The influence of fatigue load to various adhesive resin luting agents in a zirconia-reinforced lithium silicate ceramic bonded to dentin

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science in Esthetic Restorative and Implant Dentistry
Common Course
FACULTY OF DENTISTRY
Chulalongkorn University
Academic Year 2020

อิทธิพลของแรงที่เกิดจากความล้าต่อการยึดติดด้วยสารยึดติดระบบต่างๆระหว่างวัสดุลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนียกับเนื้อฟัน

น.ส.รติกร วัฒนานิยม

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม ไม่สังกัดภาควิชา/เทียบเท่า
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2563

Thesis Title	The influence of fatigue load to various adhesive resin luting agents in a zirconia-reinforced lithium silicate ceramic bonded to dentin
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Field of Study	Esthetic Restorative and Implant Dentistry
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รติกร วัฒนานิยม : อิทธิพลของแรงที่เกิดจากความล้าต่อการยึดติดด้วยสารยึดติดระบบต่างๆระหว่างวัสดุลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนียกับเนื้อฟัน. (The influence of fatigue load to various adhesive resin luting agents in a zirconia-reinforced lithium silicate ceramic bonded to dentin) อ.ที่ปรึกษาหลัก : รศ. ทพญ. ดร.ศิริวิมล ศรีสวัสดิ์

วัตถุประสงค์: เพื่อทดสอบค่าแรงที่ทำให้เกิดการแตกหักจากความล้าของสารยึดติดระบบเอชแอนด์ริ้นส์ และเซลฟ์แอดฮีซีฟ ที่ใช้ยึดลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนีย (ZLS) กับเนื้อฟัน นอกจากนั้นการศึกษานี้ยังประเมินการใช้สารเรซินที่ไม่มีวัสดุอัดแทรกบนผิวเซรามิคที่ได้รับการทาไซเลนแล้วสามารถทำให้ค่าแรงที่ทำให้เกิดการแตกหักจากความล้าเพิ่มขึ้นหรือไม่ การศึกษา: ก้อนไวต้า สุพรินิตี้ [Vita Suprinity (VS, Vita Zahnfabrik)] ถูกตัดเป็นทรงกระบอกขนาดเส้นผ่านศูนย์กลาง 5 มม. และสูง 1.5 มม. โดย VS จะเข้าสู่กระบวนการตกผลึกทั้งหมด และ พื้นผิวด้านยึดติดถูกปรับสภาพดังนี้ การทาฮีลิโอบอนด์ [Heliobond (HB, Ivoclar Vivadent)] หลังจากใช้ไซเลน และการไม่ทา HB จากนั้นแผ่น VS ถูกยึดติดกับเนื้อฟันของฟันกรามใหญ่มนุษย์ ด้วยสารยึดติดออฟติบอนด์เอฟแอลร่วมกับเน็กซัสทรี [Optibond FL (FL, Kerr) with Nexus3 (NX3, Kerr)] รีไลด์เอ็กซ์ ยูนิเซม [RelyX^{TM} Unicem (UC, 3M ESPE)] และ แม็กเซมอีลิต [Maxcem Elite (ME, Kerr)] จากนั้นทุกชิ้นงานถูกนำไปวัดค่าแรงที่ทำให้เกิดการแตกหักจากความล้าหลังจากยึดติด 24 ชั่วโมงโดยใช้วิธีทดสอบแบบขั้นบันได (staircase approach) กำหนดจำนวนรอบคงที่ที่ 500,000 รอบ และอัตราเร็วของการทดสอบ 20 รอบต่อวินาที โดยมีแรงเริ่มต้นที่ 844 นิวตัน และช่วงความกว้างของขั้นบันได 42 นิวตัน นอกจากนั้นชิ้นส่วนที่แตกหักจะถูกนำไปประเมินด้วยกล้องจุลทรรศน์อิเล็กตรอนชนิดส่องกราด (SEM) ผลการศึกษา การทดสอบทางสถิติที บีเรนส์-ฟิชเชอร์ (Behrens-Fisher T-test) พบว่า ZLS ที่ยึดกับเนื้อฟันด้วยเซลฟ์แอดฮีซีฟ (UC และ ME) มีค่าแรงที่ทำให้เกิดการแตกหักจากความล้าต่ำกว่าเอชแอนด์ริ้น (FLNX3) อย่างมีนัยสำคัญทางสถิติ (alpha = 0.05) ในขณะที่กลุ่มที่มีการใช้ HB ไม่พบความต่างอย่างมีนัยสำคัญทางสถิติเมื่อเปรียบเทียบกับกลุ่มที่ไม่ใช้ (alpha = 0.05)

สรุป สารยึดติดระบบเอชแอนด์ริ้นส์ถูกแนะนำสำหรับการยึด ZLS กับเนื้อฟัน โดยที่ไม่จำเป็นต้องใช้สารเรซินที่ไม่มีวัสดุอัดแทรกบนผิวเซรามิคด้านยึดติดของชิ้นงานหลังจากการทาไซเลน

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

6075829032 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY
KEYWORD:

Ratikorn Watananiyom : The influence of fatigue load to various adhesive resin luting agents in a zirconia-reinforced lithium silicate ceramic bonded to dentin. Advisor: Assoc. Prof. SIRIVIMOL SRISAWASDI, D.D.S., M.S., Ph.D.

Purpose. To examine fatigue failure load value of etch-and-rinse and self-adhesive luting systems used to bond ZLS to dentin. Moreover, this study seeks to evaluate whether the application of unfilled resin on silanated ceramic intaglio surface could improve fatigue failure load value. Methods. Vita Suprinity (VS, Vita Zahnfabrik) blocks were sectioned into cylindrical shape (5 mm in diameter and 1.5 mm in height). All VS were crystallized, and bonded surfaces were treated as followed: Heliobond (HB, Ivoclar Vivadent) application after silanization and non-application of HB. Each VS was cemented to each flat occlusal dentin surface of extracted human molar, following the adhesive luting systems: Optibond FL (FL, Kerr) with Nexus3 (NX3, Kerr), RelyX^{TM} Unicem (UC, 3M ESPE), and Maxcem Elite (ME, Kerr). 24-hour mean fatigue failure load was determined using a staircase approach (500,000 cycles, 20Hz, initial load = 844 N, step size = 42 N). Representatives of failed specimens were evaluated by a scanning electron microscope (SEM). Results. The Behrens-Fisher T-test revealed that ZLS cemented to dentin using self-adhesive resin luting cements (UC and ME) had a statistically significant lower mean fatigue failure load value than etch-and-rinse resin luting cement (FLNX3) (alpha = 0.05). Meanwhile, the HB application groups did not achieve statistically significant difference in fatigue failure load value when compared to non-application groups (alpha = 0.05). Conclusion. Etch-and-rinse resin luting system was recommended for cementation of ZLS to dentin, regardless the use of unfilled resin on the intaglio surface of the restoration after silanization.

Field of Study:	Esthetic Restorative and Implant Dentistry	Student's Signature ....
Academic Year:	2020	Advisor's Signature ....

ACKNOWLEDGEMENTS

A great deal of guidance and assistance from many people were needed for the completion of this thesis. I would like to take this time to express my appreciation to those who supported me throughout this research.

First of all, I would like to express my special thanks of gratitude to my advisor, Associate Professor Sirivimol Srisawasdi, Ph.D., who was incredibly supportive and gave invaluable assistance, advice and encouragement. Without her guidance and help, this research would not have been possible.

I also would like to thank to the member of supervisory committee, Prof. Mansuang Arksornnukit, Ph.D and Asst.Prof. Tool Sriamporn, Ph.D. for sharing their experience and having several useful comments that enriched the approach of this research.

I would like to share my deepest appreciation to Assist. Prof. Dr. Soranun Chantarangsu for her recommendations and help regarding statistical approaches.

Sincere gratitude is also extended to all the personnel of the CU Dental Innovation Centre, Faculty of Dentistry, Chulalongkorn University, for their laboratory assistance.

Finally, I also acknowledge with deep sense of my gratitude toward my friends and my family who have provided love, thoughtfulness and encouragement through this venture.

Ratikorn Watananiyom

TABLE OF CONTENTS

Page

LIST OF TABLES

LIST of FIGURES

CHAPTER I INTRODUCTION

Rationale and Significance of the Problem

Dental ceramic can now be fabricated by both traditional laboratory methods and computer-aided design/computer-aided manufacturing (CAD/CAM). CAD/CAM technology has enabled clinicians to provide high-quality and high precision ceramic restorations with reduced fabrication time(1). Recently, zirconia-reinforced lithium silicate glass-ceramics (ZLS) were launched in the market. These new glass-ceramics were designed to integrate high mechanical properties of zirconia and optical properties of glass-ceramics(2). ZLS, Celtra Duo® (Dentsply, Hanau-Wolfgang, Germany) was introduced in 2012, and Vita Suprinity® (VITA Zahnfabrik H. Rauter GmbH & Co. KG, Bad Säckingen, Germany) 2013. These materials contain lithium metasilicate (Li_2SO_3) crystallites and zirconia crystals in glass matrix(3). Belli et al. (2017) identified distinct microstructural differences between the two materials. Celtra Duo® had larger lithium metasilicate crystals (Li_2SO_3 phase), more than 1 micron in length, compared to Vita Suprinity®, at about 0.5 micron(4). The flexural strength of ZLS block was 370 MPa(3).

Although unsupported glass ceramics exhibited high flexural strength, they were prone to fracture under chewing loads(4, 5). Consequently, adequate clinical adhesive cementation played an important role in ensuring a long-term bond between ceramic and tooth structures(5, 6). Gold-standard protocol for glass ceramic surface treatment was etching with hydrofluoric acid (HF) followed by application of a silane coupling agent(7, 8). Meanwhile, studies(9, 10) revealed that the use of only silane was not sufficient to achieve a close contact between ceramic and resin cement. For this reason, other methods have been investigated for ceramic surface treatment**,** including applying an unfilled resin after silane application because of its low viscosity(9). Sundfeld Neto et al.(10)found that unfilled resin penetrated into pores on ceramic surface more completely than resin cement, whereas ceramic surface that was not applied with unfilled resin showed more voids and non-homogenous interface as seen in SEM image, leading to compromised bond strength(11). On the other hand, a study(12) found that unfilled resin was dispensable for significant increased bond strength of ceramic material. Therefore, the use of unfilled resin on silanated ceramic intaglio surface prior to application of resin cement is still controversial.

Selection of adhesive resin luting agent has been shown to be responsible for variation in the bond strength(6). Resin cement had not only excellent aesthetic shade matching potential, but also good adhesion to tooth structure, thus reinforcing the ceramic structure(13). Although conventional resin cement used with etch-and-rinse adhesive had been the best proven performance for all-ceramic restorations cementation(14), the system required several steps resulting in high technique sensitivity(15). Self-adhesive resin cement is developed to simplify the application of cementation procedure that do not require any pretreatment of tooth surface.

Moreover, self-adhesive resin cement was an acidic material which demineralized tooth surface and subsequently allowed for penetration of resin cement resulting in micromechanical retention(15-17).

There have been studies concerning the efficacy of adhesive resin cement including conventional resin cement used with etch-and-rinse adhesive, and self-adhesive resin luting cement on glass-ceramics(6, 18). However, the effectiveness of these cementation systems on fatigue behavior of ZLS glass-ceramics has not been thoroughly investigated. In addition, the studies(10-12) emphasizing on the use of unfilled resin on silanated ceramic surface have been rare.

Research Questions

1. What are the efficacies of etch-and-rinse resin luting cement and self-adhesive resin luting cement in terms of fatigue failure load on a zirconia-reinforced lithium silicate ceramic bonded to dentin?

2. Would an unfilled resin improve fatigue failure load when it was applied on a zirconia-reinforced lithium silicate ceramic surface after silane application?

Research Objectives

1. To examine fatigue failure load value of etch-and-rinse and self-adhesive luting systems used to bond ZLS to dentin.
2. To evaluate whether the application of an unfilled resin on silanated ceramic intaglio surface could improve fatigue failure load value.

Hypotheses

Null hypothesis

1. There was no significant difference in fatigue failure load value of ZLS cemented to dentin utilizing self-adhesive resin luting systems compared to a control three-step etch and rinse resin luting system.

2. There was no significant difference in fatigue failure load of HB application on silanated ceramic surface group when compare to non-HB application group.

Alternative hypothesis

1. There was at least one significant difference in fatigue failure load of three-step etch-and-rinse adhesive luting agent and self-adhesive resin luting agent used to bond ZLS glass-ceramic to dentin.

2.There was significant difference in fatigue failure load between HB application on silanated ceramic surface group and non-HB application group.

Conceptual Framework

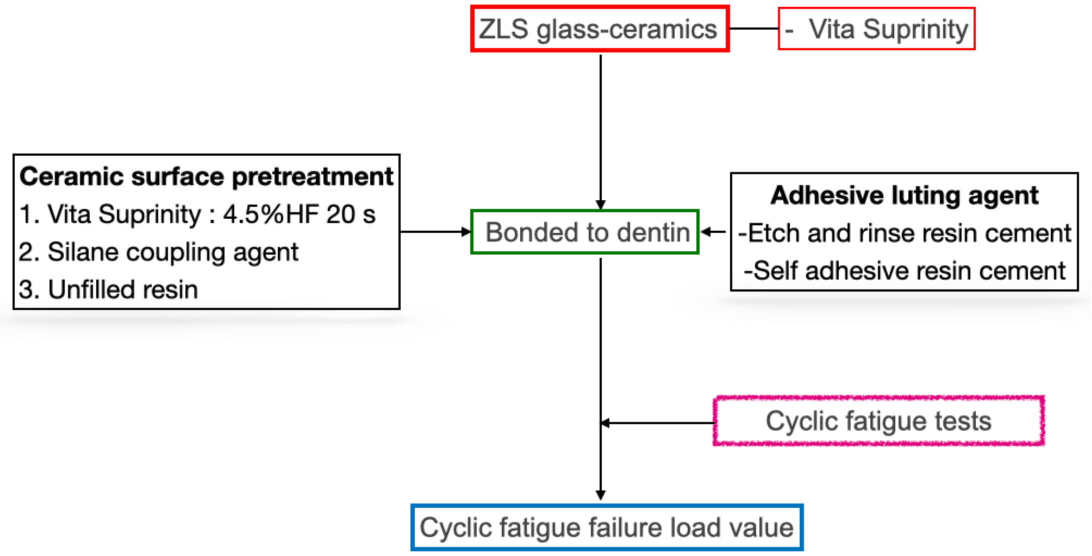


Figure 1. **Diagram of Conceptual Framework**

Keywords: Adhesive resin luting agent, Ceramic surface pretreatment, Cyclic fatigue failure load, Hydrofluoric acid (HF), Zirconia-reinforced lithium silicate (ZLS) ceramics

Expected Benefit of the Study

The outcomes of this study may provide useful clinical information for practitioners when selecting adhesive resin luting agent to carry out cementation and determining appropriate surface treatment approach for the new CAD/CAM zirconia-reinforced lithium silicate ceramic bonded to tooth structure.

CHAPTER II REVIEW OF THE LITERATURES

The literatures in these following topics have been reviewed.

Dental chairside CAD/CAM ceramic materials

Surface pretreatment of dental ceramic

Adhesive systems

Luting cements

Cyclic fatigue loading

Dental chairside CAD/CAM ceramic materials

Traditional methods to produce dental restoration have been used for decades, with long-term clinical stability and survival. However, recently, computer-aided design/computer-aided manufacturing (CAD/CAM) has introduced and enhanced dental chairside restoration. CAD/CAM technology has been shown to be user friendly, reduces the use of laboratory materials and saves time(19). Systematic review proposed that the overall survival rate of single-tooth ceramic restorations fabricated with CAD/CAM technology was similar to those conventionally manufactured following more than 3 years(20).

Moreover, CAD-CAM systems now allow practitioners to produce restorations using many kinds of materials. Feldspathic porcelains, such as Vita Mark II and Vita TriLuxe Bloc (VITA Zahnfabrik, Bad Sackingen, Germany), contain silicon oxide at about 60-64% by volume and aluminum oxide at about 20-23% by volume, while leucite-reinforced ceramic, such as Empress CAD (Ivoclar-Vivadent, Schaan, Liechtenstein) is a glass-based ceramics containing particles of the crystalline mineral leucite in a glassy matrix. Feldspathic porcelains and leucite-reinforced ceramics are ideal for fabrication of veneers, crowns and partial crowns, and restorations with high esthetic demand but cannot withstand high occlusal force (21). Consequently, lithium disilicate glass ceramic, e.max CAD (Ivoclar-Vivadent Schaan, Liechtenstein) was developed to improve mechanical properties and expand the indications of material such as inlays, onlays, crowns, and three-unit fixed dental prostheses in the anterior region(22). This material is delivered in blocks of pre-crystallized metasilicate phase or “blue” block. This block has low physical strength, but it can be milled easily. After milling, the material requires final heat treatment. The result of final crystallization firing is needle-shaped lithium disilicate crystals (Li2O·2SiO2) of 2 microns in length, randomly orientated and embedded in a 30 vol % residual Li2O·2SiO2 glass phase consisting of dissolved metasilicate crystals(4). The mechanical property of full-crystalized stage of this material increases after firing. Recently, a new ceramic material for dental restoration was introduced as a zirconia-reinforced lithium silicate (ZLS), developed to combine the positive physical properties of zirconia and the esthetic appearance of lithium disilicate glass ceramic as a lithium-metasilicate (Li2SiO3) glass ceramic reinforced with about 10% of zirconium dioxide (ZrO2). ZLS blocks are available at the pre-crystallized stage as Vita Suprinity® (VITA Zahnfabrik, Bad Sackingen, Germany) and at the fully crystallized stage as Celtra Duo® (Dentsply DeTrey). These two blocks have similar microstructures of lithium metasilicates (Li2SO3) round and slightly elongated shapes with round diminutive granules of lithium orthophosphates (Li3PO4). However, Vita Suprinity® (VITA Zahnfabrik, Bad Sackingen, Germany) (∼0.5 microns) has smaller sized lithium metasilicate (Li2SO3) glass ceramic (LMGC) than Celtra Duo (Dentsply DeTrey) (up to ∼1 micron in length). This difference can be explained by the thermal treatment parameter (time, temperature). LMGC controls the nucleation of ZLS under crystallization by heat treatment; Vita Suprinity® (VITA Zahnfabrik, Bad Sackingen, Germany) requires crystallization firing at 840°C for 25 minutes, while Celtra Duo® (Dentsply DeTrey) is ready to use after additional firing at 820°C for 8 minutes(4). After the final crystallization process, ZLS is composed of four times smaller lithium silicate crystals than lithium disilicate glass ceramic. The flexural strength of ZLS block was 370 MPa which tested by Elsaka et al.(3).

Surface pretreatment of dental ceramic

In clinical situation, glass-ceramic surface is etched by HF to create surface irregularities so that resin cement can penetrate into the pores. This procedure improves the bond strength between the tooth structure and resin cement as reported by Guarda et al. who tested lithium disilicate glass ceramic(19). Furthermore, HF etching will remove debris and unwanted oxides which increase wettability of ceramic substrates. Therefore, the selection of an appropriate surface treatment and adhesive system is important for clinical success. The etching efficiency of HF depends on its concentration and etching time. The manufacturers recommend treating the surface, Viita Suprinity® block by etching with 5% HF (IPS ceramic etching; Ivoclar Vivadent) for 20 seconds. The surface of Celtra Duo block should be etched with 5% HF for 30 seconds.

Extending the duration of etching time allowed HF more time to react with silicon-oxygen bonds (SiO_2) on glassy matrix and affected the expressiveness of microroughness of the ceramic surface(20). With increased etching time, glassy matrix lost extensively and crystal phase pulled out from the glass matrix which affected the homogeneous surface leading to compromise the strength of glass-ceramics restoration(23).

Traini et al. found that ZLS, Vita Suprinity® (Vita Zahnfabrik) etched by HF gel at 4.9% for 20 s showed the best result with preservation of microstructure, while increasing etching time to 40 s resulted in surface degradation of ZLS Vita Suprinity® (Vita Zahnfabrik) microstructure. Moreover, they found that ZLS material was largely destroyed, and surface degradation was increased when increasing HF concentration to 9.5% either for 20 s or 40s (22).

Monteiro et al. demonstrated that mean fatigue failure loads of ZLS when 5% HF etching were not affected by the etching duration (HF5-30 s = HF5-60 s = HF5-90 s), while etching time had statistically significant effect at 10% HF on fatigue failure load values(24). However, the use of HF etching requires careful attention because of potential risk of soft tissue damage (25).

After etching, ceramic surface should be treated with a silane coupling agent to improve chemical adhesion and chemical bonding durability with adhesive resin cement(26, 27). The specific silane used in dentistry is 3-methacryloxypropyltrimethoxysilane. This compound has a bifunctional reactivity including organic functional parts (e.g., vinyl $-CH=CH_2$, allyl$-CH_2CH=CH_2$, amino $-NH_2$, isocyanato $-N=C=O$) that polymerize with an organic matrix (resin cement) and the alkoxy groups (e.g., methoxy $-O-CH_3$, ethoxy $-O-CH_2CH_3$) that react with an inorganic hydroxyl-rich (-OH) surface (27, 28). Alkoxy groups are intermediates in the formation of silanol groups, $\equiv Si-OH$. Thus, ceramic surface pretreatment is responsible for chemical bonding via silane coupling agents. Sato et al. concluded that the ZLS ceramic surface should be silanized to promote high and stable bond to resin cement(29). According to the manufacturer, ZLS should be pre-treated with HF and a silane coupling agent to improve bonding performance.

However, there are some studies(10, 11) which revealed that the use of only silane was not sufficient for a close contact between ceramic and resin cement. For this reason, other options have been investigated for ceramic surface treatment including applying with unfilled resin after silane application due to its low viscosity. According to Neto et al.(10) found that the unfilled resin penetrated into pores on ceramic surface more completely than resin cement. While, the ceramic surface that unfilled resin was not applied, the SEM image showed voids without resin cement, and non-homogenous interface was exhibited. This led to negative effect on bond strength. On the other hand, there was a study (12) which found that the unfilled resin was dispensable for significant increase bond strength with ceramic material. Consequently, the use of unfilled resin is still controversial.

Adhesive system

Adequate bonding of restoration and hydrophobic resin cement to tooth substrates requires a dental adhesive agent to ensure sufficient retention and sealing. The dental adhesive procedure is a process that deals with different natures of enamel and dentin(30). Enamel is composed of high mineral content at 96% by weight of hydroxyapatite. Consequently, bonding to enamel has been proven durable without using adhesion promoting agents(30, 31). Dentin consists of 50% mineral and 50% water and protein, mainly type I collagen. It required a moist bonding technique to prevent collapse of the collagen network and maintained the strength of resin-dentin bonding (31).

The major processes of dental adhesive systems included etching, priming and bonding. Etching involved the treatment of dentin and enamel with 37% phosphoric acid removed the smear layer and demineralized the dental substrate surface. Etching increased the permeability of enamel and dentin(32). After etching, enamel showed an irregular surface which allowed resin monomers to penetrate and formed ‘prism-like’ tags. This yielded enamel bonding micromechanical. Whereas a demineralized collagen network appeared in dentin. Priming involved preparation of etched dentin with an adhesion promoting agent which transformed hydrophilic dentin to a hydrophobic state. This increased the surface energy of dentin which permitted infiltration of resin monomers to increase retention and formed a hybrid layer. Bonding involved the application of a hydrophobic resin bond adhesive over enamel and dentin (31).

Etch-and-rinse adhesive systems can be sub-classified into a three-step etch and rinse adhesive and a two-step etch and rinse adhesive(32). They required an acid-etching step using phosphoric acid and rinsing of enamel and dentin before applying adhesives agents. Three-step etch and rinse adhesives involved the application of primer and adhesive separately, while two-step etch and rinse adhesives use a self-priming adhesive. Consequently, self-priming adhesives were more permeable to water, with the possible appearance of water blisters at the resin-dentin interface(32). In vitro studies found that three-step etch and rinse adhesives bonded more effectively, with a better marginal seal than two-step etch and rinse adhesives because the latter had more difficulty in removing all residual solvent which led to increased permeability (31-33). Moreover, the excessive presence of humidity resulted in incomplete monomer polymerization and water absorption in the hybrid layer that may cause degradation via resin hydrolysis(16, 32-34).

Luting cement

Dental luting agents play an essential role in uniting the restoration to the prepared tooth. For glass-ceramic restorations, resin cements have been clinically used to obtain a strong and durable bond to dentin and enamel with extremely low solubility(35). Additionally, resin cements have better flexural and compressive strength compared with other luting cement. Moreover, Peumans et al. concluded that restorations adhered to resin cement, resulting in superior retention and fracture resistance(33).

Resin cements comprise different forms of resin composite with lower filler content and lower viscosity. They contain a resin matrix such as bis-GMA or urethane dimethacrylate and a filler of fine inorganic particles (36). Conventional resin cements can be categorized according to polymerization type: chemically-activates (self-cured), photo-activated (light-cured) and dual-cured cement (37). Self-cured cements are commonly used for fixation metallic restoration, because the light does not reach the cement material. In contrast, light-cured cements have indication restricted to thin porcelain restoration due to the reduction of light intensity during its transmission through the restoration. Moreover, the lack of tertiary amines in the cement composition provides excellent color stability (38). Dual-cured cement were developed to have material with extended working time and capable of reaching a high degree of conversion either in the presence or absence of light. They are material of choice to lute indirect tooth-colored restoration with a thickness more than 3 mm(39). Nevertheless, resin cement can be categorized by adhesive system used either etch-and-rinse adhesive system or self-etching adhesive system. The disadvantages of these cements were the multi-step application technique which was both time-consuming and technique sensitive (15). Recently, development of resin cements as self-adhesive luting agents has become popular because of their simplicity with no pretreatment requirement of the tooth surface.

Self-adhesive luting agents contain phosphorylated methacrylate monomers within the material that promote an acidic bonding environment. This environment was created subsequent to demineralization of the tooth surface and allowed the penetration of resin cement into the demineralized bonding surface. Micromechanical retention was achieved between the resin cement and tooth after polymerization (36). Moreover, the acidic functional monomer interacted with hydroxyapatite in the tooth structure to create chemical bonding. As the reaction progressed, the acidity of the cement was gradually neutralized and became more hydrophobic. Self-adhesive resin cements showed different capacities for pH neutralization during setting. In general, a faster pH-neutralization process means less susceptibility to hydrolysis over time that impacts the durability of restoration (40, 41). De Munck et al.(42) concluded that the bond strength of self-adhesive resin cements was lower than conventional multi-step luting agents as depicted by morphological SEM and TEM images of self-adhesive resin cement interface, which revealed that they superficially interacted with dentin without the presence of a hybrid layer or resin tag.

Cyclic fatigue loading

From a clinical perspective, cyclic masticatory force during chewing function in moist environment had influence on failed glass-ceramic restoration, which failure was generated from the surface flaws present within the material naturally. Subsequently, cracks propagated along the material resulting in weakening of the restoration (43). Moreover, cyclic stresses transmitted across the resin adhesive and hybrid layer may cause degradation of the interface(44). Therefore, cyclic fatigue testing was developed to simulate the failed restoration mechanism by repeated cyclic load at intensities below the material's normal strength stresses. Moreover, water molecules from the laboratory water bath diffused into the cracks and hydrolyzed siloxane bonds in ceramic material(44). In addition, loading frequency of cyclic fatigue test was determined by chewing activity of human at 0.94– 2.17 Hz(45). However, cyclic fatigue testing with low frequency was a time-consuming process. Fraga et al.(46) reported that frequency of fatigue testing up to 20 Hz could be used without compromising fatigue data. Consequently, cyclic fatigue loading was used in the present study to create the most clinically relevant fatigue approach.

CHAPTER III MATERIALS AND METHODS

Research design

This study was an in vitro experimental study. The interventions of this study were various adhesive luting agents, including resin cement used with etch and rinse adhesive, and self-adhesive resin cement, to bond a zirconia-reinforced lithium silicate ceramic (Vita Suprinity®) to tooth structure. The dependent variable was the mean value of cyclic fatigue failure load measured in Newtons (N) when the specimen was fractured or cracked.

Research methodology

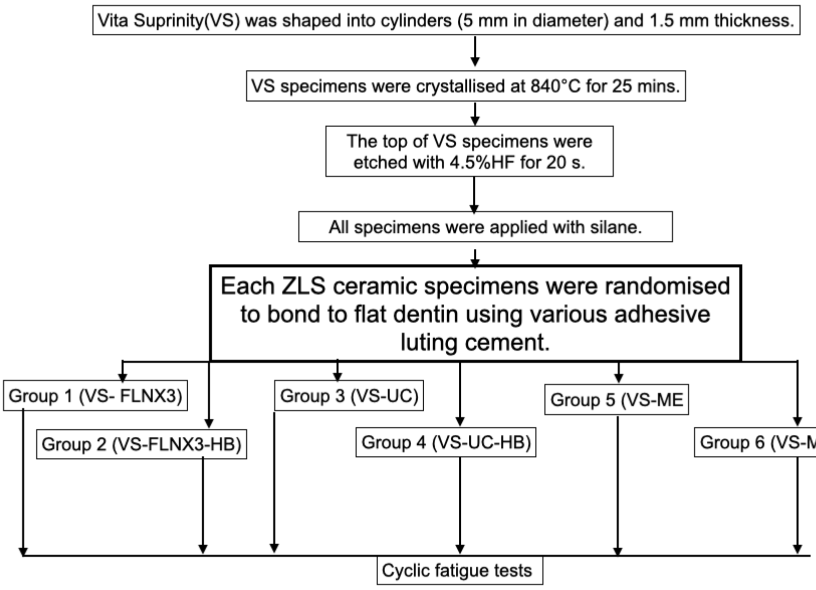


Figure 2. Diagram of Study Design

Abbreviations:

FL= Optibond FL, HB= Heliobond, HF = Hydrofluoric acid, ME= Maxcem Elite®, NX3=Nexus3, VS = Vita Suprinity®, UC = RelyX ™ Unicem, ZLS = Zirconia-reinforced lithium silicate ceramic.

Sample size description

Sample size was calculated, as previously described by Collins, to obtain a precise estimation when using staircase method(47). The value of number of specimens required (n) was calculated from

$$n = \left(\frac{1.96\sigma}{E}\right)^2$$

where

$$E = \text{width of interval}/_2$$

The fatigue data was normally distributed, it was possible to predict the width of the confidence interval as a function of a sample size. According to the formula that mentioned above, the minimum number of specimens needed to determine 95% confidence intervals of the stated width for a population mean μ, assuming the standard deviation σ was known which was determined in table 1.

Table 1. Sample size determination

Width of interval	95% Confidence limit on mean μ	Number of specimens required, n
1.0 σ	$\bar{x} \pm 0.5\sigma$	15

Sample size from calculations was determined to be at least 15 specimens for each group. The study comprises 6 experimental groups. However, the sample size per group 18 in this study were divided as follows: 3 samples per group for compressive strength test and 15 samples were used for fatigue testing.

Table 2. Datasheet of ceramics used.

Material/ Manufacturer	**Chemical composition**	**Procedure following manufacturer's instruction**
Vita Suprinity® (Vita Zahnfabrick, Bad Säckingen)/ Batch no. 78394	SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments	Crystallization in furnace (Programat P700, Ivoclar Vivadent) at 840°C for 20 mins.

Table 3. Adhesive system used in this study.

Material	**Manufacturers/Batch number**	**Chemical composition**
IPS Ceramic etching/	Ivoclar Vivadent, Schann, Liechtenstein Batch no. X 53811	4.5 % hydrofluoric acid
RelyX Ceramic Primer	3M ESPE, USA Batch no. N988623	Methacryloxypropyl trimethoxysilane, ethanol, water
Heliobond	Ivoclar Vivadent, Schann, Liechtenstein Batch no. X30679	-Bis-GMA 60 %wt. -Triethylene glycol dimethacrylate 40 %wt.
OptiBond FL	Kerr, Orange, CA, USA Batch no.7065602	**Etchant**: 37.5% phosphoric acid **Primer**: HEMA, GPDM, PAMM, ethanol, water, CQ **Bonding agent:** bis-GMA, filler (fumed SiO_2, barium aluminoborosilicate, Na_2SiF_6), CQ

NX3	Kerr, Orange, CA, USA Batch no.7090490	Resin matrix: Bis-GMA, UDMA, TEGDMA Filler: Ba-Al-borosilicate glass
RelyX ™ Unicem	3M ESPE, USA Batch no. 4927904	**Powder**: glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator **Liquid:** methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator
MaxCem Elite®	Kerr, Orange, CA, USA Batch no.7096952	GPDM, co-monomers (mono-, di-, and tri- functional methacrylate monomers, water, acetone, and ethanol.

Table 4. Instrument used in this study.

Instrument	Manufacturer
Low-Speed Cutting Machine (Isomet® 1000)	Buehler Ltd., Lake Bluff, IL, US
Ceramic furnace	Programat P700, Ivoclar Vivadent, Schaan, Liechtenstein
Automatic temperature checking set (ATK2)	Ivoclar Vivadent, Schaan, Liechtenstein
Servo Hydraulic system machine (INSTRON 8872)	Instron, England
Universal Testing Machine (E1000, INSTRON instruments)	Instron, England
Grinder-Polisher Machine (Automet® 250)	Buehler, USA
Micrometer Caliper	Mitutoyo, Japan
Durometer, ASTM D 2240 Type A	PTC Instrument, USA
Rotomix	3M ESPE, USA
LED Light-Curing System: Demi™ Plus	Kerr, USA
Radiometer: Model 100 Optilux	Kerr, USA
Stereomicroscope: ML 9300	MEIJI, Japan

Methods

This study was approved by the ethical committee of the Faculty of Dentistry, Chulalongkorn University, Thailand (approval number: HREC-DCU 2019-040).

Tooth selection

This study was performed on 135 extracted, with informed consent, human third molars, stored in a 0.1% thymol solution at 4°C in a refrigerator for no longer than 2 months. The teeth were analyzed using a stereomicroscope (ML 9300 MEIJI) at 4× magnification using the following selection criteria: no caries or previous restorations, no cracks, and the presence of completely formed apexes. After the selection process, residual soft tissue was removed by hand scaling.

Tooth preparation

Each tooth was embedded in a polyvinyl chloride (PVC) molds with 18 mm internal diameter, 22 mm external diameter, and 21 mm height. Teeth were leaving the cemento-enamel junction at the top surface of acrylic resin bases (Trey Resin II, Shofu, Kyoto, Japan), controlled by a dental surveyor (A3006 B-manual surveyor, Dentalfarm, Italy). All teeth were cut at 2 mm thickness from the central pit of occlusal surfaces using a slow-speed diamond saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) under water cooling to expose flat deep dentin surfaces. In the case of pulp exposure being detected, the tooth would be rejected.

Smear layer was standardized by grinding the dentin surface with 600 grit silicon carbide paper at 100 rpm for 30 s to produce a standard smear layer, which is comparable to dentin grinding with bur-cut surface (48, 49). The grit silicon carbide paper was changed after grinding of 10 dentin specimens. Cementation area at the center of dentin specimen was defined and isolated to a 5 mm diameter by means of perforated sticker. After that, all teeth were randomly divided into 6 groups.

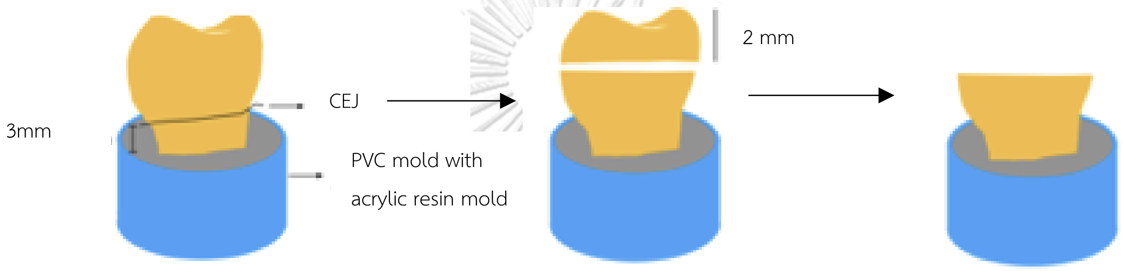


Figure 3. Preparation of dentin specimen

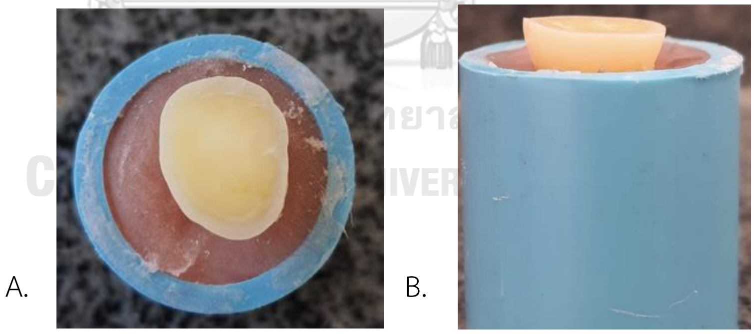


Figure 4.A.) Prepared tooth after polishing in a polyvinyl chloride tube at top view.

B.) Prepared tooth after polishing in a polyvinyl chloride tube at proximal view.

Ceramic specimen preparation

CAD-CAM pre-fabricated ceramic ingots of Vita Suprinity® (VITA Zahnfabrik) (shade A2, HT) were shaped into cylinders (5 mm in diameter) and 1.5 mm of thickness using a water-cooled precision diamond saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA). The dimensions were standardized by a caliper (Mitutoyo, Japan).

Vita Suprinity® (VITA Zahnfabrik) specimens were crystallized in a furnace according to the manufacturer's instructions (Programat P700, Ivoclar Vivadent, Schaan, Liechtenstein). The starting temperature was 400°C with holding time at the initial temperature for 8 minutes. The heating rate was 55°C/minute to reach crystallization temperature, 840°C. After that, the temperature was hold for 8 minutes. Finally, the ending temperature was 680°C. The automatic temperature checking set (ATK2) was used to check and adjust the firing temperatures in furnace with automatic calibration program for the ATK2 system before firing the ceramics.

Preparation of the ceramic surface was polished with silicon carbide paper of increasing grit-size (120-,240-,400-,600- grit) at 100 rpm under running water for 10 seconds per grit-size. The silicon carbide grit was changed after grinding of 10 ceramic blocks. This step simulated the preparation of ceramic surface with a medium-coarse diamond bur following with fine diamond(50).

The polished surface of VS was etched with a 4.5% hydrofluoric acid (HF) (IPS ceramic etching; Ivoclar Vivadent) for 20 s. The etched surfaces were then thoroughly rinsed with water for 60 s, cleaned in an ultrasonic bath with 98% alcohol for 3 mins, and air-dried. Then, ceramic primer (RelyX^{TM} Ceramic Primer, 3M ESPE) was applied to the etched ceramic surface, allowed to react for 60 s, and air dried for 5 s. Subsequently, ceramic specimens and all prepared teeth were randomly divided into 2 groups depending on Heliobond (HB, Ivoclar Vivadent, Schann, Liechtenstein) application and following 3 kinds of adhesive resin luting cements (Optibond FL with NX3, RelyX Unicem and Maxcem Elite$^{®}$).

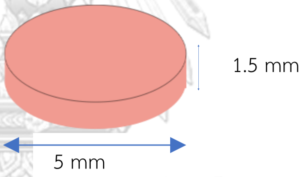


Figure 5. Preparation of ZLS blocks

Figure 6. Ceramic furnace (Programat P700, Ivoclar Vivadent)

Surface pre-treatment and cementation procedure

Groups 1 (VS-FLNX3), 3 (VS-UC) and 5 (VS-ME), HB was not applied onto silanated ceramic surfaces. Meanwhile, silanated ceramic surfaces of group 2 (VS-FLNX3-HB), 4 (VS-UC-HB), 6 (VS-ME-HB) were applied with HB for 15 s, creating a uniform thin coating with brushing motion. For group 1(VS-FLNX3) and group 2 (VS-FLNX3-HB), 37.5% phosphoric acid etching gel (Optibond FL Etchant, Kerr, Orange, CA, USA) was applied onto prepared dentin and allowed to react for 15 s. Then rinsed thoroughly with water for 15 s and blot dried with foam pellets. OptiBond FL primer was applied onto the etched dentin for 15 s with a light scrubbing motion, and gentle stream of air was blown over the liquid for about 5 s until there was no visible movement of liquid and the solvent evaporated completely. Subsequently, OptiBond FL adhesive was applied with a brushing motion for 15 s, creating a uniform thin coating followed by light-curing for 20 s using a LED light curing unit (Demi Plus, Kerr Corporation, Orange, CA, USA) with 1,100 mW/cm2 intensity. Then, NX3 resin cement (Kerr, Orange, CA, USA) was applied copiously to the prepared ceramics using auto-mix syringe. For group 2 (VS-UC) and group 3 (VS-UC-HB), RelyX^{TM} Unicem (UC, 3M ESPE) capsule was activated and mixed for 10 s (Rotomix, 3M ESPE). Then, cement was dispensed directly on to the prepared ceramics. For groups 5 (VS-ME) and group 6 (VS-ME-HB), MaxCem Elite$^{®}$ (Kerr, Orange, CA, USA) was applied copiously to the prepared ceramics using the auto-mix syringe. After loading of the luting cement in all groups, the ceramic discs were placed on the prepared dentin surface under a constant load of 1 kg, placed on the top of the ceramic disc using a custom-made loading device (Durometer, ASTM D 2240 Type A, PTC Instrument, USA). Excess cement was removed using a micro brush. A LED light curing unit (Demi Plus, Kerr Corporation, Orange, CA, USA) was used to polymerize the resin cement for 20 s per surface. Then, the load was removed, and the specimens were additionally light-cured from the top for 40 s (120 s light-curing in total). After that, the specimens were left for 10 mins and the perforated paper stickers were removed. All specimens were stored in distilled water at 37°C in an incubator (Contherm 160M, Contherm Scientific Ltd., New Zealand) for 24 hours before fatigue testing to allow possible post-cure polymerization of the luting cement. A radiometer (Model 100 Optilux, Kerr, Orange, CA, USA) was used to measure the light output of light-curing unit every 10 specimens throughout the experiment.

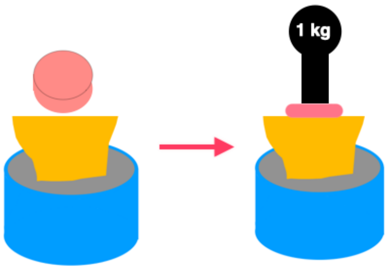


Figure 7. Cementation of the ceramic block to dentin specimen

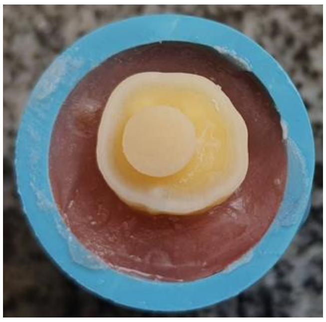


Figure 8 . ZLS disc cemented to dentin specimen.

Figure 9. Durometer (ASTM D 2240 Type A, PTC Instrument, USA)

Cyclic fatigue tests

Firstly, compressive strength tests (Servo Hydraulic system, INSTRON 8872, England) was performed by applying an increasing load (1 mm/min) for 3 specimens from each group until a radial crack fracture was occurred and the mean of compressive strength was calculated. Then, at least 15 specimens of each group were subjected to a cyclic fatigue loads (500,000 cycles at 20 Hz) in a universal testing machine (E1000, INSTRON instruments, England) with amplitudes ranging from a minimum of 10 N to the maximum load for every cycle. Fatigue testing was performed under water with a 6 mm diameter stainless steel sphere was used to apply load in the center of the ‘occlusal’ ceramic surface.

The first specimen in each group was tested at an initial load, which was calculated as 40% of the mean compressive strength, until either survival or failure at the predominate cycles. Failure of specimens included subsurface radial cracks and fracture, which the former was observed by transillumination and the later by visual inspection. Step-size load at 5% of initial load was added up or down to the next specimen according to survival or failure of the previously tested specimen39. This procedure was repeated until 15 specimens per group was submitted fatigue failure load. The staircase test was not considered to have started until the first reversal occurred (47).

Figure 10. Fatigue testing at proximal view

Figure 11. Universal Testing Machine (E1000, INSTRON instruments)

Scanning electron microscope examination

After cyclic fatigue testing, all the fractured specimens were sectioned into halves perpendicularly to the direction of the crack using a slow-speed diamond saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA). The representative specimens of each group were placed on stub with a conductive double-sided adhesive carbon tape. In addition, specimens were coated with a thin layer of gold alloy and observed in scanning electron microscope (Quanta 250, FEI, USA) at 500x, 1000x, 2500x magnification.

Data Collection and Analysis

All data were collected and sorted according to load value. The lowest load was assigned as $i = 0$, and $i = 1$ was corresponded to the followed load, and so on. The number of specimens tested using each load value was expressed as n_i.

Mean (X) and standard deviation (SD) were calculated by using the equations of Dixon and Mood method (47).

$$\mathrm{X} = \mathrm{Xo} + d\left(\frac{A}{N} \pm \frac{1}{2}\right) \qquad (1)$$

$$SD = 1.62d\left(\frac{NB-A^2}{N^2} + 0.029\right) \quad \text{if} \quad \frac{NB-A^2}{N^2} \geq 0.3 \qquad (2)$$

$$SD = 0.53d \quad \text{if} \quad \frac{NB-A^2}{N^2} < 0.3 \qquad (2)$$

where $\mathbf{Xo}$ is the lowest load value, d is the step size value, A is sum of the multiplication of in_i, B is sum of the multiplication of i^2n_i. In equation (1), the + sign was used when the more frequent event observed was survival, while the – sign was used when the more frequent event of failure.

Behrens-Fisher T-test was performed to compare means and variances among groups which determined whether they were the same or different (47).

CHAPTER IV RESULTS

The initial load for fatigue test was calculated using 40% of mean of compressive strength, which was 844 newtons (N). A step-size load of 5% of the initial load, which was 42 N, was applied to the next specimen, either up or down according to the survival or failure of the previous specimen. The staircase approach graphs are presented in Fig.12, in which survival specimens are shown as filled dot and failed specimens empty dots. Mean and standard deviation for the fatigue failure loads were in the range of 366.59 ± 37.05 N (VS-ME) to 752.99 ± 189 N (VS-FLNX3), as shown in Table 5. The Behrens-Fisher T-test revealed that ZLS cemented to dentin using self-adhesive resin luting cements (UC and ME) had a statistically significant lower mean fatigue failure load value than etch-and-rinse resin luting cement (FLNX). Meanwhile, comparison of self-adhesive resin luting cements found that UC had a significantly higher mean fatigue failure load value than ME (alpha = 0.05). Although ZLS bonded to dentin with unfilled resin application had a tendency to give higher mean fatigue failure when using self-adhesive resin cement (UC-HB and ME-HB) compared to etch-and-rinse resin luting cement group (FLNX3-HB), there was no statistically significant difference in fatigue failure load value when compared to non-application groups (alpha = 0.05). Furthermore, representative SEM images of the fractured surface were presented in Fig. 14. Failed specimens from all groups exhibited fatigue cracks in both ZLS surface and resin cement surface. However, these cracks did not propagate into the underlying tooth structure. Failure occurred in self-adhesive resin cement (UC, ME) groups demonstrated clearly that entire bonded dentin surface was exposed while no dentinal tubules were occluded by resin cement. Those failures observed in etch-and-rinse resin luting cement (FLNX3) group illustrated that in addition to the bonding of some part of ZLS to dentin, the bonding interface also presented resin tags.

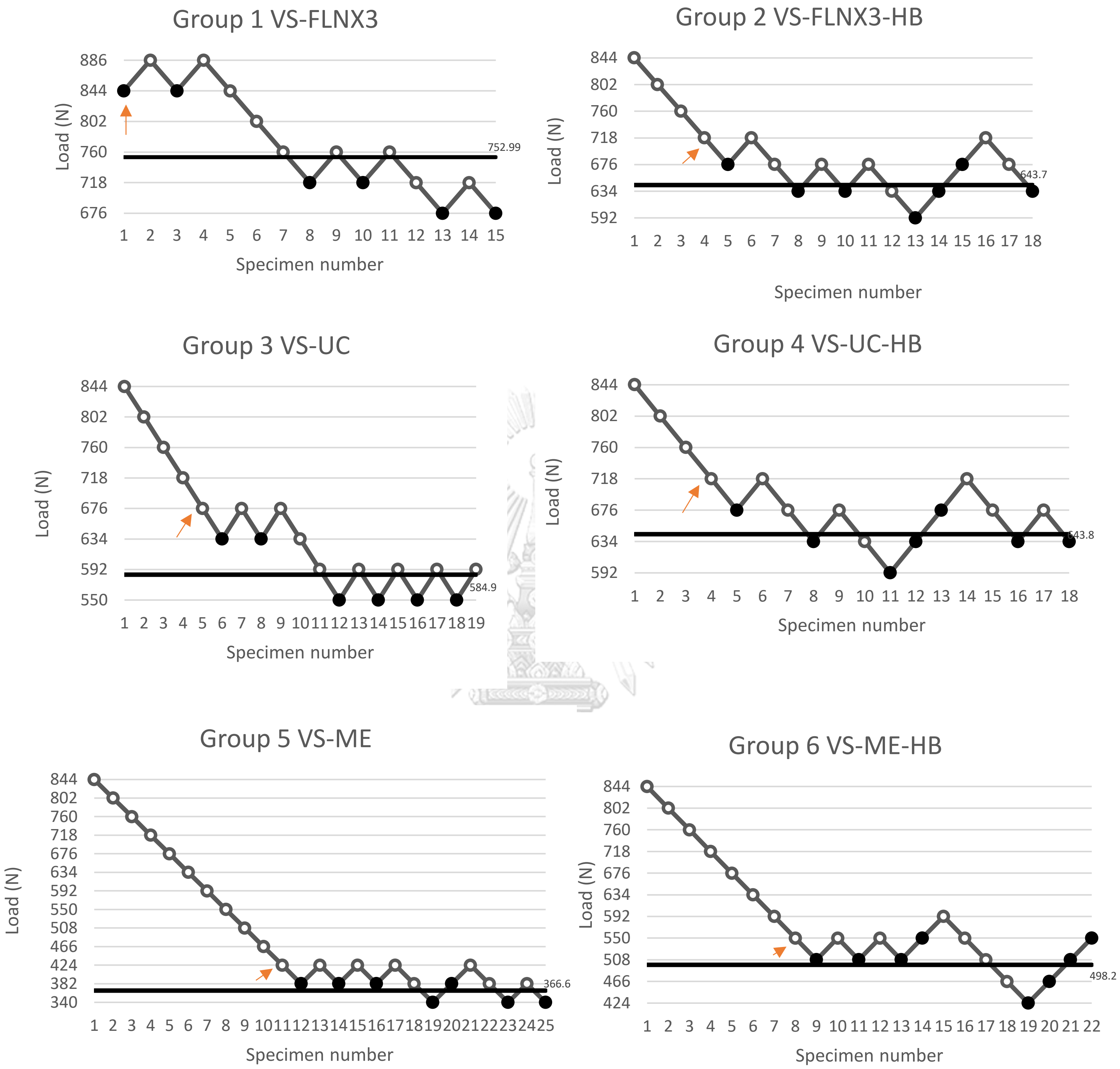


Figure 12. Staircase approach results for each group after mechanical load cycling under water (500,000 cycles at 20 Hz), the red arrows indicate the load level that the up-and-down pattern began. Horizontal lines indicate mean of fatigue failure load.

Table 5. Fatigue failure load values [mean (newton (N)) ± SD]. Behrens-Fisher T-test was used for comparison.

Groups	FLNX3	UC	ME
No HB application	752.99 ± 189 ^{Aa}	584.99 ± 80.59 ^{Ba}	366.59 ± 37.05 ^{Ca}
HB application	643.79 ± 51.5 ^{Ab}	643.79 ± 51.5 ^{Aa}	498.2 ± 69.7 ^{Ba}

Different capital letters in row indicate statistically significant difference (alpha = 0.05)

Different lowercase letters in column indicate statistically significant difference (alpha = 0. 05)

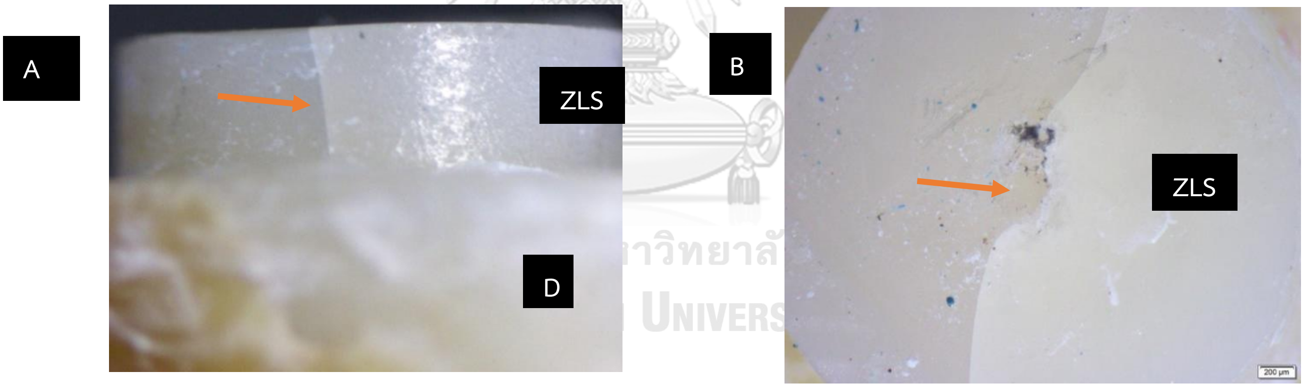
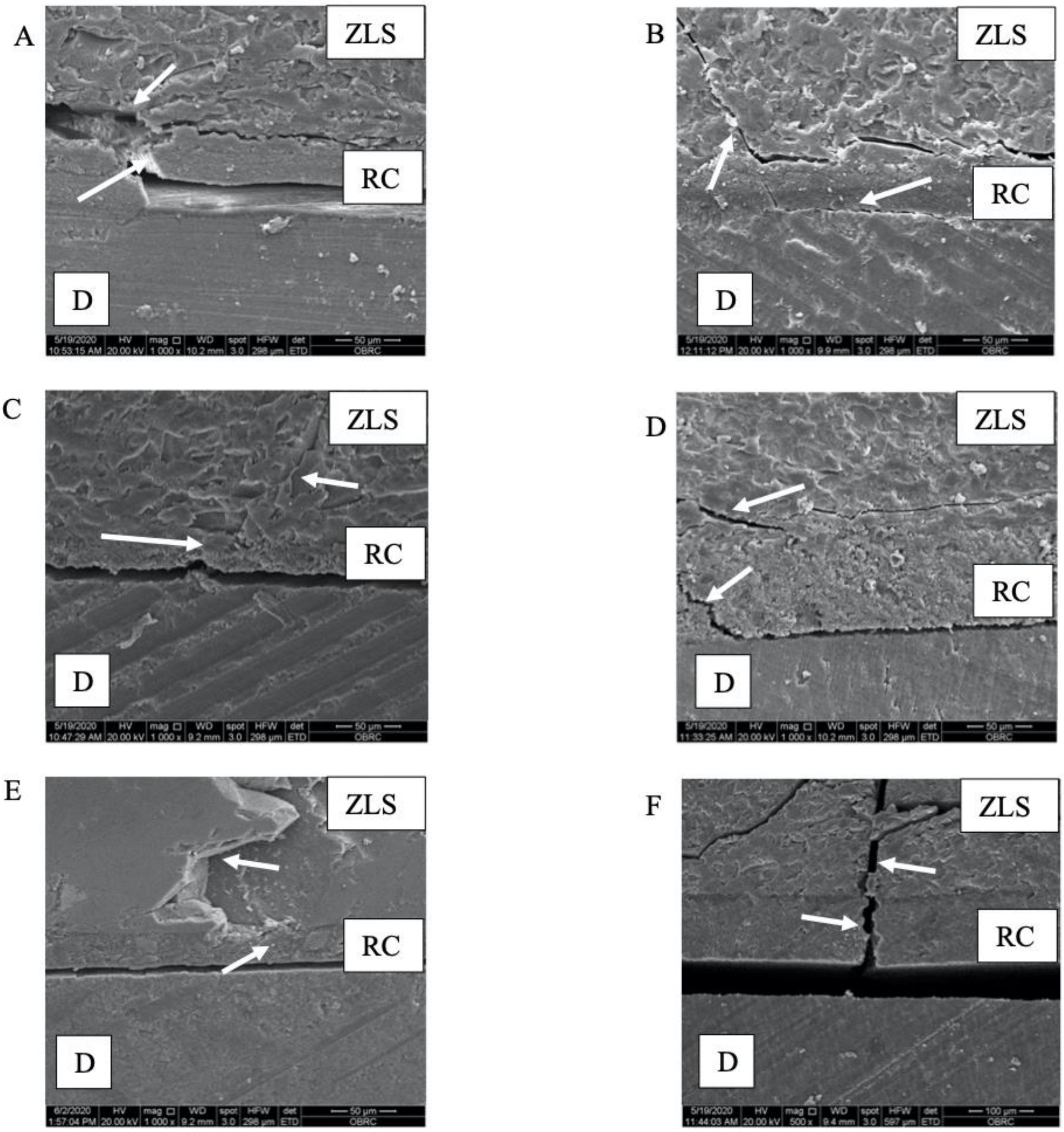


Figure 13. Example of crack tracingwith light microscope 40X. D, Dentin; ZLS, Zirconia-reinforced lithium silicate. (A) Top view (B) Proximal view. The red arrow in all images indicate crack lines.

Figure 14. SEM images of specimen failure. Images A and B show the bonding interface with resin tag, whereas, images C, D, E, F show exposed dentin without resin cement penetration. The white arrows in all images indicate crack lines which can be demonstrated in both ZLS surface and resin cement surface. D, Dentin; RC, Resin cement; ZLS, Zirconia-reinforced lithium silicate. (A) Group VS-FLNX3; (B) Group VS-FLNX3-HB; (C) Group VS-UC; (D) Group VS-UC-HB; (E) Group VS-ME; (F) Group VS-ME-HB.



CHAPTER V DISCUSSION AND CONCLUSIONS

Discussion

This *in vitro* study was performed to evaluate the fatigue failure load of a three-step etch-and-rinse resin luting system and self-adhesive resin luting systems on ZLS cemented to dentin. The first null hypothesis was rejected since self-adhesive resin luting agents showed a significantly lower mean fatigue failure load than an etch-and-rinse resin luting system. The result of high mean fatigue failure load can be explained by resin cement relying on the use of a three-step etch-and-rinse adhesive, which was Optibond FL. This adhesive system utilized phosphoric acid for etching dentin prior to primer and adhesive application. Phosphoric acid removed smear layer and created surface porosities, resulting in good permeability for monomer impregnation. However, dentin moisture control was important for this system to prevent collagen collapse and achieve complete hybridization leading to achievement of better bond strength. Meanwhile, self-adhesive luting systems used in this study had a negative effect on fatigue failure load. This was in agreement with the previous studies (51, 52) which concluded that etch-and-rinse adhesive resin cement had higher performance in fracture resistance and microshear bond strength than self-adhesive resin cement when used with glass-ceramic. This phenomenon can be explained by the fact that self-adhesive resin cement had a limited ability to demineralize dentin due to insufficient low pH. Moreover, this cement had high viscosity which may impede infiltration into dentin(53). As seen in this study, SEM images (Fig.2) demonstrated the exposed dentin without resin tags at both RelyX ^{TM} Unicem and Maxcem Elite® bonding interfaces, whereas hybrid layers were observed at Nexus3 with Optibond FL bonding interface.

In the present study, ZLS bonded to dentin using RelyX^{TM} Unicem had a significantly higher mean fatigue failure load than Maxcem Elite®. This phenomenon can be explained by the fact that both self-adhesive resin cements had different chemical compositions and physical properties, which may influence bonding ability between resin cement and dentin. RelyX^{TM} Unicem contains phosphoric acid methacrylates which react with hydroxyapatite in the tooth, whereas Maxcem Elite® also contains an acidic monomer, which is glycerol dimethacrylate dihydogen phosphate (GPDM). The pH value of RelyX^{TM} Unicem is 2.8 at the initial stage, which increases to 7 after 48 hours. Maxcem Elite® tend to maintain low pH, which was about 2.2 (54). The situation of maintaining low pH for a long time led to water absorption and solubility of the resin cement, which subsequently lowered bond strength. This is in agreement with the previous study by Liu et al.(55)who concluded that pH of acidic functional monomer containing phosphate groups had an effect on the strength of bonds formed between dentin and restorative material. Nevertheless, RelyX^{TM} Unicem (72%wt) had a higher percentage of filler than Maxcem Elite® (66%wt), which provides a better mechanical property (54).

As mentioned earlier, all specimens from both self-adhesive resin cement groups demonstrated debonding at the interface between resin cement and dentin from SEM images. Moreover, crack lines within the ceramic material were also found. The explanation could be attributed to the lower mean fatigue failure load values when ceramic was bonded to dentin poorly. Consequently, the strength of ceramic was decreased, leading to failure. As a previous study revealed that the better the bond strength between resin cement and tooth structure, the more facture resistance of a ceramic (56).

Although most manufactures recommended the use of resin cement directly on the internal silanated ceramic surface, some studies(10-12) observed that application of unfilled resin could be advantageous to increase surface energy on an etched surface. This was contrary to the present study, which found that unfilled resin could not improve mean fatigue failure load on ZLS cemented to dentin using different resin luting systems. According to SEM analysis (Fig.14), whether or not to use unfilled resin on the intaglio surface of the restoration remained ambiguous. A homogenous interface between ceramic and dentin has been observed, however, due to different ceramic materials, it could be difficult to make a direct association between studies, nevertheless, studies regarding ZLS ceramic have been scarce.

With regard to fatigue test, this approach aimed to simulate clinical conditions. The staircase approach was used in this study as it was able to identify the load at which ceramic survived in a 1-year clinical situation. Study by Wiskott et al.(57) concluded that 1,000,000 cycles of fatigue test would represent 1 year in oral function. This number was reached assuming 3 periods of chewing per day lasting 15 min each at a chewing rate of 1 cycle per second. Fatigue testing was the method of choice in this study because cyclic fatigue stress in masticatory force led to fracture of ceramic restoration. Moreover, wet conditions could influence the degradation of ceramic strength. However, all groups of testing in the present study exhibited fatigue failure load value exceeding average human masticatory force which ranged between 253.99 and 906 N.(58-60). From the perspective of a clinician, this present study showed that Optibond FL with NX3 resin cement achieved a significantly greater fatigue failure load than RelyXTM Unicem and Maxcem Elite® self-adhesive luting systems.

Limitations

This study involved several limitations, as follows:

1. This in vitro study could not fully simulate all intraoral force direction such as lateral force and sliding.
2. This study investigated only one ceramic system (ZLS) and two kinds of adhesive resin luting systems. Thus, the results might not be extended to other luting agents and ceramic systems.

Suggested further studies.

This study investigated cylindrical shaped samples, therefore, further studies should be carried out to test the fatigue failure load of ZLS produced in complex geometric design of dental restoration simulating were closely to clinical situation, such as crown, onlay etc., as well as with the use of different adhesive systems.

Conclusions

Based on the results of this in vitro study, the following conclusions were drawn:

1. When bonding ZLS to dentin, an etch-and-rinse resin luting system, Optibond FL with NX3 (752.99 ± 189 N.), had a higher fatigue failure load value than self-adhesive resin luting systems.
2. RelyX^{TM} Unicem self-adhesive luting agent (584.99 ± 80.59 N.) had a greater fatigue failure load value than Maxcem Elite$^{®}$ self-adhesive luting agent (366.59 ± 37.05 N.).
3. The use of unfilled resin on the intaglio surface of ceramic did not improve fatigue failure load value when applied to ZLS ceramic surface after the application of silane.

Clinical implication

Fatigue strength evaluation of ZLS bonded to dentin suggested that self-adhesive luting systems may not be material of choice compared to a 3-step etch and rinse luting system.

Declaration of Conflicting interest

The authors declare that there is no conflict of interest.

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APPENDICES

Appendix A. Compressive strength values of Vita Suprinity® cemented to dentin using various adhesive luting agents.

Group	Maximum load (N)
1.VS-FLNX3	2723
	3147
	1974
2.VS-FLNX3-HB	2122
	2430
	2724
3.VS-UC	1976
	1977
	2198
4.VS-UC-HB	1849
	2199
	1470
5.VS-ME	1574
	1856
	1749
6.VS-ME-HB	1974
	2072
	1949

According to this table,

Mean=2109 N

40% of the mean was 844 N which was the initial load of fatigue testing.

Appendix B. The mean (X) and standard deviation (SD) calculation of group 1 (VS-FLNX3) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	survived
2	886	Failed
3	844	Survived
4	886	Failed
5	844	Failed
6	802	Failed
7	760	Failed
8	718	Survived
9	760	Failed
10	718	Survived
11	760	Failed
12	718	Failed
13	676	survived
14	718	Failed
15	676	Survived

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by $n_{i.}$ the mean (X) and standard deviation (SD).

N	i	$n_{i.}$	in	i^2n
886	5	2	10	50
844	4	3	12	48
802	3	1	3	9
760	2	3	6	12
718	1	4	4	4
676	0	2	0	0
		15=N	35=A	123=B

where $\mathrm{X}\ o$ was the lowest load value as 676, d was the step size value as 42, A was the sum of the multiplication of in_{i}, B was the sum of the multiplication of i^2n_{i}. In equation and the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 1 were 752.99 ± 189.

Appendix C. The mean (X) and standard deviation (SD) calculation of group 2 (VS-FLNX3-HB) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	Failed
2	802	Failed
3	760	Failed
4	718	Failed
5	676	Survived
6	718	Failed
7	676	Failed
8	634	Survived
9	676	Failed
10	634	Survived
11	676	Failed
12	634	Failed
13	592	Survived
14	634	Survived
15	676	Survived
16	718	Failed
17	676	Failed
18	634	Survived

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by $n_{i.}$ the mean (X) and standard deviation (SD).

N	i	n_i	in	i^2n
718	3	3	9	27
676	2	6	12	24
634	1	5	5	5
592	0	1	0	0
		15=N	26=A	56=B

where Xo was the lowest load value as 592, d was the step size value as 42, A was the sum of the multiplication of in_{i}, B was the sum of the multiplication of i^2n_{i}. In equation, the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 2 were 643.79 ± 51.5.

Appendix D. The mean (X) and standard deviation (SD) calculation of group 3 (VS-UC) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	Failed
2	802	Failed
3	760	Failed
4	718	Failed
5	676	Failed
6	634	survived
7	676	Failed
8	634	Survived
9	676	Failed
10	634	Failed
11	592	Failed
12	550	Survived
13	592	Failed
14	550	Survived
15	592	Failed
16	550	Survived
17	592	Failed
18	550	Survived
19	592	Failed

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by $n_{i.}$ the mean (X) and standard deviation (SD).

N	i	n_i	in	i^2n
676	3	3	9	27
634	2	3	6	12
592	1	5	5	5
550	0	4	0	0
		15=N	20=A	44=B

where Xo was the lowest load value as 550, d was the step size value as 42, A was the sum of the multiplication of in_i, B was the sum of the multiplication of i^2ni. In equation, the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 3 were 584.99 ± 80.59.

Appendix E. The mean (X) and standard deviation (SD) calculation of group 4 (VS-UC-HB) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	Failed
2	802	Failed
3	760	Failed
4	718	Failed
5	676	Survived
6	718	Failed
7	676	Failed
8	634	Survived
9	676	Failed
10	634	Failed
11	592	Survived
12	634	Survived
13	676	Survived
14	718	Failed
15	676	Failed
16	634	Survived
17	676	Failed
18	634	Survived

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by n_i. the mean (X) and standard deviation (SD).

N	i	n_i	in	i^2n
718	3	3	9	27
676	2	6	12	24
634	1	5	5	5
592	0	1	0	0
		15=N	26=A	56=B

where Xo was the lowest load value as 592, d was the step size value as 42, A was the sum of the multiplication of in_i, B was the sum of the multiplication of i^2n_i. In equation, the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 4 were 643.79 ± 51.5.

Appendix F. The mean (X) and standard deviation (SD) calculation of group 5 (VS-ME) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	Failed
2	802	Failed
3	760	Failed
4	718	Failed
5	676	Failed
6	634	Failed
7	592	Failed
8	550	Failed
9	508	Failed
10	466	Failed
11	424	Failed
12	382	Survived
13	424	Failed
14	382	Survived
15	424	Failed

16	382	Survived
17	424	Failed
18	382	Failed
19	340	Survived
20	382	Survived
21	424	Failed
22	382	Failed
23	340	Survived
24	382	Failed
25	340	Survived

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by $n_{i.}$ the mean (X) and standard deviation (SD).

N	i	n_i	in	i^2n
424	2	5	10	20
382	1	7	7	7
340	0	3	0	0
		15=N	17=A	27=B

where Xo was the lowest load value as 340, d was the step size value as 42, A was the sum of the multiplication of ini, B was the sum of the multiplication of $i^2 n_i$. In equation, the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 5 were 366.59 ± 37.05.

Appendix G. The mean (X) and standard deviation (SD) calculation of group 6 (VS-ME-HB) by using to the equations of Dixon and Mood method as described on chapter V.

Specimen number	Load(N)	Status
1	844	Failed
2	802	Failed
3	760	Failed
4	718	Failed
5	676	Failed
6	634	Failed
7	592	Failed
8	550	Failed
9	508	Survived
10	550	Failed
11	508	Survived
12	550	Failed
13	508	Survived
14	550	Survived
15	592	Failed
16	550	Failed

17	508	Failed
18	466	Failed
19	424	Survived
20	466	Survived
21	508	Survived
22	550	Survived

According to staircase approach results can be used in the equation of Dixon and Mood method, all data were sorted which attribute to load value. The lowest load was assigned $i = 0$, $i = 1$ correspondent to the followed load, and so on. The number of specimens tested into each load value was expressed by $n_{i.}$ the mean (X) and standard deviation (SD).

N	i	n_i	in	i^2n
592	4	1	4	16
550	3	6	18	54
508	2	5	10	20
466	1	2	2	2
424	0	1	0	0
		15=N	34=A	92=B

where Xo was the lowest load value as 424, d was the step size value as 42, A was the sum of the multiplication of ini, B was the sum of the multiplication of i^2ni. In equation, the – sign was used because of the more frequent event of failure.

The mean (X) and standard deviation (SD) of group 6 were 498.2 ± 69.7.

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