FRACTURE RESISTANCE OF LITHIUM DISILICATE CERAMICS BONDED TO ENAMEL OR DENTIN USING DIFFERENT RESIN CEMENT TYPE AND FILM THICKNESS

Miss Thitithorn Rojpaibool

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Esthetic Restorative and Implant Dentistry
Faculty of Dentistry
Chulalongkorn University
Academic Year 2014

ผลของชนิดและความหนาของเรซินซีเมนต์ต่อความต้านทานการแตก
ชนิดแรงอัดของเซรามิกชนิดลิเทียมไดซิลิเกตที่ยึดบนผิวเคลือบฟันหรือเนื้อฟัน

นางสาวธิติธร โรจน์ไพบูลย์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2557

Thesis Title	FRACTURE RESISTANCE OF LITHIUM DISILICATE CERAMICS BONDED TO ENAMEL OR DENTIN USING DIFFERENT RESIN CEMENT TYPE AND FILM THICKNESS
By	Miss Thitithorn Rojpaibool
Field of Study	Esthetic Restorative and Implant Dentistry
Thesis Advisor	Associate Professor Chalermpol Leevailoj

Accepted by the Faculty of Dentistry, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree

_______________Dean of the Faculty of Dentistry

(Assistant Professor Suchit Poolthong, Ph.D.)

THESIS COMMITTEE

_______________Chairman

(Jaijam Suwanwela, Ph.D.)

_______________Thesis Advisor

(Associate Professor Chalermpol Leevailoj)

_______________External Examiner

(Assistant Professor Vanthana Sattabanasuk, Ph.D.)

ธิติธร โรจน์ไพบูลย์ : ผลของชนิดและความหนาของเรซินซีเมนต์ต่อความต้านทานการแตกชนิดแรงอัดของเซรามิกชนิดลิเทียมไดซิลิเกตที่ยึดบนผิวเคลือบฟันหรือเนื้อฟัน (FRACTURE RESISTANCE OF LITHIUM DISILICATE CERAMICS BONDED TO ENAMEL OR DENTIN USING DIFFERENT RESIN CEMENT TYPE AND FILM THICKNESS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ทพ. เฉลิมพล ลี้ไวโรจน์, 68 หน้า.

วัตถุประสงค์ : เพื่อศึกษาผลของความหนาของชั้นซีเมนต์และชนิดของเรซินซีเมนต์ต่อความต้านทานการแตกหักของเซรามิกที่ยึดบนผิวเนื้อฟันหรือเคลือบฟัน

วิธีการศึกษา : นำฟันกรามแท้มนุษย์ซี่ที่สามจำนวน 100 ซี่มาขัดจนได้ชิ้นงานที่มีพื้นผิวของเคลือบฟัน 50 ชิ้นและพื้นผิวของเนื้อฟัน 50 ชิ้น จากนั้นนำชิ้นงานทั้งหมดไปยึดติดกับแผ่นเซรามิกชนิดลิเทียมไดซิลิเกตที่หนา 1 มิลลิเมตร ด้วยซีเมนต์ที่มีความหนาต่างกัน (100 ไมโครเมตร หรือ 300 ไมโครเมตร) ซีเมนต์ที่ใช้ในการยึดติดชิ้นงานได้แก่ Rely X U200 และ Rely X Ultimate กลุ่มควบคุมใช้ซิงค์ฟอสเฟตซีเมนต์ในการยึดผิวฟันกับแผ่นเซรามิค(n=10) แล้วนำไปทดสอบแรงกดชนิดอัดจนเกิดการแตกหัก บันทึกค่าแรงกดเป็นนิวตัน ค่าแรงกดที่ได้นำมาวิเคราะห์ทางสถิติด้วยการวิเคราะห์ความสัมพันธ์ถดถอยเชิงเส้นตรง ที่ระดับนัยสำคัญ 0.05 หลังจากทดสอบการแตกหัก นำตัวแทนของแต่ละกลุ่มทดสอบและกลุ่มควบคุมไปวัดความหนาของชั้นซีเมนต์ภายใต้กล้องจุลทรรศน์อิเลคตรอนแบบส่องผ่าน (SEM)

ผลการทดสอบ : กลุ่มของ Rely X Ultimate ที่ยึดบนผิวเคลือบฟันด้วยความหนา 100 ไมโครเมตรให้ค่าแรงกดสูงสุด (1591±179.2) จากการวิเคราะห์ทางสถิติพบว่าค่าแรงกดที่สูงกว่าสัมพันธ์กับเรซินซีเมนต์ชนิด Rely X Ultimate เปรียบเทียบกับชนิด Rely X U200 อย่างมีนัยสำคัญ ความหนาซีเมนต์ที่หนากว่าสัมพันธ์กับค่าแรงกดที่ต่ำกว่าความหนาซีเมนต์ที่บางกว่าอย่างมีนัยสำคัญ และเซรามิกที่ยึดกับผิวเนื้อฟันสัมพันธ์กับค่าแรงกดที่ต่ำกว่าเซรามิกที่ยึดบนผิวเคลือบฟัน

สรุป : ความหนาของชั้นซีเมนต์ที่บางและเรซินซีเมนต์ชนิด RelyX Ultimate สัมพันธ์กับค่าแรงกดที่สูงกว่าอย่างมีนัยสำคัญ และการยึดเซรามิกบนผิวเนื้อฟันสัมพันธ์กับค่าแรงกดที่ต่ำกว่าอย่างมีนัยสำคัญเมื่อเทียบกับการยึดเซรามิกบนผิวเคลือบฟัน

สาขาวิชา	ทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม	ลายมือชื่อนิสิต	....
		ลายมือชื่อ อ.ที่ปรึกษาหลัก	....
ปีการศึกษา	2557		

5475828532 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY
KEYWORDS: LITHIUM DISICATE CERAMICS, FRACTURE RESISTANCE, RESIN CEMENTS, ENAMEL BONDING, DENTIN BONDING, 68 THICKNESS

THITITHORN ROJPAIBOOL: FRACTURE RESISTANCE OF LITHIUM DISILICATE CERAMICS BONDED TO ENAMEL OR DENTIN USING DIFFERENT RESIN CEMENT TYPE AND FILM THICKNESS. ADVISOR: ASSOC. PROF. CHALERMPOL LEEVAILOJ, pp.

Objective : The objective of this study was to investigate the influence of cement film thickness, cement type, and substrate (enamel or dentin) on fracture resistance of ceramic.

Materials and Methods : One hundred extracted human third molars were polished to obtain fifty enamel and fifty dentin samples. To these samples 1-mm thick lithium disilicate ceramic plates were cemented with different cement film thicknesses (100 and 300 μm) using metal strips as spacers. The cements used were etch-and rinse (RelyX Ultimate) and self-adhesive (RelyX U200) resin cements. Compressive load was applied on ceramic plates using a universal testing machine and fracture loads were recorded in Newton (N). Statistical analysis was performed by multiple regression ($P < .05$). The representative specimens were evaluated by scanning electron microscopy (SEM) in order to control the cement film thickness.

Results : The group with a cement thickness of 100-μm cemented to enamel with Rely X ultimate showed the highest mean fracture load (MFL; 1591 ±172.59 N). Higher MFL significantly related to RelyX Ultimate comparing to RelyX U200 and thinner film cement ($P < .05$). Bonding to dentin resulted in lower MFL comparing to enamel ($P < .001$).

Conclusion : The Higher fracture loads were related to thinner cement film thickness and RelyX Ultimate resin cement. Bonding to dentin resulted in lower fracture loads than bonding to enamel.

Field of Study:	Esthetic Restorative and Implant Dentistry	Student's Signature	....
		Advisor's Signature	....
Academic Year:	2014		

ACKNOWLEDGEMENTS

I would like to express my deepest gratitude to my advisor, Assoc. Prof. Chalermpol Leevailoj, for his excellent guidance, caring, patience, and providing me with an excellent atmosphere for doing research. I would never have been able to finish my master degree without his dedication to our program.

I would also like to express my sincere thanks to the members of the Thesis Supervisory Committee, Jaijam Suwanwela, PhD. and Asst. Prof. Vanthana Sattabanasuk. PhD., who share expertise, and sincere and valuable guidance to me.

My greatest appreciation extended to Assst. Prof. Tewarit Somkotra, PhD. His enthusiasm and encouragement have been extremely useful. He gave generously of his time and vast knowledge.

I am also grateful to all the staffs of Dental Research center, Chulalongkorn University and Mahidol University for provision of expertise, and technical support in the implementation. Without their superior knowledge and experience, the Project would not like in quality of outcomes.

Special thanks to all the staffs of Esthetic Restorative and Implant Dentistry clinic for providing their assistance and co-operation throughout my graduate training program.

This thesis would not be achieved without the great kind of my family. They were always supporting me and encouraging me with their best wishes. I would also thank to my friends in this program for their care and precious friendship during training in the program.

Finally, I also gratefully acknowledge Ivoclar Vivadent. Co. Ltd., and 3M ESPE Co. Ltd. for their kindly support regarding to the supply of materials used in this study and thanks to the Graduated School of Chulalongkorn University for the grant on this thesis.

CONTENTS

Page

Page

Page

LIST OF TABLES

LIST OF FIGURES

CHAPTER I

INTRODUCTION

Rationale and Significance of the Problem

All-ceramic restorations are considered a promising treatment modality for esthetic dentistry. Their performance provides more natural appearance compared with that of other tooth-colored restorations. Lithium disilicate glass ceramics were developed to increase mechanical properties and improve translucency. As a result, the indications for usage range from single-tooth restorations to prostheses in the anterior and premolar regions, including minimally invasive inlays and onlays. According to clinical studies, a cumulative survival rate of 93% after 8 years of service has been reported for lithium disilicate crowns and three-unit fixed dental prostheses (FDPs).[1-4] The minimum thickness of material required is 0.8 mm. The majority of restoration failures were due to fracture. To increase the fracture resistance of ceramic, restorations should be cemented to tooth structure.[5, 6] The longevity of glass-ceramic restorations that luted with resin composite revealed more favorable survival rates than those luted with glass ionomers or zinc phosphate cement.[7] Resin-based luting material may reduce the potential for crack propagation and strengthen the porcelain due to polymerization shrinkage of resin cement.[8]

Therefore, resin cements are strongly recommended for the cementation of glass-ceramic materials.

Preparation depth for all ceramic restorations involve tooth structures both enamel and dentin. Dentin exposure has significantly association with failure rate of porcelain veneers.[9] The different characteristics of enamel and dentin as the bonding substrate affect the strength and reliability of the bond. Bonding to dentin is more complicated because of the cements' susceptibility to variations in the degree of dentin moisture. To simplify bonding procedures, self-adhesive resin cements have been introduced to prevent the collapse of demineralized dentin conditioned with phosphoric acid and reduce the number of bonding steps.[10] A previous study reported that the bond strength of self-adhesive resin cements to enamel is lower than those to dentin.[11] However, the effect of fracture resistance of ceramics luted with self-adhesive resin cement has not been well-established.

The longevity of ceramic restorations also depends on the close proximity between restoration and tooth structure.[12] The resistance of cemented crowns has been related to cement film thickness subjected to lateral and compressive loading.[13] ISO standards require a cement film thickness for resin-based cements of no greater than 50 μm.[14] However, there are several factors that might influence the thickness of resin cement. Internal adaptation of ceramic restorations is significantly influenced by the accuracy of fabrication process used. As a result of the manufacturing process, mean cement film thicknesses of 106.74 μm for the pressable technique and 340.35 μm for the machinable CAD/CAM technique were observed.[15] Further, variations in finger pressure during cementation procedures, coupled with die spacer application, could generate a layer thickness of more than 100 μm.[16, 17]

The effect of resin cement thickness on the fracture resistance of ceramic has not been clarified in the available literature. The study by Prakki et al revealed that higher resin cement film thickness tended to increase the fracture resistance of 1-mm ceramic plates cemented to dentin.[18] In contrast, Scherrer et al reported that increased resin cement thickness reduced the fracture resistance of glass-ceramic plates bonded to resin composite blocks.[19] This study aimed to determine whether cement film thickness, cement type, and bonded substrate influence fracture resistance loads of lithium disilicate ceramic plates. The null hypothesis was that the variables in cement film thickness, cement type, and substrate would be no significant correlation to fracture loads.

Research Questions

Is there any significant correlation between the variables and fracture loads of lithium disilicate ceramics?

Objective of the Study

The objective of study was to investigate the influence of cement film thicknesses, cement types, and substrate (enamel or dentin) on fracture resistance of ceramic.

Statement of Hypothesis

Null Hypothesis:

There is no significant correlation between the variables (cement film thickness, cement type and substrate) and fracture loads of lithium disilicate ceramics

Alternative Hypothesis:

There is a significant correlation between the variables (cement film thickness, cement type and substrate) and fracture loads of lithium disilicate ceramics

Conceptual Framework

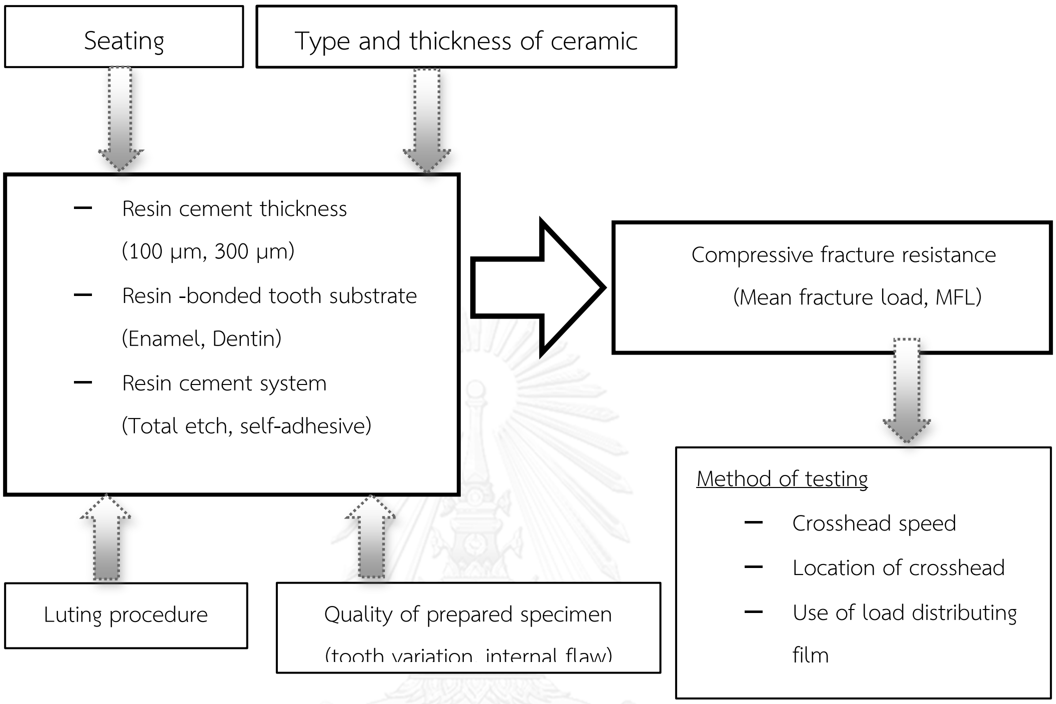

Figure 1 Diagram of conceptual framework

Basis Assumption

1. All procedures were performed under well-controlled conditions by one operator and evaluated by one examiner.

2. Ceramic plates were fabricated according to manufacturer's instruction by one technician

3. Lithium disilicate ceramics (IPS e.max) and resin cements (RelyX U200, RelyX Ultimate) were chosen in this study based on popularity using in Thailand.

4. In order to control the comparable thickness of restorations and minimize the distortion from several firing cycles, the ceramic plates which used in this study were not glazed

5. The metal strips that used as spacers were evaluated in pilot study and

Study Limitation

1. This study focuses on the effect of compressive force on ceramics which is one of masticatory forces. In clinical situation, there are other masticatory forces that involved the fracture of ceramic restorations.

2. This experimental study is designed to reduce the difficulty of complex geometry of a full molar crown. Therefore, the flat plane of specimens was prepared for compressive testing.

Keywords

Lithium disilicate ceramic/ Fracture resistance/ Cement film thickness/ Resin cement systems/ Enamel bonding/ Dentin bonding

The Expected Benefits

The results of this study might indicate whether the thicker resin cement thickness and resin cement systems have any effect to strength of ceramic restorations. For clinical application, it can be basic knowledge for improve the longevity of all ceremic restorations in associated with resin cement film thickness and substrate.

CHAPTER II

Review of Literatures

Dental Ceramics

Recently, many different type of ceramics system have been introduced for indirect restoration. According to McLaren et al., dental ceramics can be classified into 4 categories[20]

Category 1 - Glass-based systems (mainly silica)

Category 2 –Glass-based system with fillers usually csystalline

Category 3 – Crytalline-based systems with glass fillers (mainly alumina)

Category 4 – Poly-crytalline solids (alumina and zirconia)

<u>Category 1: Glass–based Systems, Amorphous Glass</u>

The majority component of this system is silicon dioxide which contains of different amounts of alumina. The synthetic forms of aluminosilicate glassed were used for fabricating dental restorations. This material has good performance in translucency, therefore it were used as veneering materials for metal or ceramic substructures. However, its mechanical properties are low (flexural strength 60-70 MPa)

Category 2: Glass-based System with Crystalline Second Phase

The main composition in this category is silicon dioxide which acts as glassy matrix. This matrix is added with filler which is leucite, lithium disilicate, or fluoroapatite. This category can divided into 3 subgroups depend on glass-crystalline ratios and crystal types.

Subcategory 2.1: Low-to-moderate leucite containing feldspathic glass

Subcategory 2.2: High-leucite (approximately 50%) – containing glass, glass-ceramics

Subcategory 2.3: Lithium disilicate glass-ceramics

The glassy matrix contains of high percentage (approximately 70%) of lithium-orthophosphate crystals. The flexural strength is approximately 360 MPa which is greater than others subgroups in this category. Its strength is contributed to highly content and needle-like in shape of crystals which create the interlocking between crystals. This material is also very translucent due to the relatively low refractive index of the lithium-disilicate crystals.

IPS e.max® pressable and machinable ceramics were introduced to the market by Ivoclar Vivadent. The processing technique to fabricate the material can be both pressable and machinable systems. From clinical guidelines for material selection, it suggests that IPS e.max ceramics can be used in high-loaded areas such as molars. The clinical study reported that the cumulative survival rate of lithium disilicate crown was 94.8% after 8 years of service even on posterior crowns.[1] However, the optimal space requirement is more than 0.8 mm which can allow sufficient room for workability and desired esthetics. The exception for this is marginal areas which minimum thickness can be gradually thin to 0.3 mm. [21]

Category 3: Crystalline Based Systems with Glass Fillers (Mainly Alumina)

This category is also known as interpenetrating phase ceramics. The first phase of fabrication process is creating pores in crystalline matrix which is mainly alumina. Then, a second-phase materials which is a lanthanum aluminosilicate glass were filled in a porous matrix. This material possesses high flexural strength which ranges from 350 to 650 MPa. According to the clinical guideline for material selection, it should be used in areas which unfavorable risk for flexure and stress distribution is founded.

Category 4: Polycrystalline Solids

Alumina or zirconia is sintered without any intervening matrix in order to fabricate solid-sintered monophase materials. Flexural strength of zirconia is approximately 900-1000 MPa which is twice greater strength than alumina. In clinical study, Zirconia-based restorations which included 3-units FPD and single crowns revealed high survival rates. The most complications were chipping and cracking of veneering material.

Ceramic Surface Treatment

The method of ceramic surface treatment is one of crucial factosr which has influence to bond strength between porcelain and tooth structure. Sandblasting, etching technique and silane coupling agents are the most common procedures that can improve the result. However, the suitable surface treatment of ceramic restorations depends on the composition of ceramics material. According to the study of Soares et al.2005, surface treatment protocol in the cementation process of lithium disilicate reinforced ceramics is application of 9.5% hydrofluoric acid for 20 seconds, 1 minute washing followed by silane application.[22] Airborne particle abrasion did not recommend for cementing silica-based all-ceramic restorations because chipping of ceramic material could be induced. The study of Guarda et al. investigated the effect of two surface treatments on microtensile bond strength of IPS e.max®. The results showed that etching with 10% hydrofluoric acid for 20 seconds significantly increased microtensile bond strength when compared to sandblast with 50-μm aluminum oxide particles for 5 seconds.[23] The application of silane coupling agents is important to the adhesion of ceramic restorations. Silane is bifunctional molecule which response for creating covalent bond (Si-O-Si) between the inorganic ceramic phase and the organic phase of the composite cement. The study of bella dona et al. demonstrated that silane application on lithium disilicate ceramic surface revealed low adhesive resistance to resin composite.[24] Additionally, heat application associating silanization process promoted greater adhesion of ceramic restorations than those without heat application.[25, 26]

Tooth Surface Treatment

To achieve the long-term success of ceramic restorations, it is necessary to provide good adhesion to tooth substrate. Bonding to enamel is different from bonding to dentin which is attributed to the difference of composition between enamel and dentin.

1 Bonding to Enamel

Enamel contains approximately 86 vol % of an inorganic matrix, 2 vol% of an organic matrix, and 12 vol% of fluid.[27] Structurally, enamel is composed of millions of enamel rods or prisms. The acid etching procedures using 35-50% of phosphoric acid creates an irregular and pitted surface with numerous microscopic undercuts. This appearance can be observe under scanning electron microscope which caused by an uneven dissolution of enamel rod heads and tails. The microretention is provided by interlocking between resin tags and surface irregularities which created by etching procedure. Currently, etching procedure using 30-40% of phosphoric acid for 15-20 seconds was recommended which achieved sufficient resin-enamel bond strength (25-30 MPa).[28]

2 Bonding to Dentin

Dentin contains approximately 45 vol% of an inorganic matrix, 33 vol% of an organic matrix and 22 vol % of fluid by volume.[27] Dentine is less mineralized than enamel. The majority of mineralized tissue is hydroxyapatite crystallites. Dentinal tubules are small canals which extend from the dentinoenamel junction to the pulp. Tubules in superficial dentin close to the DEJ are smaller than those close to pulp. They are filled odontoblast process and dentinal fluid. The humidity of dentin is one of important factors that reduces reliability of dentin bonding. Mechanism of dentin bonding can be classified generally into 2 strategies using different approaches to smear layer.[29]

a) Complete Removal of Smear Layer

Bonding procedures are conducted in 3 seperated steps involving etching, priming and bonding. Phosphoric acid solutions are utilized to remove the smear layer on enamel and dentin (total-etch). Infiltration of resin monomers enables micromechanical locking of the resin via the formation of hybrid layer and resin tags. Total etch bonding system provided greater bonding effectiveness comparing to other systems. According to Peumans et al.1999, FE-SEM imaging of the tooth/luting composite/porcelain interface illustrated no separation at the adhesive interface when using multi-step total etch adhesive system. The authors also mentioned that no de-bonding area was observed although cervical enamel and exposed dentin were less potential for resin impregnation and bonding.[30]

b) Modification of Smear Layer

This latter approach utilizes monomers acids to disrupt the smear layer, dissolve hydroxyapatites and incorporate as part of a hybrid zone. Self-etching bonding system was proposed in order to deal the difficulty in determining the optimal level of dentin moisture and incomplete hybrid layer. Overdrying or overwetting problems which occur in total etch bonding system are possibly eliminated due to non-rinsing procedure.[29] Subsequently, all-in-one adhesive systems were launched in 1997. This bonding system combined all three stages of dentin bonding in to a single package. However, the study of Tay et al. investigated micromorphological spectrum at the resin-dentin interface when single-bottle primer/adhesives were used. The results demonstrated that the blister-like spaces formed on the dentin surface and resin globules were found around the tubular orifices and on the surface of the hybrid layer.[31]

Resin Luting Cement

The longevity of the adhesive restorations is contributed to the strength and durability of the interface between resin cements and the bondable surface of restorations. It also involves in conditioning of the bonded surfaces. Resin cements were introduced to use for cementation of all-ceramic restoration in order to improve the bond strength. There are many characteristics that resin cement was superior than other luting cements. The classification of resin cements could use their polymerization mechanism or their adhesive schemes.[32]

Classification by adhesive schemes

1. Total-etch resin cements – This resin cement system utilizes the etching procedure to remove the smear layer and open the dentinal tubules. Total-etch resin cements provide the highest cement-to-tooth bond but it requires several steps in cementation procedure. The disadvantage of etch-and-rinse adhesive is their susceptibility to variations in the degree of dentin moisture. Dentin demineralized using phosphoric acid is easily collapsed when air-dried. It results in poor permeability for impregnating monomers.

2. Self-etch resin cement – To prevent the collapse of demineralized dentin and reduce the numbers of bonding step, resin cements that incorporate self-etching primers were developed. Self-etching primer diffuse through smear layer and into tooth substrates. Therefore, the reliable and durable bonds depend on the characteristics and thickness of the smear layer.[33]

3. Self-adhesive resin cements – This category of resin cements is delivered as one component without the need for separate bonding agents. These cements contain phosphoric acid which reacts with filler particles and

dentin in the presence of water. The bond was formed by polymerizing of resin and created a cross-linked polymer.[34]

Recently, universal or multimode one-bottle adhesives have been launched that can be used as self-etch or as etch and rinse adhesives. Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA) contains of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer which is utilized to provide acidity for self-etching capability. The polyalkeonic acid copolymer is also contained in this system in order to provide chemical bonds to the calcium in hydroxyapatite. (Table 1) Chemical bonding ability of both the 10-MDP and copolymer to hydroxyapatite may play an important role that results in stable and durable interface. Clinical studies have shown reliable performance when used in noncarious cervical lesions and may not depend on the bonding strategy.[35] However, the microtensile bond strengths of Scotchbond Universal Adhesive was lower than two-steps etch-and-rinse adhesive (Optibond Solo Plus), but higher than two-steps self-etch adhesive (Clearfil SE Bond).[36]

Table 1 Composition of materials used in study according to manufacturer's data

Adhesive systems	Composition
Single Bond Universal Adhesive (3M ESPE, St. Paul, MN, USA)	1. Scotchbond Universal Etchant: 34% phosphoric acid 2. Adhesive: - methacryloyloxydecyl dihydrogen phosphate (MDP) phosphate monomer, dimethacrylate resins, hydroxyethyl methacrylate (HEMA), methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane
Rely™ X U200 (3M ESPE, St. Paul, MN, USA)	1.Base paste - methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components stabilizers 2. Catalyst paste - methacrylate monomers, alkaline (basic) fillers silanated fillers, initiator components,stabilizers pigments
Rely™ X Ultimate (3M ESPE, St. Paul, MN, USA)	1. Base paste - methacrylate monomers, radiopaque, silanated fillers, initiator components 2. Catalyst paste - methacrylate monomers, Radiopaque alkaline (basic) fillers, Initiator components, Pigments, Rheological additives, Fluorescence dye, Dark cure activator for Scotchbond Universal adhesive

**Scotchbond™ Universal and Single Bond Universal are the same adhesive with different product names that are sold in different regions of the world*

The Adhesion Complex: Tooth/Luting Composite/Porcelain

The proper internal fit is an essential part of long-term success of metal-free restoration. According to Magne et al, the ratio of the thickness of ceramic and luting composite was defined as the parameter associated with the development of cracks in ceramic restorations. There were significant results of crack propensity when the ratio of the measurements of the ceramic and luting composite thickness (CER/CPR) were greater than 3.0.[12] Lower ratio of CER/CPR produced higher compression forces in ceramics. In finite element analysis, this ratio appeared to have a relevant influence on stress distribution due to the curing contraction of the luting composite and thermal expansion coefficient mismatch of the restorative materials. Cementation procedures and laboratory process were determined as factor associated to control resin film thickness.

The cement film thickness of lithium disilicate restoration using different fabrication procedure were evaluated in the study by Abousehib et al. Pressable ceramic restorations (106.74 µm) has significantly lower mean cement thickness than CAD/CAM machinable ceramics (340.37 µm).[15] Mean cement film thickness of all ceramic crowns that ranged from 262 µm to 310 µm were observed with variation of finger load application.[12] The results of both studies found that the film thickness of cervical one-third area was significantly thinner than those of incisal one-third area.

The die spacers are generally used for allowing proper seating of a restoration. Cho et al. aimed to study the effect of die spacer thickness on the shear bond strength of porcelain veneers. Thicker die spacer reduced the mean values of shear bond strengths. They concluded that 2-coat application provided suitable space (12.8 μm) for cement thickness regarding to shear bond strengths.[16] Furthermore, the storage time also influenced to the film thickness of die spacer. The increasing of die spacer thickness was observed when bottles were stored for a period of three to six months before using. Sunil et al. reported that the thickness of die spacer application after storing for six months ranged from 34.11 μm to 121.80 μm regarding to number of coatings.[17]

Few studies reported the effect of resin cement film thickness on fracture resistance of ceramics. Prakki et al found that fracture resistance for the 1-mm thickness of ceramic plates was increased when higher cement film thickness was used.[18] In this study, three thicknesses of cement film, 100, 200 and 300 μm including uncemented control group were luted to two thicknesses of ceramic, 1.0 and 2.0 mm. The ceramics plates were cemented to bovine dentin. The statistical difference in fracture load between groups with 100 and 300 μm cement thickness were observed. Molin et al evaluated the influence of different film thicknesses of resin composite luting agents on the joint bend strength of a ceramic/resin interface. The result revealed that lower bond strength was observed in 20 μm-resin luting thickness compared to the thicker ones (50 μm, 100 μm, 200 μm).[37] In contrast, the result of Scherrer et al. concluded that there was gradual decrease of the fracture strength of ceramic plate cemented with the resin composite cement. Fracture loads showed significant differences between the 26- and 297-μm groups, but not with other cement thicknesses.[19]

Compressive Fracture Resistance Test

Compressive strength test have been a one of common laboratory tests for mechanical properties of dental materials. Compressive stress which is one of the major components of masticatory forces could induce fracture of ceramic restorations. According to ISO 9917-1:2003, the mechanical test maching (e.g., Instron mechanical test system) was used for examining the compressive strength of dental materials. A constant crosshead speed of 0.75 ± 0.30 mm/min or a rate of loading of 50 ± 16 N/min are provided for testing.[38]

In previous study, the compressive tests that evaluated fracture resistance associated to resin cement thickness were similar. Universal testing machine applied compressive loading at a crosshead speed of 0.5 mm/min. The steel indenter was contacted to the center of specimens. The crosshead of the testing machine was stopped when the first discontinuity of the chart recording appeared. Prakki et al reported that mean fracture loads for 1-mm ceramic plates were higher in thicker resin cement comparing with thinner resin cement.[18] Another study resulted in slight downward trend of the fracture load with increasing cement thickness.[39] The study of Piemjai et al exhibited that fracture resistance of ceramic plates bonded to enamel was significant higher than those bonded to dentin. However, the authors suggested that bonding technique and curing system of resin cements influenced the fracture resistance of porcelain laminates.[40]

CHAPTER III

Materials and Methods

Operational Definition

1. Rely X™ U200 Self-adhesive universal resin cement (UN) (3M ESPE, St. Paul, MN, USA) – self-adhesive resin cement
2. Rely X™ Ultimate Adhesive Resin Cement (UL) (3M ESPE, St. Paul, MN, USA) – adhesive resin cement with separated bonding system
3. Zinc Phosphate cement (ZN) (Zinc cement improved, SS White, Gloucester, UK) – Zinc phosphate cement
4. Single Bond Universal Adhesive (SU) (3M ESPE, St. Paul, MN, USA) - universal multimode adhesive
5. Scotchbond™ Universal Etchant (ET) (3M ESPE, St. Paul, MN, USA) - Etching Gel
6. Compressive strength test – The compressive strength test is conducted by using the universal testing machine (Instron model 5566, Canton, MA, USA) at a cross-head speed of 0.5 mm/min

Research Design

This study was an experimental study which extracted human third molars were used to investigate. Interventions of this study were thickness of resin cement (100 and 300 μm), resin-bonded tooth substrate (enamel and dentin) and adhesive resin cement system (total-etch and self-adhesive resin cement). Zinc phosphate cement was used as control. Dependent variable was load to crack or fracture of the ceramic specimens, measured in Newton. Compressive strength test was used to evaluate.

Research Methodology

Diagram of study design

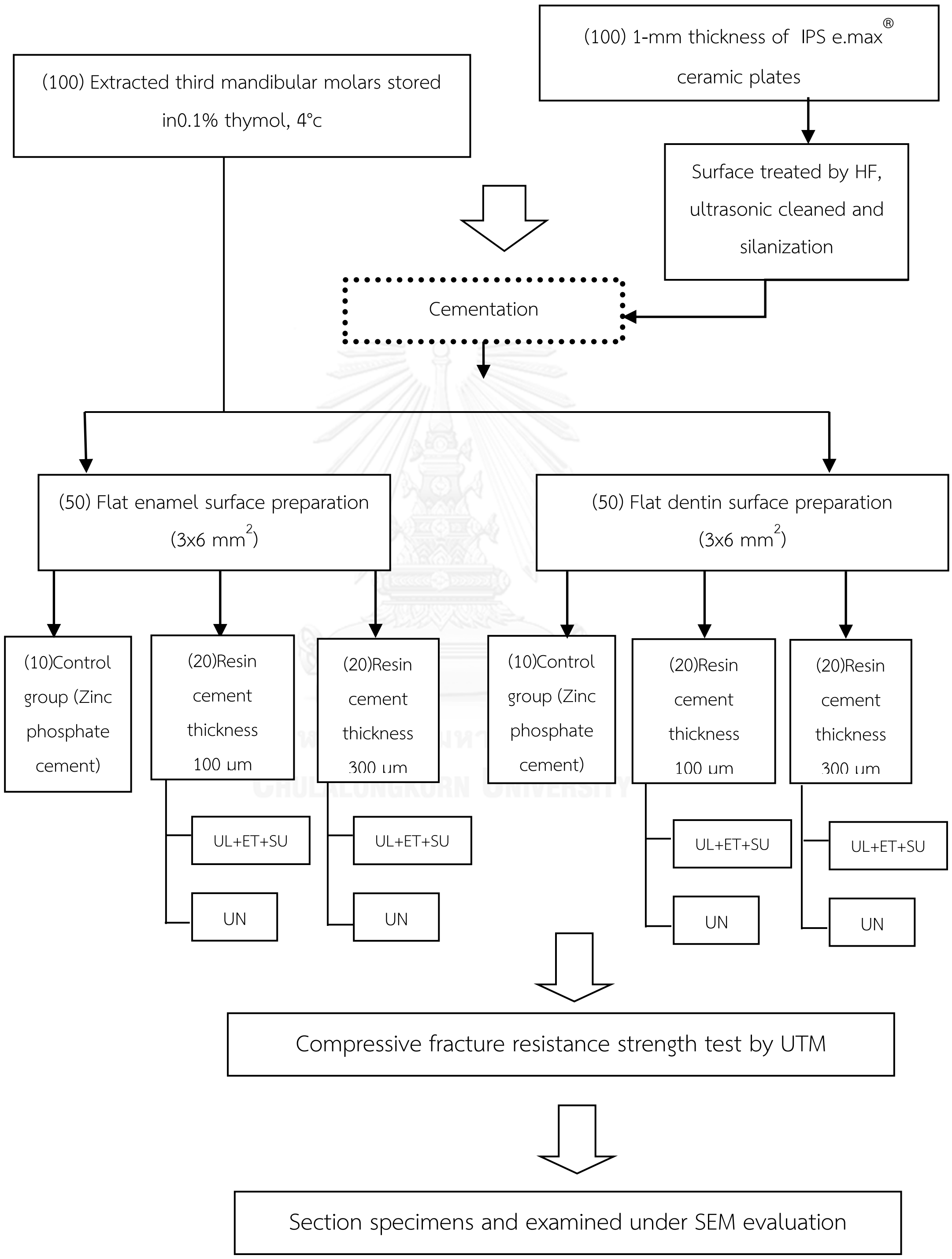

Figure 2 Diagram of study design

Ethical Consideration

This study had been approved by the ethical committee of the Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand. The study reference ID was HREC-DCU 2013-006. (Appendix A)

Sample Description

Samples of this study were thirty selected non-carious and non-restored extracted human molars.

Sample size estimation (n per group) was calculated from this formula;

$$n = \frac{2\sigma^2(Z_\alpha + Z_\beta)^2}{(\mu_1 - \mu_2)^2}$$

$$\sigma^2 = \frac{(n_1 - 1)S_1^2 + (n_2 - 1)S_2^2}{n_1 + n_2 - 2}$$

Where:

Z_α is the value of the standardized score cutting off $\alpha/2$ proportion of each tail of a standard normal distribution (for a two-tailed hypothesis test)

Z_β is the value of the standardized score cutting off the upper proportion

α^2 is the assumed common variance in the two groups (S)

$\mu_1 - \mu_2$ is the difference in means of the two groups

In this study determines

Z_α = 1.96 at 95 % confidence interval

Z_β = 0.84 at 80% power of test

According to the result of pilot study, mean and standard deviation was applied to calculate the sample size using STATA software (version 10). The number of sample size estimation was 7.87. Therefore, the number of specimens per group should be more than 7.

Materials

Table 2 Materials used in this study

Materials	Manufacturer	Batch code
IPS e.max Press Ingot LT A1	Ivoclar Vivadent	S02097
Rely X Ultimate	3M ESPE	499133
Rely X U200	3M ESPE	513888
Zinc Cement Improved	SS WHITE	324708
Single bond Universal Adhesive	3M ESPE	507334
Scotchbond Etchant	3M ESPE	N432275
Monobond N Universal Primer	Ivoclar Vivadent	R71491
IPS Ceramic Etching Gel (5%HF)	Ivoclar Vivadent	S13497

Preparation of Enamel and Dentin Specimens

One hundred extracted human third molars with anatomical width greater than 10 mm were included. Visual examination showed all selected teeth to be free of caries, restorations, and defects. The teeth were stored in a solution of 0.1% thymol for 24 hours, followed by a solution of normal saline. The buccal aspects of molars were polished to achieve a flat area of at least 3 x 6 mm^2 under water coolant in an automatic polishing machine (DPS 3200, IMTECH, Durban, South Africa).

Each tooth was sectioned by means of a low-speed cutting machine (Isomet 1000, Buehler, Lake Bluff, Ill., USA) to obtain identical 4-mm-thick specimens. The flat surface and thickness of each specimen were measured with a digital caliper (Mitutoyo, Kanagawa, Japan) to ensure the desired dimensions. For enamel specimens, the horizontal flat area was polished with 800- to 1000-grit silicon carbide paper. The flat surfaces were examined by microscopy at 20x magnification (SZ 61, Olympus, Tokyo, Japan). The specimens with exposed dentin were excluded from the study. The enamel specimens were randomly divided into 5 groups of 50 specimens. For dentin specimens, the superficial flat dentin surface was prepared and polished by the same procedure as used for enamel specimen preparation. Fifty dentin specimens were divided randomly into each group. All dentin specimens provided at least 3 x 6 mm^2 of surface area when examined by microscopy at 20x magnification. All tooth specimens were embedded in unfilled resin with the flat polished surface exposed as shown in Fig.3

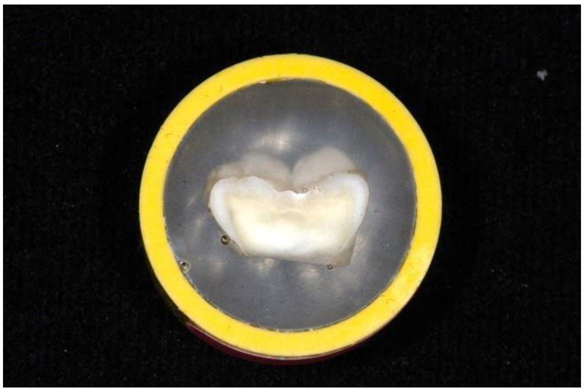

Figure 3 specimen

Fabrication of Ceramic Plates

Standardized wax patterns with dimensions of 3 x 6 mm^2 and thickness of 1 mm were fabricated in metallic molds (Fig.4) and measured by means of digital calipers (Mitutoyo). IPS e.max Press ingots LT A1 (Ivoclar Vivadent) were used. Spruing, investing, pressing, divesting, and finishing of the ceramic plates were processed according to manufacturer's recommendations. One technician performed the entire laboratory procedure. The thickness of ceramic plates was measured with a precimeter (Aura-Dental, Euerdorf, Germany) at three locations to ensure uniform thickness of each specimen (Fig.5). The center of each ceramic plate was marked to illustrate where the indenter of the testing machine came into contact with the ceramic plate. All specimens were stored at room temperature until their surfaces were treated in the cementation procedure. For surface treatment of ceramic plates, IPS Ceramic Etching Gel 5% HF acid (Ivoclar Vivadent) was applied to the inner surface of specimens for 20 seconds. The gel was then rinsed with copious water and air-dried. Monobond N Universal Primer (Ivoclar Vivadent) was applied in one stroke to the etched surface with a microbrush and allowed to dry completely.

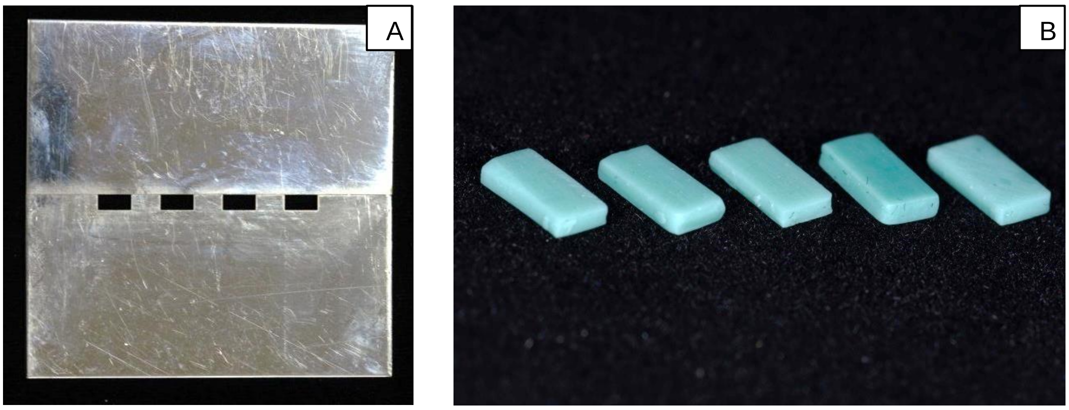

Figure 4 Metallic molds (A) for fabricating specimens (B)

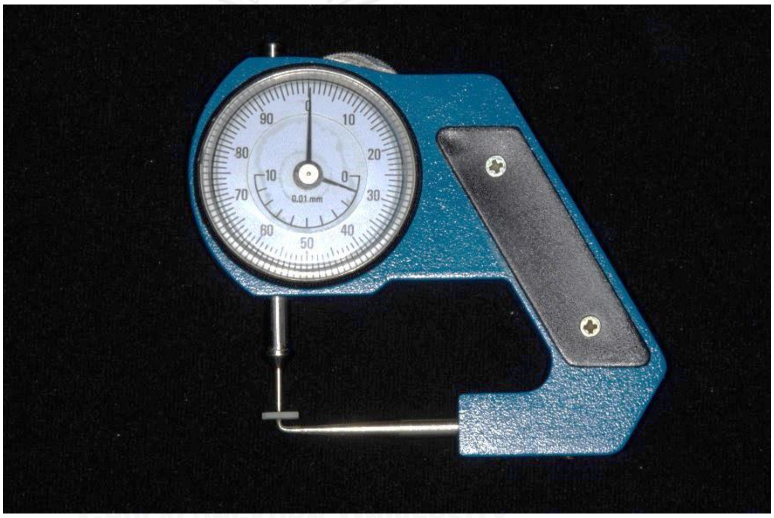

Figure 5 Measuring ceramic plate with precimeter

Cementation Procedure

Both tooth specimens and ceramic plates were randomly assigned to eight groups of 10 for luting with resin cement. Two different types of resin cements (RelyX U200, 3M ESPE; and RelyX Ultimate, 3M ESPE) were applied to the inner surface of ceramic plates. Each type of resin cement was performed according to the manufacturer’s instructions for each individual group (Table 3). Metal strips of 100-

and 300-μm thicknesses were used as spacers in each group. The metal strips were positioned at the ends of each side of the ceramic plates. The inner surface of ceramic plates was seated on either enamel or dentin specimens. A 10-N force was loaded by means of a durometer for 10 seconds. The excessive cement was removed with a dry microbrush. Resin cements were polymerized with a visible-light-polymerization unit (Demi Plus, Kerr Corporation, Orange, Calif.; operating at 1100 mW/cm^2) for 2 minutes as follows: directly on top for 40 seconds and at a 45° angle on each of the four sides for 20 seconds each (Fig.6). A light intensity meter (100 Optilux; Kerr Corporation) was used to monitor the light intensity throughout the study. All specimens were stored in deionized water at 37 °C ± 2°C for 24 hours before compressive fracture testing. For control groups, zinc phosphate cement (Zinc Cement Improved, SS White, Gloucester, UK) was used for cementation. The mixing procedure was performed according to the manufacturer's instructions (Table 3). The freshly mixed cement was applied to specimens before testing.

Table 3 Summary of resin cement manipulations according to manufacturers' instructions

Type of resin cement	**Procedure**
RelyX Ultimate (3M ESPE, St. Paul, MN, USA)	Application Mode : Etch-and-rinse - Apply etchant for 15 s - Rinse for 10 s - Air dry to remove excess water - Apply the adhesive for 20 s with vigorous agitation - Gently air thin for 5 s - Mix equal amounts of base and catalyst paste for 15 s - Apply resin cement on inner surface of ceramic plates - Remove excess cement - Light-cure for 20 s per surface
RelyX U200 (3M ESPE, St. Paul, MN, USA)	- Mix equal amounts of base and catalyst paste for 10 s - Apply resin cement on inner surface of ceramic plates - Remove excess cement - Light-cure for 20 s per surface
Zinc Cement Improved (SSWHITE, Gloucester, England)	- Mix 1.20 g of powder an 0.5 ml of liquid for 1½ min - Apply zinc phosphate cement on inner surface of ceramic plates - Hold the pressure for seating 7 min

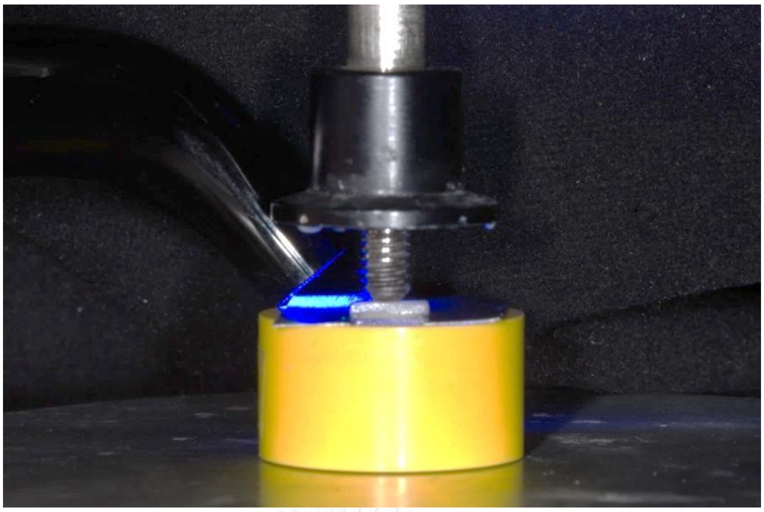

Figure 6 Polymerizing resin cement during specimen were pressed with durometer

Compressive Fracture Resistance Test and Data Analysis

Each specimen was aligned to the center of the universal testing machine (Instron model 5566, Canton, Mass., USA). The crosshead speed of 0.5 mm/min was applied to the center of each ceramic plate. A round 2-mm-diameter steel indenter was loaded in compressive force (Fig.7). When the first discontinuity of the chart recording appeared, the testing machine crosshead was stopped. All loads to fracture were recorded in Newton (N).

The data were analyzed with STATA software, version 10. Multiple linear regression analysis was used to determine if any correlation of fracture load existed among the cement film thickness, resin cement type, substrate variables. The level of significance was determined at 5% ($P<.05$).

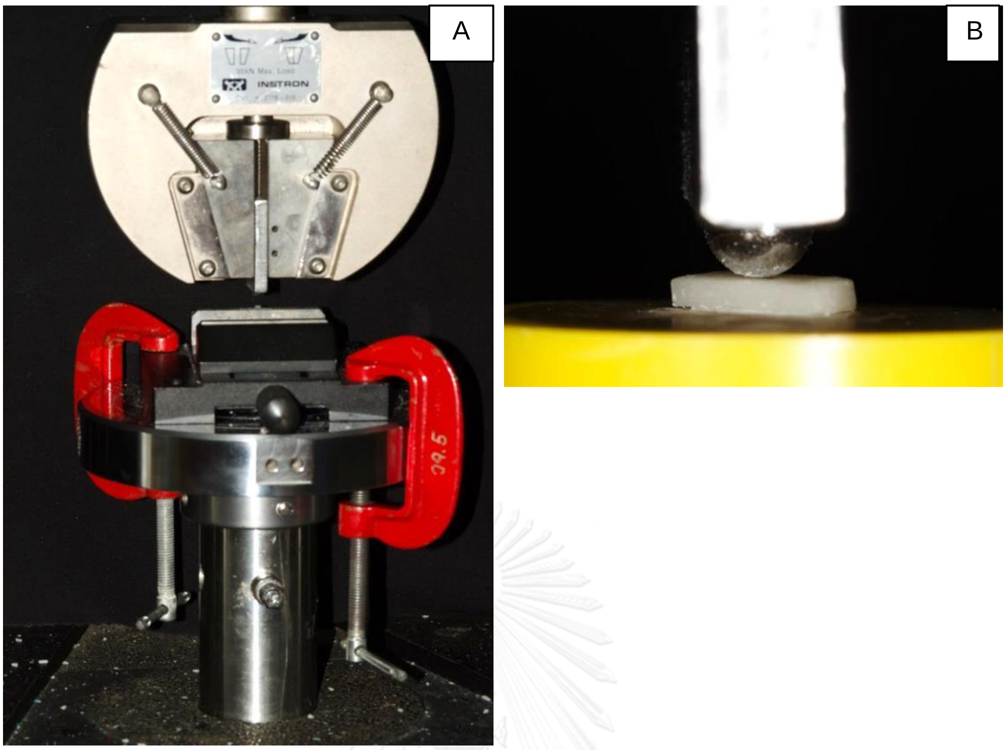

Figure 7 Apparatus for holding specimens during compressive test (A) Round 2-mm in diameter indenter was applied load at the center of ceramic plate (B)

Failure Types Evaluation

After compressive testing, all specimens were examined under a stereomicroscopy (SZ 61, Olympus, Tokyo, Japan) for failure type evaluation. Failure types were classified into 4 categories (Fig.8). Type I – adhesive failure was that the failure was occurred at the interface between cement layer and tooth or ceramic plate. Type II – cohesive failure was that the fracture was observed in cement layer. Type III – mixed failure was classified when adhesive and cohesive failures were presented in same specimen. Type IV – partial failure was that some parts of fractured ceramic plate attached to the bonded substrate.

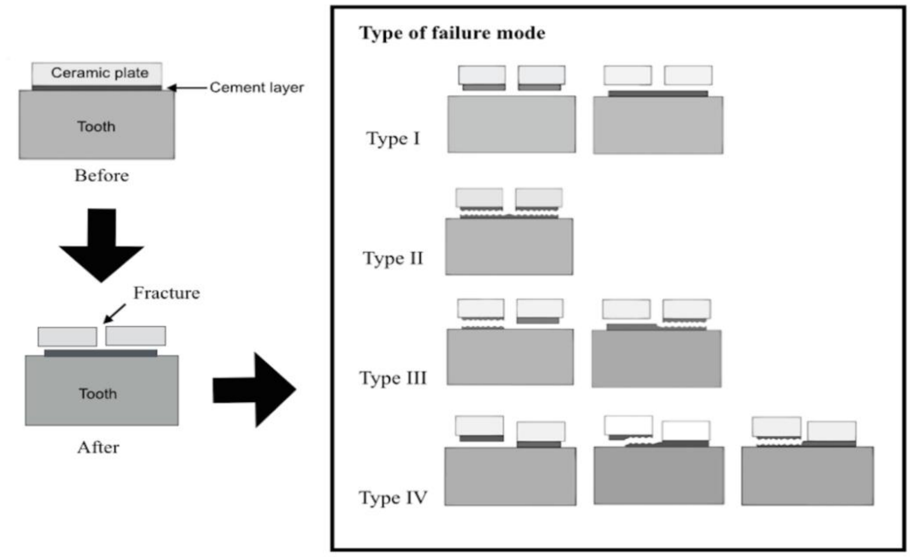

Figure 8 Schematic illustration and type of failure mode

Scanning Electron Microscopic Observations

To measure cement film thickness, five fractured specimens of each group were embedded in autopolymerizing clear acrylic and sectioned with a low speed-cutting machine (Isomet 1000, Buehler). The surfaces of the prepared specimens were then polished with 800- to 1000-grit silicon carbide papers and ultrasonically cleaned for 1 minute. The specimens were allowed to dry completely in a desiccator for 24 hours before SEM analysis. Specimens were then mounted on stubs and sputter-coated with gold coater, then examined by SEM at 15 kV and 150x magnification. The measurement of cement thickness was performed at 3 locations, at both ends of the specimen and at the middle.

CHAPTER IV

Results

Mean Fracture Loads

The mean fracture loads (MFL) and standard deviations for specimens cemented with the different cement film thicknesses and resin cement types are shown in Fig.9 and Table 4. The group with 100-µm of cement thickness bonded to enamel with RelyX Ultimate presented the highest MFL (1591 ±172.59 N), followed by group of those bonded to dentin (1414.13 ±157.21 N). MFL of groups with 300-µm of RelyX Ultimate cement thickness bonded to enamel (1176.02 ±159.81 N) was slightly higher than those to bonded to dentin (1155.47 ±110.92 N). For RelyX U200, MFL of group with 100-µm cement thickness bonded to enamel revealed the higher MFL (1262.48 ±158.97 N) than those of group with 300-µm cement thickness bonded to enamel (874.65 ±83.83 N). The group with 100-µm of cement thickness bonded to dentin resulted in MFL of 842.13 ±92.16 N, followed the group with 300-µm cement thickness bonded to dentin (618.14 ±70.23 N). The lowest MFL was founded in dentin control group (565.19 ±54.50 N).

Table 4 Mean fracture load ± SD (N)

Substrate	Cement	Mean fracture load ± SD (N)		
		Cement thickness 42.6 µm	Cement thickness 100 µm	Cement thickness 300 µm
Enamel	Rely X Ultimate		1591.98 ±172.59	1176.02 ±159.81
Enamel	Rely X U200		1262.48 ±158.97	874.65 ±83.83
Enamel	Zinc phosphate (Control)	879.45 ±84.72		
Dentin	Rely X Ultimate		1414.13 ±157.21	1155.47 ±110.92
Dentin	Rely X U200		842.13 ±92.16	618.14 ±70.23
Dentin	Zinc phosphate (Control)	565.19 ±54.50		

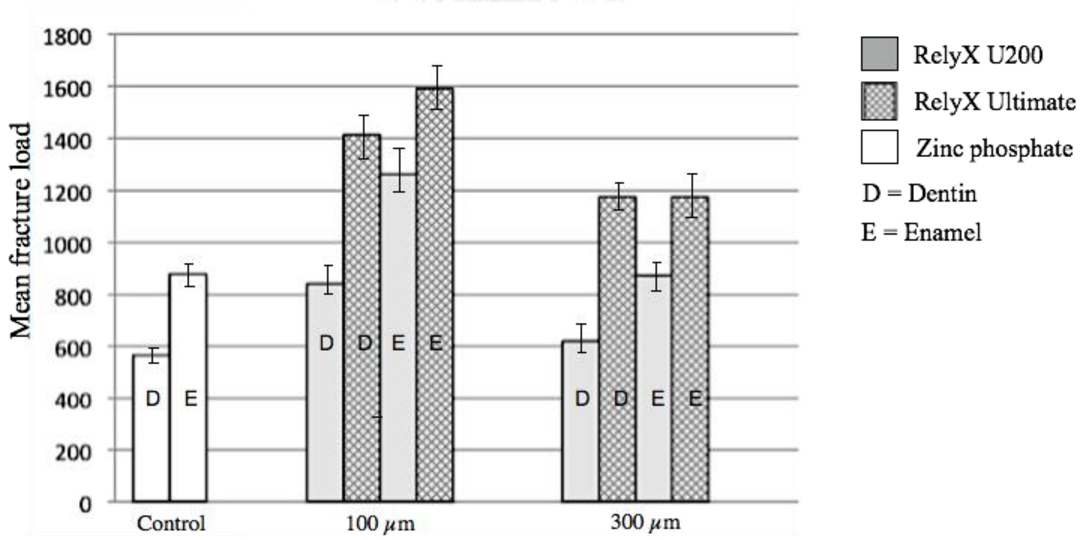

Figure 9 Mean fracture loads (MFL) of ceramic associated with different cement types, cement film thicknesses and substrates

The data of the fracture loads were normally distributed and homogeneity of variance was shown using Kolmogorov-Smirnov analysis. Therefore, the multiple regression analysis was used to test the relationship between the variables (cement film thickness, resin cement type and substrate) and fracture resistance loads (Table 5). There was strong correlation between the variables and fracture loads (Adjusted R^2 = .837). With regard to cement film thicknesses, higher fracture loads of lithium disilicate ceramic were significantly related to a thinner cement thickness (P = .018). RelyX Ultimate significantly correlated with higher fracture loads (P = .004) when compared with RelyX U200. The ceramic plates that cemented on dentin revealed significantly related to lower fracture loads than the groups cemented on enamel (P < .001).

Table 5 Multiple regression analysis among variables (cement thickness, cement type, substrate) and fracture loads

Variables	Beta coefficients (β)	95% CI	*P*-value
Cement thickness			
- 100 µm	349.80	(60.94, 638.66)	.018
- 300 µm	27.89	(-254.25, 310.02)	.845
- Zn phosphate (as reference)			
Cement type			
- RelyX Utimate	423.24	(142.71, 703.76)	.004
- RelyX U200	-12.11	(-292.88, 268.66)	.932
- Zn phosphate (as reference)			
Substrate			
- Dentin	-238.15	(-293.24, -183.06)	<.001
- Enamel (as reference)			

Adjusted R^2 = .837

CI = Confidence Interval

Failure Types Evaluation

The failure pattern distribution (%) as analyzed by stereomicroscopy and SEM is shown in Table 6. RelyX U200 cement presented the highest percentage of adhesive failure (Type I) independent of cement film thickness and bonded substrate. Partial failure (Type IV) occurred with RelyX Ultimate resin cement, and the failure pattern of zinc phosphate cement was type I.

The Type I failure in RelyX U200 is demonstrated that the failure of specimen was occurred at the surface between resin cement and dentin (Fig. 10A). Dentin surface was exposed entire the bonded area. Higher magnifications using SEM illustrated the fractured ceramic and bonded surfaces of tooth specimens. The dentin surface revealed the occluded dentinal tubules by smear plugs, while some dentinal tubules were exposed without any resin cement (Fig. 10B). The mean thickness of the cement layer at the inner surface of ceramic was measured in both groups (Fig 10C, 10D). Air bubbles were apparent in 300-μm cement thickness.

The morphologic aspect of the type IV failure patterns for RelyX Ultimate cement was illustrated in Fig. 11. Under the stereomicroscope, the image showed a general view of fractured specimens in which partial of ceramic plate was attached to bonded substrate (Fig. 11A). At a higher magnification (Figs. 11B, 11C), SEM images exhibited the mixed failure of fracture – ceramic, cement, and adhesive failure between the tooth surface and the cement. The cohesive fracture of cement was characterized by an irregular surface of the cement layer.

Table 6 Proportional prevalence (%) of failure types

Cement type	**Substrate**	**Failure modes**			
		Type I	**Type II**	**Type III**	**Type IV**
Zinc phosphate	Enamel	100%			
	Dentin	100%			
RelyX U200	Enamel	60%		20%	20%
	Enamel	50%			50%
	Dentin	70%		10%	20%
	Dentin	60%		20%	20%
RelyX Ultimate	Enamel				100%
	Enamel				100%
	Dentin				100%
	Dentin				100%

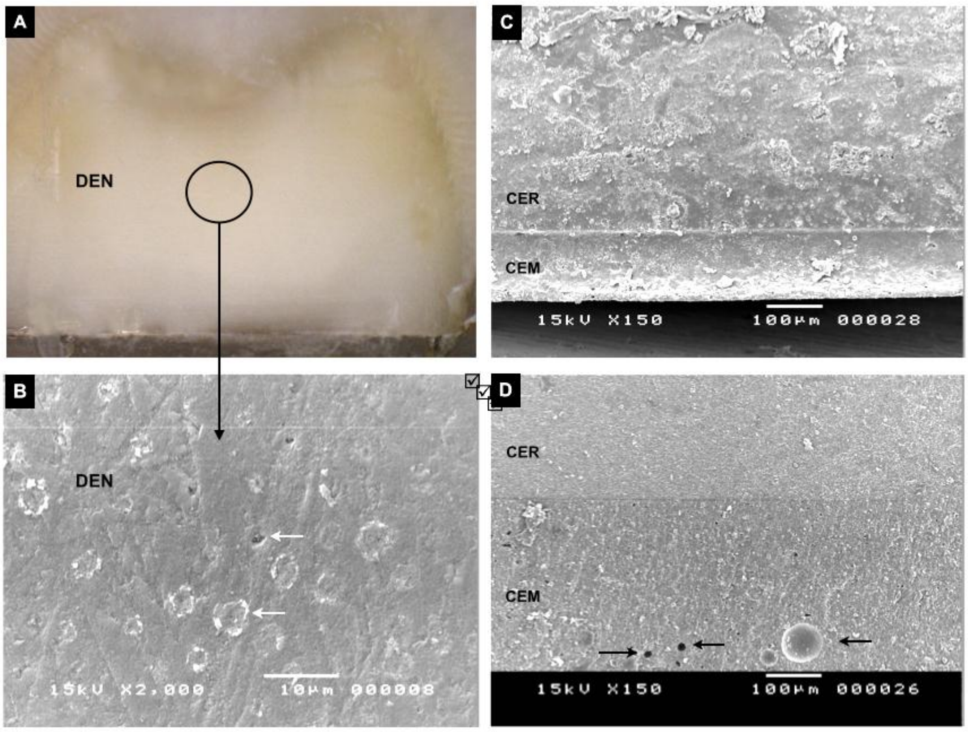

Figure 10 Photomicrographic analysis of RelyX U200 cement after being tested. (A) Type I failure modes can be seen by stereomicroscopy (at 20x). The circled region is shown in (B). The dentin surface of the specimen was exposed (at 2000x), and both open and occluded dentinal tubules by smear plugs can be noted (white arrows). (C, D) SEM images of ceramic plates show that fractured adhesively occurred between interface of the tooth and the cement layer. The cement film thicknesses were measured at 150x magnification (mean thicknesses of 104.3 µm for C and 301.7 µm for D). (D) Air bubbles resulting from mixing can be observed in cement layers (black arrows). CER, ceramic; CEM, cement; DEN, dentin.

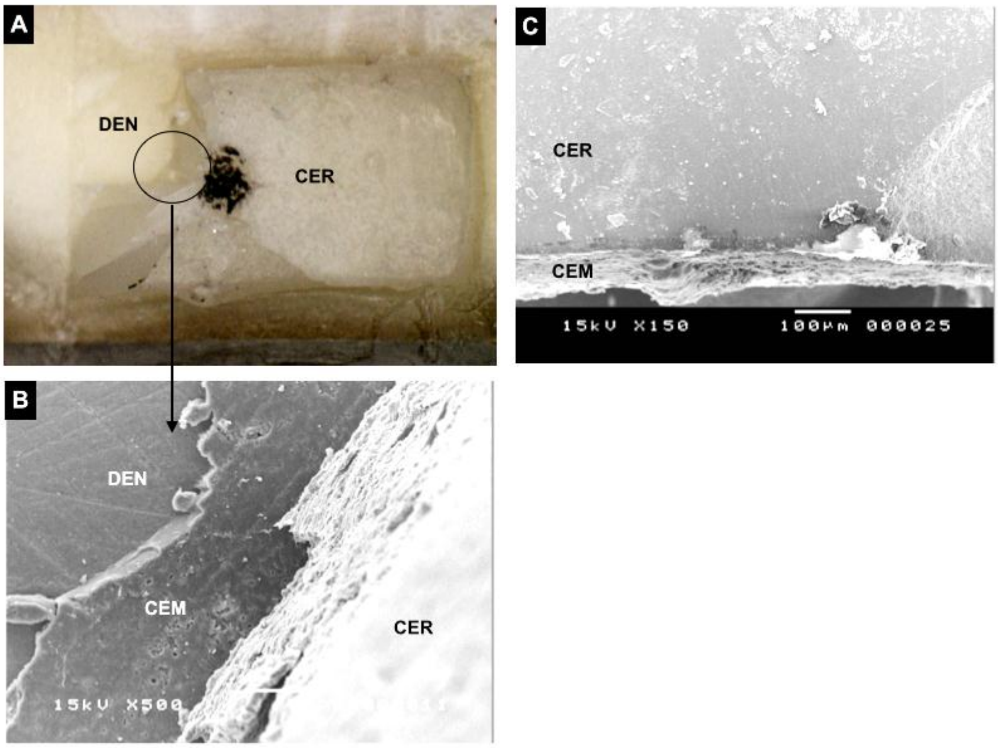

Figure 11 Photomicrographic analysis of RelyX Ultimate cement after being tested. (A) Photomicrograph showing Type IV failure modes by stereomicroscopy (at 20x). (B) At higher magnification (500x), the circled area shows the fracture patterns in ceramic plates that fractured adhesively between the tooth and the cement layer. (C) The cohesive fracture in cement can be observed in this SEM image of fractured ceramic (at150x). CER, ceramic; CEM, cement; DEN, dentin.

Scanning Electron Microscopic Observations

After compressive testing, cement film thickness of representative specimens was measure under SEM at 150x magnification as shown in Table 7. The SEM images showed that the cement film thicknesses fabricated with 100-µm (Fig. 12C) and 300-µm (Fig. 12B) spacers were the desired thickness. The mean film thickness of the zinc phosphate cement was 41.67 µm (Fig. 12D).

Table 7 Mean cement film thickness after compressive fracture resistance testing

Cement type	Substrate	Mean Cement film thickness (±SD)
Zinc phosphate	Enamel	41.6 (±3.5)
	Dentin	43.9 (±5.5)
RelyX U200	Enamel	105.2 (±3.9)
	Enamel	306.5(±4.1)
	Dentin	101.9 (±4.0)
	Dentin	309.5 (4.5)
RelyX Ultimate	Enamel	97.6 (7.6)
	Enamel	311 (±6.5)
	Dentin	102.1 (1.8)
	Dentin	308.9 (±2.6)

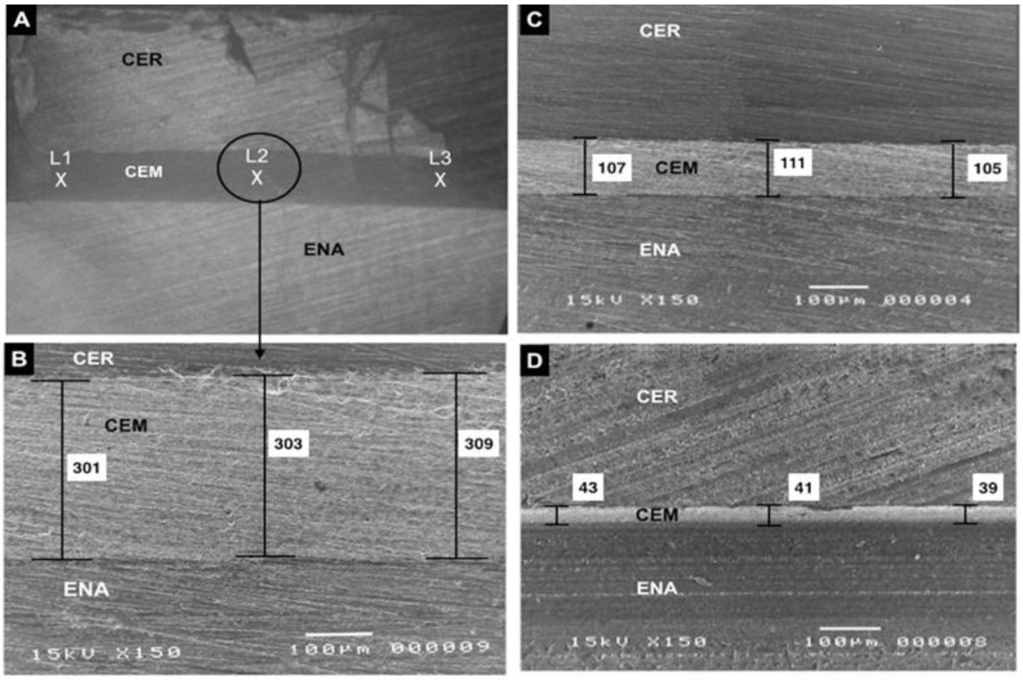

Figure 12 Photomicrography images of cement layers of representative specimens. (A) Stereomicroscopic image (at 45x) illustrates the marked locations for measuring cement film thickness under SEM. (B) SEM image of the circled area in A, showing the cement film thickness of the specimen fabricated with 300-μm spacers at higher magnification (150x). (C) SEM image (at150x) showing the cement film thickness of the specimen fabricated with 100-μm spacers. (D) SEM image showing zinc phosphate cement thickness at 150x. CER - ceramic; CEM - cement; ENA – enamel.

CHAPTER V

DISCUSSIONS AND CONCLUSIONS

Discussions

Ceramics should be adhesively bonded to teeth to increase fracture resistance. This study aimed to evaluate the factors that affected fracture resistance of ceramics under compressive load. This study resulted that cement film thickness, resin cement type, and substrate significantly influenced fracture loads. Therefore, the null hypothesis of this study was rejected. The thicker cement was statistically significantly related to a decreased fracture resistance of ceramics. This is in agreement with results of a previous study by Scherrer et al., these authors showed significant differences between the 26- and 297-μm groups, but not with other cement thicknesses.[19] The manufacturing process for lithium disilicate ceramic could produce cement film thicknesses ranging from 106 to 340 μm after thermocycling.[15] These findings contributed to the two different resin cement film thicknesses that were chosen in the present study. SEM microscopy illustrated the minimal variation of desired cement layer. In contrast, the study by Prakki et al. demonstrated higher fracture resistance of the feldspathic ceramic when resin cement film thickness increased.[18] Because of different ceramic materials, it could be difficult to make direct association between the two studies. The flexural strength of lithium disilicate ceramic (400 MPa) which used in the present study has approximately 5 times greater than those of feldspathic porcelain (60-70 MPa).[20] Based on a study by Molin et al., the result showed the influence of the film thickness of resin cement on the joint bend strength.[37] The authors suggested that the air incorporated during the mixing procedure for a dual-cure resin cement might influence the degree of conversion which may result in a reduction of material properties. The porosity of materials could be relatively prominent in thicker layers of resin cement.

The stiffness of supporting structures has a significant impact on the fracture of ceramics when the ceramic thickness decreases to 1 mm. Flexural radial fracture is influenced by the relative moduli of supporting structures, that is, the cement layer and remaining tooth structure.[41] The higher the elastic modulus of the supporting structure, the higher the load to failure. As a result of this study, reducing fracture loads of ceramics has been shown to be significantly related to the dentin substrate. This could result from a lower elastic modulus of dentin (16 GPa) compared with that of enamel (70-80 GPa).[42] Regarding to resin cement types, RelyX Ultimate cement and RelyX U200 have different chemical composition and mechanical properties. RelyX Ultimate (7.7 GPa) has higher elastic modulus than RelyX U200 (6.6 GPa) according to manufacturer’s data. The higher modulus support could provide a greater fracture loads. Further, increasing the thickness of the cement layer could magnitude the effect of a low elastic modulus of cement. Therefore, a thinner cement thickness is significantly related to greater fracture loads.

Resin luted to ceramics increased the fracture loads when compared with the control groups, this was apparent for both enamel and dentin. The strengthening effect of resin cements on porcelain surfaces have been investigated in the study of Fleming et al. The results revealed that resin cements significantly increased the strength of porcelain independent of surface flaws.[8] The authors proposed that a combination of surface preparation and the luting cement acted to move the fracture origin from the porcelain/cement interface to the cement surface.

Resin bonding systems and bonded substrates were significantly influenced in fracture resistance loads of ceramic plates in the present study. It has been reported that resin bonded to enamel provided better support for porcelain than did resin bonded to dentin.[40] Moreover, controlling of degree of dentin moisture has been the disadvantage of etch-and-rinse system. Overdrying of acid-etched dentin may lead to collapse of collagen fibrils, resulting in poor permeability for impregnating monomers. In contrast, the use of phosphoric acid increased the micromechanical adhesion between enamel and resin cement that promotes the better bond strength.[43]

RelyX Ultimate resin cement demonstrated significant correlation with higher fracture loads of ceramic plates compared with RelyX U200. These results suggest that resin cements relying on the use of etch-and-rinse adhesives achieved higher fracture resistance values. In a study by Vaz et al., the authors suggested that the bond strength responded to the quality of the hybrid layer. Their results showed a significant decrease in bond strength values for the self-adhesive system compared with the etch-and-rinse system.[44] According to SEM analysis of the self-adhesive resin cement by Goracci et al., no hybrid layer or resin tags were observed at the interface of RelyX Unicem and dentin.[45] Close proximity of the resin cement with the dentin tissue was apparent. The study revealed that the heavier seating forces significantly promoted greater bond strength of RelyX Unicem. The authors stated that reducing cement thickness and porosity could result in an increase in bond strength.[45, 46] In the present study, relatively small differences (< 10%) between the group with 300-µm film thickness of self-adhesive resin cement and the control groups were observed in the same substrates. The explanation could be the decreased fracture resistance due to the magnitude of cement film thickness and the self-adhesive resin cement system.

The failure pattern distributions revealed that adhesive fractures between the cement layer and the tooth were noticeable in the RelyX U200 cement. SEM images demonstrated both exposed and occluded dentinal tubules by smear plugs. This could be suggestive of a limited potential for removal or modification of smear layer and resin infiltration into the underlying dentin. In contrast, samples with RelyX Ultimate were categorized primarily as partial and mixed failures, including cohesive fracture of cement. This failure type might represent that cohesive resistance of the cement rather than the magnitude of the bonding material to the enamel or dentin. Increased bond strength could result from the use of acid-etching on tooth surfaces, since hybrid layers were formed. Based on the result of this study, better bond strength between resin cement and tooth substrate resulted in greater fracture resistance of ceramics. Therefore, etch-and-rinse resin cement associated significantly with greater fracture load compared to self-adhesive resin cement. It should be noted that zinc phosphate cement did not lute to ceramic plates, since it presented 100% adhesive failure at the interface between the cement layer and the ceramic.

Within the limitations of this study, a simple geometric configuration was designed to achieve identical and uniform cement film thicknesses supporting rectangular plates.[18, 19] Compressive fracture testing was the method of choice in this study because compressive stress in masticatory force leads to fracture of ceramic restorations. The results of this study could enable clinicians to predict the critical load of ceramic restorations in high-stress areas. However, there are many factors that involve failure of restorations in a physiological environment.[47] High numbers of low cyclic load and wet conditions could affect the degradation of ceramic strength. The different elastic moduli between extracted human teeth (E=6 GPa) and vital teeth (E=15 GPa) should be considered.[48] Clinical evaluations should be conducted to investigate the fracture resistance of restorations as a function of cement film thickness and bonding systems.

Conclusions

Within the limitation of this study, the following conclusions can be drawn:

1. The variables (resin cement film thicknesses, resin cement types, and substrates) had a strong correlation with fracture resistance of lithium disilicate ceramics.

2. Higher fracture resistance of ceramic significantly related to thinner cement film thickness and RelyX Ultimate comparing to RelyX Unicem.

3. Bonding to dentin significantly related to lower fracture resistance of ceramics when compared to bonding to enamel.

4. Partial failure mode in RelyX Ultimate could be suggested that better bond strength provided greater fracture load than RelyX U200

Clinical Implications

Reduced resin film thickness could benefit lithium disilicate restorations. Etch-and-rinse resin cements are recommended in cementation procedures on either enamel or dentin, compared with self-adhesive resin cement, for improved fracture resistance.

REFERENCES

1. Gehrt M, Wolfart S, Rafai N, Reich S, Edelhoff D. Clinical results of lithium-disilicate crowns after up to 9 years of service. Clin Oral Investig 2013;17(1):275-84.
2. Wolfart S, Eschbach S, Scherrer S, Kern M. Clinical outcome of three-unit lithium-disilicate glass-ceramic fixed dental prostheses: up to 8 years results. Dent Mater 2009;25(9):e63-71.
3. Etman MK, Woolford MJ. Three-year clinical evaluation of two ceramic crown systems: a preliminary study. J Prosthet Dent 2010;103(2):80-90.
4. Dhima M, Paulusova V, Carr AB, et al. Practice-based clinical evaluation of ceramic single crowns after at least five years. J Prosthet Dent 2014;111(2):124-30.
5. Burke FJ. Fracture resistance of teeth restored with dentin-bonded crowns constructed in a leucite-reinforced ceramic. Dent Mater 1999;15(5):359-62.
6. Burke FJ. The effect of variations in bonding procedure on fracture resistance of dentin-bonded all-ceramic crowns. Quintessence Int 1995;26(4):293-300.
7. Malament KA, Socransky SS. Survival of Dicor glass-ceramic dental restorations over 16 years. Part III: effect of luting agent and tooth or tooth-substitute core structure. J Prosthet Dent 2001;86(5):511-9.

8. Fleming GJ, Maguire FR, Bhamra G, Burke FM, Marquis PM. The strengthening mechanism of resin cements on porcelain surfaces. J Dent Res 2006;85(3):272-6.

9. Gurel G, Sesma N, Calamita MA, Coachman C, Morimoto S. Influence of enamel preservation on failure rates of porcelain laminate veneers. Int J Periodontics Restorative Dent 2013;33(1):31-9.

10. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. J Adhes Dent 2008;10(4):251-8.

11. Reis A, Pellizzaro A, Dal-Bianco K, et al. Impact of adhesive application to wet and dry dentin on long-term resin-dentin bond strengths. Oper Dent 2007;32(4):380-7.

12. Magne P, Kwon KR, Belser UC, Hodges JS, Douglas WH. Crack propensity of porcelain laminate veneers: A simulated operatory evaluation. J Prosthet Dent 1999;81(3):327-4.

13. Wiskott HW, Belser UC, Scherrer SS. The effect of film thickness and surface texture on the resistance of cemented extracoronal restorations to lateral fatigue loading. Int J Prosthodont 1999;12(3):255-62.

14. International Organization for Standardization. ISO no.4049:2000: Dentistry-polymer-based filling restorative and luting materials.

15. Aboushelib MN, Elmahy WA, Ghazy MH. Internal adaptation, marginal accuracy and microleakage of a pressable versus a machinable ceramic laminate veneers. J Dent 2012;40(8):670-7.

16. Cho SH, Chang WG, Lim BS, Lee YK. Effect of die spacer thickness on shear bond strength of porcelain laminate veneers. J Prosthet Dent 2006;95(3):201-8.

17. Jacob SJ, Hegde C, Prasad KD, Shetty M. An in vitro study to evaluate the effect of storage time and application of subsequent layers on the variation in thickness of three commercially available die spacers. Indian J Dent Res 2010;21(1):92-7.

18. Prakki A, Cilli R, Da Costa AU, et al. Effect of resin luting film thickness on fracture resistance of a ceramic cemented to dentin. J Prosthodont 2007;16(3):172-8.

19. Scherrer SS, de Rijk WG, Belser UC, Meyer JM. Effect of cement film thickness on the fracture resistance of a machinable glass-ceramic. Dent Mater 1994;10(3):172-7.

20. Giordano R, McLaren EA. Ceramics overview: classification by microstructure and processing methods. Compend Contin Educ Dent 2010;31(9):682-70.

21. McLaren EA, Whiteman YY. Ceramics: rationale for material selection. Compend Contin Educ Dent 2010;31(9):666-80.

22. Soares CJ, Soares PV, Pereira JC, Fonseca RB. Surface treatment protocols in the cementation process of ceramic and laboratory-processed composite restorations: a literature review. J Esthet Restor Dent 2005;17(4):224-35.

23. Guarda GB, Correr AB, Goncalves LS, et al. Effects of surface treatments, thermocycling, and cyclic loading on the bond strength of a resin cement bonded to a lithium disilicate glass ceramic. Oper Dent 2013;38(2):208-17.

24. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. Dent Mater 2004;20(4):338-44.

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

26. Roulet JF, Soderholm KJ, Longmate J. Effects of treatment and storage conditions on ceramic/composite bond strength. J Dent Res 1995;74(1):381-7.

27. Marshall GW, Jr., Marshall SJ, Kinney JH, Balooch M. The dentin substrate: structure and properties related to bonding. J Dent 1997;25(6):441-58.

28. Nordenvall KJ, Brannstrom M, Malmgren O. Etching of deciduous teeth and young and old permanent teeth. A comparison between 15 and 60 seconds of etching. Am J Orthod 1980;78(1):99-108.

29. Van Meerbeek B, Perdigao J, Lambrechts P, Vanherle G. The clinical performance of adhesives. J Dent 1998;26(1):1-20.

30. Peumans M, Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G. Porcelain veneers bonded to tooth structure: an ultra-morphological FE-SEM examination of the adhesive interface. Dent Mater 1999;15(2):105-19.

31. Tay FR, Gwinnett JA, Wei SH. Micromorphological spectrum from overdrying to overwetting acid-conditioned dentin in water-free acetone-based, single-bottle primer/adhesives. Dent Mater 1996;12(4):236-44.

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

33. Koibuchi H, Yasuda N, Nakabayashi N. Bonding to dentin with a self-etching primer: the effect of smear layers. Dent Mater 2001;17(2):122-6.

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

35. Perdigao J, Kose C, Mena-Serrano A, et al. A New Universal Simplified Adhesive: 18-Month Clinical Evaluation. Oper Dent 2013.

36. Perdigao J, Sezinando A, Monteiro PC. Laboratory bonding ability of a multi-purpose dentin adhesive. Am J Dent 2012;25(3):153-8.

37. Molin MK, Karlsson SL, Kristiansen MS. Influence of film thickness on joint bend strength of a ceramic/resin composite joint. Dent Mater 1996;12(4):245-9.

38. American Dental Association. ADA Professional Product Review Core Materials: Laboratory Testing Methods. 2008;3(4).

39. Scherrer SS, de Rijk WG, Belser UC, Meyer JM. Effect of cement film thickness on the fracture resistance of a machinable glass-ceramic. Dent Mater 1994;10(3):172-7.

40. Piemjai M, Arksornnukit M. Compressive fracture resistance of porcelain laminates bonded to enamel or dentin with four adhesive systems. J Prosthodont 2007;16(6):457-64.

41. Thompson VP, Rekow DE. Dental ceramics and the molar crown testing ground. J Appl Oral Sci 2004;12(spe):26-36.

42. Lawn BR, Pajares A, Zhang Y, et al. Materials design in the performance of all-ceramic crowns. Biomaterials 2004;25(14):2885-92.

43. Shinchi MJ, Soma K, Nakabayashi N. The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel. Dent Mater 2000;16(5):324-9.

44. Vaz RR, Hipolito VD, D'Alpino PH, Goes MF. Bond strength and interfacial micromorphology of etch-and-rinse and self-adhesive resin cements to dentin. J Prosthodont 2012;21(2):101-11.

45. Goracci C, Cury AH, Cantoro A, et al. Microtensile bond strength and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. J Adhes Dent 2006;8(5):327-35.

46. De Munck J, Vargas M, Van Landuyt K, et al. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater 2004;20(10):963-71.

47. Anusavice KJ, Kakar K, Ferree N. Which mechanical and physical testing methods are relevant for predicting the clinical performance of ceramic-based dental prostheses? Clin Oral Implants Res 2007;18 Suppl 3:218-31.

48. Scherrer SS dRW, Belser UC. Fracture resistance of three all-ceramic crown systems, compard to extracted teeth. J Dent Res 1994;73:190.

APPENDIX

Appendix A. Study protocol and consent form approval

No. 070/2013

Study Protocol and Consent Form Approval
Certificate of Exemption

The Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand has approved the following study to be carried out according to the protocol and patient/participant information sheet dated and/or amended as follows in compliance with the **ICH/GCP with exemption**

Study Title	: Effect of cement types and film thickness on compressive fracture resistance of lithium disilicate ceramics bonded to enamel or dentin
Study Code	: HREC-DCU **2013-047**
Study Center	: Chulalongkorn University
Principle Investigator	: Dr. Thitithorn Rojpaibool
Protocol Date	: August 27, 2013
Date of Approval	: October 1, 2013
Date of Expiration	: September 30, 2015

(Associate Professor Dr. Veera Lertchirakarn)
Chairman of Ethics Committee

(Assistant Professor Dr. Kanokporn Bhalang)
Associate Dean for Research

*A list of the Ethics Committee members (names and positions) present at the Ethics Committee meeting on the date of approval of this study has been attached (upon requested). This Study Protocol Approval Form will be forwarded to the Principal Investigator.

Approval is granted subject to the following conditions: (see back of the approval)

Appendix B. Fracture loads and Type of failure mode

No. of specimen	Fracture load (N)	Mode of failure
Group of 100-µm cement film thickness/ RelyX U200/ enamel		
1	1098.15	Type I
2	1313.63	Type I
3	1332.5	Type III
4	1093.36	Type I
5	1145.15	Type I
6	1218.29	Type I
7	1070.7	Type IV
8	1449.1	Type I
9	1502.8	Type III
10	1401.26	Type IV
Group of 300-µm cement film thickness/ RelyX U200/ enamel		
11	821.25	Type I
12	758.84	Type I
13	925.12	Type IV
14	902.45	Type I
15	866.72	Type IV
16	998.73	Type I
17	761.09	Type IV
18	866.76	Type IV
19	995.64	Type IV
20	849.94	Type I

No. of specimen	Fracture load (N)	Mode of failure
Group of 100-µm cement film thickness/ RelyX U200/ dentin		
21	830.15	Type I
22	845.3	Type I
23	935.63	Type I
24	891.43	Type I
25	734.45	Type I
26	880.06	Type III
27	702.51	Type IV
28	934.2	Type I
29	943.23	Type IV
30	724.31	Type I
Group of 300-µm cement film thickness/ RelyX U200/ dentin		
31	584.28	Type I
32	583.24	Type I
33	524.84	Type I
34	626.19	Type I
35	554.49	Type I
36	603.99	Type IV
37	766.42	Type I
38	700.11	Type I
39	606.66	Type I
40	631.13	Type I

No. of specimen	Fracture load (N)	Mode of failure
Group of 100-μm cement film thickness/ RelyX Ultimate/ enamel		
41	1519.35	Type IV
42	1362.08	Type IV
43	1429.8	Type IV
44	1808.81	Type IV
45	1412.4	Type IV
46	1534.2	Type IV
47	1602.52	Type IV
48	1655.07	Type IV
49	1719.46	Type IV
50	1876.07	Type IV
Group of 300-μm cement film thickness/ RelyX Ultimate/ enamel		
51	1115.87	Type IV
52	1344.06	Type IV
53	1534.2	Type IV
54	1032.09	Type IV
55	1171.35	Type IV
56	1203.3	Type IV
57	1018.47	Type IV
58	1094.45	Type IV
59	1197.9	Type IV
60	1048.49	Type IV

No. of specimen	Fracture load (N)	Mode of failure
Group of 100-µm cement film thickness/ RelyX Ultimate/ dentin		
61	1389.05	Type IV
62	1422.29	Type IV
63	1615.29	Type IV
64	1552.8	Type IV
65	1410.26	Type IV
66	1413.38	Type IV
67	1468.66	Type IV
68	1016.38	Type IV
69	1442.9	Type IV
70	1410.26	Type IV
Group of 300-µm cement film thickness/ RelyX Ultimate/ dentin		
71	1203.3	Type IV
72	1027.55	Type IV
73	1344.06	Type IV
74	1049.37	Type IV
75	1271.75	Type IV
76	1206.89	Type IV
77	1227.22	Type IV
78	1105.46	Type IV
79	1098.6	Type IV
80	1020.53	Type IV

No. of specimen	Fracture load (N)	Mode of failure
Control group for enamel (Zinc phosphate cement)		
81	752.94	Type I
82	982.79	Type I
83	867.21	Type I
84	881.06	Type I
85	879.97	Type I
86	949.4	Type I
87	872.21	Type I
88	886.68	Type I
89	987.77	Type I
90	734.43	Type I
Control group for dentin (Zinc phosphate cement)		
91	524.84	Type I
92	643.48	Type I
93	539.46	Type I
94	647.48	Type I
95	532.36	Type I
96	469.43	Type I
97	575.25	Type I
98	549.9	Type I
99	575.25	Type I
100	594.45	Type I

Appendix C. Measurement of film thickness under SEM

No. specimen	L1	L2	L3	Mean(μm)	Group mean ±SD (μm)
Group of 300-μm cement film thickness/ RelyX U200/ enamel					
1	312.9	300.7	305.6	306.4	
2	300.7	303.2	303.1	302.3	
3	306.7	302.3	310.5	306.5	
4	320.3	315.3	308	314.5	
5	304.3	300.7	302.7	302.6	306.5 ±4.1
Group of 300-μm cement film thickness/ RelyX U200/ dentin					
1	305.1	315.3	308	309.5	
2	305.6	304.3	306.7	305.5	
3	306.2	307.4	310.5	308.0	
4	309.5	308.7	311	309.7	
5	315.3	309.4	320.2	315.0	309.5 ±4.5
Group of 100-μm cement film thickness/ RelyX U200/ enamel					
1	109.7	110.8	106.4	109.0	
2	112	100.4	92.9	101.8	
3	105	104.2	106.9	105.4	
4	104	107	109.5	106.8	
5	102.3	102.7	105	103.3	105.2 ±3.9
Group of 100-μm cement film thickness/ RelyX U200/ dentin					
1	100.2	102.8	99.4	100.8	
2	102.7	100.2	100.2	101.0	
3	110.1	107.3	102.3	106.6	
4	92.6	107.3	106	102.0	
5	100.3	100.2	100.2	100.2	101.9 ±4.0

No. specimen	L1	L2	L3	Mean(μm)	Group mean ±SD(μm)
Group of 300-μm cement film thickness/ RelyX Ultimate/ enamel					
1	315	310.4	312.9	312.8	
2	322.7	322.5	322.2	322.5	
3	301	308.5	305.6	305.0	
4	310.5	315.3	315.3	313.7	
5	308.6	310.5	308.5	309.2	311 ±6.5
Group of 300-μm cement film thickness/ RelyX Ultimate/ dentin					
1	309.4	314.1	309.4	311.0	
2	308	308	303.1	306.4	
3	300.7	303.1	300.7	301.5	
4	310.6	307.8	310.1	309.5	
5	315.3	310.5	315.3	313.7	308.9 ±2.6
Group of 100-μm cement film thickness/ RelyX Ultimate/ enamel					
1	102.7	97.78	97.78	99.4	
2	95.44	100.2	102.7	99.4	
3	85.6	88.6	82.4	85.5	
4	98	98	98.7	98.2	
5	108.4	98.9	107.1	104.8	97.6 ±7.6
Group of 100-μm cement film thickness/ RelyX Ultimate/ dentin					
1	110.2	105.1	107.6	107.6	
2	100.2	105.1	100.4	101.9	
3	95.3	95.3	97.8	96.1	
4	98	102.1	96.4	98.8	
5	100.2	100.6	110	103.6	102.1 ±1.8

No. specimen	L1	L2	L3	Mean(µm)	Group mean ±SD(µm)
Control group of dentin					
1	38.4	33.9	42.2	38.2	
2	47.6	40.2	49.8	45.9	
3	40	48.2	44.5	44.2	
4	35.6	42	49.7	42.4	
5	42.5	47.8	47.7	46.0	43.9 ±5.5
Control group of enamel					
1	42.7	40.4	41.8	41.6	
2	34	43.1	37.8	38.3	
3	44.9	42	37.9	41.6	
4	45.1	46.9	41.8	44.6	
5	46.7	40.9	36.9	41.5	41.6 ±3.5

VITA

NAME Thitithorn Rojpaibool

DATE OF BIRTH September 07, 1985

PLACE OF BIRTH Bangkok, Thailand

ADDRESS 23 Onnut 17 yak 15 Road, Suanluang, Bangkok, Thailand

INSTITUTIONS ATTENDED

- 2004 - 2010 Doctor of Dental Surgery, Faculty of Dentistry, Mahidol University, Bangkok, Thailand

PROFESSIONAL PRACTICE

- 2010 - 2011 General practitioner, Plubplachai Hospital, Buriram, Thailand
- 2011 - 2012 General practitioner, Nangrong Hospital, Buriram, Thailand

RESEARCH GRANT

- The Graduated School of Chulalongkorn University, Bangkok, Thailand