THE SHEAR BOND STRENGTH OF LITHIUM DISILICATE GLASS-CERAMIC BONDED WITH TITANIUM USING FOUR RESIN CEMENTS

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กำลังยึดเฉือนของลิเทียมไดซิลิเกตเซรามิกและไททาเนียมที่ยึดด้วยเรซินซีเมนต์ 4ชนิด

นางสาวธนัชชา วนิชย์ถนอม

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Thesis Title	THE SHEAR BOND STRENGTH OF LITHIUM DISILICATE GLASS-CERAMIC BONDED WITH TITANIUM USING FOUR RESIN CEMENTS
By	Miss Thanatcha Wanichtanom
Field of Study	Esthetic Restorative and Implant Dentistry
Thesis Advisor	Jaijam Suwanwela, Ph.D.

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ธนัชชา วนิชย์ถนอม : กำลังยึดเฉือนของลิเทียมไดซิลิเกตเซรามิกและไททาเนียมที่ยึดด้วยเรซินซีเมนต์ 4ชนิด. (THE SHEAR BOND STRENGTH OF LITHIUM DISILICATE GLASS-CERAMIC BONDED WITH TITANIUM USING FOUR RESIN CEMENTS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: อ. ทญ. ดร.ใจแจ่ม สุวรรณเวลา, 69 หน้า.

การศึกษาเชิงทดลองในห้องปฏิบัติการนี้ มีวัตถุประสงค์เพื่อหาค่าและเปรียบเทียบกำลังยึดเฉือนของลิเทียมไดซิลิเกตเซรามิกและไททาเนียมที่ยึดด้วยเรซินซีเมนต์ 4 ชนิด คือ มัลติลิงค์อิมพลาน รีไลเอ็กซ์ยูนีเซ็ม พานาเวียเอฟสอง และ ซุปเปอร์บอนด์ซีแอนด์บี นำลิเทียมไดซิลิเกตเซรามิกชนิดกดอัดยึดกับไททาเนียม 60 ชิ้น ยึดกันด้วยเรซินซีเมนต์ 4 กลุ่ม 15 ชิ้นเท่ากัน แล้วนำมาทดสอบแรงยึดเฉือนด้วยเครื่องทดสอบแบบเอนกประสงค์ เปรียบเทียบค่าเฉลี่ยกำลังยึดเฉือนด้วยสถิติการวิเคราะห์ความแปรปรวนแบบทางเดียวและสถิติทดสอบที พบว่าค่าแรงยึดเฉือนของซีเมนต์แต่ละชนิดเรียงตามลำดับจากมากไปน้อยดังนี้ มัลติลิงค์อิมพลาน 58.27±2.05 เมกะปาสคาล รีไลเอ็กซ์ยูนีเซ็ม 45.81±1.88 เมกะปาสคาล ซุปเปอร์บอนด์ซีแอนด์บี 39.55±2.29 เมกะปาสคาล และ พานาเวียเอฟสอง 39.42±2.11 เมกะปาสคาล พบความแตกต่างกันอย่างมีนัยสำคัญทางสถิติระหว่างค่าเฉลี่ยแรงยึดเฉือนของซีเมนต์แต่ละกลุ่ม ยกเว้นซีเมนต์ซุปเปอร์บอนด์ซีแอนด์บีและพานาเวียร์เอฟสอง

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5376150732 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY
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THANATCHA WANICHTANOM: THE SHEAR BOND STRENGTH OF LITHIUM DISILICATE GLASS-CERAMIC BONDED WITH TITANIUM USING FOUR RESIN CEMENTS. ADVISOR: JAIJAM SUWANWELA, Ph.D., 69 pp.

The purpose of this experimental study was to determine and compare the shear bond strength of pressed lithium disilicate ceramic bonded with titanium disc by using four cements; Multilink implant® (MI), RelyX unicem® (RU), Panavia F2® (PF) and Superbond C&B® (SB). Sixty pressed lithium disilicate discs were luted to 60 titanium discs with 1 of 4 luting cements. The bonded materials were subjected for shear bond strength testing by a universal testing machine. Comparison of mean shear bond strength was evaluated by one-way ANOVA and t-test analysis. Shear bond strength of each cement ranked from the highest to the lowest are following; MI was 58.27±2.05 MPa, RU was 45.81±1.88 MPa, SB was 39.55±2.29 MPa, and PF was 39.42±2.11 MPa. Significant differences were observed statistically among groups ($p<0.001$), except SB and PF. Based on the result of this study, Multilink implant® provided the highest shear bond strength, followed by RelyX Unicem®, Superbond C&B®, and Panavia F2.0®, respectively.

Field of Study:	Esthetic Restorative and Implant Dentistry	Student's Signature	....
		Advisor's Signature	....
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CHAPTER I

INTRODUCTION

Rationale and significance of the problem

High esthetic demands have led to the development of new cement retained implant supported restorations, made of ceramic materials luted to titanium bases, as a treatment option to achieve better results in esthetically compromised areas. The introduction of hybrid implant abutments and hybrid implant abutment crowns (IPS e.max® Press, Ivoclar Vivadent AG) for implant-supported restorations provided new options in which the shape, emergence profile, and esthetic properties can be ideally adjusted to the specific clinical situation. A hybrid implant abutment is composed of a customized pressed lithium disilicate abutment which then connected to a titanium base with resin cement and subsequently received an all-ceramic crown. A monolithically pressed hybrid abutment crown joined the abutment and the monolithic crown in one piece. Although the innovative implant-supported restorations were fabricated highly efficiently, it was important to understand the mechanical properties of the new restorations using scientific studies to determine their appropriate use in the clinic. This study was conducted to evaluate the shear bond strength between pressed lithium disilicate ceramic and titanium disc bonded by four cements.

Research question

Which cement used to lute a pressed lithium disilicate glass-ceramic to a titanium disc that shows the highest level of shear bond strength?

Objectives of the study

The purpose of this *in vitro* study was to evaluate the shear bone strength of pressed lithium disilicate ceramic bonded to titanium disc by four different luting cements.

Statement of hypothesis

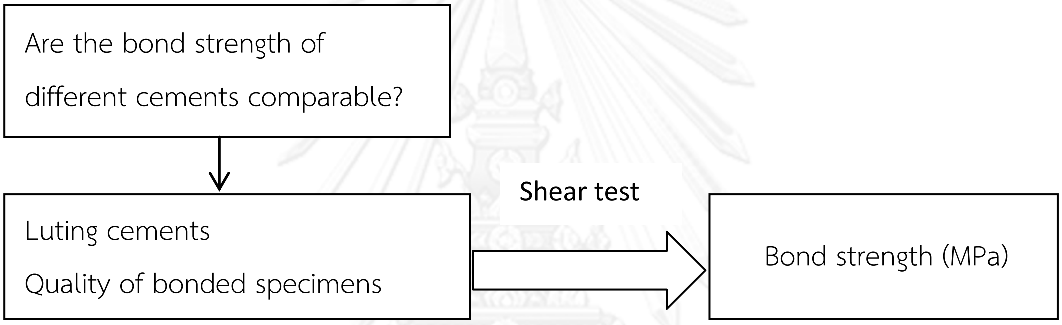
Null hypothesis

There was no significant difference in bond strength between the materials tested.

Alternative hypothesis

There was a significant difference in bond strength between the materials tested.

Conceptual framework



Plane surface	force(N)
Surface treatment	interface area (mm^2)
Bonding technique	

Study limitations

1. This study used an *in Vitro* technique which might not reflex confounding factors in clinical treatment

2. This study was only present the shear bone strength of four different cements, it could not refer to the appropriate cement type to use in clinical treatment

3. This study investigated only 1 type of ceramic.

Keywords

Shear bond strength, Cements, Titanium, Lithium disilicate ceramic, Implant

The expected benefits

1. The results of this study will be a benefit for further study.
2. The results from this study might draw a clinical suggestion for the selection of resin cements.

CHAPTER II

REVIEW OF THE LITERATURE

Dental implants require transmucosal abutments to support the implant restoration. Currently, porcelain fused to metal crowns are used with titanium abutments for the prosthetic phase of implant dentistry. However, these may result in less natural looking esthetic integration with the gray metal color being visible through the peri-implant tissue. With patient's increasing esthetic demands, new dental implant abutments made of ceramic materials luted to titanium bases have been introduced as an alternative.

The majority of dental implants and their abutments are fabricated from titanium and titanium alloy. The modern dentistry is increasing play attention to titanium-porcelain restorations because the benefit from having two similar materials contacted directly. One of the benefits could possible to reduce the corrosion and wear, the other is benefit to tissue healing[1]. Also, the properties of the Titanium metal alloys can descript as, titanium metal alloys are used as a replacement for noble metal alloys. Titanium and its alloys also have a low specific gravity (4.52 g/cm^3; compared to 18.3 g/cm^3 for high noble metal alloy for porcelain-fused-to-metal (PFM) restorations)[2], biocompatibility[3], high strength, heat resistance, corrosion resistance[4], low cost, availability[5], and titanium has a high dampening capacity, quickly absorbing impact force. However, the disadvantages of titanium when used in PFM restorations and bonded with porcelain are: 1) high melting point (1,668°C; compared to 1,063°C for gold)[2], 2) strong affinity for oxygen, nitrogen and carbon at high temperatures[5], 3) requiring casting to be done in a vacuum or inert gas chamber, 4) a poorly adherent oxide layer if heated above 883°C [6], [7], and 5) use of low fusing porcelains[8]. King et al. suggested enhancing the wettability of the metal surface through increasing the surface energy to achieve optimal contact between the metal and porcelain[9]. One of the recommended methods is sandblasting with alumina (Al_2O_3) to create surface irregularities allowing for mechanical interlocking by the porcelain.

Ceramic

Ceramic offers excellent esthetics, chemical stability, and biocompatibility. The conventional glass ceramics are silica-based ceramics, leucite glass-ceramic, and lithium disilicate ceramic. Ceramics can be classified by composition into three main divisions of dental ceramics.

1. Predominantly glassy ceramics are amorphous, or without form, in structure. The glass is derived from a group of mined minerals referred to as feldspar and these are composed of silica (silicon oxide) and alumina (aluminum oxide)[10]. Feldspathic porcelains are prepared from aluminosilicate and are used in high esthetic areas. The main disadvantages of feldspathic glasses are low mechanical properties with a flexural strength of approximately 69.74±5.47 MPa[11]. Predominantly glassy ceramics best mimic the natural appearance of enamel and dentin.

2. Particle-filled glasses, which are generally crystalline and contain particles of a higher melting glass, are addition to the base glass-ceramic to enhance its mechanical properties and control the optical properties. The flexural strength of these materials is 182 MPa[12].

3. Polycrystalline ceramics are aluminum oxide ceramics and zirconium oxide ceramics, which contain no glassy materials[13]. These materials are more difficult for a crack to propagate through and thus are much tougher and stronger than conventional or glassy ceramics. The flexural strength and fracture toughness of aluminum oxide ceramics are 547 MPa and 3.55 $MPa.m^{1/2}$, respectively[12, 14].

Lithium disilicate ceramic

Lithium disilicate ceramic is a particle-filled glass with a flexural strength of 400±40 MPa and is considered an enhanced-press-ceramic material with better physical properties and esthetics. Its production process generates homogeneous ingots with higher strength.

Lithium disilicate ceramic with a titanium base

Lithium disilicate ceramic has been increasing used in combination with titanium base for delivering esthetic results. However, there is currently no scientific evidence to validate its use in terms of shear bond strength. The shear bond strength of lithium disilicate luted on titanium base may be altered with the use of different surface treatments or luting agents.

Preparation of the bonding surfaces of the abutment and restoration prior to the application of the bonding resin, is a normal practice. This includes a combined physical and chemical approach; for example, sandblasting+silanation. Sandblasting with alumina particles has become a common technique in dentistry serving many purposes, which are the removal of contaminants, increasing the effective surface area[15], and improvement of the wetting ability of porcelain. Lavine et al. discovered that roughening a cast surface by stone could enhance the bond strength of the porcelain to metal compared with non-roughened samples. This is because the resultant increased surface area, leading to an improvement in metal wettability. This allows for the diffusion of porcelain particles into the metal[16]. In addition, the large particle size of alumina can be used to increase the surface roughness and promote mechanical interlocking of titanium with porcelain[17, 18]. According to Tsuchimoto et al., for a resin-based material bond to a substrate to be strong and durable depends on both micromechanical retention and chemical bonding[19]. In cementation with adhesive resin cements, increasing surface roughness can be achieved by airborne-particle abrasion with aluminum-oxide (Al_2O_3) particles. Both bonding mechanisms must be employed when using the non-adhesive resin cements. Metal surface treatments used for this purpose can be classified into: 1) promoters of micromechanical retention, 2) promoters of chemical bonding, and 3) promoters of micromechanical retention and chemical bonding[20]. Among these treatments, airborne-particles abrasion with Al_2O_3 particles is widely used[21] with the procedure cleaning the surface, increasing the surface area[15], decreasing surface tension, and creating a highly activated surface[22]. Other surface treatments such as roughening with a diamond bur[23] and chemical etching[19, 24], can also be employed. Adhesives offer greater contact between the cement and substrate, favoring micromechanical retention because of their lower viscosity in comparison with the viscosity of resin cements[20]. Chemical bonding can be accomplished with metal primers. Metal primers contain active monomers such as MDP, MPES, 4-META and others that react chemically with the oxides present on the metal surface[25, 26] and silanes create a chemical bond between the resin matrix and the metal surface[15, 27]. The third category of surface treatment provides both micromechanical retention and chemical bonding. Tribochemical silica coating is one method to achieve both bonding mechanisms using airborne-particle abrasion with silica-modified Al_2O_3 particles in conjunction with silanization (Cojet Sand and

Rocatec; 3M ESPE, Seefeld, Germany)[22]. Cojet Sand has a 30 µm particle size, is applied in a single step, and used chairside with the use of a chairside air abrasion device. Rocatec has a 110µm particle size and requires pre-treatment by airborne-particle abrasion with Al_2O_3 particles and is for laboratory use, not in the clinical setting[20].

Luting cements

The longevity of fixed dental prostheses can be affected by multiple factors, including the mode of cementation. Luting cement primarily creates reliable retention and durable seal of the space between abutment and titanium base. There are various types of luting cements are provided with different properties. The selection of the luting cement is crucial for the clinical success of definitive restorations because this influences the retention of the restoration and its long-term durability. One conventional luting cements that is widely used is zinc phosphate, which has the advantages of good handling characteristics, biocompatibility, the disadvantages of high solubility, lack of adhesion[28], and absence of a chemical bond to the substrate[29]. Another commonly used cement is glass-ionomer, which is favored because it bonds to tooth structure[30] and releases fluoride[31]. Disadvantages include low tensile strength, low fracture resistance, and susceptibility to moisture during the early stages of the setting process[32]. Hybrids of glass ionomer cement and resin composite became available in the late 1980's, termed "resin-modified glass ionomer cements (RMGI)[33]. RMGI offer advantages, including greater compressive and diametral tensile strength than zinc phosphate and some conventional glass ionomers[34]. Its disadvange is its hydrophilic nature arising from the formation of poly-(hydroxyethyl methacrylate) during the setting reaction, leading to increased water absorption and hygroscopic expansion[35].

Dual-polymerizing resin cements can be polymerized by either chemical polymerization or by light. These mechanisms allow for the widespread use of luting materials in the definitive cementation of all ceramics, composite, and metal-based indirect restorations. Dual-polymerizing resin cements are also characterized by high mechanical strength and excellent esthetic properties[36] because their chemical compositions provide adherence to many dental substrates[37, 38]. Nevertheless, skillful handling is needed for resin cements during the bonding procedure and during the removal of excess cement, which is time consuming[39]. A self-adhesive, dual-polymerizing universal resin cement has been introduced with easy manipulation and none of the pretreatment steps required from the glass ionomer cements with favorable mechanical properties[40], attractive esthetics, and good tooth adhesion. Bonding to the tooth structure can be completed without any pre-treatment steps such as etching, priming, or bonding as described by the manufacturer. This self-adhesive universal resin cement is composed of a new proprietary monomer, filler, and initiator technology[41].

Resin based luting cements are recommended for luting ceramic restorations due to improved retention, marginal adaptation of the restorations, low solubility in the oral environment, and less microleakage compared to conventional cements[42, 43]. Resin cements are divided into three groups based on their bonding systems.

1. Total etch resin cements require the enamel and dentin to be etched using phosphoric acid before cementation. Then, the etchant is rinsed off and the tooth is dried until it is only slightly moist. Next, primer and adhesive are applied and the resin cement is used. The resin cements in this group are Variolink and Variolink II (Ivoclar Vivadent AG), Calibra (Dentsply Caulk), and Nexus (Kerr)[44]. Their bonding effectiveness may be compromised due to technique sensitive and multi-step application process[45].

2. Self etch resin cements are developed to reduce the number of procedural steps by combining the acidic primer and adhesive. The self-etching primer is applied on the tooth structure to prepare the enamel and dentin. Examples for this type of cement are Panavia F21, Panavia F, Panavia F2.0 (Kuraray), and Multilink (Ivoclar Vivadent AG)[44].

3. Self adhesive resin cements contain multifunctional monomers with phosphoric acid groups, which simultaneously demineralize and infiltrate the enamel and dentin. The polymerization of dual cured resin cements can be initiated by light exposure or their self-curing mechanism.

Thermocycling

Restorations are subjected to thermal change in the mouth thus, thermocycling is a technique commonly used to stimulate aging. Mair, L. and P. Padipatvuthikul found that the oral temperature changed between 0°C to 60-65°C when eating ice cream and then eating a hot cheese sandwich and this temperature is rarely exceeded by food[46]. Normally, restorations experience only a very small variation in temperature when patients are not eating or drinking[47]. It is also typical that change in mouth’s temperature is relatively slow. Thermocycling by alternately plunging the test pieces in water baths at 0°C and 60°C can result in thermal shock from the expansion or contraction of the surface layer over the unaffected bulk of material. While this situation arises on the wings of fighter aircraft based on acceleration, it rarely occurs in the mouth as patients typically do not bite into ice cream and then abruptly drink hot coffee. Thermocycling can cause spontaneous debonding of specimens but does not have to have a deleterious effect. There are no differences seen in dye penetration between cycled and uncycled samples resin bonded composite restorations by Rossomando and Wendt[48]. However, it is more important to know in thermal conductivity between amalgam and tooth is different than between amalgams in contacting teeth[47].

Despite these shortcomings, thermocycling is a common way to access bond durability which simulates the thermal changes that occur in the oral cavity caused by eating, drinking, and breathing[49]. These thermal changes can reduce bond strength values[50] by inducing repetitive contraction and expansion stress at the tooth-material interface. This can lead to crack propagation along the bonded interface, which may cause gap formation[51] allowing the passage of fluid through the interface[50]. Adhesive failure may occur between the bonding resin and dentin after thermocycling. The thermocycling regimens experimentally employed vary in the number of cycles, temperatures, and dwelling times used. Some studies suggest that 500 cycles are not effective to simulate long-term bond durability[50, 52, 53], while other studies propose that the thermocycling protocol does not influence the bond strength and microleakage of adhesive systems[50, 52, 54, 55]. A large number of cycles has a drawback to the restoration interface[56, 57] and accelerates the aging process by the deleterious effect of water[58]. Regarding temperature, the ranges of the temperatures used for thermocycling are varied, such as 4 and 60°C, 5 and 55 °C, 15 and 45 °C, and 5 and 60 °C[59]. However, alternating the temperature between 5 and 55 °C simulates the actual temperature range that occurs in the oral cavity[60]. While the ISO standard recommends a dwell time of at least 20 seconds in each bath[61], patients usually cannot tolerate the direct contact of a vital tooth with extremely hot and cold substances for an extended period of time. Thus, a short dwell time (no longer than 15 seconds) has been suggested to simulate the clinical situation[54, 62]. In addition, shorter intervals may cause more immediate changes temperature that occur in the oral cavity[60].

Piwowarczyk et al.[41] studied the shear bond strength of luting cements to high strength aluminum oxide ceramics after thermocycling for 10,000 cycles in water at 5/55 °C. Their report concluded that the resin cements, Panavia F and Rely X Unicem, demonstrated high bond strength to high-purity aluminum oxide. In contrast, zinc phosphate cements, glass-ionomer cements, and resin-modified glass-ionomer cements did not provide a stable bond to high-purity aluminum oxide ceramics under these conditions. LÜthy et al[63] studied the shear bond strength of different cements to densely sintered zirconia ceramic after thermocycling using the same conditions as Piwowarczyk et al. They reported that Ketac-Cem had the weakest bond strength when the specimens are subjected to thermocycling. Thermocycling had significantly impact to the bond strength of Ketac-Cem, Nexus, and Superbond C&B. However, the bond strength of Panavia F21 significantly increased, while the bond strength of Panavia F and Rely X Unicem did not change significantly. Superbond C&B cement contains META-4, which bonds chemically to ceramic. However, the bond strength of this resin cement is significantly decreased after thermocycling[64]. The reduction of the bond strength resulted from water resorption by poly(methyl methacrylate) (PMMA) during the aging process[64] and a stable bond strength between Superbond C&B and alloy is not achieved after water storage[65].

CHAPTER III

RESEARCH METHODOLOGY

Materials

1. Multilink implant (Ivoclar Vivadent AG., Liechtenstein)
2. Panavia F2.0 (Kuraray, Japan)
3. Superbond C&B (Sun Medical, Japan)
4. RelyX unicem (3M-ESPE)
5. 5% IPS ceramic etching gel (Ivoclar Vivadent AG., Liechtenstein)
6. Monobond S (Ivoclar Vivadent AG)
7. Metal/Zirconia Primer (Ivoclar Vivadent AG)
8. RelyX™ ceramic primer (3M-ESPE)
9. V-PRIMER (Sun medical, Japan)
10. Porcelain Liner M (Sun medical, Japan)
11. Clearfil™ ceramic primer (Kuraray, Japan)
12. Alloy primer (Kuraray, Japan)
13. IPS e.max® LT Ingot (Ivoclar Vivadent AG., Liechtenstein)
14. Cylindrical refractory investment molds
15. Putty silicone and light body silicone impression materials
16. Ivory inlay wax
17. Titanium discs
18. Milled stainless steel
19. PVC tubes (22 millimeter diameter and 25 millimeter length)
20. Round transparent stickers
21. Stopwatch
22. Distilled water
23. Ultrasonic model (TP 680DH)
24. Durometer model (Durometer model 471, Pacific transducer, CA, USA)

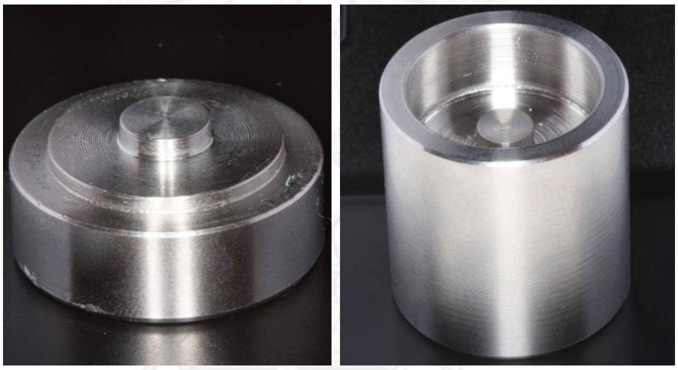
25. Sandblasting equipment (Penblaster II, Shofu Inc, Kyoto, Japan)
26. Polishing machine (Polishmachine DPS 3200, Imptech, Sunward Park, South Africa)
27. Thermo cycling Unit (King Mongkut's University of Technology Thonburi (KMUTT), Thailand)
28. Light curing unit (Demi plus, Ker)
29. Shear bond test - Using universal testing machine (Instron corp., Canton, MA, USA)
30. Stereo microscope (ML9300, Meiji)
31. Universal testing machine (LR10K, LLOYD Instrument, England)
32. Abrasive products with Pressure sensitive adhesive backing (3M)
33. Incubator 8-100°C (Contherm160M, Contherm Scientific Ltd., New Zealand)
34. Polymethyl methacrylate acrylic resin (UnifastTM trad, GC America Inc, USA)
35. Low speed cutting machine (ISOMET 1000, USA)

This experimental study was modified from the International Organization for Standardization (ISO/TR 11405:1994).

Specimen preparation

Sixty titanium discs were prepared as 7 mm in diameter and 3 mm in thickness by cutting technique. The titanium discs were embedded into a polyvinyl chloride (PVC) tube (22 mm diameter and 25 mm height), using polymethyl methacrylate acrylic resin (UnifastTM trad, GC America Inc, USA). In order to prepare the specimens standardize, the milled stainless steel (Figure 1A) are used to specify the correct position between titanium disc and PVC tube. All specimens bonded surfaces were smoothed with 400, 600, 800 and 1200– grit silicon carbide paper (Abrasive products with Pressure sensitive adhesive backing, 3M). Polishing pressure was adjusted at 2 bars and speed was at 100 rounds per minute on counter clockwise direction. The specimens became plane surface which was perpendicular of the long axis of specimen. The surface treatment of titanium discs were airborne-particle abrasion with 50 μm alumina particles. The index acrylic were used to specify the position of a thin adhesive tape adhered. A thin adhesive non-reactive tape which have 5 mm diameter of hole were adhered into the center of the polished specimens. All specimens were treated with four commercially available silane systems recommended by each cement manufacturer (Table 1).

The test porcelain samples were made of IPS e.max® LT A3 Ingots (Ivoclar Vivadent AG., Liechtenstein) using the lost wax and high temperature injection molding techniques. A test mold were prepared from a milled stainless steel cylinder (Figure 1B) and by taking an impression of this using putty silicone impression and light body silicone as shown in Figure 2. Then, molten wax was poured into the mold to generate 60 cylinder shaped wax patterns.



(A) (B)

Figure 1 The milled stainless steel cylinder

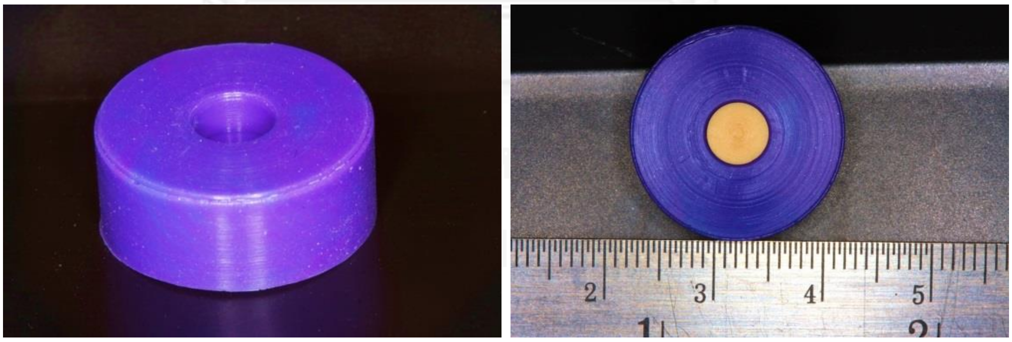


Figure 2 The tested mold with a 5-millimeter diameter and a 3-millimeter depth

Sixty pressed lithium disilicate discs (5 mm in diameter and 3 mm thickness) were fired in cylindrical refractory investment molds, according to the manufacturer's instructions. The investment blocks were bench cooled and divested by airborne-particle. All ceramic specimens bonded surfaces were smoothed with 400, 600, 800 and 1200–grit silicon carbide paper (Abrasive products with Pressure sensitive adhesive backing, 3M ESPE). Polishing pressure was adjusted at 2 bars and speed was at 100 rounds per minute on counter clockwise direction. The specimens became plane surface which was perpendicular of the long axis of specimen. The surface treatment of the lithium disilicate discs were airborne-particle abrasion at 2 bars pressure with 50 µm alumina, 10 mm of distances particles and then all specimens were then decontaminated by soaking in distilled water in an ultrasonic bath for 10 minutes and left to dry at room temperature. The tested ceramics were etched with hydrofluoric acid (5% IPS ceramic etching gel) for 20 seconds, rinsed and dried. All specimens were treated with four commercially available silane systems recommended by each cement manufacturer (Table 1).

Consequently, lithium disilicate discs were luted to titanium discs with different four luting cements, i.e., Multilink implant® (MI), RelyX Unicem® (RU), Panavia F2.0® (PF), and Superbond C&B® (SB). The details of materials tested in this study were shown in table 2. The luting agents were applied according to the manufacturer’s recommendations and each pair of specimens was pressed using a durometer (Figure 3) for 10 minutes. The procedure for preparation of the specimens and shear testing were shown in figure 4.

Table 1 Description of silane systems used

Materials	Composition	Manufacturer
Monobond S	ɣ-MPS*, ethanol.*	Ivocar Vivadent AG., Liechtenstein
Metal/Zirconia Primer	tert-Butylalkohol, Methylisobutylketon, phosphonic acid acrylate, dibenzoyl peroxide	
RelyX™ ceramic primer	Methacryloxy propyl trimethoxysilane, ethanol	3M-ESPE
V-PRIMER	VTD, acetone	Sun medical, Japan
Porcelain Liner M	Liquid A: MMA , 4-META Liquid B: MMA, ɣ -MPS	
Clearfil™ ceramic primer	*MDP, ɣ - MPS, ethanol*	Kuraray, Japan
Alloy primer	*VTD, MDP, acetone*	

MMA : methylmethacrylate

ɣ - *MPS* : 3-(trimethoxysilyl) propyl methacrylate

4-META : 4-methacryloxyethyl trimellitate anhydride

MDP : *10-methacryloyloxydecyl dihydrogen phosphate*

VTD : 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol

Table 2 Description of materials used

Materials	Curing mode	Composition	Manufacture
MI	Dual-cure	Monomer: dimethacrylates, HEMA	Ivocar Vivadent AG., Liechtenstein
RU	Dual-cure	Glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, methacrylated phosphoric ester, dimathacrylate	3M-ESPE
SB	Self-cure	MMA, 4-META, TBB, polymethymethacrylate	Sun medical, Japan
PF	Dual-cure	Paste A: Quartz glass, microfiller, MDPB, methacrylates, photoinitiator Paste B: Barium glass, sodium fluoride, methacrylates, chemical initiator	Kuraray, Japan
IPS e.max®	Ceramic ingot LT A3	Lithium disilicate glass ceramic	Ivoclar Vivadent AG., Liechtenstein

MI : Multilink implant®

RU : RelyX Unicem®

SB : Superbond C&B®

PF : Panavia F2.0®

HEMA : 2-hydroxyethyl methacrylate

MMA : methylmethacrylate

4-META : 4-methacryloxyethyl trimellitate anhydride

MDPB : 10-methcryloyloxydecyl dihydrogen phosphate bromide

TBB : tri-n-butylborane

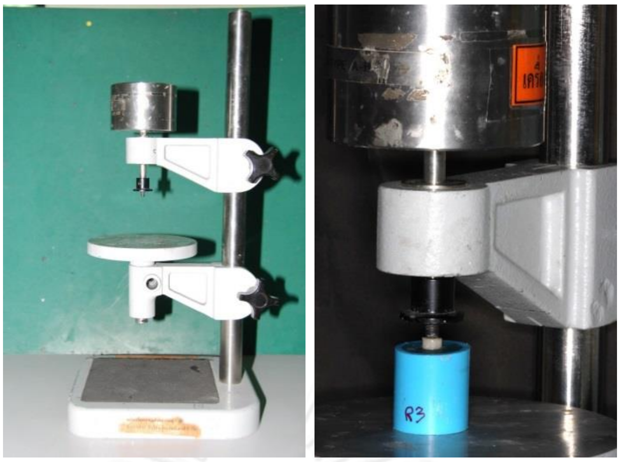


Figure 3 Pressing procedure using the Durometer

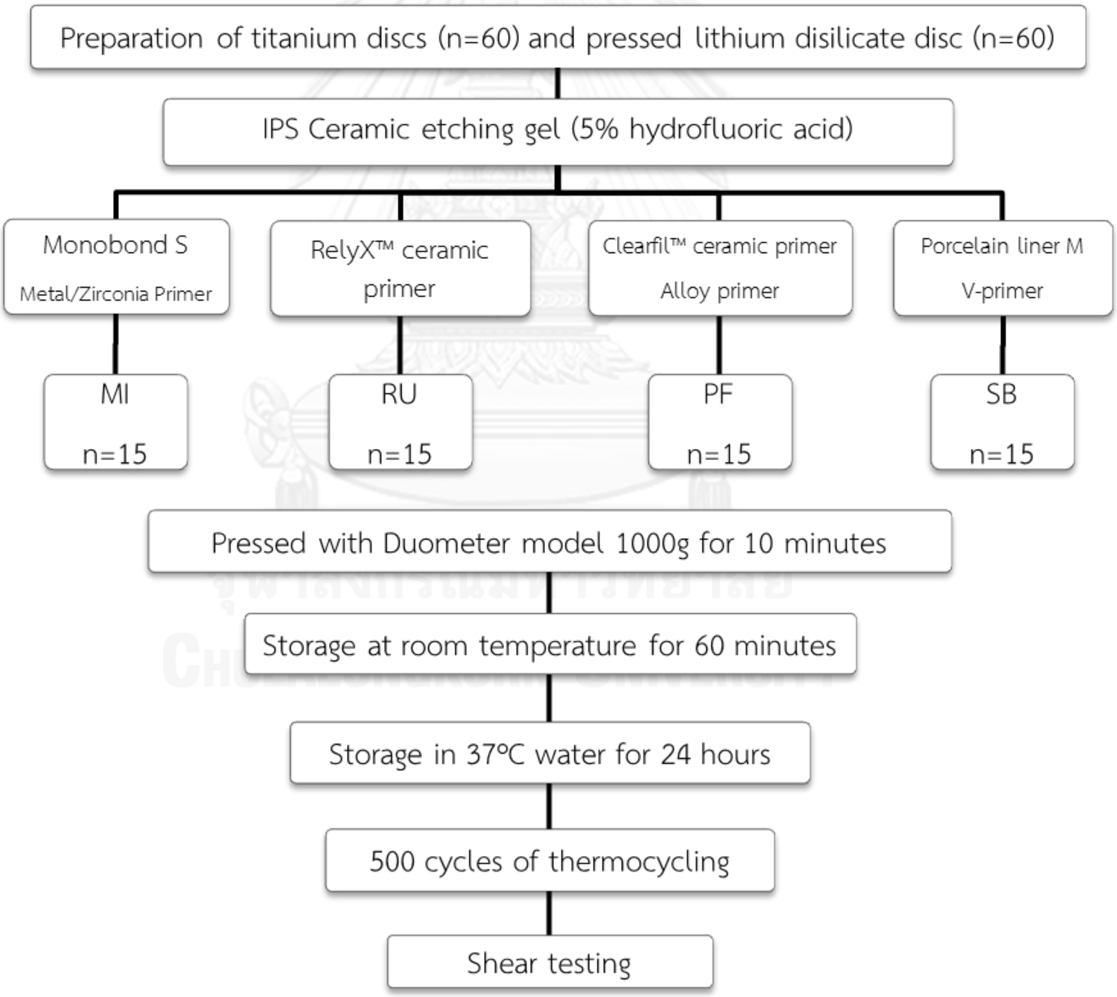


Figure 4 Procedure for specimens preparation and shear testing

(MI: Multilink implant® RU: RelyX Unicem® SB: Superbond C&B® PF: Panavia F2.0®)

Subsequently, excess cement were removed, and the test samples were stored in dry conditions for 60 minutes at room temperature and then stored in tap water for 24 hours at 37°C. All of the specimens were placed in the thermocycling apparatus. The samples were submitted to 500 cycles of thermocycling with a different temperature among 5°C to 55°C water with 20 seconds dwell time.

In order to evaluate that whether the interfaces were smoothed and plane surface, The specimen from each group was cross-sectionally cut (Figure 5) and SEM was used to analyze.

Figure 5 Cross-sectional tested specimen

Each specimen were embedded in a resin mold and seated in a shear-testing jig (Figure 6). Shear bond strength were then determined on a universal testing device (LR10K, LLOYD Instrument, England) at a crosshead speed of 0.5 mm/min. Shear load at failure were recorded and converted to strength. For each condition, the shear bond strength mean and standard deviation (SD) of the 15 specimens were calculated. Difference of the shear bond strength values between groups were determined by one-way analysis of variance (ANOVA) and t-test analysis with p value = 0.05.

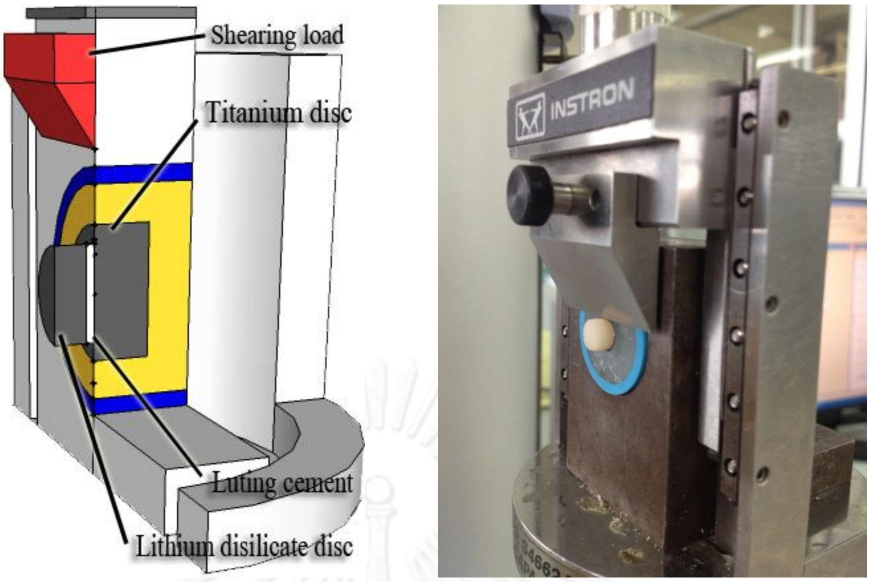


Figure 6 Assembly used for determination of shear bond strength.

After shear testing, the deboned surfaces were observed through a stereo microscope (ML9300, Meiji, Japan) at 15X magnification to calculate the debonded area and to identified failure modes as mixed of cohesive and adhesive failures, cohesive failure and adhesive failure. Representative samples were examined in a scanning electron microscope (SEM, XL 30 CP, Philips) with and acceleration voltage of 15 KeV after sputtering using a gold alloy conductive layer of approximately 15 nm.

CHAPTER IV

RESULTS

The results of this study revealed that mean shear bond strength of MI was the highest (58.27±2.05 MPa). The second shear bond strength was RU (45.81±1.88 MPa) followed by SB (39.55±2.29 MPa). The lowest shear bond strength was PF (39.42±2.11 MPa). Significant differences were observed statistically among groups ($p<0.001$), except the difference between SB and PF (Table 3).

Table 3 Mean, standard deviation, statistical comparison and p value

Cement types	N	Mean [SD]	Statistical comparison among materials*	p value
MI	15	58.27 [2.05]	A	< 0.001
RU	15	45.81 [1.88]	C	
PF	15	39.42 [2.11]	B	
SB	15	39.55 [2.29]	B	

Degree of freedom=3

*Identical upper case letters define no significant differences

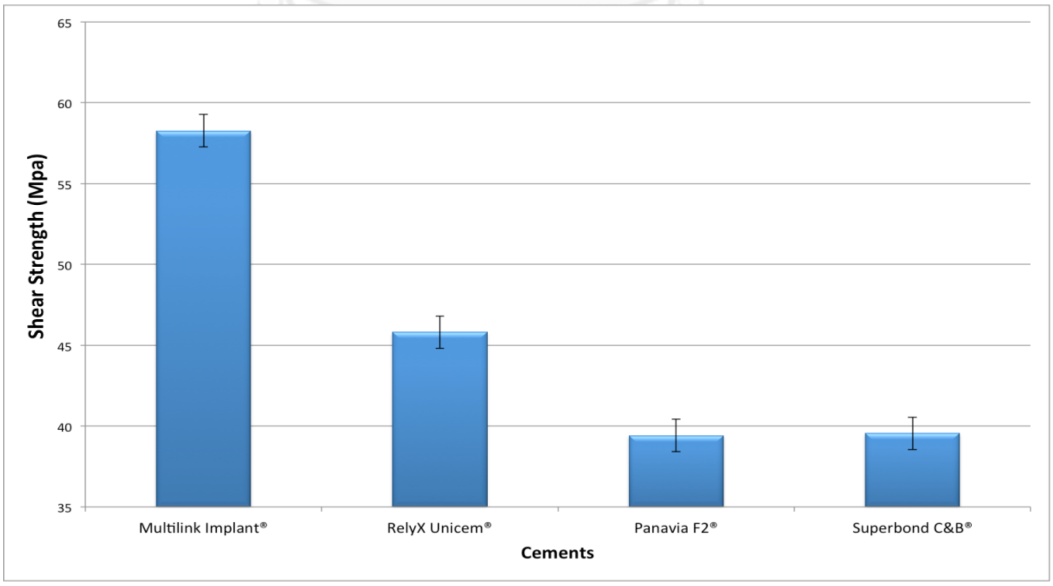


Figure 7 Mean shear bond strength of four luting cements.

Examination of failure mode

Mode of failure was categorized into 3 types as follows:

(1) Mixed of cohesive and adhesive failures, (2) Cohesive failure and (3) Adhesive failure. The percentage distributions of failure modes were recorded by using stereomicroscope at a magnification of 15X as shown in table 4. Typical samples in the SEM were examined to verify the failure modes detected with the stereomicroscope in all groups. Figures 9-11 show SEM photographs with typical examples of mixed of cohesive and adhesive failures mode, completely cohesive failures mode, and completely adhesive failure mode.

Table 4 Distribution of failure mode

Group	Failure Mode		
	(1)Mixed	(2)Cohesive	(3)Adhesive
MI	15	-	-
RU	12	2	1
PF	10	3	2
SB	11	2	2

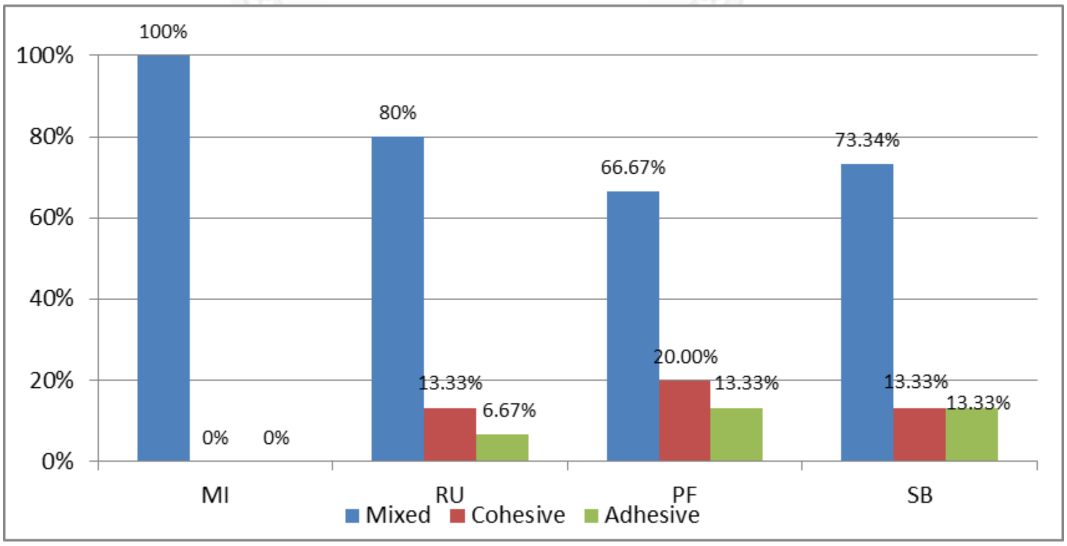
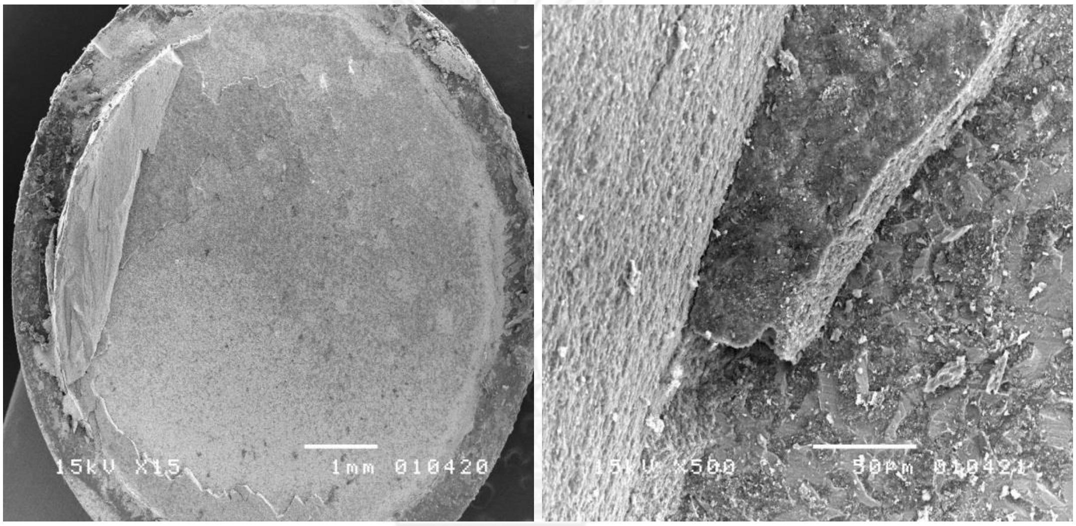


Figure 8 Types of bonding failure mode as identified with a stereo microscope at 15X magnification and calculated in percentage of the bonding area.

The majority of the failures were predominantly mixed failure. MI showed the highest mixed failure (100%), followed by RU (80.00%), SB (73.34%) and PF (66.67%). PF showed the highest percent of cohesive failure (20.00%), followed by RU (13.33%), SB (13.33%). Moreover, adhesive failure of PF and SB showed the same result (13.33%), followed by RU (6.67%). MI showed none of cohesive or adhesive failure.

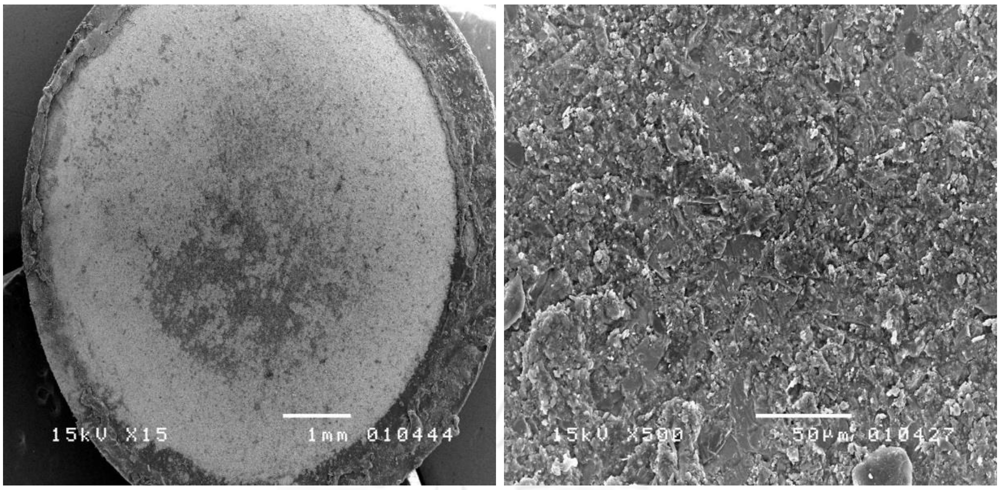
Examples for the three type of failure mode as follow:



(A) 15X Magnification **(B) 500X Magnification**

Figure 9 SEM photographs from representatives illustrating the fractured surfaces of the specimen (mixed of cohesive and adhesive failures) from MI group.

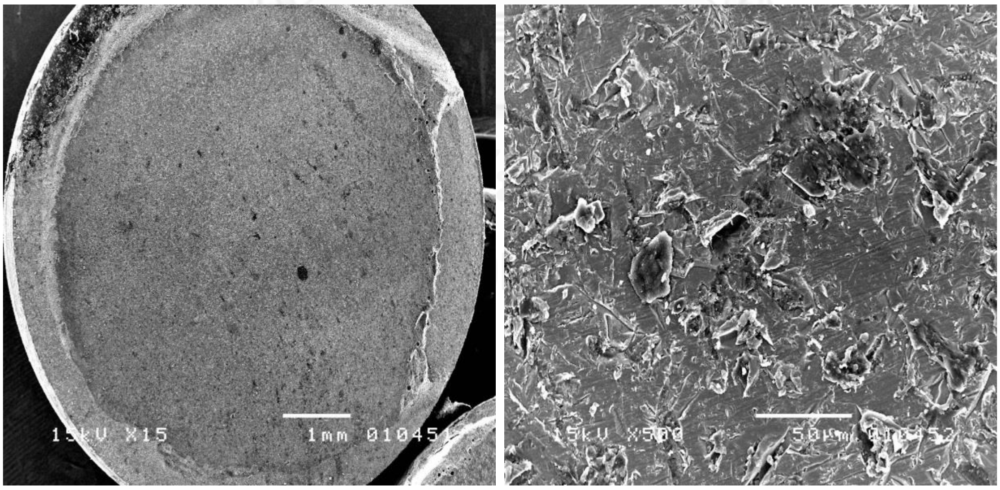
The SEM photograph at 15 magnification showed mixed failure found in MI group (Figure 9(A)). The crack occured at the interface both ceramic and titanium and small piece of ceramic remained on the titanium surface. There were parts of cement and ceramic remained on the titanium surface (Figure 9(B)).



(A) 15X Magnification (B) 500X Magnification

Figure 10 SEM photographs from representatives illustrating the fractured surfaces of the specimen (cohesive failure) from PF group.

The SEM photograph at 15 magnification showed cohesive failure found in PF group which the crack occured completely in cement layer (Figure 10(A)). Cement was observed more than 90% of the titanium surface (Figure 10(B)).

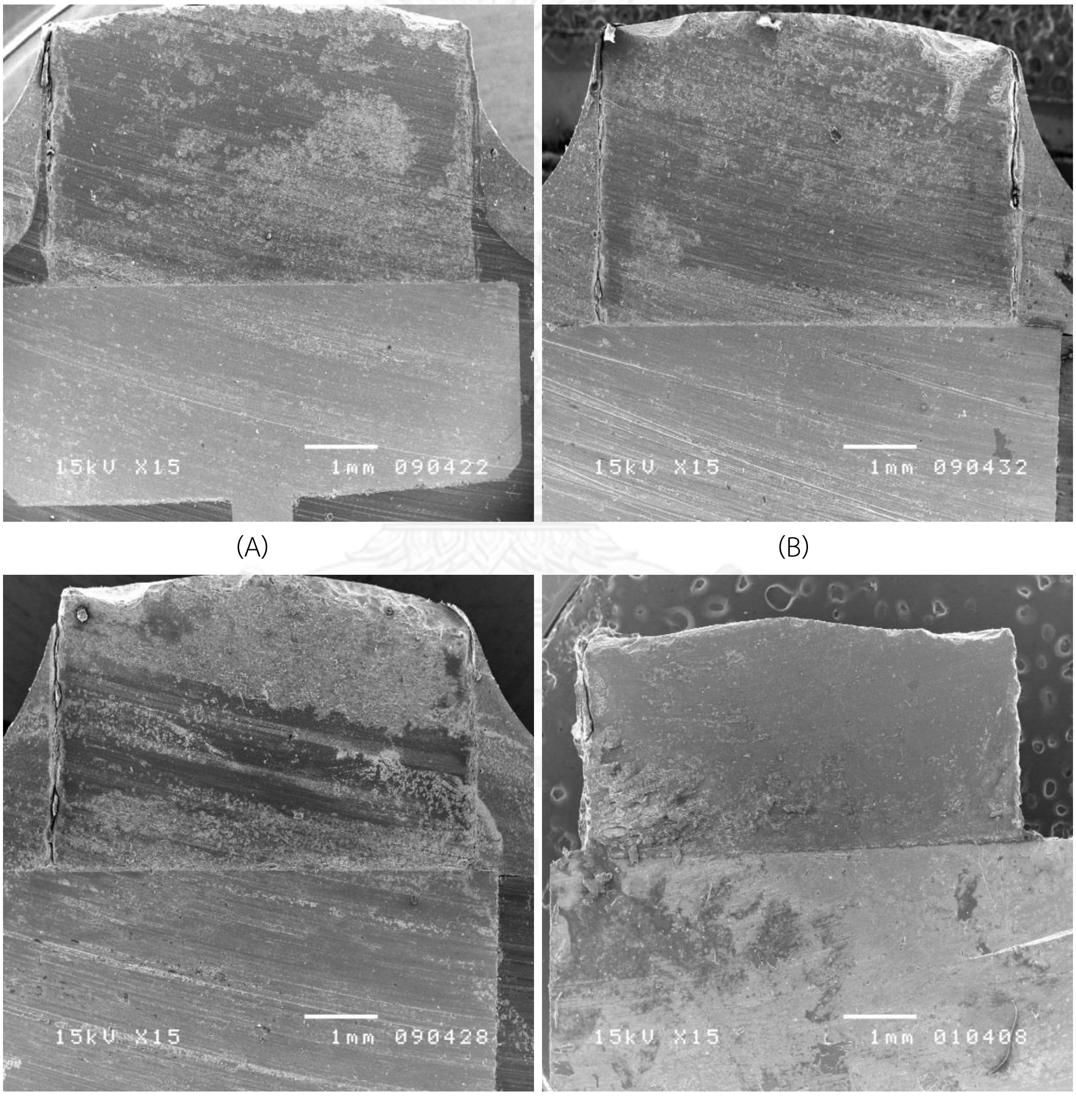


(A) 15X Magnification (B) 500X Magnification

Figure 11 SEM photographs from representatives illustrating the fractured surfaces of the specimen (adhesive failure) from RU group.

The SEM photograph at 15 magnification showed adhesive failure found in RU group which the crack occured in cement titanium interface (Figure 11(A)). The titanium surface was clearly observed however a little of remaining cement was observed (Figure 11(B)).

SEM photographs of cross sectional tested specimens showed the interfaces were smoothed and in horizontal plane (Figure 12, 13). Thicknesses of the cements were quite even within sample.



(A) (B)

(C) (D)

Figure 12 SEM micrograph of cross sectional tested specimens (magnification 15x). (A)MI (B)RU (C)PF (D)SB

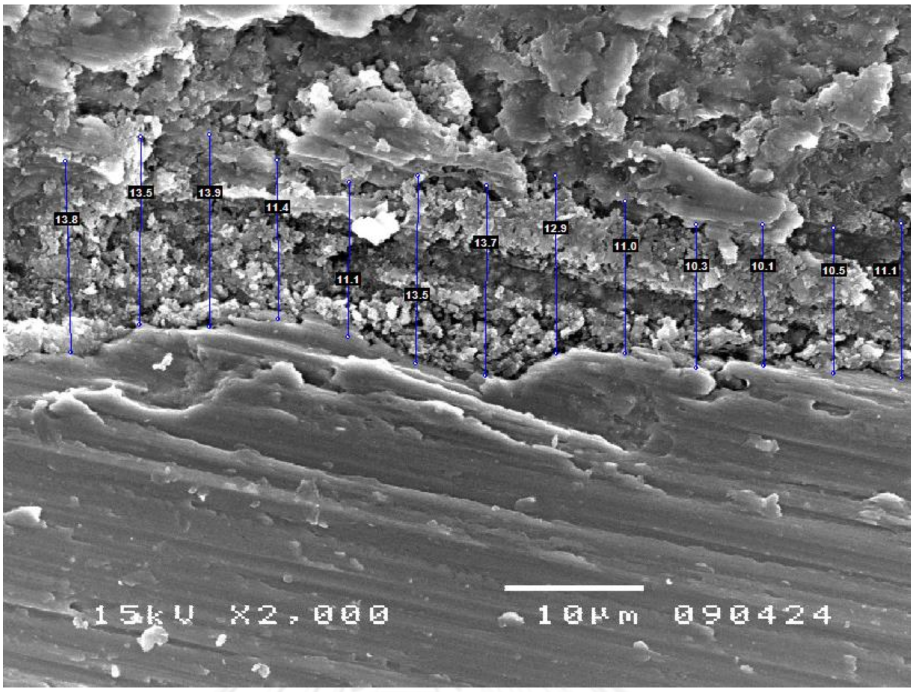


Figure 13 SEM micrograph of cross sectional MI specimen (magnification 2000x).

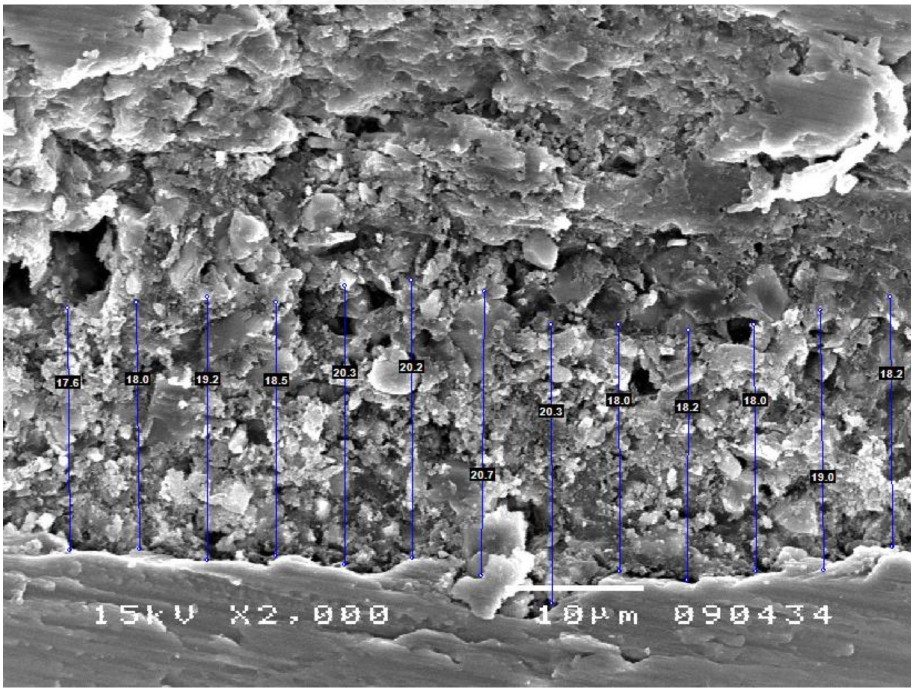


Figure 14 SEM micrograph of cross sectional RU specimen (magnification 2000x).

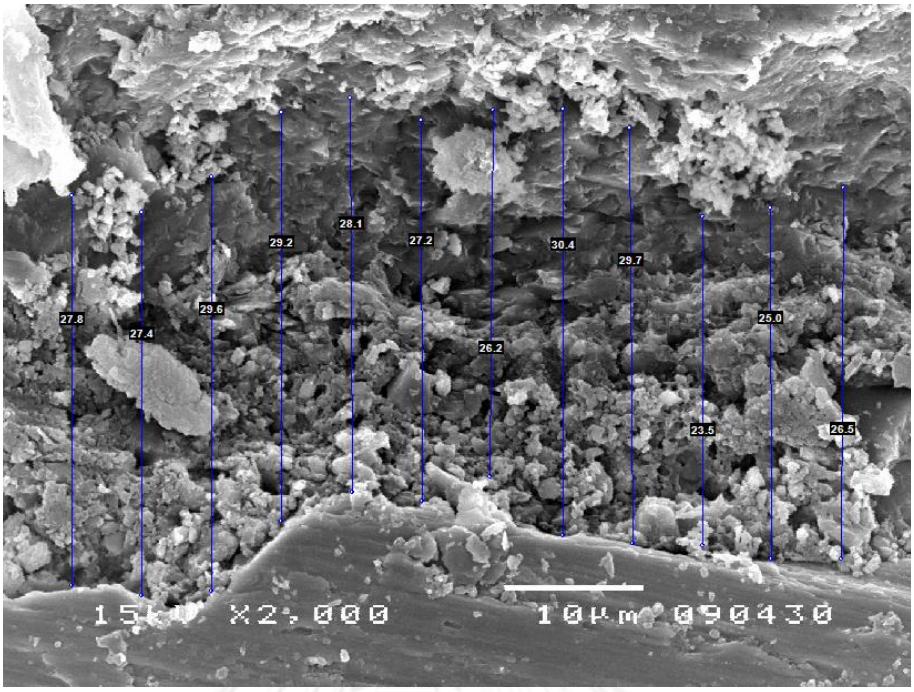


Figure 15 SEM micrograph of cross sectional PF specimen (magnification 2000x).

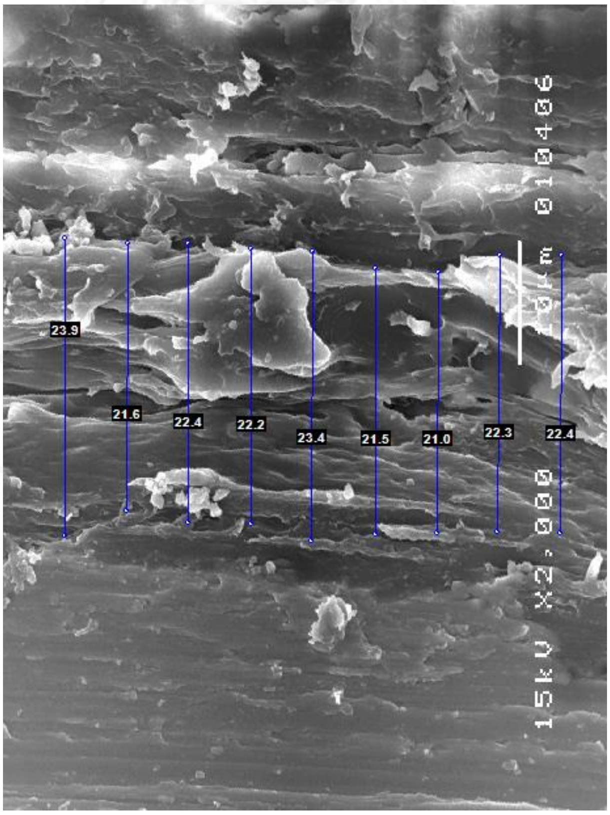


Figure 16 SEM micrograph of cross sectional SB specimen (magnification 2000x).

The specimens were analyzed thickness of cement by using SEMafore image analysis program. Cement thickness of sample from MI group range from 10-14 micron and the average is approximately 12 micron (Figure 13). Cement thickness of sample from RU group range from 17-21 micron and the average cement thickness is approximately 19 micron (Figure 14). Cement thickness of sample from PF group range from 23-30 micron. The average cement thickness of PF group is approximately 28 micron (Figure 15). Cement thickness found in SB group range from 23-30 micron and the average cement thickness of SB group is approximately 23 micron (Figure 16).

CHAPTER V

DISCUSSION AND CONCLUSION

Discussion

The objective of this study was to determine and compare the shear bond strength of pressed lithium disilicate ceramics bonded with titanium discs using four resin cements. The null hypothesis which proposed that the types of cements would not influence the bond strength was rejected. There were significant differences in shear bond strength when using different cements. Significant differences were observed statistically among groups ($p<0.001$), except the difference between SB and PF. This is a first study to report the bond strength of pressed lithium disilicate ceramic and titanium disc bonded by cements. The results cannot be compared with other studies due to the lack of evidence for the four resin cement groups. The studies on luting agents used to cement the restorations onto implant abutments are inadequate to provide an information for the cement selections because the tested protocols of different studies were variety [66].

There were many treatment procedures such as sandblasting, tin plating, silicoating, and metal primer application that were used to produce irregularities on the internal surface of the casting and abutment. Sandblasting creates irregularities on the metal surfaces, increases the surface area, and mechanically removes debris [67, 68]. During sandblasting, alumina particles become encrusted on the metal surface since the velocity and pressure hit the surface, and they cannot be removed even by ultrasonic cleaning or acid etching. Thus, these non-removable alumina particles cause the chemical bonds of the alloy primer and silane agents to themselves, eventually increasing the bond strength of resin cements. The size of the aluminum oxide particles differed according to the authors[68]. In our study, sandblasting was applied using 50-μm aluminum oxide at 2 bar pressure and 10-mm distance. Sandblasting is the easiest and most inexpensive method of surface treatment. Treatment with different chemical components such as tin plating and silicoating is not commonly used to increase bonding because of their requirement for additional equipment [69]. Metal primer application is an easy method of surface treatment for metal substructures.

A combination of resin cement/adhesive system has an ability to adhere to dental ceramics depends on the microstructure of the esthetic restoration and the surface treatment applied [70]. While roughening the surface by grinding or airborne particle abrasion is can improve adhesion for most of the esthetic materials, which appears to be only effective for silica-based ceramics [50]. A durable and reliable bond for dental resin-bonded ceramics is usually applied via two principal mechanisms: (1) micromechanical attachment to porosities originated from hydrofluoric acid (HF) etching or (2) grit blasting, associated with a silane-coupling agent. Evaluations of bond strength between ceramic and resin composite have derived different conclusions about the effect of surface treatments. Controversy in the literature [71, 72] relies on possible inefficacy or inactivity of the silane-coupling agent applied and operator's handling of the procedure. Meng and colleagues [73] recently demonstrated that hydrofluoric acid treatment could enhance the bond durability of resin/silanated glass ceramics, increasing the chemical adhesion area on the ceramic rough surface and subsequently reducing degradation speed of the silane coupler, rather than the mechanical retention of the ceramic rough surface [73].

The tested ceramics were pressed lithium disiligate which have a high flexural strength, high esthetic and, simple production process [74]. The surfaces were treated by etching with hydrofluoric acid to enchant the bond strength. Since IPS e.max® Press hybrid restoration was cement-retained restoration, cemented to titanium abutments and/or pressed lithium disilicate abutments therefore, well treatment of ceramic and titanium surface and selection of cements are important factors for determining the longevity of restoration. The surface conditioning of ceramic and titanium was favorably used by sandblasting, etching and silanizing [75]. However, various choices of cements that can impact on final outcome of the ceramic restoration were debatable in clinical applications [76, 77]. Thus, the handling of the lute during mixing, placement, setting and the number of steps in the bonding procedure should be taken into consideration when cements were selected [78].

In order to evaluate the difference between luting cement, two factors could be considered as the luting resin system and the silane coupling agent. As a matter of luting resin system fact, the flexural strength is the resistance of a test sample against flexural stress at the point of breaking. In addition to the compressive strength and tensile strength, it is a significant parameter describing the mechanical strength of a material. According to the information provided by the manufacturer, MI has higher flexural strength than RU. In terms of flexural strength, MI had higher flexural strength than other cements however it was not corresponding to the filler loading of MI which had weight percent of filler less than the others. Furthermore, the main components of RU are methacrylate monomers, partially containing phosphoric acid groups and alkaline fillers. Water product that comes from the neutralization through the reactions between phosphoric acid groups and alkaline fillers is claimed to provide the cement's initially hydrophillicity. According to previous study, RU group had higher water absorption and water solubility than MI group [79]. The higher hydrophilicity of the composite, the higher its tendency to absorb water and to swell. The absorption of water might affect the bond strength of cement. This explained the shear bond strength of RU group that lower than MI group. However, study had shown that some resin monomers including phosphoric acid group are able to react chemically and bond with the superficial oxide layer of base metal alloys [80]. This might explained the failure mode in RU group that appeared mostly mixed failure and only one adhesive failure in between cement and titanium was found. Another factor could be considered when evaluating the difference between luting cement materials is the silane coupling agent. One study showed the shear bond strength of MI on titanium higher than that of RU. The result of this study showed the same direction that the shear bond strength of MI group higher than RU group. In one study, shear bond strength values of MI group showed the similar bond function to titanium and pressed lithium disiligate [79], corresponding to the mixed failure found in this study. Focusing on the individual primer recommended by the manufacturer, Monobond plus comprises three different active ingredients in an ethanolic solution. The silane methacrylate group establishes a bond to silicate ceramic materials. The phosphoric acid methacrylate group is responsible for bonding to base metals. The use of a three different active ingredients in an ethanolic solution was a precondition to obtaining durable bonding between resin cement and ceramic in a previous study [81] and in the current study. The most commonly applied silane use in dental laboratories and chairside applications is 3-trimethoxysilylpropyl methacrylate (MPS). The MPS silane used in this study were monobond s and ceramic primer which were used with MI and RU respectively. Any resin composite material that contains methacrylate groups in their composition could therefore be used in conjunction with MPS silanes. This explained the shear bond strength of MI and RU which statistically significant difference from SB and PF. While the primers using with PF group and SB group contains VTD that did not have affinity to titanium. This explained the statistically lower shear bond strength of SB and PF than MI and RU. It was likely due to ineffective of primer for bonding base metal alloys.

The nature of adhesive strength of the materials at the interface was depicted by the shear bond strength. Bonding of a restorative material yields greatest significance, which directly indicates the clinical success. Shear bond strength can be assumed clinical important to restorative materials because the major dislodging forces at the restoration interface have shearing effect.

The importance of resin cement film thickness currently relies on the fact that most of the ceramic materials have considerable internal space. The effect of resin cement thickness on the fracture resistance of all-ceramic restorations is not completely established yet, but some studies have found some correlation between these factors. In 1994, Scherrer and colleagues [82] reported that when resin cement thickness of 300 mm or more was present, a gradual decrease of the fracture strength was observed [83] demonstrating that thick ceramic combined with minimal thickness of luting composite provided restorations with a favorable configuration with regard to prevent cracking [83]. It was also demonstrated that glass–ceramics with thick cement layers exhibit significantly lower reliability after water aging [84]. It seems that a thin cement thickness and proper bond to the ceramic structure is necessary for improved support and increased fracture resistance of all-ceramic crowns. Thicker cement layers have also been related to decreased bond strength of ceramic systems [85]. Thus, clinicians are strongly advised to maintain a minimal film thickness (approximately 50 mm) to minimize the effects of water sorption and its consequences to the properties of the cement and respective support for the ceramic restoration.

In addition, the cement thickness of representatives of each group in this study had the different cement thickness and difference shear bond strength. The shear bond strength values of pressed lithium disilicate glass-ceramic bonded with titanium tended to decrease when the film thickness increased. There were studies mentioned the influence of the film thickness of resin luting agents on strength [86, 87]. The cement thickness might influence the shear bond strength, which should be evaluated in further study

Conclusion

Multilink implant$^{®}$ showed superior bond strength to prosthodontics substrates and thus it was more applicable to clinical situations. However, selection of the cements should be determined by using all related suitable criteria for the specific need. These in vitro results need to be further studies in a clinical context.

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APPENDIX

Appendix A. The data of shear bond strength test form MI group

Specimen	Shear strength	Mode of failure	Characteristics
1	54.25	1	A
2	59.83	1	B
3	55.67	1	A
4	57.99	1	A
5	59.12	1	B
6	60.88	1	CF
7	60.52	1	CF
8	54.86	1	B
9	56.85	1	B
10	60.01	1	B
11	58.30	1	A
12	59.29	1	B
13	58.49	1	B
14	60.02	1	B
15	57.98	1	B

Mean of shear bond strength 58.2707

Standard deviation 2.05179

Mode of failure

(1) Mixed of cohesive and adhesive failures,

(2) Cohesive failure, and

(3) Adhesive failure

Characteristics = Characteristics of remnant cement

(A) Cement remaining adhere to ceramic specimen more than 50%

(B) Cement remaining adhere to titanium specimen more than 50%

(AB) Cement remaining adhere to both side of specimen more than 50%

(CF) Ceramic fracture

Appendix B. The data of shear bond strength test form RU group

Specimen	Shear strength	Mode of failure	Characteristics
1	43.99	3	A
2	49.16	1	B
3	48.87	1	B
4	45.18	1	A
5	45.41	1	A
6	43.66	1	B
7	44.05	2	AB
8	45.89	1	A
9	46.05	1	A
10	44.72	1	B
11	44.66	1	B
12	46.66	1	A
13	49.11	1	A
14	44.03	2	AB
15	45.77	1	A

Mean of shear bond strength 45.8140

Standard deviation 1.88096

Mode of failure

(1) Mixed of cohesive and adhesive failures,

(2) Cohesive failure, and

(3) Adhesive failure

Characteristics = Characteristics of remnant cement

(A) Cement remaining adhere to ceramic specimen more than 50%

(B) Cement remaining adhere to titanium specimen more than 50%

(AB) Cement remaining adhere to both side of specimen more than 50%

(CF) Ceramic fracture

Appendix C. The data of shear bond strength test form PF group

Specimen	Shear strength	Mode of failure	Characteristics
1	42.13	1	B
2	36.00	3	A
3	38.56	1	A
4	42.81	1	B
5	41.55	1	B
6	41.14	1	AB
7	37.06	2	A
8	39.45	1	B
9	41.29	1	B
10	38.08	2	AB
11	36.99	3	A
12	37.57	2	AB
13	40.67	1	B
14	38.11	1	B
15	39.90	1	B

Mean of shear bond strength 39.4207

Standard deviation 2.11233

Mode of failure

(1) Mixed of cohesive and adhesive failures,

(2) Cohesive failure, and

(3) Adhesive failure

Characteristics = Characteristics of remnant cement

(A) Cement remaining adhere to ceramic specimen more than 50%

(B) Cement remaining adhere to titanium specimen more than 50%

(AB) Cement remaining adhere to both side of specimen more than 50%

(CF) Ceramic fracture

Appendix D. The data of shear bond strength test form SB group

Specimen	Shear strength	Mode of failure	Characteristics
1	41.43	1	B
2	37.09	3	A
3	37.18	2	AB
4	40.43	1	B
5	45.54	1	B
6	36.53	3	A
7	40.54	1	B
8	39.71	1	B
9	41.34	1	B
10	38.77	1	B
11	37.89	2	AB
12	39.11	1	B
13	37.99	1	A
14	40.84	1	B
15	38.86	1	A

Mean of shear bond strength 39.5500

Standard deviation 2.28846

Mode of failure

(1) Mixed of cohesive and adhesive failures,

(2) Cohesive failure, and

(3) Adhesive failure

Characteristics = Characteristics of remnant cement

(A) Cement remaining adhere to ceramic specimen more than 50%

(B) Cement remaining adhere to titanium specimen more than 50%

(AB) Cement remaining adhere to both side of specimen more than 50%

(CF) Ceramic fracture

Appendix E. MI case processing summary

		Cases					
		Valid		Missing		Total	
	group	N	Percent	N	Percent	N	Percent
shear	MI	15	100.0%	0	.0%	15	100.0%

Descriptives

	group			Statistic	Std. Error
shear	MI		Mean	58.2707	.52977
		95% Confidence Interval for Mean	Lower Bound	57.1344	
			Upper Bound	59.4069	
			5% Trimmed Mean	58.3491	
			Median	58.4900	
			Variance	4.210	
			Std. Deviation	2.05179	
			Minimum	54.25	
			Maximum	60.88	
			Range	6.63	
			Interquartile Range	3.16	
			Skewness	-.763	.580
			Kurtosis	-.393	1.121

Tests of Normality

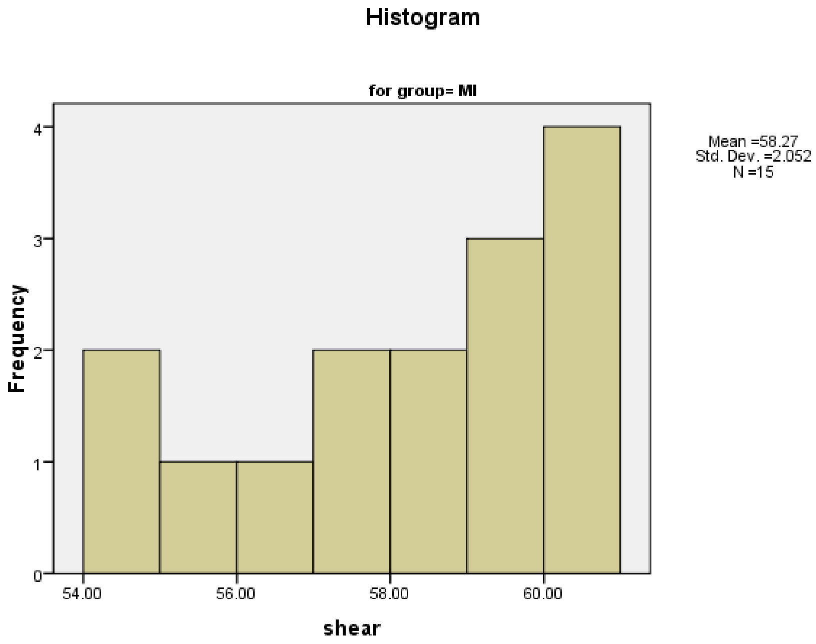
		Kolmogorov-Smirnova			Shapiro-Wilk		
	group	Statistic	df	Sig.	Statistic	df	Sig.
shear	MI	.177	15	.200*	.920	15	.195

a. Lilliefors Significance Correction

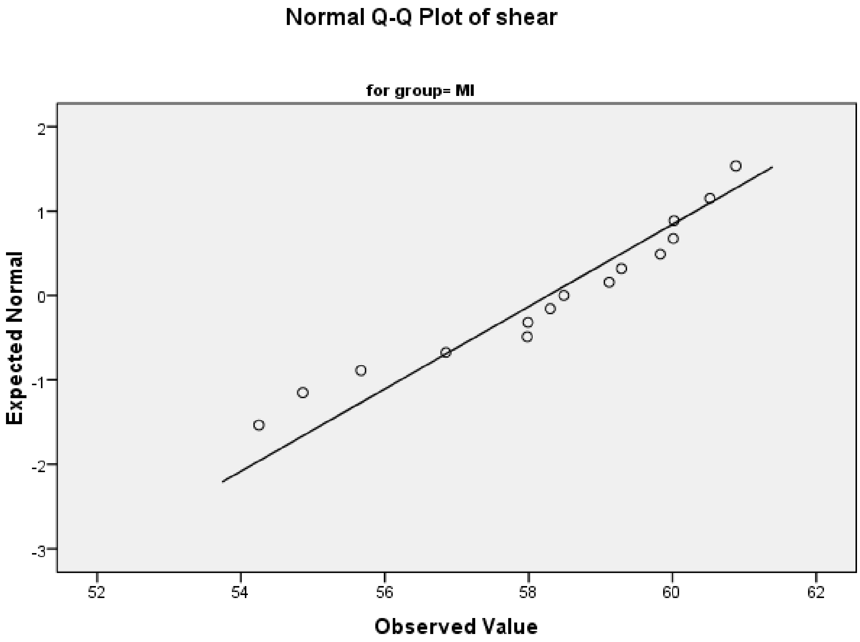
*. This is a lower bound of the true significance.

shear

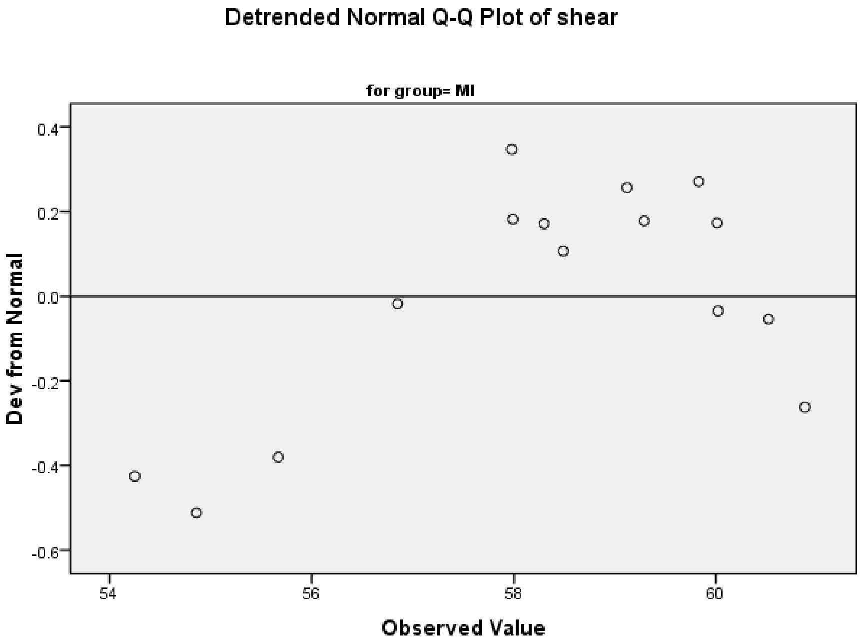
Histograms

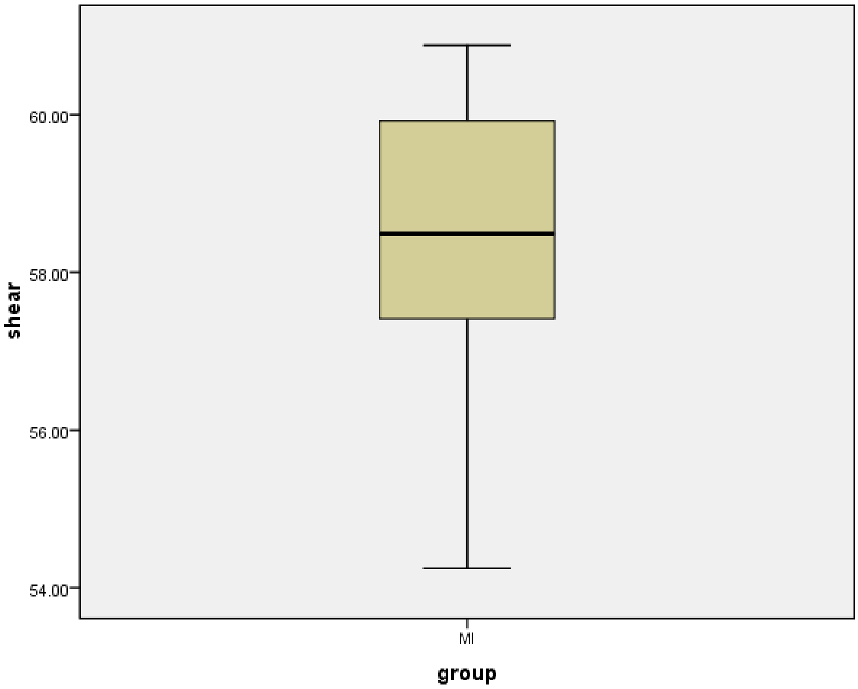


Normal Q-Q Plots



Detrended Normal Q-Q Plots




shear
60.00
58.00
56.00
54.00
MI
group

Appendix F. RU case processing summary

		Cases					
		Valid		Missing		Total	
	group	N	Percent	N	Percent	N	Percent
shear	RU	15	100.0%	0	.0%	15	100.0%

Descriptives

	group			Statistic	Std. Error
shear	RU		Mean	45.8140	.48566
		95% Confidence Interval for Mean	Lower Bound	44.7724	
			Upper Bound	46.8556	
			5% Trimmed Mean	45.7478	
			Median	45.4100	
			Variance	3.538	
			Std. Deviation	1.88096	
			Minimum	43.66	
			Maximum	49.16	
			Range	5.50	
			Interquartile Range	2.61	
			Skewness	.892	.580
			Kurtosis	-.373	1.121

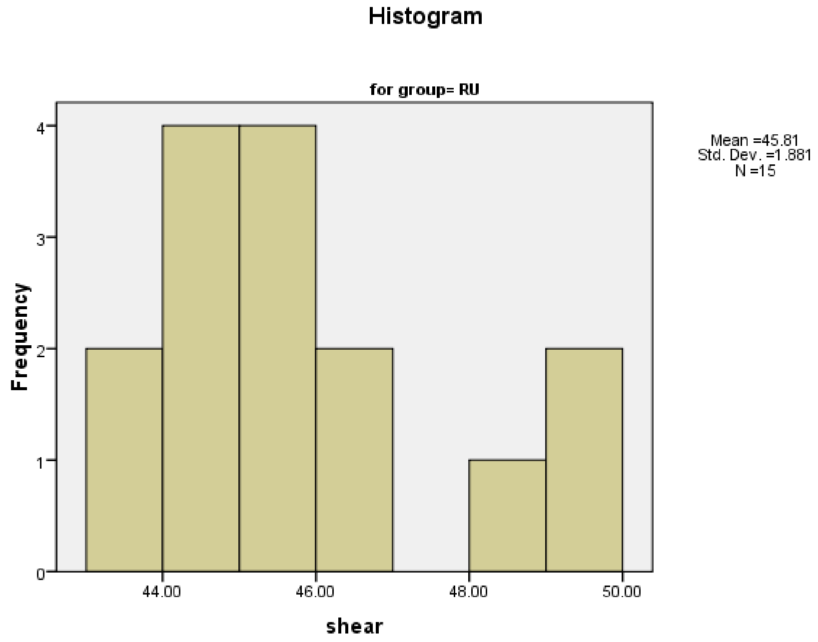
Tests of Normality

	Kolmogorov-Smirnova			Shapiro-Wilk		
group	Statistic	df	Sig.	Statistic	df	Sig.
shear RU	.183	15	.187	.863	15	.027

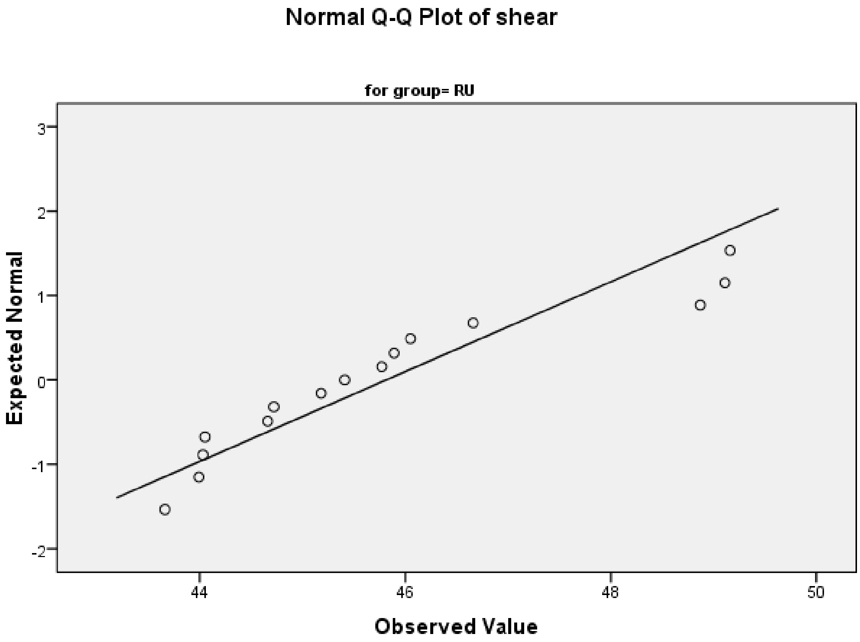
a. Lilliefors Significance Correction

shear

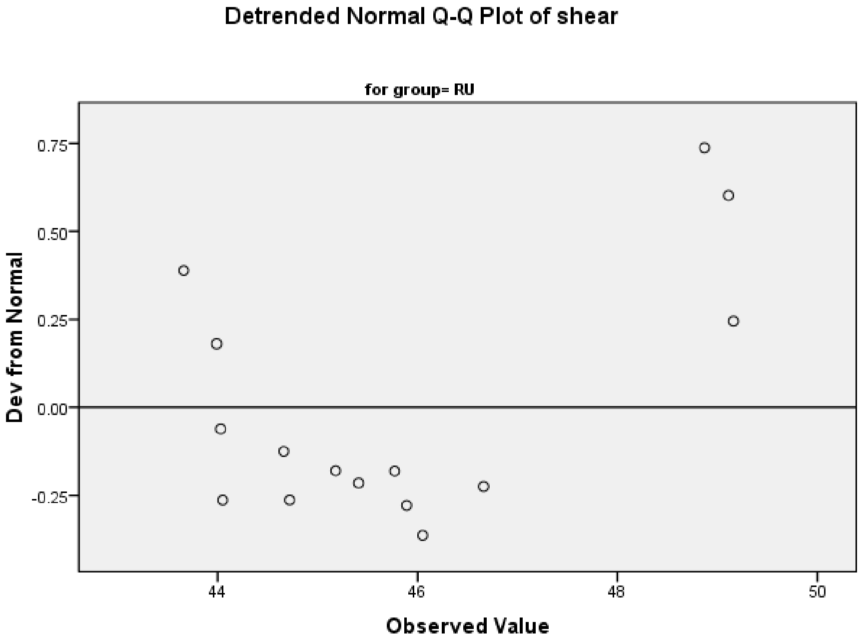
Histograms

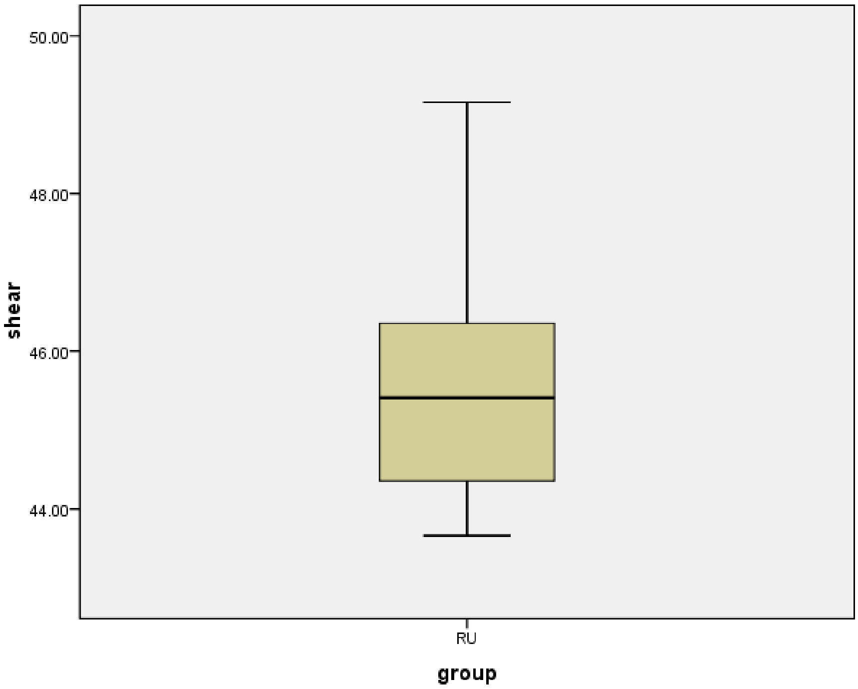


Normal Q-Q Plots



Detrended Normal Q-Q Plots




50.00
48.00
46.00
44.00
shear
RU
group

Appendix G. PF case processing summary

		Cases					
		Valid		Missing		Total	
	group	N	Percent	N	Percent	N	Percent
shear	PF	15	100.0%	0	.0%	15	100.0%

Descriptives

	group			Statistic	Std. Error
shear	PF		Mean	39.4207	.54540
		95% Confidence Interval for Mean	Lower Bound	38.2509	
			Upper Bound	40.5904	
			5% Trimmed Mean	39.4224	
			Median	39.4500	
			Variance	4.462	
			Std. Deviation	2.11233	
			Minimum	36.00	
			Maximum	42.81	
			Range	6.81	
			Interquartile Range	3.72	
			Skewness	.026	.580
			Kurtosis	-1.279	1.121

Tests of Normality

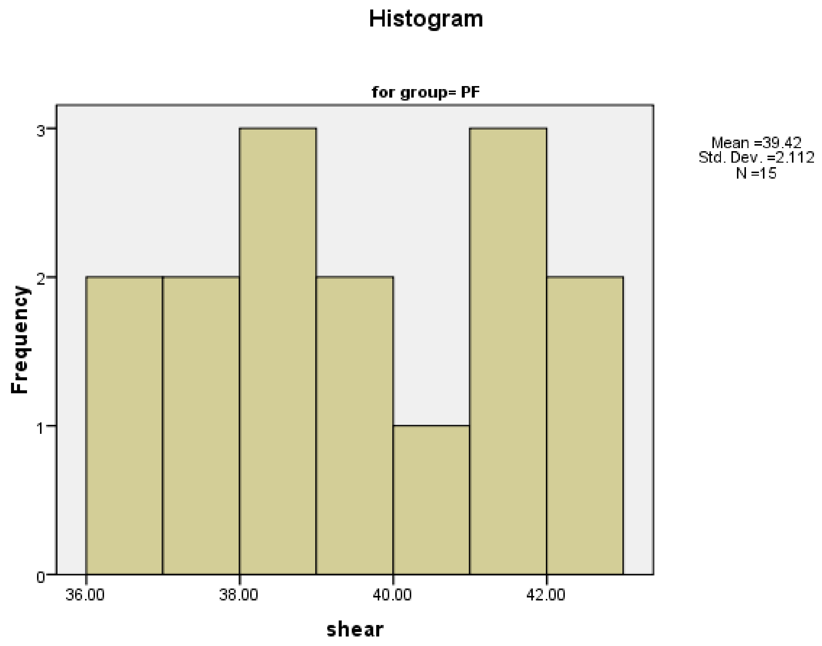
group		Kolmogorov-Smirnova			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
shear	PF	.133	15	.200*	.955	15	.604

a. Lilliefors Significance Correction

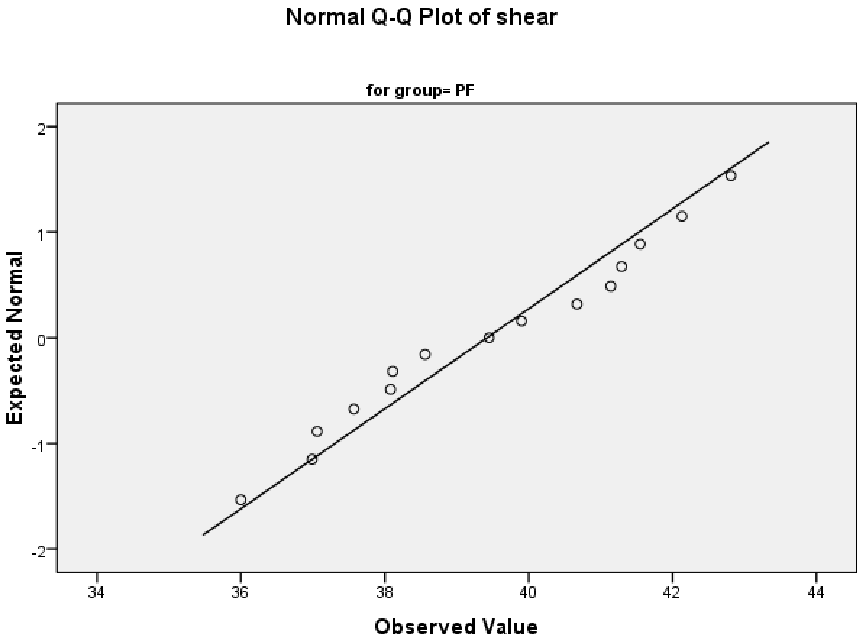
*. This is a lower bound of the true significance.

shear

Histograms



Normal Q-Q Plots



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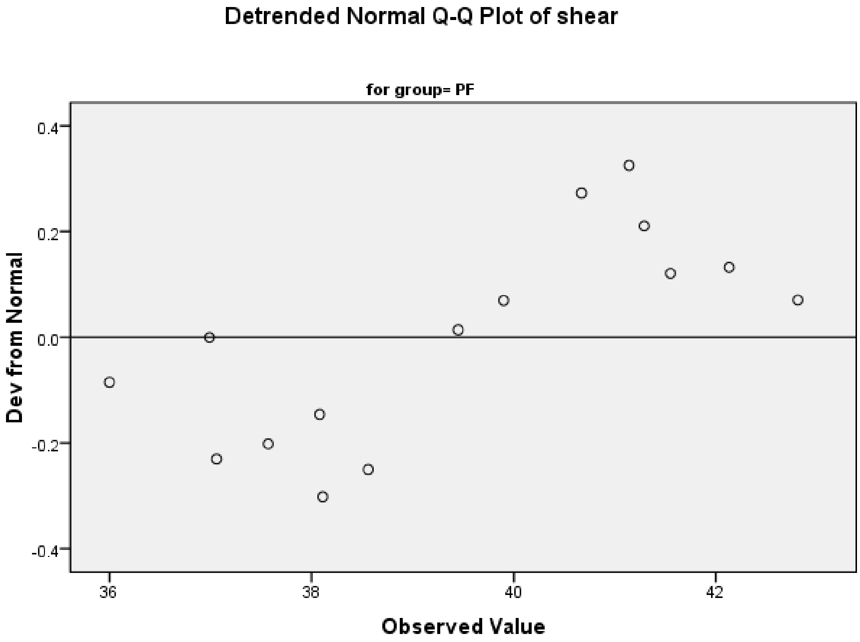
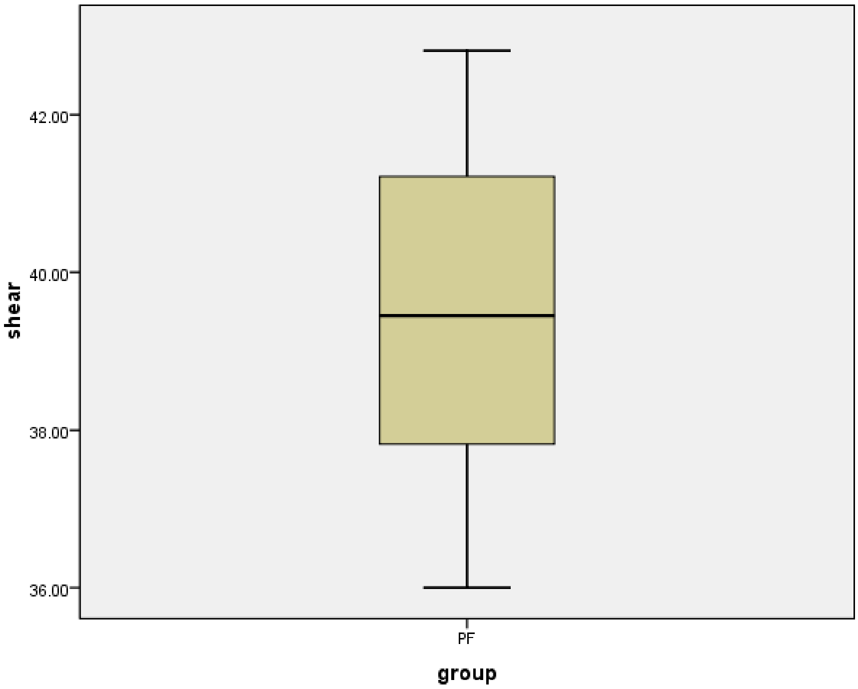



42.00
40.00
38.00
36.00
shear
PF
group

Appendix H. SB case processing summary

		Cases					
		Valid		Missing		Total	
	group	N	Percent	N	Percent	N	Percent
shear	SB	15	100.0%	0	.0%	15	100.0%

Descriptives

	group			Statistic	Std. Error
shear	SB		Mean	39.5500	.59088
		95% Confidence Interval for Mean	Lower Bound	38.2827	
			Upper Bound	40.8173	
			5% Trimmed Mean	39.3850	
			Median	39.1100	
			Variance	5.237	
			Std. Deviation	2.28846	
			Minimum	36.53	
			Maximum	45.54	
			Range	9.01	
			Interquartile Range	2.95	
			Skewness	1.142	.580
			Kurtosis	2.161	1.121

Tests of Normality

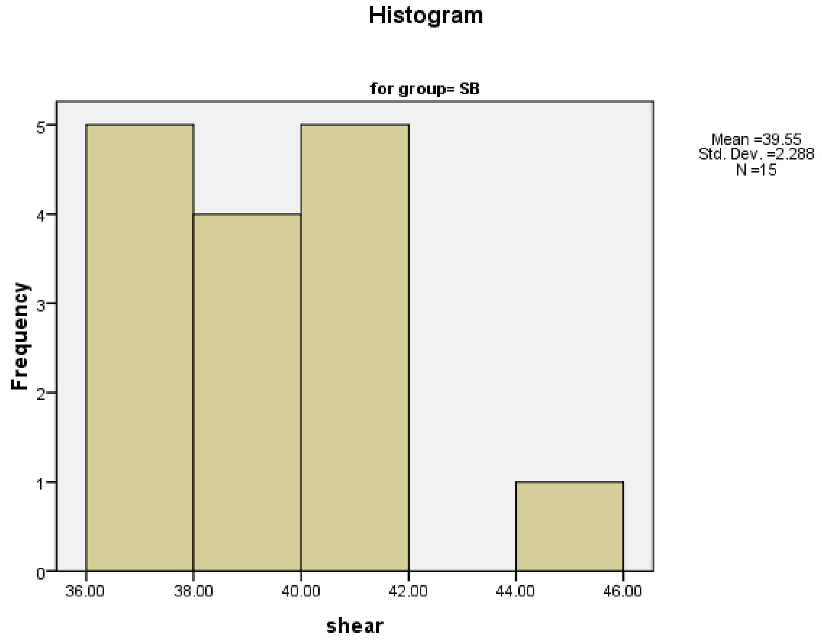
		Kolmogorov-Smirnova			Shapiro-Wilk		
	group	Statistic	df	Sig.	Statistic	df	Sig.
shear	SB	.139	15	.200*	.916	15	.167

a. Lilliefors Significance Correction

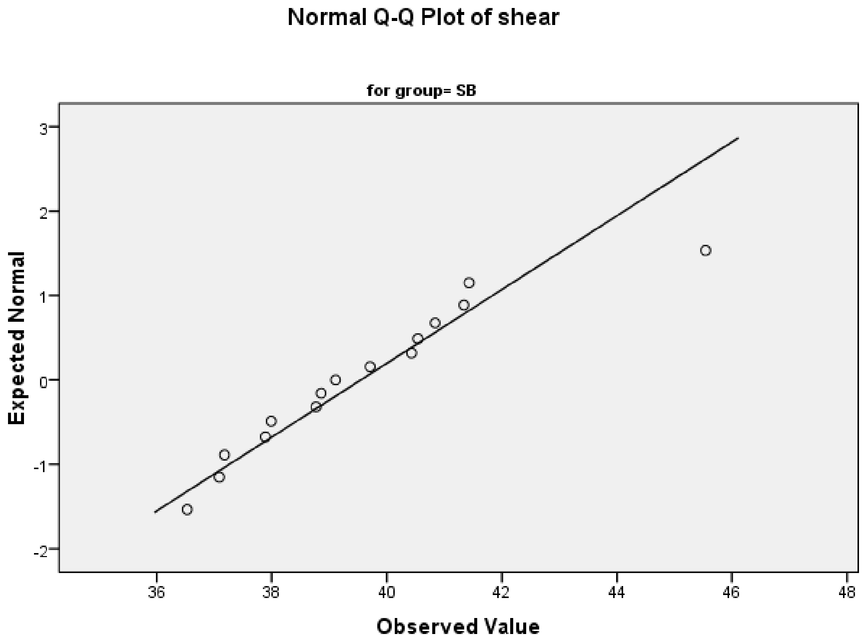
*. This is a lower bound of the true significance.

shear

Histograms



Normal Q-Q Plots



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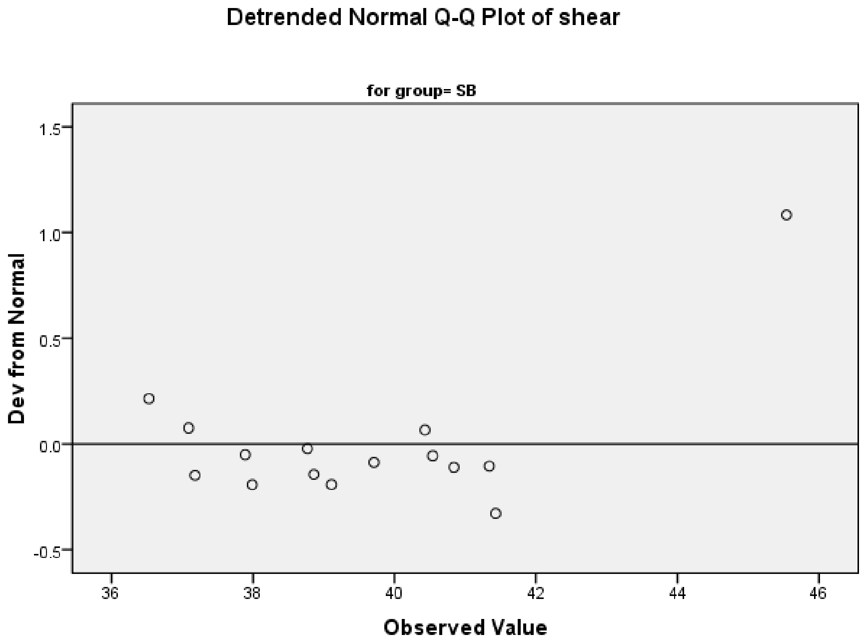

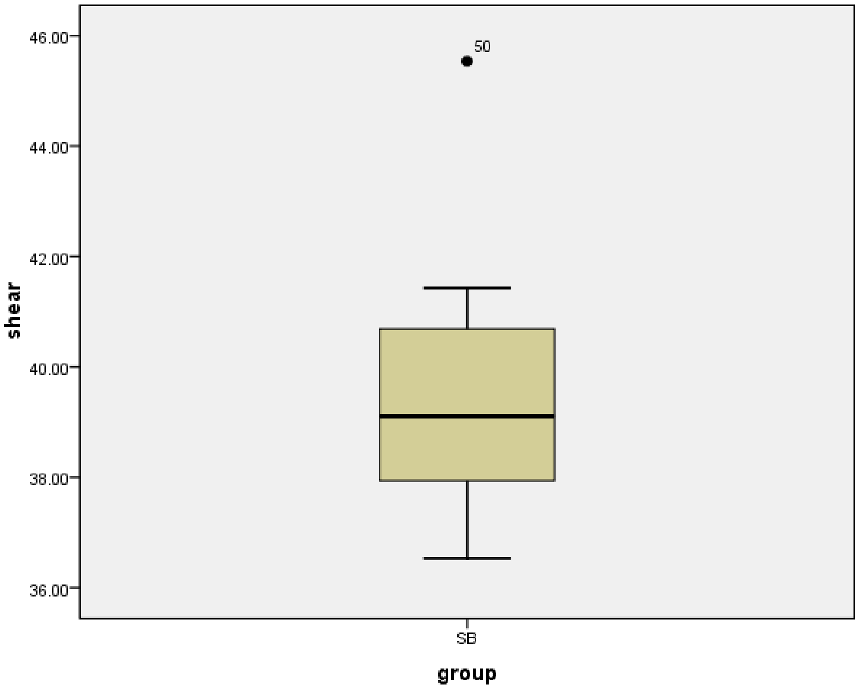
46.00
44.00
42.00
40.00
38.00
36.00
shear
50
SB
group

Appendix I. One-way ANOVA

Descriptives

shear

					95% Confidence Interval for Mean	
	N	Mean	Std. Deviation	Std. Error	Lower Bound	Upper Bound
MI	15	58.2707	2.05179	.52977	57.1344	59.4069
RU	15	45.8140	1.88096	.48566	44.7724	46.8556
PF	15	39.4207	2.11233	.54540	38.2509	40.5904
SB	15	39.5500	2.28846	.59088	38.2827	40.8173
Total	60	45.7638	7.99716	1.03243	43.6979	47.8297

Descriptives

shear

	Minimum	Maximum
MI	54.25	60.88
RU	43.66	49.16
PF	36.00	42.81
SB	36.53	45.54
Total	36.00	60.88

Test of Homogeneity of Variances

shear

Levene Statistic	df1	df2	Sig.
.263	3	56	.852

ANOVA

shear

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3529.063	3	1176.354	269.700	.000
Within Groups	244.256	56	4.362		
Total	3773.319	59			

Appendix J. Post Hoc Test

Multiple Comparisons

shear

LSD

(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
MI	RU	12.45667*	.76260	.000	10.9290	13.9843
	PF	18.85000*	.76260	.000	17.3223	20.3777
	SB	18.72067*	.76260	.000	17.1930	20.2483
RU	MI	-12.45667*	.76260	.000	-13.9843	-10.9290
	PF	6.39333*	.76260	.000	4.8657	7.9210
	SB	6.26400*	.76260	.000	4.7363	7.7917
PF	MI	-18.85000*	.76260	.000	-20.3777	-17.3223
	RU	-6.39333*	.76260	.000	-7.9210	-4.8657
	SB	-.12933	.76260	.866	-1.6570	1.3983
SB	MI	-18.72067*	.76260	.000	-20.2483	-17.1930
	RU	-6.26400*	.76260	.000	-7.7917	-4.7363
	PF	.12933	.76260	.866	-1.3983	1.6570

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

VITA

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