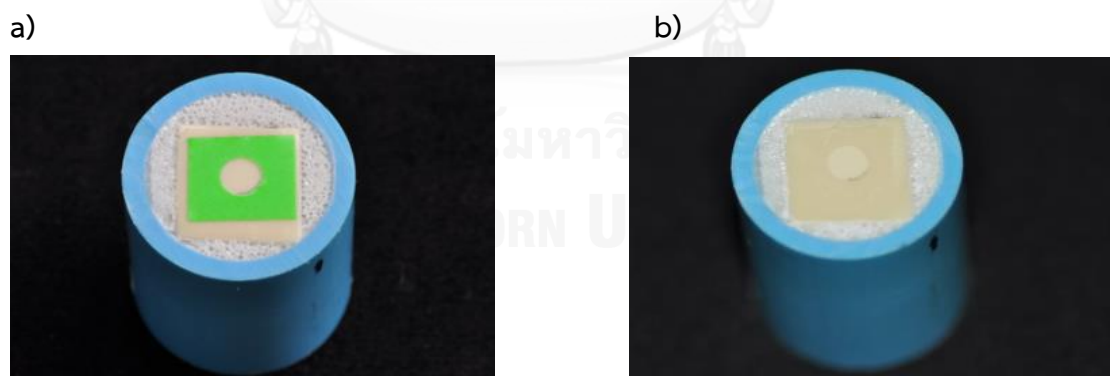
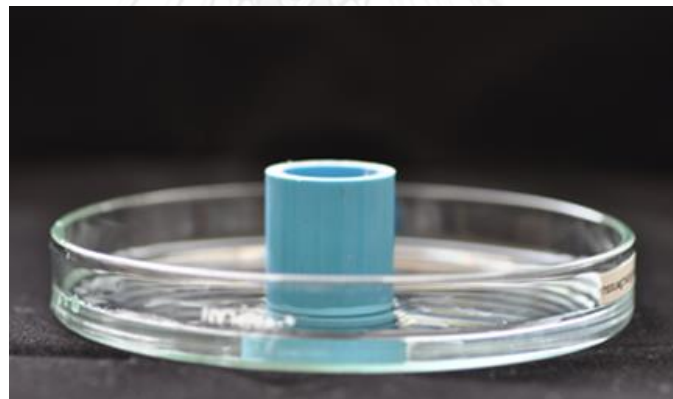


Figure 5 Air abrasion with  $\text{Al}_2\text{O}_3$  on zirconia surface a) sticker punched hole diameter 4 mm attached to zirconia before air abrasion b) zirconia after air abrasion with  $\text{Al}_2\text{O}_3$

## 2. Saliva contamination

Stimulated saliva is collected for 5 min between 10.00 and 11.00 a.m. by habitual chewing paraffin. Saliva was collected from one healthy author who had refrained from eating and drinking 1.5 hours prior to the collection procedure and using fresh saliva collected on the same occasion.<sup>[9-11, 14, 15]</sup> The specimens were immersed in saliva for one minute except uncontaminated controlled group before bonding. After saliva immersion, rinsed with deionized water for 15 seconds and air dried for 15 seconds.



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Chulalongkorn University  
Figure 6 Specimen was immersed in saliva for one minute

### 3. Cleaning methods

After saliva contamination, 50 specimens of each materials were designated into five experiment groups (n=10) according to different cleaning methods. Ten specimens without contamination process were served as a control group. The specimens were cleaned according to cleaning method :

- **Deionized water** : rinsed with deionized water for 15 seconds and air dried for 15 seconds.
- **37% phosphoric acid** : etched with 37% phosphoric acid gel (Email Preparator; Vivadent AG, Schaan, Liechtenstein) for 30 seconds then rinse with deionized water for 30 seconds and air dried 15 seconds twice. <sup>[14]</sup>
- **5% hydrofluoric acid** : etched with 5% hydrofluoric acid gel (IPS Ceramic Etching gel; Ivoclar Vivadent AG, Schaan, Liechtenstein) for 20 seconds then rinsed with deionized water for 15 seconds and air dried for 15 seconds (Manufacturer's instruction)
- **Ivoclean** : applied the cleaning paste (Ivoclean; Ivoclar Vivadent AG, Schaan, Liechtenstein) for 20 seconds then rinsed with deionized water for 15 seconds and air dried for 15 seconds (Manufacturer's instruction)
- **30% Sodium silicate solution** : applied the cleaning solution for 20 seconds then rinse with deionized water for 15 seconds and air dried for 15 seconds.

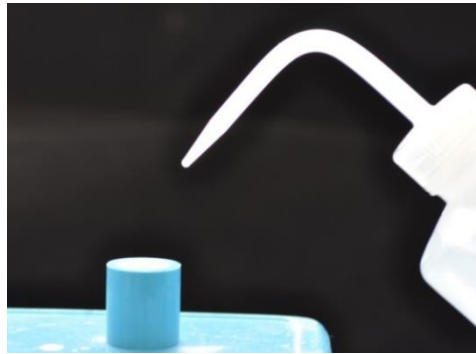


Figure 7 Contaminated specimen cleaned with deionized water

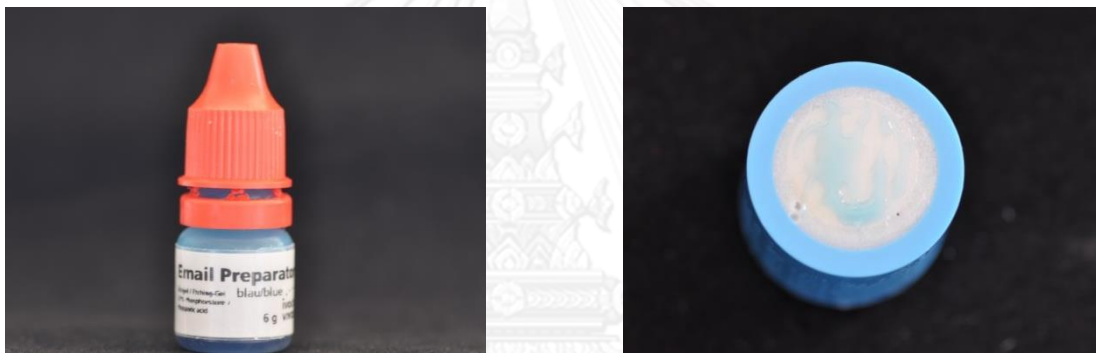


Figure 8 Contaminated specimen cleaned with 37% phosphoric acid

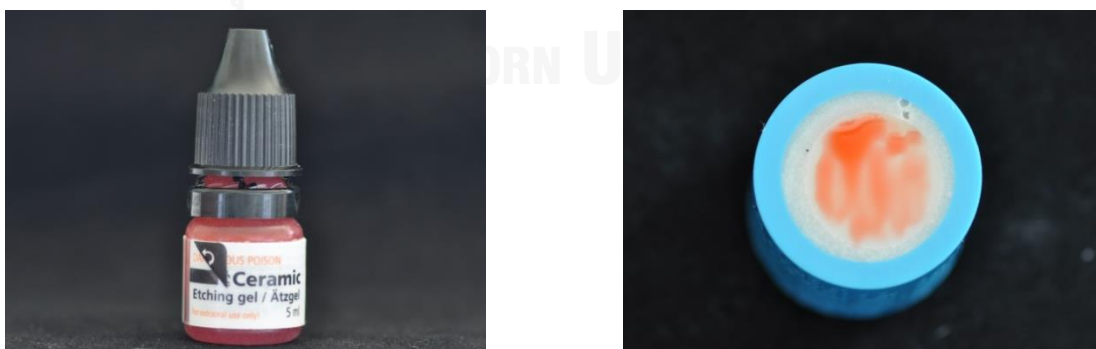


Figure 9 Contaminated specimen cleaned with 5% hydrofluoric acid



Figure 10 Contaminated specimen cleaned with Ivoclean



Figure 11 Contaminated specimen cleaned with 30% sodium silicate solution

#### 4. Shear bond strength testing (SBS)

Silane solution (Monobond-S; Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied on lithium disilicate ceramic specimens for both control group and experimental groups and left dry for 1 minute. Cylindrical dual-cured composite resin blocks (Clearfil DC core automix; Kuraray Medical Inc., Osaka, Japan), 3 ×3 mm., were prepared by using 600 mw/cm<sup>2</sup> light-activated (Elipar™ S10 LED Curing Light; 3M ESPE, St. Paul, MN, USA). Resin cements (Super bond C&B; Sun Medical CO., Shiga, Japan), Panavia F2.0 and Oxyguard II; (Kuraray Medical Inc., Osaka, Japan) and RelyX



U200;(3M ESPE, St. Paul, MN, USA)) were used to bond composite resin block to lithium disilicate ceramics and zirconia surface using alignment apparatus under a static load of 1000 gram.<sup>[54]</sup> After excess cement removal, the specimens were light-activated using (Elipar™ S10 LED Curing Light; 3M ESPE, St. Paul, MN, USA) with intensity of 600 mw/cm<sup>2</sup> for 20 seconds from two opposite sides (except Superbond C&B group, composite resin blocks were bonded to ceramic surface and left for 8 minutes). After Panavia F2.0 cement bonded specimen was light-activated, an Oxyguard II gel was applied around the bonding margins 3 minutes. The specimens were stored in 37°C water. After 24 hours storage, the shear bond strength was evaluated using a universal testing machine (EZ-S 500 N; Shimadzu, Osaka, Japan) with a cross head speed of 0.5 mm. per minute.

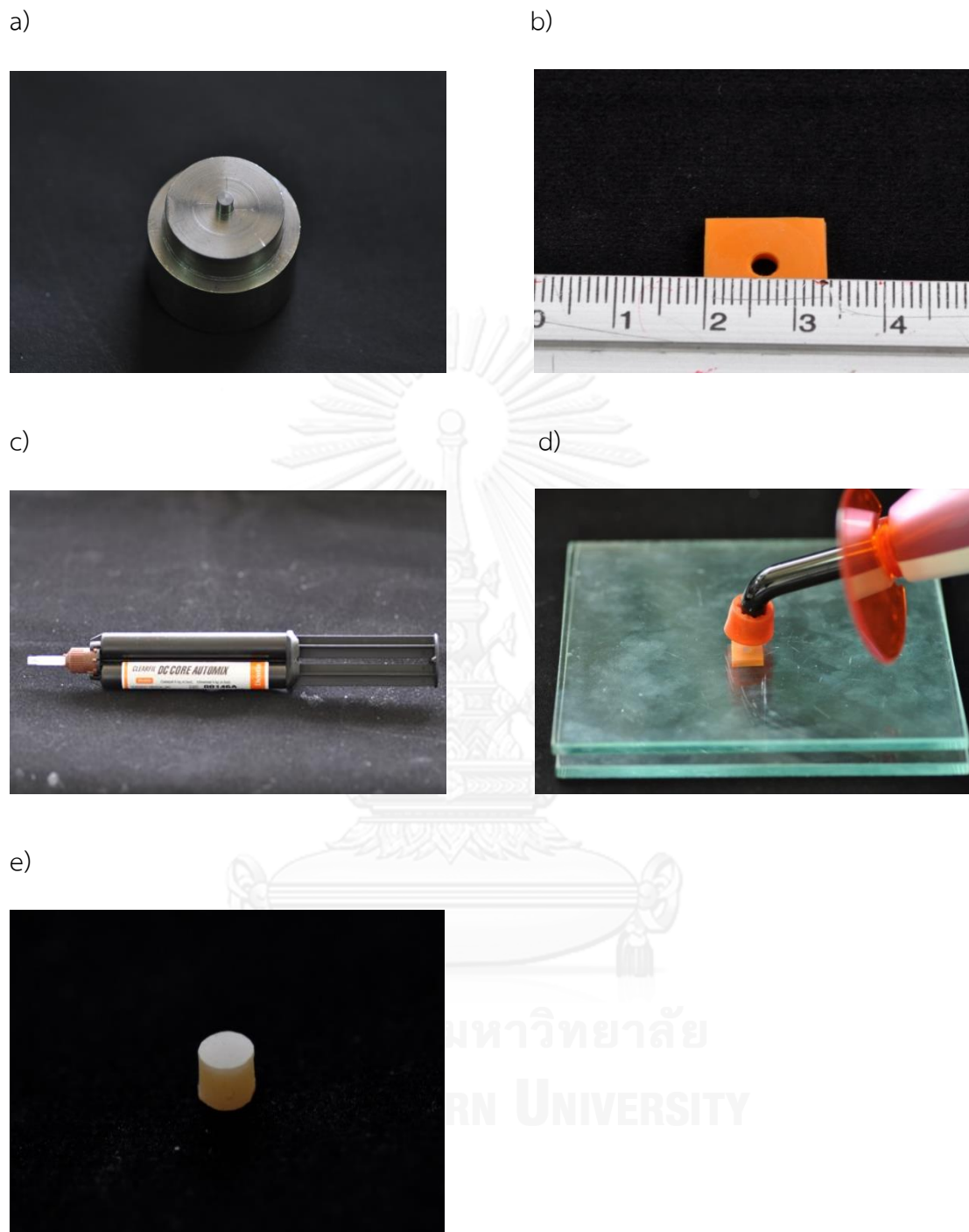


Figure 12 The procedure of making composite resin block a) stainless steel jig with diameter of 3 mm and a height of 3 mm b) silicone mold with diameter of 3 mm and a height of 3 mm was produced from stainless steel jig c) composite resin core build-up material d) technique to made composite resin block e) composite resin block with diameter of 3 mm and a height of 3 mm.



Figure 13 Bonding procedure a) stainless steel jig with diameter of 3 mm and a height of 3 mm b) acrylic jig with diameter of 3 mm and a height of 2 mm was produced from stainless steel jig c) sticker punched hole diameter 3 mm attached to ceramic after cleaning process d) acrylic jig placed on specimen aligned with the hole of sticker and then composite resin block that applied resin cement was placed on ceramic e) using alignment apparatus under a load of 1000 gram.

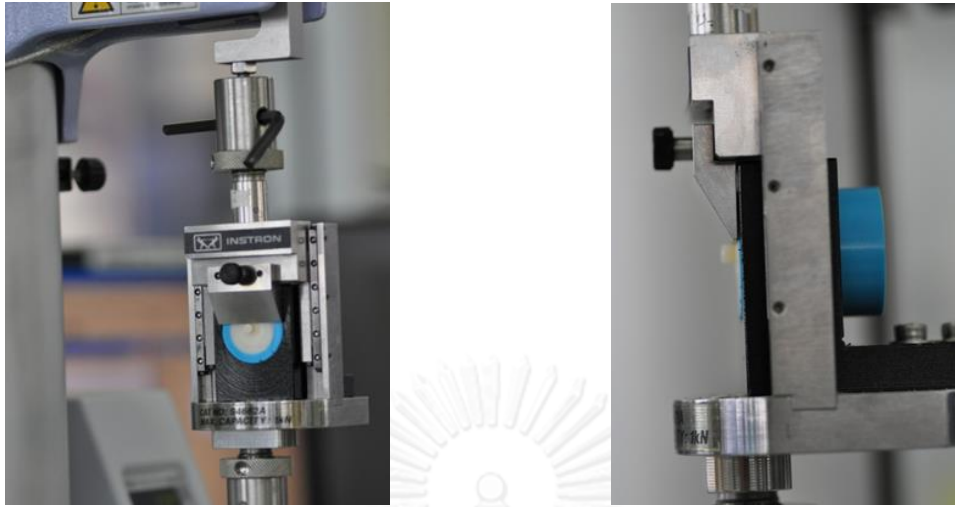


Figure 14 Shear bond strength testing by universal testing machine

## 5. Evaluation of mode of failure

After shear bond strength testing, all specimens and composite resin blocks were investigated by stereomicroscope at 30x magnification to evaluate failure mode [A, Adhesive failure between resin cement and ceramic; B, mixed failure of adhesive failure (adhesive failure between resin cement and ceramic and adhesive failure between resin cement and resin composite); C, Cohesive failure of cement; D, Adhesive failure between resin cement and composite].

## 6. Statistical analysis

One-way analysis of variance (ANOVA) followed by Tukey's HSD test and Tamhane's T2 test at a significance level of  $\alpha = 0.05$  (SPSS statistics ver.20, SAS, Cary, USA) was used to test for between-group significance of difference in mean shear bond strength.

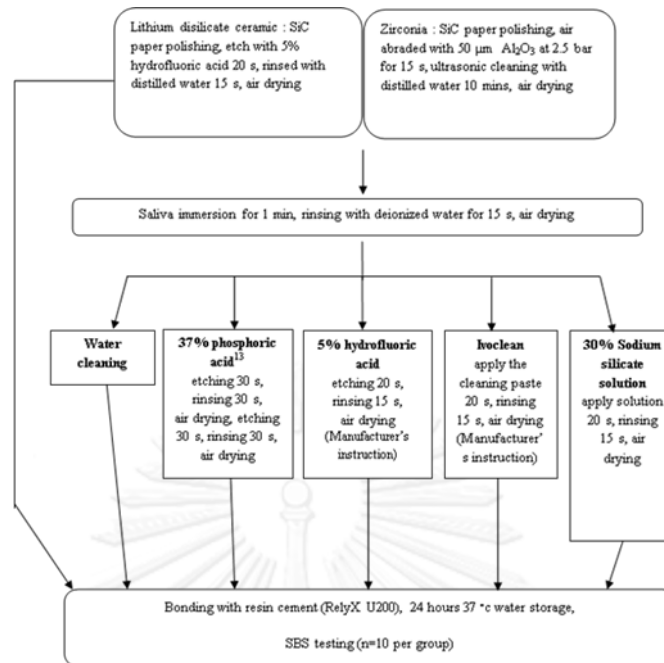


Figure 15 Study design for shear bond strength testing

Table 1 Materials and composition were used in this study

Material	Main composition <sup>a</sup>	Manufacturer
IPS e.max press (lot R59415 )	>57% SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, P <sub>2</sub> O <sub>5</sub> , ZrO <sub>2</sub> , ZnO, Al <sub>2</sub> O <sub>3</sub> , MgO, La <sub>2</sub> O <sub>3</sub> cont. ceramic	Ivoclar Vivadent, Schaan, Liechtenstein
Cercon (lot 18011411)	ZrO <sub>2</sub> (94%) , Y <sub>2</sub> O <sub>3</sub> (5%) , Al <sub>2</sub> O <sub>3</sub> (<1%), Si <sub>2</sub> O <sub>3</sub> (<1%) cont. ceramic	DeguDent, Hanau, Germany
IPS ceramic Etching-gel (lot R05327)	5% hydrofluoric acid, water cont. ceramic etching gel	Ivoclar Vivadent
Email Preparator Total Etch (lot P71444)	37% phosphoric acid, water cont. gel	Ivoclar Vivadent
Ivoclean (lot P75582)	10-15% ZrO <sub>2</sub> , NaOH, polyethylene glycol, water cont.	Ivoclar Vivadent
Sodium silicate solution	30% sodium silicate powder, deionized water cont.	
Clearfil DC core automix (lot 00146A)	BisGMA, TEGDMA, Hydrophobic aromatic dimethacrylate	Kuraray Medical, Osaka, Japan
Monobond-S (lot R03109)	Ethanol, water, silane cont. bonding agent	Ivoclar Vivadent
RelyX U200 (lot 450831)	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, alkaline fillers, silanated fillers	3M ESPE, St. Paul, MN, USA
Panavia F2.0 (lot 051381)	Paste A : MDP, DMA, BPEDEMA Paste B : Ba-B-Si-glass, silica cont. composite	Kuraray Medical
Superbond C&B (lot GG1)	Oxyguard II : Polyethyleneglycol, glycerin gel PMMA, 4-META/MMA monomer, TBB catalyst	Sun Medical CO., Shiga, Japan
BisGMA, bisphenol-A-diglycidylmethacrylate; TEGDMA, triethyleneglycol dimethacrylate; MDP, 10-methacryloyloxydecyl dihydrogenphosphate; DMA, aliphatic dimethacrylate; BPEDEMA, bisphenol-A-polyethoxy dimethacrylate; Li, lithium; K, potassium; P, phosphorus; Zn, zinc; Mg, magnesium; La, lanthanum; Al, aluminium; B, boron; Ba, barium; Si, silicium; Zr, zirconium; Y, yttrium; cont., containing.		
<sup>a</sup> According to the information provided by the manufacturers		

## CHAPTER IV

### RESULTS

#### 1. Shear bond strength of saliva contaminated lithium disilicate ceramic

For RelyX U200 group, decontaminated lithium disilicate ceramics with Ivoclean or 30% sodium silicate solution yielded the highest shear bond strength, which was not significantly different from control group. When using 37% phosphoric acid or 5% hydrofluoric acid as cleaning agent, shear bond strength were significantly reduced from that of control group. Whereas cleaning with only deionized water showed minimum shear bond strength. For Panavia F2.0 and Superbond C&B groups, decontaminated lithium disilicate ceramics with Ivoclean, 30% sodium silicate or 5% hydrofluoric acid demonstrated in higher shear bond strength than the other methods. Whereas cleaning with 37% phosphoric acid or deionized water showed minimum shear bond strength. Results are shown in Table 2 and Figure 16.

#### 2. Shear bond strength of saliva contaminated zirconia

For RelyX U200 group, decontaminated zirconia with Ivoclean or 30% sodium silicate solution yielded the highest shear bond strength, which was not significantly different from control group. When using 37% phosphoric acid or deionized water as cleaning agent, shear bond strength were significantly reduced from that of control group, whereas cleaning with 5% hydrofluoric acid showed minimum shear bond strength. For Panavia F2.0 group, decontaminated zirconia with Ivoclean and 30% sodium silicate solution yielded higher shear bond strength than the other methods. When using 37% phosphoric acid or deionized water as cleaning agent, shear bond strength were significantly reduced from that of Ivoclean and 30% sodium silicate solution groups, whereas cleaning with 5% hydrofluoric acid showed minimum shear bond strength. For Superbond C&B group, decontaminated zirconia with Ivoclean yielded the highest shear bond strength, which was not significantly different from control group. When using 30% sodium silicate solution and 37%

phosphoric acid as cleaning agent, shear bond strength were significantly reduced from that of control group, whereas cleaning with deionized water or 5% hydrofluoric acid showed minimum shear bond strength. Results are shown in Table 3 and Figure 17.



Table 2 Shear bond strength of saliva contaminated lithium disilicate ceramic (MPa  $\pm$ SD).

Cements	Cleaning agents					
	Uncontamination	Deionized water	37% phosphoric acid	5% hydrofluoric acid	Ivoclean	30% sodium silicate
RelyX U200	22.82 $\pm$ 2.22***	10.22 $\pm$ 1.36*	16.87 $\pm$ 2.45**	19.07 $\pm$ 1.84**	24.10 $\pm$ 2.98***	24.40 $\pm$ 3.80***
Panavia F2.0	14.61 $\pm$ 2.92***	7.59 $\pm$ 1.54*	8.43 $\pm$ 0.83*	10.08 $\pm$ 1.13**	11.96 $\pm$ 1.22**	10.76 $\pm$ 1.38**
Superbond C&B	26.50 $\pm$ 2.81***	18.75 $\pm$ 1.16**	15.15 $\pm$ 2.29*	27.98 $\pm$ 2.28***	27.41 $\pm$ 2.61***	28.72 $\pm$ 1.58***

n=10 for each group. Difference asterisk symbols denoted statistically significant differences at  $p < 0.05$ .

Table 3 Shear bond strength of saliva contaminated zirconia (MPa  $\pm$ SD).

Cements	Cleaning agents					
	Uncontamination	Deionized water	37% phosphoric acid	5% hydrofluoric acid	Ivoclean	30% sodium silicate
RelyX U200	9.56 $\pm$ 1.03***	7.11 $\pm$ 1.33**	7.88 $\pm$ 1.33**	5.63 $\pm$ 0.80*	10.71 $\pm$ 1.57***	9.24 $\pm$ 1.53***
Panavia F2.0	12.77 $\pm$ 1.16***	7.19 $\pm$ 0.85**	8.22 $\pm$ 0.55**	6.41 $\pm$ 1.14*	10.55 $\pm$ 1.22***	10.06 $\pm$ 1.73***
Superbond C&B	26.09 $\pm$ 1.73***	15.15 $\pm$ 2.14**	18.11 $\pm$ 1.60*	14.64 $\pm$ 1.98*	27.06 $\pm$ 2.17***	20.12 $\pm$ 2.07***

n=10 for each group. Difference asterisk symbols denoted statistically significant differences at  $p < 0.05$ .



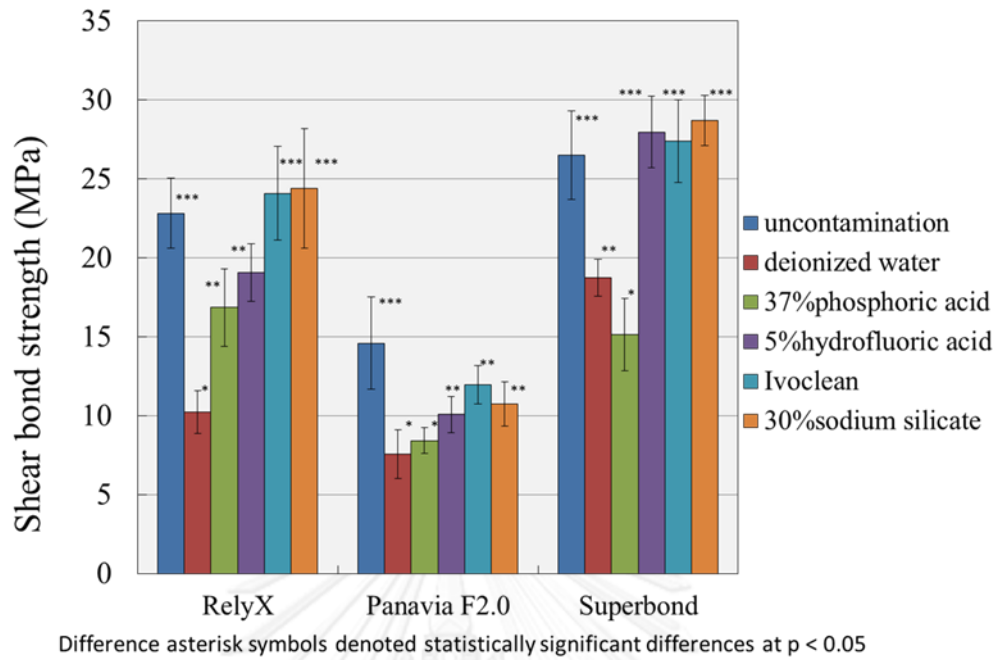


Figure 16 Shear bond strength of saliva contaminated lithium disilicate ceramic

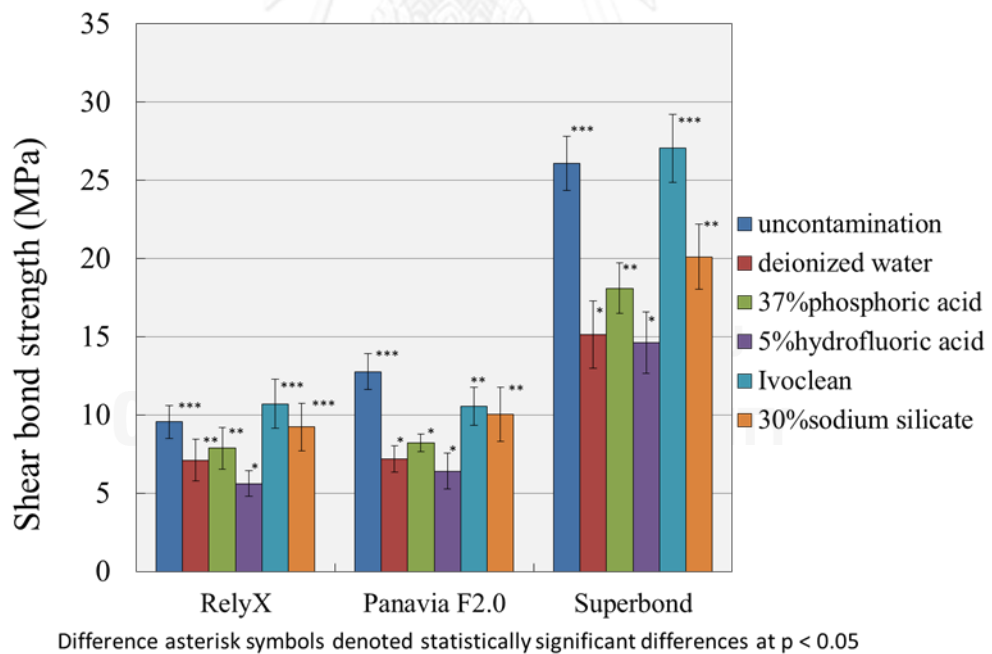


Figure 17 Shear bond strength of saliva contaminated zirconia

## Mode of failure

Almost saliva-contaminated lithium disilicate ceramic and zirconia specimens demonstrated adhesive failure between resin cement and ceramic surface more than mixed mode of failure between two interfaces (adhesive failure on ceramic surface and adhesive failure on resin composite) for all cleaning methods and resin cements. Some saliva contaminated lithium disilicate ceramic demonstrated mixed mode of failure between two interfaces more than saliva contaminated zirconia. The failure of saliva-contaminated lithium disilicate ceramics and zirconia cleaned with Ivoclean and 30% sodium silicate solution demonstrated mixed failure of adhesive failure more than the other methods for all resin cements. Results are shown in Table 4 and Table 5.

Table 4 Mode of failure of lithium disilicate ceramic

Cements	Cleaning agents	Number of specimens	Failure mode	
			A	B
RelyX U200	Uncontamination	10	8	2
	Water	10	9	1
	37% phosphoric acid	10	10	0
	5% hydrofluoric acid	10	10	0
	Ivoclean	10	8	2
	30% sodium silicate	10	4	6
Panavia F2.0	Uncontamination	10	5	5
	Water	10	7	3
	37% phosphoric acid	10	5	5
	5% hydrofluoric acid	10	5	5
	Ivoclean	10	5	5
	30% sodium silicate	10	5	5
Superbond C&B	Uncontamination	10	6	4
	Water	10	8	2
	37% phosphoric acid	10	6	4
	5% hydrofluoric acid	10	6	4
	Ivoclean	10	5	5
	30% sodium silicate	10	5	5
A, Adhesive failure between resin cement and ceramic; B, mixed failure of adhesive failure (adhesive failure between resin cement and ceramic, adhesive failure between resin cement and composite)				

Table 5 Mode of failure of zirconia

Cements	Cleaning agents	Number of specimens	Failure mode	
			A	B
RelyX U200	Uncontamination	10	9	1
	Water	10	9	1
	37% phosphoric acid	10	8	2
	5% hydrofluoric acid	10	10	0
	Ivoclean	10	9	1
	30% sodium silicate	10	8	2
Panavia F2.0	Uncontamination	10	3	7
	Water	10	7	3
	37% phosphoric acid	10	8	2
	5% hydrofluoric acid	10	9	1
	Ivoclean	10	5	5
	30% sodium silicate	10	6	4
Superbond C&B	Uncontamination	10	6	4
	Water	10	7	3
	37% phosphoric acid	10	7	3
	5% hydrofluoric acid	10	10	0
	Ivoclean	10	5	5
	30% sodium silicate	10	7	3
A, Adhesive failure between resin cement and ceramic; B, mixed failure of adhesive failure (adhesive failure between resin cement and ceramic, adhesive failure between resin cement and composite)				

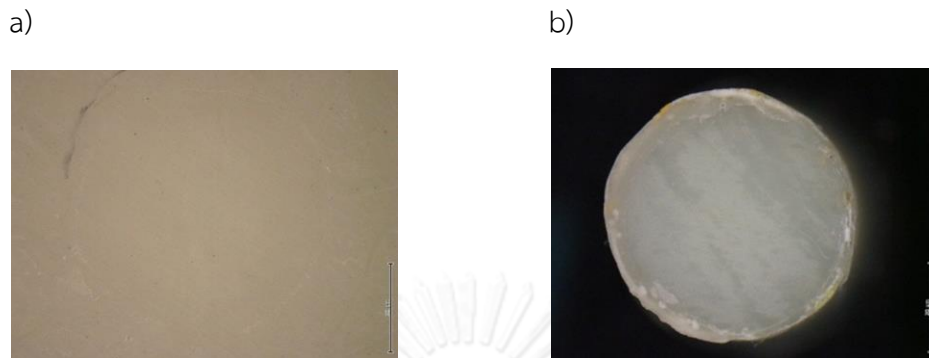


Figure 18 Stereomicroscope micrograph of interface of fractured surface demonstrates adhesive failure between resin cement and lithium disilicate ceramic. a) lithium disilicate bonding surface b) resin composite bonding surface.

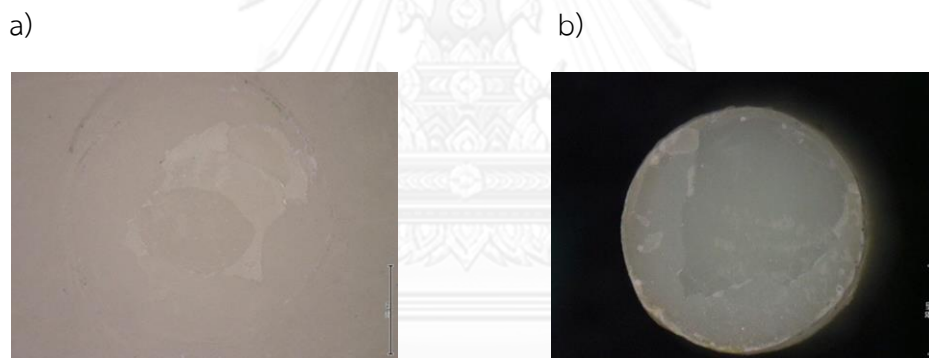


Figure 19 Stereomicroscope micrograph of interface of fractured surface demonstrates mixed failure of adhesive failure of lithium disilicate ceramic (adhesive failure between resin cement and ceramic and adhesive failure between resin cement and resin composite). a) lithium disilicate bonding surface b) resin composite bonding surface.

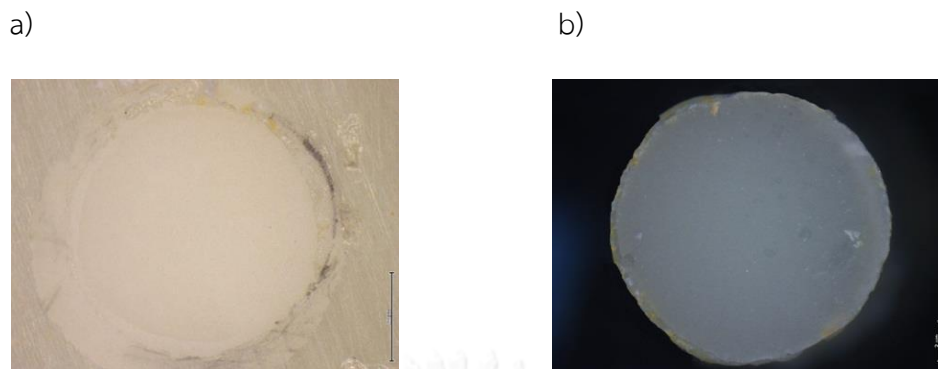


Figure 20 Stereomicroscope micrograph of interface of fractured surface demonstrates adhesive failure between resin cement and zirconia. a) zirconia bonding surface b) resin composite bonding surface.

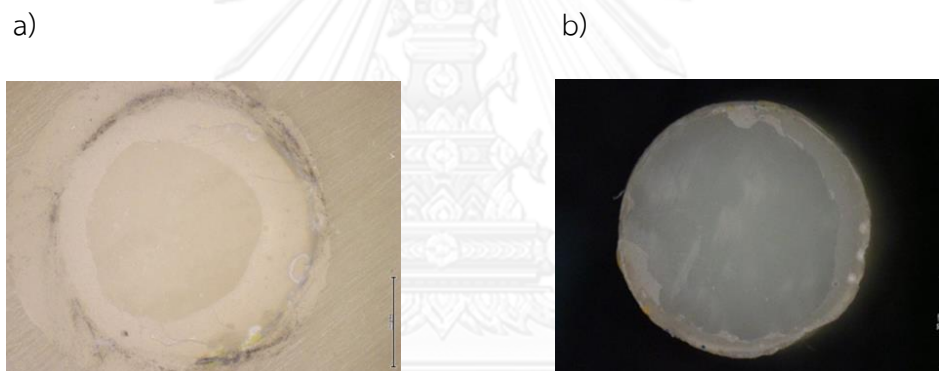


Figure 21 Stereomicroscope micrograph of interface of fractured surface demonstrates mixed failure of adhesive failure of zirconia (adhesive failure between resin cement and ceramic and adhesive failure between resin cement and resin composite). a) zirconia bonding surface b) resin composite bonding surface.

## CHAPTER V

### DISCUSSION AND CONCLUSION

#### DISCUSSION

In this investigation, the different cleaning methods influence shear bond strength to saliva contaminated lithium disilicate ceramic and zirconia surface. Therefore, the proposed null-hypothesis that there was no difference in the effect of different cleaning agents to shear bond strength of saliva contaminated lithium disilicate ceramic was rejected. The second null-hypothesis that there was no difference in the effect of different cleaning agents to shear bond strength of saliva contaminated zirconia was also rejected.

In this study, five different methods were used in an attempt to remove the saliva coating on lithium disilicate ceramic and zirconia. The uncontaminated surfaces were used as the control groups. The results showed that water rinsing alone was not sufficient in removing saliva coating, as the shear bond strength was significantly decreased in both lithium disilicate ceramic and zirconia groups for all resin cements, which was in agreement with previous reports.<sup>[9-11]</sup> The shear bond strength of the lithium disilicate ceramics cleaned with 37% phosphoric acid was lower than using 5% hydrofluoric acid, Ivoclean or 30% sodium silicate solution as cleaning agents. Thirty-seven percent of phosphoric acid showed similar shear bond strength as 5% hydrofluoric acid for RelyX U200 group. Therefore, phosphoric acid demonstrated slightly cleaning effects on saliva-contaminated lithium disilicate ceramic surfaces. This might attribute to their acidic property, which can remove some organic residues.<sup>[10, 11, 14, 15]</sup> Phosphoric acid can penetrate through saliva film into the lithium disilicate ceramic surface underneath, thereby removing the film.<sup>[4]</sup> However, the result showed that phosphoric acid was not sufficient in removing saliva contaminated lithium disilicate ceramic because some phosphoric acid gel might be remained on the ceramic surface, which decreased surface energy, wettability and silane coupling activity.<sup>[10, 49]</sup> While the shear bond strength of the lithium disilicate

ceramics cleaned with 5% hydrofluoric acid was higher than the 37% phosphoric acid and water-rinsing groups for all resin cement groups. This result might be that 5% hydrofluoric acid was effective in removing saliva from lithium disilicate ceramic. Due to its acidic property, which can resolve organic residues.<sup>[10, 11, 14, 15]</sup> Hydrofluoric acid re-etching of lithium disilicate ceramic provided more surface roughness, which might cause better resin penetration, mechanical interlocking and hence increased the bond strength. This was contrary to the previous study which found that 5% hydrofluoric acid re-etching on lithium disilicate ceramic surface after saliva contamination did not affect the bonding surface, even increasing the etching time up to 40 seconds.<sup>[9]</sup> However, mechanical properties of lithium disilicate ceramic after hydrofluoric acid re-etching should be further investigated.

In addition, 5% hydrofluoric acid had no effect on saliva-contaminated zirconia. The result showed significantly low shear bond strength for all resin cement groups. Five percent hydrofluoric acid could not break the zirconium-phosphate bond and remove phosphate from zirconia surface. Moreover, hydrofluoric acid etch did not make any changes to the surface morphology of zirconia.<sup>[55, 56]</sup> While the shear bond strength of the zirconia cleaned with water or 37% phosphoric acid was higher than 5% hydrofluoric acid, but still lower than the zirconia using Ivoclean or 30% sodium silicate solution as cleaning agents for all resin cement groups. This demonstrated that only water and phosphoric acid could not remove saliva from zirconia. Phosphoric acid might decrease surface energy at activated zirconia surface.<sup>[10, 15, 57]</sup> It might remain on zirconia surface due to the durable bond between phosphate group and zirconium oxide. In addition, residual phosphorus might also influence bonding.<sup>[57, 58]</sup> Therefore, conventional 5% hydrofluoric acid and 37% phosphoric acid etching have no positive influence on the resin bond to zirconia.<sup>[2]</sup>

This present study demonstrated that Ivoclean and sodium silicate solution were the most effective cleaning agents on decontaminate saliva-contaminated lithium disilicate ceramic and zirconia surfaces. While saliva contaminated zirconia cleaned with 30% sodium silicate solution using Superbond C&B group showed lower shear bond strength than Ivoclean. Ivoclean is an alkali paste and its major

composition is zirconium oxide particle. Alkalinity is effective in removing proteins, oil substances and contaminations. Previous work suggested that alkaline cleaning process could optimize adhesive bonding.<sup>[59]</sup> Zirconium oxide particles can interact strongly with phosphate group<sup>[12]</sup>, causing the removal of saliva phosphate from ceramic surfaces. The high concentration of zirconium oxide particles in the Ivoclean, act as a sponge and bind to the phosphate groups.<sup>[51]</sup> In addition, sodium hydroxide in Ivoclean might increase the presence of hydroxyl groups on zirconia surface. This favored acid-base reaction between metal oxides on zirconia surface with resin cement and might increase surface energy and increase the wettability of zirconia surface.<sup>[60]</sup> Sodium silicate solution is also basic, so it can be used as alkaline cleaning agent. Sodium silicate solution can be easily rinsed off from ceramic surface. Moreover, with lower pH than Ivoclean, it might be considered more safety to be used as an alternative cleaning agent.

Almost saliva contaminated lithium disilicate ceramic and zirconia demonstrated adhesive failure between resin cement and ceramic surfaces for all cleaning methods. This can be assumed that almost the bonding failure occur on resin cement-ceramic interface. The resin cement-ceramic bonding was important for durability of all-ceramic restoration. The failure of saliva-contaminated lithium disilicate ceramics demonstrated mixed failure of adhesive failure more than zirconia. Therefore, lithium disilicate ceramic might be effective in bonding with resin cements more than zirconia, which might cause better resin penetration, mechanical interlocking from surface treatment and silane activity than zirconia. The failure of saliva-contaminated lithium disilicate ceramics and zirconia cleaned with Ivoclean and 30% sodium silicate solution demonstrated mixed failure of adhesive failure more than the other methods for all resin cements. Therefore, sodium silicate solution might be an effective cleaning agent in removing saliva from lithium disilicate surface and enhance resin cement bonding to lithium disilicate ceramics. This corresponded to the highest shear bond strength value of saliva contaminated lithium disilicate ceramics and zirconia in Ivoclean and 30% sodium silicate groups. However, further study on Ivoclean and sodium silicate is recommended.



## CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn.

1. Saliva contaminated lithium disilicate ceramic and zirconia decreased the shear bond strength between resin cement and ceramic.
2. Ivoclean and 30% sodium silicate solution were effective in decontaminating the saliva from lithium disilicate ceramic. Ivoclean was the most effective in decontaminating the saliva from zirconia.
3. Five percent of hydrofluoric acid was moderate effective in decontaminating the saliva from lithium disilicate ceramic but not zirconia.
4. Thirty-seven percent of phosphoric acid was not sufficient in decontaminating the saliva from lithium disilicate ceramic and zirconia similar to water-rinsing.

### Clinical implication

Saliva contamination statistically significant reduced bond strengths of resin cements bonded to ceramics. Ivoclean and 30% sodium silicate solution were effective in decontaminating the saliva from lithium disilicate ceramic than the other methods for all resin cements. Ivoclean was the most effective in decontaminate the saliva from zirconia than the other methods for all resin cements.

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APPENDIX

จุฬาลงกรณ์มหาวิทยาลัย  
**CHULALONGKORN UNIVERSITY**

Statistical analysis for shear bond strength test of contaminated lithium disilicate ceramic

RelyX U200 cement

One-Sample Kolmogorov-Smirnov Test

cleaning		shear	
uncontaminati on	N	10	
	Normal Parameters <sup>a,b</sup>	Mean	22.81749
		Std. Deviation	2.215335
		Most Extreme Differences	Absolute
	Positive		.153
	Negative		-.176
	Kolmogorov-Smirnov Z	.555	
	Asymp. Sig. (2-tailed)	.917	
deionized water	N	10	
	Normal Parameters <sup>a,b</sup>	Mean	10.22499
		Std. Deviation	1.357305
		Most Extreme Differences	Absolute
	Positive		.204
	Negative		-.140
	Kolmogorov-Smirnov Z	.644	
	Asymp. Sig. (2-tailed)	.801	
phosphoric acid	N	10	
	Normal Parameters <sup>a,b</sup>	Mean	16.86508
		Std. Deviation	2.453017



hydrofluoric acid	Most Extreme Differences	Absolute	.154	
		Positive	.154	
		Negative	-.139	
	Kolmogorov-Smirnov Z		.488	
	Asymp. Sig. (2-tailed)		.971	
	N		10	
	Normal Parameters <sup>a,b</sup>	Mean		19.07121
		Std. Deviation		1.842359
	Ivoclean	Most Extreme Differences	Absolute	.221
		Positive	.141	
		Negative	-.221	
Kolmogorov-Smirnov Z			.698	
Asymp. Sig. (2-tailed)			.715	
N			10	
Normal Parameters <sup>a,b</sup>		Mean		24.09882
		Std. Deviation		2.977205
sodium silicate N		Most Extreme Differences	Absolute	.213
		Positive	.124	
		Negative	-.213	
	Kolmogorov-Smirnov Z		.672	
	Asymp. Sig. (2-tailed)		.757	
	N		10	
	Normal Parameters <sup>a,b</sup>	Mean		24.40441
		Std. Deviation		3.799439
	Most Extreme	Absolute	.150	

Differences	Positive	.150
	Negative	-.146
Kolmogorov-Smirnov Z		.474
Asymp. Sig. (2-tailed)		.978

a. Test distribution is Normal.

b. Calculated from data.



Panavia F2.0

## One-Sample Kolmogorov-Smirnov Test

cleaning		shear
uncontaminati on	N	10
	Normal Parameters <sup>a,b</sup>	
	Mean	14.60863
	Std. Deviation	2.923493
	Most Extreme Differences	Absolute Positive Negative
		.159 .159 -.133
	Kolmogorov-Smirnov Z	.504
	Asymp. Sig. (2-tailed)	.961
deionized water	N	10
	Normal Parameters <sup>a,b</sup>	
	Mean	7.59371
	Std. Deviation	1.536486
	Most Extreme Differences	Absolute Positive Negative
		.132 .113 -.132
	Kolmogorov-Smirnov Z	.418
	Asymp. Sig. (2-tailed)	.995
phosphoric acid	N	10
	Normal Parameters <sup>a,b</sup>	
	Mean	8.42546
	Std. Deviation	.827442
	Most Extreme Differences	Absolute Positive
		.191 .191

		Negative	-.109
	Kolmogorov-Smirnov Z		.603
	Asymp. Sig. (2-tailed)		.860
hydrofluoric acid	N		10
	Normal Parameters <sup>a,b</sup>	Mean	10.08243
		Std. Deviation	1.131997
	Most Extreme Differences	Absolute	.190
		Positive	.183
		Negative	-.190
	Kolmogorov-Smirnov Z		.600
	Asymp. Sig. (2-tailed)		.864
lvoclean	N		10
	Normal Parameters <sup>a,b</sup>	Mean	11.95873
		Std. Deviation	1.216175
	Most Extreme Differences	Absolute	.195
		Positive	.126
		Negative	-.195
	Kolmogorov-Smirnov Z		.617
	Asymp. Sig. (2-tailed)		.841
sodium silicate	N		10
	Normal Parameters <sup>a,b</sup>	Mean	10.75568
		Std. Deviation	1.383673
	Most Extreme Differences	Absolute	.150
		Positive	.089
		Negative	-.150

Kolmogorov-Smirnov Z	.475
Asymp. Sig. (2-tailed)	.978

a. Test distribution is Normal.

b. Calculated from data.



## Superbond C&amp;B cement

## One-Sample Kolmogorov-Smirnov Test

cleaning			shear
uncontaminati on	N		10
	Normal Parameters <sup>a,b</sup>	Mean	26.49611
		Std. Deviation	2.813681
	Most Extreme Differences	Absolute Positive	.256 .256
		Negative	-.181
	Kolmogorov-Smirnov Z		.809
	Asymp. Sig. (2-tailed)		.530
deionized water	N		10
	Normal Parameters <sup>a,b</sup>	Mean	18.74501
		Std. Deviation	1.164431
	Most Extreme Differences	Absolute Positive	.127 .127
		Negative	-.108
	Kolmogorov-Smirnov Z		.400
	Asymp. Sig. (2-tailed)		.997
phosphoric acid	N		10
	Normal Parameters <sup>a,b</sup>	Mean	15.15118
		Std. Deviation	2.285546
	Most Extreme Differences	Absolute Positive	.210 .136

		Negative	-.210
	Kolmogorov-Smirnov Z		.664
	Asymp. Sig. (2-tailed)		.769
hydrofluoric acid	N		10
	Normal Parameters <sup>a,b</sup>	Mean	27.98401
		Std. Deviation	2.275393
	Most Extreme Differences	Absolute	.228
		Positive	.135
		Negative	-.228
	Kolmogorov-Smirnov Z		.720
	Asymp. Sig. (2-tailed)		.678
lvoclean	N		10
	Normal Parameters <sup>a,b</sup>	Mean	27.40553
		Std. Deviation	2.612135
	Most Extreme Differences	Absolute	.162
		Positive	.162
		Negative	-.134
	Kolmogorov-Smirnov Z		.513
	Asymp. Sig. (2-tailed)		.955
sodium silicate	N		10
	Normal Parameters <sup>a,b</sup>	Mean	28.71608
		Std. Deviation	1.575415
	Most Extreme Differences	Absolute	.128
		Positive	.128
		Negative	-.118

Kolmogorov-Smirnov Z	.405
Asymp. Sig. (2-tailed)	.997

a. Test distribution is Normal.

b. Calculated from data.





Statistical analysis for shear bond strength test of contaminated zirconia  
RelyX U200 cement

One-Sample Kolmogorov-Smirnov Test

cleaning	shear
uncontaminatioN	10
n	
Normal Parameters <sup>a,b</sup>	
Mean	9.56489
Std. Deviation	1.026009
Most Extreme Differences	
Absolute	.167
Positive	.150
Negative	-.167
Kolmogorov-Smirnov Z	.529
Asymp. Sig. (2-tailed)	.942
deionized waterN	10
Normal Parameters <sup>a,b</sup>	
Mean	7.11222
Std. Deviation	1.329353
Most Extreme Differences	
Absolute	.151
Positive	.106
Negative	-.151
Kolmogorov-Smirnov Z	.479
Asymp. Sig. (2-tailed)	.976
phosphoric acidN	10
Normal Parameters <sup>a,b</sup>	
Mean	7.87783
Std. Deviation	1.328828
Most Extreme	
Absolute	.177

hydrofluoric acid	Differences	Positive	.167	
		Negative	-.177	
	Kolmogorov-Smirnov Z		.559	
	Asymp. Sig. (2-tailed)		.913	
	N		10	
	Normal Parameters <sup>a,b</sup>	Mean	5.62985	
		Std. Deviation	.795384	
		Most Extreme Differences	Absolute Positive Negative	.145 .145 -.110
	Kolmogorov-Smirnov Z		.457	
	Asymp. Sig. (2-tailed)		.985	
Ivoclean	N		10	
	Normal Parameters <sup>a,b</sup>	Mean	10.71032	
		Std. Deviation	1.570802	
		Most Extreme Differences	Absolute Positive Negative	.178 .178 -.127
	Kolmogorov-Smirnov Z		.564	
	Asymp. Sig. (2-tailed)		.908	
	sodium silicate	N		10
		Normal Parameters <sup>a,b</sup>	Mean	9.23937
			Std. Deviation	1.525089
			Most Extreme Differences	Absolute Positive

	Negative	.180
	Kolmogorov-Smirnov Z	.640
	Asymp. Sig. (2-tailed)	.808

a. Test distribution is Normal.

b. Calculated from data.



Panavia F2.0 cement

## One-Sample Kolmogorov-Smirnov Test

cleaning	shear
uncontaminatioN	10
n	
Normal Parameters <sup>a,b</sup>	
Mean	12.77300
Std. Deviation	1.159739
Most Extreme Differences	
Absolute	.220
Positive	.165
Negative	-.220
Kolmogorov-Smirnov Z	.697
Asymp. Sig. (2-tailed)	.716
deionized waterN	10
Normal Parameters <sup>a,b</sup>	
Mean	7.18841
Std. Deviation	.846979
Most Extreme Differences	
Absolute	.147
Positive	.147
Negative	-.117
Kolmogorov-Smirnov Z	.464
Asymp. Sig. (2-tailed)	.983
phosphoric acidN	10
Normal Parameters <sup>a,b</sup>	
Mean	8.21824
Std. Deviation	.547936
Most Extreme Differences	
Absolute	.197
Positive	.197

		Negative	-.127
	Kolmogorov-Smirnov Z		.622
	Asymp. Sig. (2-tailed)		.834
hydrofluoric acid	N		10
	Normal Parameters <sup>a,b</sup>	Mean	6.40535
		Std. Deviation	1.144051
	Most Extreme Differences	Absolute	.141
		Positive	.141
		Negative	-.118
	Kolmogorov-Smirnov Z		.446
	Asymp. Sig. (2-tailed)		.989
Ivoclean	N		10
	Normal Parameters <sup>a,b</sup>	Mean	10.54622
		Std. Deviation	1.224618
	Most Extreme Differences	Absolute	.204
		Positive	.204
		Negative	-.157
	Kolmogorov-Smirnov Z		.646
	Asymp. Sig. (2-tailed)		.799
sodium silicate	N		10
	Normal Parameters <sup>a,b</sup>	Mean	10.05614
		Std. Deviation	1.729751
	Most Extreme Differences	Absolute	.260
		Positive	.260
		Negative	-.172

Kolmogorov-Smirnov Z	.823
Asymp. Sig. (2-tailed)	.507

- a. Test distribution is Normal.
- b. Calculated from data.



## Superbond C&amp;B cement

## One-Sample Kolmogorov-Smirnov Test

cleaning	shear
uncontaminatioN	10
n	
Normal Parameters <sup>a,b</sup>	
Mean	26.09419
Std. Deviation	1.726218
Most Extreme Differences	
Absolute	.154
Positive	.122
Negative	-.154
Kolmogorov-Smirnov Z	.488
Asymp. Sig. (2-tailed)	.971
deionized waterN	10
Normal Parameters <sup>a,b</sup>	
Mean	15.14512
Std. Deviation	2.143195
Most Extreme Differences	
Absolute	.134
Positive	.134
Negative	-.130
Kolmogorov-Smirnov Z	.424
Asymp. Sig. (2-tailed)	.994
phosphoric acidN	10
Normal Parameters <sup>a,b</sup>	
Mean	18.10883
Std. Deviation	1.603070
Most Extreme Differences	
Absolute	.224
Positive	.189

		Negative	-.224
		Kolmogorov-Smirnov Z	.708
		Asymp. Sig. (2-tailed)	.698
hydrofluoric acid	N		10
	Normal Parameters <sup>a,b</sup>	Mean	14.64366
		Std. Deviation	1.979404
	Most Extreme Differences	Absolute	.165
		Positive	.165
		Negative	-.134
		Kolmogorov-Smirnov Z	.522
		Asymp. Sig. (2-tailed)	.948
Ivoclean	N		10
	Normal Parameters <sup>a,b</sup>	Mean	27.05959
		Std. Deviation	2.173663
	Most Extreme Differences	Absolute	.200
		Positive	.200
		Negative	-.134
		Kolmogorov-Smirnov Z	.633
		Asymp. Sig. (2-tailed)	.818
sodium silicate	N		10
	Normal Parameters <sup>a,b</sup>	Mean	20.12474
		Std. Deviation	2.066021
	Most Extreme Differences	Absolute	.142
		Positive	.142
		Negative	-.101



Kolmogorov-Smirnov Z	.450
Asymp. Sig. (2-tailed)	.987

- a. Test distribution is Normal.
- b. Calculated from data.



Saliva contaminated lithium disilicate ceramic

Oneway : RelyX U200 cement

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
1.835	5	54	.121

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1493.219	5	298.644	45.408	.000
Within Groups	355.149	54	6.577		
Total	1848.368	59			

## Multiple Comparisons

Dependent Variable: shear

Tukey HSD

(I) cleaning	(J) cleaning	Mean Difference (I- J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatiodeionized n	water	12.592499 <sup>*</sup>	1.146895	.000	9.20402	15.98098
	phosphoric acid	5.952412 <sup>*</sup>	1.146895	.000	2.56393	9.34089
	hydrofluoric acid	3.746280 <sup>*</sup>	1.146895	.022	.35780	7.13476
	Ivoclean	-1.281330	1.146895	.872	-4.66981	2.10715
	sodium silicate	-1.586920	1.146895	.736	-4.97540	1.80156
deionized water	Uncontaminati on	-12.592499 <sup>*</sup>	1.146895	.000	-15.98098	-9.20402
	phosphoric acid	-6.640087 <sup>*</sup>	1.146895	.000	-10.02857	-3.25161
	hydrofluoric acid	-8.846219 <sup>*</sup>	1.146895	.000	-12.23470	-5.45774
	Ivoclean	-13.873829 <sup>*</sup>	1.146895	.000	-17.26231	-10.48535
	sodium silicate	-14.179419 <sup>*</sup>	1.146895	.000	-17.56790	-10.79094
phosphoric acid	uncontaminatio n	-5.952412 <sup>*</sup>	1.146895	.000	-9.34089	-2.56393
	deionized water	6.640087 <sup>*</sup>	1.146895	.000	3.25161	10.02857
	hydrofluoric acid	-2.206132	1.146895	.399	-5.59461	1.18235
	Ivoclean	-7.233742 <sup>*</sup>	1.146895	.000	-10.62222	-3.84526
	sodium silicate	-7.539332 <sup>*</sup>	1.146895	.000	-10.92781	-4.15085

hydrofluoric acid	uncontaminatio n	-3.746280 *	1.146895	.022	-7.13476	-3.5780
	deionized water	8.846219 *	1.146895	.000	5.45774	12.23470
	phosphoric acid	2.206132	1.146895	.399	-1.18235	5.59461
	Ivoclean	-5.027610 *	1.146895	.001	-8.41609	-1.63913
	sodium silicate	-5.333200 *	1.146895	.000	-8.72168	-1.94472
Ivoclean	uncontaminatio n	1.281330	1.146895	.872	-2.10715	4.66981
	deionized water	13.873829 *	1.146895	.000	10.48535	17.26231
	phosphoric acid	7.233742 *	1.146895	.000	3.84526	10.62222
	hydrofluoric acid	5.027610 *	1.146895	.001	1.63913	8.41609
	sodium silicate	-3.05590	1.146895	1.000	-3.69407	3.08289
sodium silicate	uncontaminatio n	1.586920	1.146895	.736	-1.80156	4.97540
	deionized water	14.179419 *	1.146895	.000	10.79094	17.56790
	phosphoric acid	7.539332 *	1.146895	.000	4.15085	10.92781
	hydrofluoric acid	5.333200 *	1.146895	.000	1.94472	8.72168
	Ivoclean	.305590	1.146895	1.000	-3.08289	3.69407

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

shear

Tukey HSD

cleaning	N	Subset for alpha = 0.05		
		1	2	3
deionized water	10	10.22499		
phosphoric acid	10		16.86508	
hydrofluoric acid	10		19.07121	
uncontaminati on	10			22.81749
Ivoclean	10			24.09882
sodium silicate	10			24.40441
Sig.		1.000	.399	.736

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.



Oneway : Panavia F2.0 cement

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
3.270	5	54	.012

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	319.686	5	63.937	23.582	.000
Within Groups	146.406	54	2.711		
Total	466.092	59			

## Robust Tests of Equality of Means

shear

	Statistic <sup>a</sup>	df1	df2	Sig.
Brown-Forsythe	23.582	5	27.506	.000

a. Asymptotically F distributed.



## Multiple Comparisons

Dependent Variable: shear

Tamhane

(I) cleaning	(J) cleaning	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatio n	deionized water	7.014917 <sup>*</sup>	1.044395	.000	3.32091	10.70892
	phosphoric acid	6.183168 <sup>*</sup>	.960805	.001	2.56337	9.80297
	hydrofluoric acid	4.526203 <sup>*</sup>	.991374	.010	.89539	8.15701
	Ivoclean	2.649896	1.001294	.276	-.98896	6.28875
	sodium silicate	3.852947 <sup>*</sup>	1.022808	.035	.19033	7.51556
deionized water	uncontaminatio n	-7.014917 <sup>*</sup>	1.044395	.000	-10.70892	-3.32091
	phosphoric acid	-.831749	.551856	.919	-2.77806	1.11457
	hydrofluoric acid	-2.488714 <sup>*</sup>	.603507	.011	-4.54906	-.42837
	Ivoclean	-4.365021 <sup>*</sup>	.619667	.000	-6.46948	-2.26057
	sodium silicate	-3.161970 <sup>*</sup>	.653861	.002	-5.36878	-.95516
phosphoric acid	uncontaminatio n	-6.183168 <sup>*</sup>	.960805	.001	-9.80297	-2.56337
	deionized water	.831749	.551856	.919	-1.11457	2.77806
	hydrofluoric acid	-1.656965 <sup>*</sup>	.443405	.025	-3.17170	-.14223
	Ivoclean	-3.533272 <sup>*</sup>	.465160	.000	-5.13253	-1.93401
	sodium silicate	-2.330221 <sup>*</sup>	.509824	.006	-4.10679	-.55366
hydrofluoric acid	uncontaminatio n	-4.526203 <sup>*</sup>	.991374	.010	-8.15701	-.89539
	deionized water	2.488714 <sup>*</sup>	.603507	.011	.42837	4.54906



	phosphoric acid	1.656965 *	.443405	.025	.14223	3.17170
	Ivoclean	-1.876307 *	.525404	.032	-3.64805	-.10456
	sodium silicate	-.673256	.565329	.987	-2.58932	1.24281
Ivoclean	uncontaminatio n	-2.649896	1.001294	.276	-6.28875	.98896
	deionized water	4.365021 *	.619667	.000	2.26057	6.46948
	phosphoric acid	3.533272 *	.465160	.000	1.93401	5.13253
	hydrofluoric acid	1.876307 *	.525404	.032	.10456	3.64805
	sodium silicate	1.203051	.582549	.564	-.76470	3.17081
sodium silicate	uncontaminatio n	-3.852947 *	1.022808	.035	-7.51556	-.19033
	deionized water	3.161970 *	.653861	.002	.95516	5.36878
	phosphoric acid	2.330221 *	.509824	.006	.55366	4.10679
	hydrofluoric acid	.673256	.565329	.987	-1.24281	2.58932
	Ivoclean	-1.203051	.582549	.564	-3.17081	.76470

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

Oneway : Superbond C&B cement

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
2.057	5	54	.085

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1618.172	5	323.634	67.007	.000
Within Groups	260.811	54	4.830		
Total	1878.983	59			

## Multiple Comparisons

Dependent Variable: shear

Tukey HSD

(I) cleaning	(J) cleaning	Mean Difference (I- J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatiodeionized n	water	7.751100 <sup>*</sup>	.982836	.000	4.84733	10.65487
	phosphoric acid	11.344930 <sup>*</sup>	.982836	.000	8.44116	14.24870
	hydrofluoric acid	-1.487900	.982836	.657	-4.39167	1.41587
	Ivoclean	-.909420	.982836	.938	-3.81319	1.99435
	sodium silicate	-2.219970	.982836	.229	-5.12374	.68380
deionized water	uncontaminatio n	-7.751100 <sup>*</sup>	.982836	.000	-10.65487	-4.84733
	phosphoric acid	3.593830 <sup>*</sup>	.982836	.007	.69006	6.49760
	hydrofluoric acid	-9.239000 <sup>*</sup>	.982836	.000	-12.14277	-6.33523
	Ivoclean	-8.660520 <sup>*</sup>	.982836	.000	-11.56429	-5.75675
	sodium silicate	-9.971070 <sup>*</sup>	.982836	.000	-12.87484	-7.06730
phosphoric acid	uncontaminatio n	-11.344930 <sup>*</sup>	.982836	.000	-14.24870	-8.44116
	deionized water	-3.593830 <sup>*</sup>	.982836	.007	-6.49760	-.69006
	hydrofluoric acid	-12.832830 <sup>*</sup>	.982836	.000	-15.73660	-9.92906
	Ivoclean	-12.254350 <sup>*</sup>	.982836	.000	-15.15812	-9.35058
	sodium silicate	-13.564900 <sup>*</sup>	.982836	.000	-16.46867	-10.66113

hydrofluoric acid	uncontaminatio n	1.487900	.982836	.657	-1.41587	4.39167
	deionized water	9.239000*	.982836	.000	6.33523	12.14277
	phosphoric acid	12.832830*	.982836	.000	9.92906	15.73660
	Ivoclean	.578480	.982836	.991	-2.32529	3.48225
	sodium silicate	-.732070	.982836	.975	-3.63584	2.17170
Ivoclean	uncontaminatio n	.909420	.982836	.938	-1.99435	3.81319
	deionized water	8.660520*	.982836	.000	5.75675	11.56429
	phosphoric acid	12.254350*	.982836	.000	9.35058	15.15812
	hydrofluoric acid	-.578480	.982836	.991	-3.48225	2.32529
	sodium silicate	-1.310550	.982836	.765	-4.21432	1.59322
sodium silicate	uncontaminatio n	2.219970	.982836	.229	-.68380	5.12374
	deionized water	9.971070*	.982836	.000	7.06730	12.87484
	phosphoric acid	13.564900*	.982836	.000	10.66113	16.46867
	hydrofluoric acid	-.732070	.982836	.975	-2.17170	3.63584
	Ivoclean	1.310550	.982836	.765	-1.59322	4.21432

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

shear

Tukey HSD

cleaning	N	Subset for alpha = 0.05		
		1	2	3
phosphoric acid	10	15.15118		
deionized water	10		18.74501	
uncontaminati on	10			26.49611
Ivoclean	10			27.40553
hydrofluoric acid	10			27.98401
sodium silicate	10			28.71608
Sig.		1.000	1.000	.229

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.



Saliva contaminated zirconia

Oneway : RelyX U200 cement

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
1.218	5	54	.313

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	169.921	5	33.984	20.367	.000
Within Groups	90.104	54	1.669		
Total	260.026	59			

## Multiple Comparisons

Dependent Variable: shear

Tukey HSD

(I) cleaning	(J) cleaning	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatio n	deionized water	2.452673 *	.577685	.001	.74591	4.15943
	phosphoric acid	1.687056	.577685	.054	-.01970	3.39382
	hydrofluoric acid	3.935042 *	.577685	.000	2.22828	5.64180
	Ivoclean	-1.145433	.577685	.365	-2.85219	.56133
	sodium silicate	.325522	.577685	.993	-1.38124	2.03228
deionized water	uncontaminatio n	-2.452673 *	.577685	.001	-4.15943	-.74591
	phosphoric acid	-.765617	.577685	.770	-2.47238	.94114
	hydrofluoric acid	1.482369	.577685	.124	-.22439	3.18913
	Ivoclean	-3.598106 *	.577685	.000	-5.30487	-1.89135
	sodium silicate	-2.127151 *	.577685	.007	-3.83391	-.42039
phosphoric acid	uncontaminatio n	-1.687056	.577685	.054	-3.39382	.01970
	deionized water	.765617	.577685	.770	-.94114	2.47238
	hydrofluoric acid	2.247986 *	.577685	.004	.54123	3.95475
	Ivoclean	-2.832489 *	.577685	.000	-4.53925	-1.12573
	sodium silicate	-1.361534	.577685	.190	-3.06829	.34523
hydrofluoric acid	uncontaminatio n	-3.935042 *	.577685	.000	-5.64180	-2.22828
	deionized water	-1.482369	.577685	.124	-3.18913	.22439

	phosphoric acid	-2.247986*	.577685	.004	-3.95475	-.54123
	Ivoclean	-5.080475*	.577685	.000	-6.78723	-3.37372
	sodium silicate	-3.609520*	.577685	.000	-5.31628	-1.90276
Ivoclean	uncontaminatio n	1.145433	.577685	.365	-.56133	2.85219
	deionized water	3.598106*	.577685	.000	1.89135	5.30487
	phosphoric acid	2.832489*	.577685	.000	1.12573	4.53925
	hydrofluoric acid	5.080475*	.577685	.000	3.37372	6.78723
	sodium silicate	1.470955	.577685	.129	-.23580	3.17771
sodium silicate	uncontaminatio n	-.325522	.577685	.993	-2.03228	1.38124
	deionized water	2.127151*	.577685	.007	.42039	3.83391
	phosphoric acid	1.361534	.577685	.190	-.34523	3.06829
	hydrofluoric acid	3.609520*	.577685	.000	1.90276	5.31628
	Ivoclean	-1.470955	.577685	.129	-3.17771	.23580

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



shear

Tukey HSD

cleaning	N	Subset for alpha = 0.05			
		1	2	3	4
hydrofluoric acid	10	5.62985			
deionized water	10	7.11222	7.11222		
phosphoric acid	10		7.87783	7.87783	
sodium silicate	10			9.23937	9.23937
uncontaminatio n	10			9.56489	9.56489
Ivoclean	10				10.71032
Sig.		.124	.770	.054	.129

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.



Oneway : Panavia F2.0 cement

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
1.769	5	54	.135

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	281.320	5	56.264	41.354	.000
Within Groups	73.469	54	1.361		
Total	354.789	59			

## Multiple Comparisons

Dependent Variable: shear

Tukey HSD

(I) cleaning	(J) cleaning	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatio n	deionized water	5.584593 <sup>*</sup>	.521638	.000	4.04342	7.12576
	phosphoric acid	4.554763 <sup>*</sup>	.521638	.000	3.01359	6.09593
	hydrofluoric acid	6.367647 <sup>*</sup>	.521638	.000	4.82648	7.90882
	Ivoclean	2.226780 <sup>*</sup>	.521638	.001	.68561	3.76795
	sodium silicate	2.716856 <sup>*</sup>	.521638	.000	1.17569	4.25802
deionized water	uncontaminatio n	-5.584593 <sup>*</sup>	.521638	.000	-7.12576	-4.04342
	phosphoric acid	-1.029830	.521638	.370	-2.57100	.51134
	hydrofluoric acid	.783054	.521638	.665	-.75811	2.32422
	Ivoclean	-3.357813 <sup>*</sup>	.521638	.000	-4.89898	-1.81664
	sodium silicate	-2.867737 <sup>*</sup>	.521638	.000	-4.40891	-1.32657
phosphoric acid	uncontaminatio n	-4.554763 <sup>*</sup>	.521638	.000	-6.09593	-3.01359
	deionized water	1.029830	.521638	.370	-.51134	2.57100
	hydrofluoric acid	1.812884 <sup>*</sup>	.521638	.012	.27172	3.35405
	Ivoclean	-2.327983 <sup>*</sup>	.521638	.001	-3.86915	-.78681
	sodium silicate	-1.837907 <sup>*</sup>	.521638	.011	-3.37908	-.29674
hydrofluoric acid	uncontaminatio n	-6.367647 <sup>*</sup>	.521638	.000	-7.90882	-4.82648
	deionized water	-.783054	.521638	.665	-2.32422	.75811

	phosphoric acid	-1.812884*	.521638	.012	-3.35405	-.27172
	Ivoclean	-4.140867*	.521638	.000	-5.68204	-2.59970
	sodium silicate	-3.650791*	.521638	.000	-5.19196	-2.10962
Ivoclean	uncontaminatio n	-2.226780*	.521638	.001	-3.76795	-.68561
	deionized water	3.357813*	.521638	.000	1.81664	4.89898
	phosphoric acid	2.327983*	.521638	.001	.78681	3.86915
	hydrofluoric acid	4.140867*	.521638	.000	2.59970	5.68204
	sodium silicate	.490076	.521638	.934	-1.05109	2.03124
sodium silicate	uncontaminatio n	-2.716856*	.521638	.000	-4.25802	-1.17569
	deionized water	2.867737*	.521638	.000	1.32657	4.40891
	phosphoric acid	1.837907*	.521638	.011	.29674	3.37908
	hydrofluoric acid	3.650791*	.521638	.000	2.10962	5.19196
	Ivoclean	-.490076	.521638	.934	-2.03124	1.05109

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

shear

Tukey HSD

cleaning	N	Subset for alpha = 0.05			
		1	2	3	4
hydrofluoric acid	10	6.40535			
deionized water	10	7.18841	7.18841		
phosphoric acid	10		8.21824		
sodium silicate	10			10.05614	
Ivoclean	10			10.54622	
uncontaminatio n	10				12.77300
Sig.		.665	.370	.934	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.



Oneway : Superbond C&B cement

Test of Homogeneity of Variances

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Levene Statistic	df1	df2	Sig.
.321	5	54	.898

ANOVA

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	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1425.988	5	285.198	74.224	.000
Within Groups	207.488	54	3.842		
Total	1633.476	59			

## Multiple Comparisons

Dependent Variable: shear

Tukey HSD

(I) cleaning	(J) cleaning	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
uncontaminatio n	deionized water	10.949072 <sup>*</sup>	.876627	.000	8.35910	13.53905
	phosphoric acid	7.985363 <sup>*</sup>	.876627	.000	5.39539	10.57534
	hydrofluoric acid	11.450532 <sup>*</sup>	.876627	.000	8.86056	14.04051
	Ivoclean	-.965400	.876627	.879	-3.55538	1.62458
	sodium silicate	5.969450 <sup>*</sup>	.876627	.000	3.37947	8.55943
deionized water	uncontaminatio n	-10.949072 <sup>*</sup>	.876627	.000	-13.53905	-8.35910
	phosphoric acid	-2.963709 <sup>*</sup>	.876627	.016	-5.55369	-.37373
	hydrofluoric acid	.501460	.876627	.992	-2.08852	3.09144
	Ivoclean	-11.914472 <sup>*</sup>	.876627	.000	-14.50445	-9.32450
	sodium silicate	-4.979622 <sup>*</sup>	.876627	.000	-7.56960	-2.38965
phosphoric acid	uncontaminatio n	-7.985363 <sup>*</sup>	.876627	.000	-10.57534	-5.39539
	deionized water	2.963709 <sup>*</sup>	.876627	.016	.37373	5.55369
	hydrofluoric acid	3.465169 <sup>*</sup>	.876627	.003	.87519	6.05515
	Ivoclean	-8.950763 <sup>*</sup>	.876627	.000	-11.54074	-6.36079
	sodium silicate	-2.015913	.876627	.212	-4.60589	.57406
hydrofluoric acid	uncontaminatio n	-11.450532 <sup>*</sup>	.876627	.000	-14.04051	-8.86056
	deionized water	-.501460	.876627	.992	-3.09144	2.08852

	phosphoric acid	-3.465169 <sup>*</sup>	.876627	.003	-6.05515	-.87519
	Ivoclean	-12.415932 <sup>*</sup>	.876627	.000	-15.00591	-9.82596
	sodium silicate	-5.481082 <sup>*</sup>	.876627	.000	-8.07106	-2.89111
Ivoclean	uncontaminatio n	.965400	.876627	.879	-1.62458	3.55538
	deionized water	11.914472 <sup>*</sup>	.876627	.000	9.32450	14.50445
	phosphoric acid	8.950763 <sup>*</sup>	.876627	.000	6.36079	11.54074
	hydrofluoric acid	12.415932 <sup>*</sup>	.876627	.000	9.82596	15.00591
	sodium silicate	6.934850 <sup>*</sup>	.876627	.000	4.34487	9.52483
sodium silicate	uncontaminatio n	-5.969450 <sup>*</sup>	.876627	.000	-8.55943	-3.37947
	deionized water	4.979622 <sup>*</sup>	.876627	.000	2.38965	7.56960
	phosphoric acid	2.015913	.876627	.212	-.57406	4.60589
	hydrofluoric acid	5.481082 <sup>*</sup>	.876627	.000	2.89111	8.07106
	Ivoclean	-6.934850 <sup>*</sup>	.876627	.000	-9.52483	-4.34487

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



shear

Tukey HSD

cleaning	N	Subset for alpha = 0.05		
		1	2	3
hydrofluoric acid	10	14.64366		
deionized water	10	15.14512		
phosphoric acid	10		18.10883	
sodium silicate	10		20.12474	
uncontaminatio n	10			26.09419
Ivoclean	10			27.05959
Sig.		.992	.212	.879

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.



## VITA

Mr. Jitti Doungsri was born on May 25,1982 in Ratchaburi, Thailand. He received degree of Doctor of Dental Surgery (D.D.S.) from Mahidol University in 2006 . He worked as a general dentist at Srimuangmai Hospital, Ministry of Public health in 2007 - 2008 and then he worked at private dental clinic from 2009 to the present day.





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