MICROSHEAR BOND STRENGTH AND MICROLEAKAGE
OF GLASS IONOMER CEMENT ON AGED DENTIN

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A Thesis Submitted in Partial Fulfillment of the Requirements
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ความแข็งแรงยึดติดแบบเฉือนระดับจุลภาคและการรั่วซึมระดับจุลภาคของวัสดุกลาสไอโอโนเมอร์กับ
เนื้อฟันในฟันผู้สูงอายุ

น.ส.ชุติมา เตชอังกูร

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ชุติมา เตชอังกูร : ความแข็งแรงยึดติดแบบเฉือนระดับจุลภาคและการรั่วซึมระดับจุลภาคของวัสดุกลาสไอโอโนเมอร์กับเนื้อฟันในฟันผู้สูงอายุ. (MICROSHEAR BOND STRENGTH AND MICROLEAKAGE OF GLASS IONOMER CEMENT ON AGED DENTIN) อ.ที่ปรึกษาหลัก : รศ. ทญ. ดร.รังสิมา สกุลณะมรรคา

วัตถุประสงค์ เพื่อศึกษาคุณสมบัติการยึดติดและการรั่วซึมของวัสดุกลาสไอโอโนเมอร์แต่ละชนิดกับเนื้อฟันในฟันผู้สูงอายุ

วิธีการ เก็บข้อมูลอายุของผู้ป่วย และตัวอย่างฟันจำนวน 78 ซี่ที่ได้รับการถอนจากการเป็นโรคปริทันต์อักเสบ หรือด้วยเหตุผลทางการแพทย์ ของผู้ป่วยที่มารับการรักษาทางทันตกรรมที่คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย ซึ่งแบ่งเป็น 2 กลุ่มอายุ คือ อายุ 16-30 ปี และอายุมากกว่า 65 ปีขึ้นไป โดยนำไปศึกษาประสิทธิภาพการยึดติดกับวัสดุอุดกลาสไอโอโนเมอร์ 3 ชนิด (Fuji II LC, Equia Forte Fil และ Fuji BULK) ด้วยวิธีการทดสอบความแข็งแรงยึดติดแบบเฉือนระดับจุลภาค และการรั่วซึมระดับจุลภาค โดยการทดสอบความแข็งแรงยึดติดแบบเฉือนระดับจุลภาค นำฟันมาตัดแนวระนาบ และฝังในเรซิน หลังจากนั้นนำท่อพอลิเอทิลีนที่ตัดไว้แล้ว มายึดบนเนื้อฟัน 3 จุดด้วยขี้ผึ้งแบบเหนียว และใส่วัสดุลงในท่อ หลังจากที่วัสดุแข็งตัวแล้วจึงใช้ใบมีดตัดท่อออก และแช่ในน้ำปราศจากไอออนเป็นเวลา 24 ชั่วโมงที่อุณหภูมิ 25 องศาเซลเซียส แล้วจึงนำไปทดสอบความแข็งแรงยึดติดแบบเฉือนด้วยเครื่อง Universal testing machine ในส่วนของการทดสอบการรั่วซึมระดับจุลภาค กรอเตรียมโพรงฟัน Class V ที่ด้านแก้มของฟันแต่ละซี่ ทำการบูรณะด้วยวัสดุทั้ง 3 ชนิด และแช่ในน้ำปราศจากไอออนเป็นเวลา 24 ชั่วโมงที่อุณหภูมิ 25 องศาเซลเซียส หลังจากนั้นจึงนำฟันมาขัดและเคลือบด้วยยาทาเล็บยกเว้นบริเวณวัสดุบูรณะ แล้วจึงนำไปแช่ใน basic fuchsin ความเข้มข้นร้อยละ 0.5 เป็นเวลา 24 ชม. เมื่อครบเวลานำฟันมาขัดทำความสะอาด ทำการตัดฟันที่กึ่งกลางของวัสดุอุด และนำไปส่องกล้องจุลทรรศน์แบบสเตอริโอเพื่อดูการรั่วซึม และคัดเลือกเพื่อนำไปส่องกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด

ผลการศึกษา จากการทดสอบความแข็งแรงยึดติดแบบเฉือนระดับจุลภาค พบว่าวัสดุ Fuji II LC มีค่าความแข็งแรงยึดเฉือนมากที่สุด (เนื้อฟันอายุน้อย, 7.29 MPa; เนื้อฟันคนสูงอายุ, 8.59 MPa; $P < 0.001$) และเมื่อดูที่อายุพบว่าไม่มีความแตกต่างอย่างมีนัยสำคัญ ส่วนการทดสอบการรั่วซึมระดับจุลภาค พบว่า ฟันที่บูรณะด้วย Fuji LC มีการรั่วซึมของสีย้อมมากอย่างมีนัยสำคัญ ($P < 0.01$) และพบว่าเนื้อฟันคนสูงอายุมีการรั่วซึมน้อยกว่าเนื้อฟันอายุน้อยอย่างมีนัยสำคัญ (ด้านบดเคี้ยว: $P = 0.007$, ด้านเหงือก: $P = 0.02$)

สรุปผลการศึกษา การยึดของวัสดุกลาสไอโอโนเมอร์กับเนื้อฟันในฟันผู้สูงอายุไม่แตกต่างอย่างมีนัยสำคัญเมื่อทดสอบความแข็งแรงยึดติดแบบเฉือน แต่มีการรั่วซึมระดับจุลภาคน้อยกว่าเนื้อฟันอายุน้อย

สาขาวิชา	ทันตกรรมผู้สูงอายุและการดูแลผู้ป่วยพิเศษ	ลายมือชื่อนิสิต ....
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KEYWORD: Aged dentin, Glass ionomer cement, Microleakage, Microshear bond strength

Chutima Techa-ungkul : MICROSHEAR BOND STRENGTH AND MICROLEAKAGE OF GLASS IONOMER CEMENT ON AGED DENTIN. Advisor: Assoc. Prof. RANGSIMA SAKOOLNAMARKA, D.D.S., Grad. Dip., Ph.D.

Objective: To compare the microshear bond strength and microleakage of glass ionomer cements when bonded to aged and young dentin.

Background: Root caries commonly occurs in elderly patients. Glass ionomer cement (GIC) is frequently used to restore root caries. Many studies of GIC have been conducted using young dentin, however few studies have assessed adhesion and microleakage of GICs to aged dentin.

Materials and methods: Seventy-eight non-carious human molars (patient age 16-30 = 39; patient age ≥ 65 = 39) were tested with three commercial GICs (Fuji II LC, Equia Forte Fil, and Fuji BULK). For microshear bond strength (μSBS), teeth were horizontally sectioned and embedded in resin and three tubes attached to the sectioned surface. Materials were mixed and injected into the tubes, allowed to set and the tubes removed leaving the GIC cylinders. Specimens were stored in deionized water for 24 h at 25°C and subjected to a shear force in a universal testing machine. For microleakage, a buccocervical cavity was prepared, restored with GIC, and stored in deionized water for 24 h at 25°C. The specimens were polished, coated with nail varnish, placed in 0.5% basic fuchsin for 24 h, sectioned at the midpoint and evaluated for microleakage under a stereomicroscope and scanning electron microscope (SEM).

Results: For μSBS, Fuji II LC showed the highest bond strength among all three products (young dentin, 7.29 MPa; aged dentin, 8.59 MPa; $P < 0.001$). There was no significant difference between age groups. For microleakage, Fuji II LC had more dye penetration ($P < 0.01$) and there was significant difference between age groups. (Occlusal: $P = 0.007$, Gingival: $P = 0.02$)

Conclusion: After bonding of GICs, aged dentin showed no difference in μSBS but less microleakage when compared to young dentin.

Field of Study:	Geriatric Dentistry and Special Patients Care	Student's Signature ....
Academic Year:	2019	Advisor's Signature ....

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CHAPTER 1

INTRODUCTION

Background and rationale

Aging is a physiological and dynamic process that occurs in all events of life, including the biological, psychological and social events.(1) Old age is defined as the final stage of the aging process, ending in death. The United Nation (UN) determines that old age begins from 65, while the World Health Organization (WHO) defines aged as 60 years old. In Thailand, the number of elderly people (defined as aged 60 and over) has increased rapidly and will continue to inflate in future decades.(2) The effects of aging on properties of dentin are increasing occluded dentinal tubules, hardness and mineral contents and reducing fracture toughness.(3)

The dental problem that frequently occurs in the elderly is dental caries, especially root surface caries.(4) The prevalence of dental caries has generally increased with age.(5, 6) It is a major cause of tooth loss in old adults.(7) There are many different treatment modalities to treat dental caries. If the lesion is cavitated on root surface, it can be restored with glass ionomer cement (GIC) as it can release fluoride to the oral cavity and protect the tooth surface against bacteria.(4, 8) Conventional glass ionomer cement (CGIC) has many advantages, such as relatively ease of use, chemically bond to tooth substrate, long-term fluoride ion release, low coefficient of thermal expansion and acceptable esthetic. However, it is sensitive to moisture and desiccation during the initial setting stage, and the physical properties are relatively poor.(9, 10) From its limitation, this material has been improved by incorporating resin monomers, which can polymerize under an action of a light curing unit. This material is known as resin modified glass ionomer cement (RMGIC).

Many studies of GIC have been conducted using young dentin. However, there are limited studies about adhesion and microleakage of GICs to aged dentin. Thus, this research aims to study microshear bond strength and microleakage of glass ionomer cement to aged dentin.

Research questions

Is bonding efficacy of glass ionomer cement different when bonded to aged dentin and young dentin?

Research objectives

1. To compare microshear bond strength of glass ionomer cement when bonded to aged dentin and young dentin and compare between each type of glass ionomer cement
2. To compare microleakage of glass ionomer cement when bonded to aged dentin and young dentin and compare between each type of glass ionomer cement.

Research hypothesis

Ho: There are no differences in microshear bond strengths for each type of glass ionomer cement when bonded to aged dentin compare to young dentin.

H1: There are differences in microshear bond strengths for each type of glass ionomer cement when bonded to aged dentin compare to young dentin.

Ho: Microleakage of glass ionomer cement bonded to aged dentin is not different from young dentin.

H1: Microleakage of glass ionomer cement bonded to aged dentin is different from young dentin.

Scope of research

The research aims to study microshear bond strength and microleakage in extracted teeth divided into 2 age groups, collected from private dental clinic and the hospital at Faculty of Dentistry, Chulalongkorn University.

Limitation

The number of molar teeth obtained from aged patients were limited.

Expected outcomes

1. Understand the bonding properties of different glass ionomer cements.
2. Obtain the data that can help deciding what materials to be used in elderly patients.

Keywords

Aged dentin, Glass ionomer cement, Microleakage, Microshear bond strength

Research design

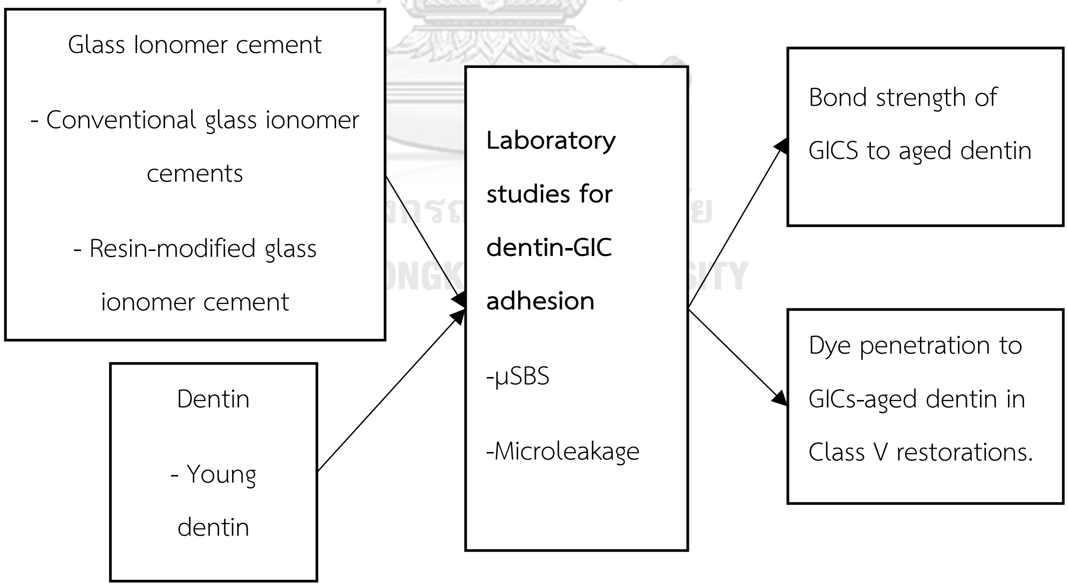
Laboratory research and experimental research

Obstacles and strategies

Obstacle: Glass ionomer cements were not strong and microshear specimens were small

Strategies: Specimens were carefully prepared for microshear bond strength test to prevent the pre-test failure.

Conceptual framework



CHAPTER II

LITERATURE REVIEW

Dentin structure and composition

The compositions of dentin are approximately 70% inorganic material, 20% organic material and 10% water. It acts as the elastic foundation for the enamel and protects pulp tissue. The composition of dentin is similar to bone that comprises of type 1 collagen fibrils. But only dentin has a network of long tubules that extend outward between the pulp and the dentin-enamel junction (DEJ). In young dentin, the dentinal tubules have a diameter approximately 1 to 2 μm. After the third decade of life, there is a continuous deposition of mineral which causes a gradual reduction of tubule dimensions. When the tubules become occluded and appear transparent, it is called 'sclerotic'. The amount of sclerotic dentin increases with age and is common at the apical third of the root and the middle half of dentin in the crown.(11)

In human dentin with increasing age, it easily increases crack growth. The fracture toughness of aged dentin is lower than young dentin.(12) Moreover, the occluded dentinal tubules, hardness and mineral content are found to increase in adult patients with increasing age. This increase is most evident in the outer coronal dentin. For mineral content, the differences of mineral-to-collagen ratio from different regions of dentin in young and old patients, were reported 40% for middle dentin and 70% for outer dentin whereas 4% for inner dentin. The largest proportion of obliterated dentinal tubules is near the DEJ and decreases toward the pulp.(3) In term of mechanical property, aged dentin is found to exhibit higher hardness and elastic modulus than young dentin only in the mantle dentin which is the region within 5 µm from the DEJ.(13)

Glass ionomer cement (GIC)

Glass ionomer cement was first introduced by Wilson and Kent in 1972.(14) It composes of 2 components including the powder which is fluoroaluminosilicate glass powder, and the liquid containing polyalkenoic acid.(8) When the powder is mixed with the liquid, the acid-base reaction starts by neutralizing acid and producing cross-linked polyacid chains. The setting reaction of GIC composes of 3 phases including dissolution, gelation, and hardening phase. For dissolution phase, aluminium, calcium, sodium and fluoride ions are released. Later, the hydrogen ions from acid will diffuse to the glass to replace aluminium, calcium sodium and fluoride ions. The setting reaction is a slow process. It takes 24 h to stabilize the translucency and color of the GIC after placement. The final physical and mechanical properties can be achieved after a month. After the initial set, the gelation phase begins. It depends on rapid reaction of the calcium ions which react with the carboxyl groups of the acid to form a cross-linked polyacid chains. Subsequently, there is a hardening phase which can be as long as 7 days. It takes 30 min to uptake aluminum ions that provide the final strength to the cement.(15) For restorative GIC, the liquid is approximately 60% water, which plays an important role in the setting reaction and in the final structure of the set cement.(8)

Conventional glass ionomer cements have advantages such as adhesion to tooth structure, release of fluoride ions, biocompatibility, and similar coefficient of thermal expansion to tooth structure. However, they have many disadvantages such as short working time and long setting time, brittle, low fracture toughness, poor wear resistance, susceptible to moisture contamination or dehydration during the early stage of the setting reaction. Their properties critically depend on several factors, such as powder/ liquid ratio and amount of water gain or loss during setting reaction. The increased of power/ liquid ratio can reduce working time. In addition, if the water is lost in the reaction, the material will be weak.(15)

Attempts have been made to improve the performance of GIC by adding inorganic or organic components into glass powder or polyacrylates. Although there is continuous improvement, the two remaining problems of CGIC are moisture sensitivity and lack of controlled cure. To overcome these problems, attempts have been made to combine glass ionomer cement with resin composite. Therefore, resin modification of glass ionomer cement was designed to produce favorable physical properties similar to resin composite while maintaining the basic features of the conventional glass ionomer cement. This material is called resin modified glass ionomer cement (RMGIC). Its fundamental acid/ base reaction is supplemented by a second curing process, which is initiated by light. It has many advantages including greater working time, command set on application of visible light, good adhesion, acceptable fluoride release, esthetics similar to those of resin composites, and higher strength characteristics.(10)

The interesting characteristic of GIC is the adhesion to tooth structure. The bond to dentin may be a hydrogen bond to the collagen combined with an ionic bond to the apatite within the dentin structure. The bond strength, as measured by tensile bond strength test (TBS), suggests that it is not strong (2-7 MPa), but clinical experience indicates that it is durable when the material is used to restore non-carious cervical lesion. However, when a GIC is debonded, the fracture will generally run through the GIC (cohesive failure), not along the interface (adhesive failure). The nature of this material is brittle and low tensile bond strength.(15)

Equia Forte Fil is a bulk fill glass hybrid restorative system which contains ultrafine, highly reactive glass particles, disperse within the CGIC structure. It can provide improved physical properties, optimal marginal seal, wear and acid resistance, high fluoride release and no polymerization shrinkage.(16, 17)

Fuji BULK is a new acid-resistant and rapid setting CGIC that consists of high reactivity glass fillers and high molecular weight polyacrylic acid. This material has the high acid erosion resistance and fast setting because of the low phosphate components in glass fillers. (Data from the GC Corporation)

Microshear bond strength test

The rapid progress in dental adhesive technology has extensively influenced modern restorative dentistry. Nevertheless, the bonded interface is the weakest point of an adhesive restoration. Nowadays, micro-bond tests are more reliable than macro-bond tests because of the lower probability of presence of critical sized defects. The bond area of micro-bond strength test is less than 3 mm^2.(18, 19) Micro-bond strength test can be generate higher bond strength value than macro-bond test because the defect concentration in the small cross-sectional areas is lower. Smaller test specimen apparently has less flaws which leads to increase bond strength and reduce the variation.(18, 20, 21) However, the bond strength test has no standard format for reporting which could lead to misinterpretation of the data and bonding ability of adhesives. Micro-bond strength tests are categorized into three types based on the stresses exerted on the test specimens: microshear bond strength (μSBS), microtensile bond strength (μTBS) and micro-push out bond strength (μPBS) tests. μSBS test has lower stress than μTBS test. This test has lower pre-test failure than μTBS testing because it only receives stress before testing when a mold is removed. Whereas μTBS test has more stress before testing. Therefore, μSBS is useful for low bond strength substrate such as glass ionomer cement.(18, 20, 22) Bonded surfaces of μSBS and μTBS tests are exceedingly small. The μTBS test and μSBS test are able to test many specimens on a single surface but specimens used in μTBS test are easily damaged. The other advantage of μSBS test is ease of specimen preparation. It permits regional mapping or depth profiling of different tooth substrates.(23, 24)

Despite all the advantages of μSBS, there is different methodological standardization among the different studies which cannot be compared. There are 2 types of devices that are widely used in a test: knife-edge-chisel and a wire loop. When compared with a wire loop, a knife-edge-chisel caused more stress concentration at the load area. This may lead to the separation of the adhesive interface and dislodgement of dentin substrate.(25) Although a wire loop shows better stress distribution, a knife-edge-chisel is more sensitive to detect subtle differences among materials and techniques that produce higher bond strength values. Moreover, a wire loop also has disadvantage when a loop is stretched during load application, this may cause dislodgement from the bonded interface.(23) The ISO (International Organization for Standardization) recommends the use of a chisel.(26) The cross-head speed using in the chisel method should be at 0.5 and 1.0 mm/min. Therefore, the chisel method is preferable due to easy handling, higher frequency of adhesive failures and lower sensitivity for detecting differences among groups.(23)

Microleakage

In spite of the improvement in adhesive restorative materials, major causes of failure in dental restoration are poor adaptation and microleakage.(27) When a material is placed in a prepared cavity, there are dimensional changes due to variations in the oral environment and placement techniques.(28) Microleakage can occur at the interface of the cavity wall and the restoration.(29)

A microleakage test is a method to evaluate the seal of restorations placed in extracted teeth.(30) This technique involves placement of the bonded specimen in a dye solution for a predetermined period, followed by washing and sectioning of the specimen and examining under microscope to determine the extent of leakage around the tooth/restoration interface. Dyes are most frequently used in dental research. There are either solutions or particle suspensions which have different particle sizes depending on manufacture and the individual behavior of the dye.(31) In 2001, Raskin et al. reviewed 144 studies on microleakage and reported that three most frequently used dyes/tracers were basic fuchsin, methylene blue, and silver nitrate respectively.(32) The three tracers are statistically significantly higher penetrating at the dentin than at the enamel margin. Tracer penetration with fuchsin or silver nitrate showed a moderate correlation with SEM quantitative marginal analysis data at dentin margins, but not at enamel margins. Methylene blue has disadvantages. It is a tracer which is not stable at room temperature and when it is exposed to light. Furthermore, it easily changes to colorless.(33) The advantages of dye penetration method in microleakage were easy, inexpensive and nontoxic.(34)

Microleakage test is often tested in class V cavity, following by class II cavity. Cavities are preferentially located at the cementoenamel junction. Class V dimensions are frequently 2 mm high, 3 mm wide, and 1.5 mm deep.(32)

CHAPTER III

RESEARCH METHODOLOGY

Sample size calculation

The pilot study was performed using 32 molar teeth (young = 16, aged = 16) and the tested materials were Fuji II LC and Equia Forte Fil. The pilot methods were as same as the actual methods. The differences were after a tooth was horizontal sectioned, the occlusal part was used for microshear bond strength test (3 bonded areas/specimen). The other part was used for microleakage test.

<u>For the microshear bond strength test:</u>

We calculated a sample size by using the G*Power program (35) for testing four independent means (ANOVA test).

The values from pilot study were used in the program as followed:

Mean in group1 (μ_1) = 15.85, Size (N) = 9

Mean in group2 (μ_2) = 22.08, Size (N) = 9

Mean in group3 (μ_3) = 8.09, Size (N) = 9

Mean in group4 (μ_4) = 7.98, Size (N) = 9

SD within each group = 9.85

Alpha (α) = 0.05, Z(0.975) = 1.959964

Beta (β) = 0.2, Z(0.8) = 0.842

The sample size calculated by G*Power program were 9 in each group.

For the microleakage test:

The data from the microleakage test is an ordinal scale which there is no specific formula to calculate the sample size. Indirectly, we determined a sample size calculation by using the G*Power program (35) for testing four independent means (ANOVA test).

The values from pilot study were used as followed:

Mean in group1 (μ_1) =0.38, Size (N) = 8

Mean in group2 (μ_2) = 1.88, Size (N) = 8

Mean in group3 (μ_3) = 2.25, Size (N) = 8

Mean in group4 (μ_4) = 2.38, Size (N) = 8

SD within each group = 1.202

Alpha (α) = 0.05, Z (0.975) = 1.959964

Beta (β) = 0.2, Z (0.8) = 0.842

The sample size calculated from G*Power program is 8 in each group.

Materials and methods

Seventy-eight non-carious and unrestored human molars, extracted within 6 months, were collected by age of patients; 39 young molars (patients from 16-30 years old) and 39 aged molars (patients ≥ 65 years old), and stored in 0.1% thymol solution at 25°C. Eighteen teeth (young molars = 9, aged molars = 9) were used for microshear bond strength testing, and sixty teeth (young molars = 30, aged molars = 30) were used for microleakage testing. In each test, the teeth were divided to test with the three GIC products: RMGIC (Fuji II LC, GC Corporation, Tokyo, Japan), and two CGICs (Equia Forte Fil, GC Corporation; Fuji BULK, GC Corporation). The composition of the materials and the manufacturers' instructions are presented in Table 1. Thus, there were six groups in each test (Figure 1): Group S1 or L1, young molars/Fuji II LC; Group S2 or L2, young molars/Equia Forte Fil; Group S3 or L3, young molars/Fuji BULK; Group S4 or L4, aged molars/Fuji II LC; Group S5 or L5, aged molars/Equia Forte Fil; Group S6 or L6, aged molars/Fuji BULK (S = microshear bond strength test; L = microleakage test).

Table 1. *Material's composition and manufacturer*

Materials	Product description	Composition	Manufacturers	Application procedures
GC dentin conditioner Lot 1703081	Dentin conditioner	Polyacrylic acid	GC Corporation Tokyo, Japan	- Apply dentin conditioner to the surface for 20 s using a cotton

				pellet or sponge. - Rinse thoroughly with water and dry. Do not desiccate.
Fuji II LC Lot 1801121	RMGI	Powder: aluminofluorosilicated glass Liquid: polyacrylic acid, tartaric acid, distilled water, camphorquinone, dibutyl hydroxyl toluene, three-resin complex (HEMA)	GC Corporation Tokyo, Japan	- Apply GC Dentin Conditioner for 20 s, rinse and dry gently. - Mix capsule for 10 s. - Dispense into the cavity - Light cure for 20 s. - Polish and apply a final coat of Equia Forte Coat - Light cure for 20 s.
Equia Forte Fil Lot 1701241	CGIC	Powder: 95% strontium fluoroaluminosilicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid	GC Corporation Tokyo, Japan	- Apply GC dentin conditioner for 20 s, rinse and dry gently. - Mix capsule for 10 s - Dispense into the cavity within 10 s. - Final polish after 2 min 30 s from the start of the mix.

				- Apply Equia Forte Coat -Light cure for 20 s.
Fuji BULK Lot 1801261	CGIC	Powder: ultrafine highly reactive glass particles Liquid: high molecular weight polyacrylic acid	GC Corporation Tokyo, Japan	- Apply GC conditioner for 20 s, rinse and dry gently. - Mix capsule for 10s - Dispense into the cavity within 10 s. - Final polish after 2 min 30 s from the start of the mix. - Apply Equia Forte Coat -Light cure for 20 s.
Equia Forte Coat Lot 1612061	Low-viscosity nanofilled surface coating resin	50% methyl methacrylate, 0.09% camphorquinone	GC Corporation Tokyo, Japan	- Dispense a few drops of Equia Forte Coat into a disposable dish. - Immediately apply (within 1 min) to the surfaces using the disposable micro-tip applicator. - Light cure for 20 s.

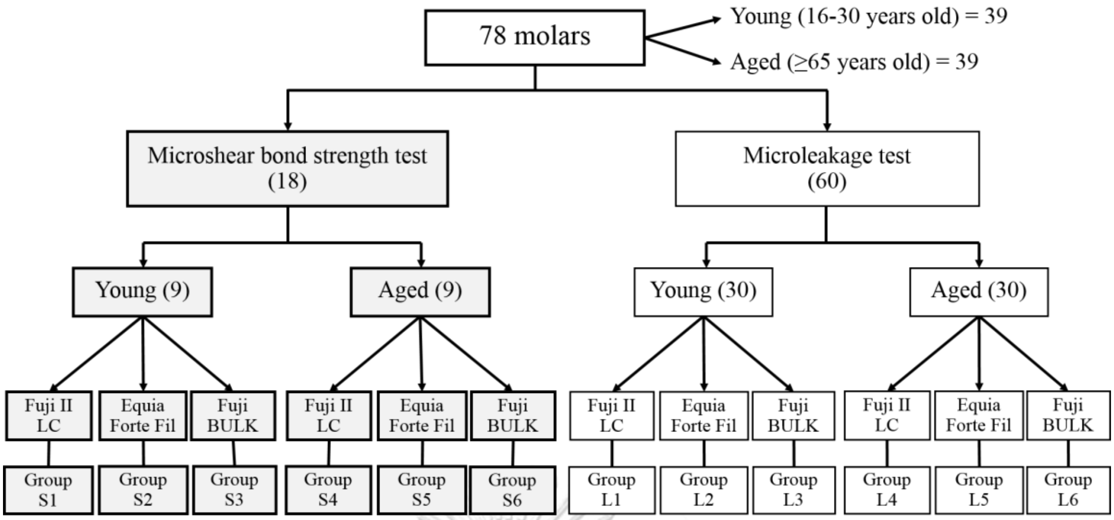


Figure 1: A schematic diagram showing test groups' catagorization.

Parenthesis in text box = numbers of teeth

Microshear bond strength test

Each tooth was horizontally sectioned 3 mm below marginal ridge using low speed cutting machine (Isomet 1000, Buehler, Lake Bluff, IL) under water cooling. The lower part (remaining crown and roots) was used and embedded vertically in self-cured resin. The sectioned dentin surface was used for testing. The specimens were polished with wet 600-grit silicon carbide paper to make a uniform smear layer by polishing machine (Minitech233; Presi, Eybens, France). The presence of dentin was verified under a stereomicroscope (SZ 61, Olympus Corporation, Tokyo, Japan) at x25 magnification.

The sectioned surface was applied with dentin conditioner (GC Corporation) following the manufacturers' instructions. The polyethylene tube with 1.13 mm in diameter was cut 2 mm in length. Then, three tubes were attached on each dentin surface with sticky wax at middle dentin region to obtain 9 specimens in each group. After mixing of CGIC and RMGIC capsules using an amalgamator (ProMix 402E, Dentsply, York, PA), the GICs were injected into the tubes. For RMGIC, the restoration was light cured from the top of specimen for 20 s. Light intensity (>500 mW/cm^2) was periodically checked using a radiometer (100 Optilux, KaVo Kerr, Brea, CA). The tubes were removed, and the specimens were stored in deionized water at 25°C for 24 h. (Figure 2)

Microshear bond strength testing was performed using a knife-edge-chisel with universal testing machine (EZ-S, Shimadzu, Tokyo, Japan). The specimen was stressed in shear using a load cell of 5 kN at a crosshead speed of 0.5 mm/min. The shear force at failure was recorded in newtons and divided by the bonding area (mm^2) to calculate the shear stress in MPa.

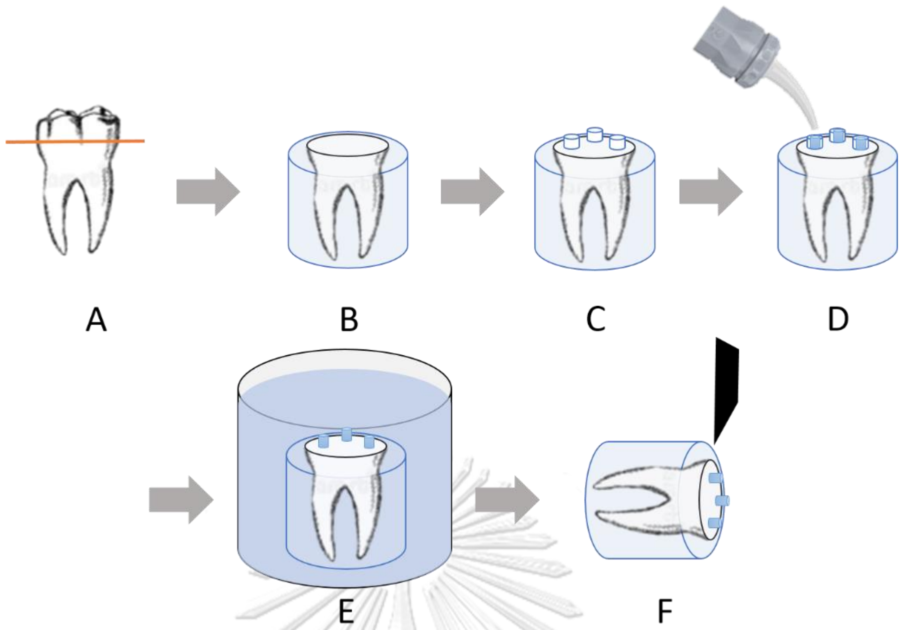


***Figure* 2**: *Microshear bond strength test process.*

Molar tooth was horizontally section (A), embedded in self-cure resin and the surface was polished (B). The surface was treated with dentin conditioner and three tubes were attached on the surface (C). GICs were injected into tubes following the manufacturers' instructions (D). Tubes were removed, and the bonded specimens were stored in deionized water for 24 h (E). The specimens were subjected to test shear bond strength.

After bond strength testing, all specimens were examined using a stereomicroscope (SZ61, Olympus Corporation) at ×25 magnification to evaluate the fracture mode. Failures were classified as type I: interfacial failure (adhesive failure) between dentin and the GIC (no GIC remaining on the dentin surface), type II: mixed failure (combinations of cohesive failure in the GIC and adhesive, shown by the presence of GIC at the dentin surface < 50%) and type III: cohesive failure within the GIC. (failure in GIC or present of GIC at the dentin surface > 50%) (36, 37)

Microleakage test

The teeth were soaked in distilled water at 25°C for a minimum of 12 h. (26) Then, teeth were cleaned with a rubber cup and pumice slurry. A trapezoidal class V cavity preparation, 5 mm wide x 3 mm occluso-gingivally x 1.5 mm deep, was placed at the buccal surface at the cemento-enamel junction using a no. 835 cylindrical diamond bur (ISO No. 806 31 4 107 524 008, Komet dental, Brasseler, Lemgo, Germany) with a high-speed handpiece (Synea Fusion Turbine TG-97, W&H, Bürmoos, Austria). The University of North Carolina probe (UNC15, Hu-friedy, Chicago, IL) was used to measure the dimensions of the cavity.

The teeth were restored with Fuji II LC, Equia Forte Fil or Fuji BULK following the manufacturer's instructions. The unpolished restorations were immediately coated with the Equia Forte Coat (GC Corporation), light cured, and the tooth returned to deionized water storage at 25°C for 24 h. The restoration was polished with coarse, medium, fine, and super fine polishing discs (Sof-Lex discs, 3M Dental Products), respectively, using a low-speed handpiece (Alegra WE-56, W&H, Bürmoos, Austria).

The teeth were prepared for microleakage evaluation by double coating the entire tooth with nail varnish, except for 1 mm around the restoration margins. Then, the teeth were placed in a solution of 0.5% basic fuchsin dye (Fuchsin C.I. 42510, Merck, Darmstadt, Germany) at 25°C for 24 h. After removal of the specimens from the dye solution, the superficial dye was removed with a pumice slurry and rubber cup. The teeth were mounted in a self-cure resin to facilitate handling during sectioning. The resin was cured for 24 h, then teeth were sectioned longitudinally with a low speed cutting machine (Isomet 1000, Buehler) at the midpoint of the restoration to evaluate the dye penetration. (31) The specimen was examined at the occlusal and gingival margins with a stereomicroscope (SZ61, Olympus Corporation) at 30x magnification. The specimen examination was undertaken at random by the examiners who were unaware of the type restorative material. (Figure 3)

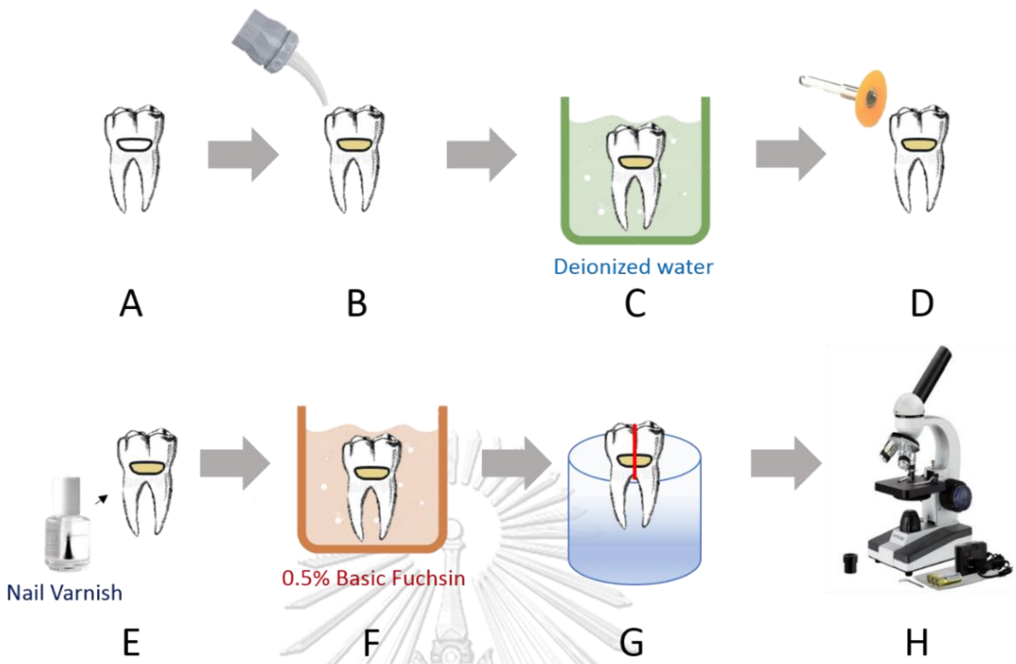


Figure 3: Microleakage test procedure (A-H)

Staining along the tooth restoration interface was recorded at occlusal margin and gingival margin by 2 examiners, according to the following criteria: 0 = no dye penetration; 1 = penetration not more than the half way of the cavity wall; 2 = penetration more than half way of the cavity wall, but no pulpal wall involvement; and 3 = penetration including the pulpal floor of the cavity. If disagreement occurred between the examiners, a consensus was obtained after re-examination of the specimen by both examiners.

Six microleakage test specimens were randomly selected to analyze interface structure. The sectioned surface of each specimen was finished using polishing machine with wet 1200 grit silicon carbide paper, followed by velvet polishing cloths with aluminum oxide paste (0.05μm). The specimens were placed into an ultrasonic cleaner (VGT-1990QTD, GT Sonic, Shenzhen, China) in distilled water for 5 min to remove the smear layer. Specimens were air dried, examined without gold sputtering using scanning electron microscope (Quanta 250, ThermoFisher Scientific, Hillsboro, OR) under low vacuum mode. The representative scanning electron photomicrographs of the bonding interface were obtained at ×1500 magnification.

Statistical analysis

Age data were described in mean, maximum and minimum. Comparison of microshear bond strength was analyzed using Two-way ANOVA and Tukey's post hoc analysis for pairwise comparison, and the relationship between μSBS and fracture mode was determined using the Mann-Whitney U test. Comparison of microleakage test was performed using Kruskal-Wallis H test and Dunn test for pairwise comparison. Data were analyzed using IBM SPSS Statistics for Windows, Version 22.0 (IBM, Armonk, NY). A *P*-value ≤ 0.05 was considered statistically significant.

Ethical consideration

The study protocol and ethics approved by The Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University.

CHAPTER IV

RESULTS

Microshear bond strength test

The mean age of all groups was 48.28 years. The mean age of young and aged group was 24.78 years and 71.78 years, respectively. The maximum age was 80 years and minimum age was 22 years.

Table 2. Means±SD of μSBS values in MPa of all tested groups

Restoration	Age group	
	Young	Aged
Fuji II LC	7.29 ± 2.05 $^{A, a}$	8.59 ± 3.09 $^{A, a}$
Equia Forte Fil	3.43 ± 2.43 $^{B, a}$	2.43 ± 1.63 $^{B, a}$
Fuji BULK	3.58 ± 2.40 $^{B, a}$	3.11 ± 1.92 $^{B, a}$

*Significant at $P \leq 0.05$. Superscripts with different letters are statistically significant. Superscripts with uppercase letters are used for comparison between materials in the same column. Superscripts with lower case letters are used for comparison between age group within the same row.

Two-way ANOVA revealed that there was significant difference between Fuji II LC and other GIC ($P < 0.001$). No significant difference was found between young dentin and aged dentin ($P = 0.93$). No interaction was found between restoration and age group (P = 0.30). Mean and standard deviation of the tested groups are presented in Table 2. It was evident that Fuji II LC showed the highest microshear bond strength (7.29 MPa for young dentin; 8.59 MPa for aged dentin) compared to Equia Forte Fil (3.43 MPa for young dentin; 2.43 MPa for aged dentin) and Fuji BULK (3.58 MPa for young dentin; 3.11 MPa for aged dentin).

Fracture mode analysis results are given in Table 3. Fuji II LC and Fuji BULK showed the highest per cent of type I failure in young dentin (55.6% and 66.7% respectively) and aged dentin (77.8% and 55.6% respectively), while Equia Forte Fil had the highest per cent of type II failure (77.8% both young and aged dentin). Comparison of shear bond strength in fracture mode was not significant (P = 0.051).

Table 3*. The percentages of fracture modes of all tested groups*

Substrate	**Type**	**Material**		
		Fuji II LC	Equia Forte Fil	Fuji BULK
Young dentin	Type I	55.6	22.2	66.7
	Type II	44.4	77.8	33.3
	Type III	0	0	0
Aged dentin	Type I	77.8	22.2	55.6
	Type II	22.2	77.8	44.4
	Type III	0	0	0

Microleakage test

The mean age of all groups was 46.53 years. The mean age of young and aged group was 22.73 and 70.33 years, respectively. The maximum age was 80 years and minimum age was 18 years.

Table 4. Distribution of microleakage in different groups

Group	Dye leakage at occlusal margin				Dye leakage at gingival margin			
	0	1	2	3	0	1	2	3
Group L1	4	4	1	1	0	1	0	9
Group L2	10	0	0	0	0	0	0	10
Group L3	10	0	0	0	2	4	1	3
Group L4	9	1	0	0	5	3	0	2
Group L5	10	0	0	0	4	3	1	2
Group L6	10	0	0	0	4	2	0	4

The distribution of microleakage at the occlusal (enamel) and gingival (dentin) margins are shown in Tables 4. Kruskal-Wallis one-way ANOVA showed statistically significant difference in dye leakage between all the restorative materials for occlusal margins ($P < 0.01$) and gingival margins ($P < 0.01$). Fuji II LC showed more dye penetration than other materials at occlusal margin. At gingival margin, aged dentin was less leakage than young dentin among 3 materials. Examples of stereomicroscope images are shown in Figure 4.

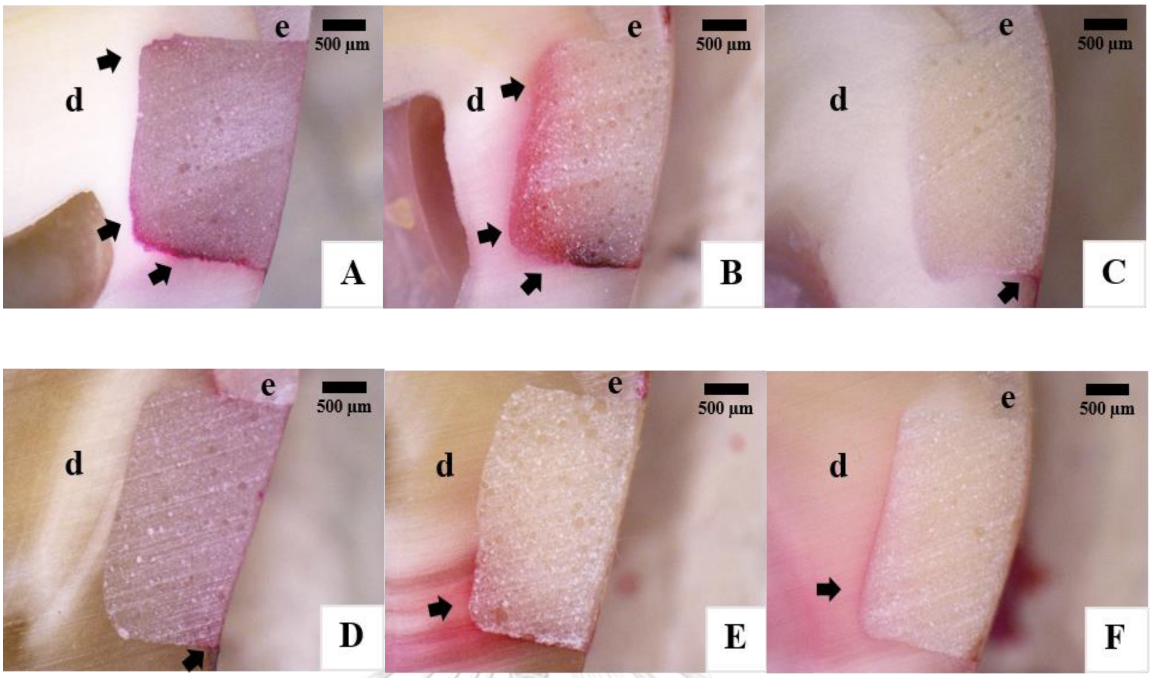


***Figure 4**: Stereomicroscope images of six microleakage specimen groups.*

Arrows show extent of dye penetration. Dentin (d); enamel (e); bar = 500 µm. A and B; Group 1 and 2: dye penetration along pulpal floor at both occlusal and gingival margins, scored '3'. C and D; Group 3 and 4: dye penetration not more than half way along the cavity wall at gingival margin, score '1'. E and F, Group 5 and 6: dye penetration along pulpal floor at gingival margin, score '3'.

For pairwise comparison (Table 5), Fuji II LC revealed statistically significant differences between young and aged dentin, both for the occlusal (P = 0.007) and gingival (P = 0.017) margins. Equia Forte Fil showed statistically significant difference between young and aged dentin only at gingival margin (P = 0.015). No significant difference found between Fuji BULK in young and aged dentin. Dye leakages at occlusal margin and gingival margin showed positive correlation (P = 0.04). When considering the type of material factor, there were significant differences between Fuji II LC and other materials at occlusal margins in young dentin ($P < 0.001$). There were significant differences between young-Fuji II LC and aged-Equia Forte Fil at gingival margin and between young-Equia Forte Fil and aged-Fuji II LC ($P < 0.046$ and $P < 0.005$, respectively). Meanwhile, Fuji II LC and Fuji BULK showed significant difference only at occlusal margin in both young and aged dentin ($P < 0.001$).

Table 5. Intergroup comparison

Groups	Occlusal margins	Gingival margins
Group L1 and group L2	<0.001*	1.000
Group L1 and group L3	<0.001*	0.373
Group L1 and group L4	0.007*	0.017*
Group L1 and group L5	<0.001*	0.046*
Group L1 and group L6	<0.001*	0.239
Group L2 and group L3	1.000	0.153
Group L2 and group L4	1.000	0.005*
Group L2 and group L5	1.000	0.015*
Group L2 and group L6	1.000	0.093
Group L3 and group L4	1.000	1.000
Group L3 and group L5	1.000	1.000
Group L3 and group L6	1.000	1.000

Group L4 and group L5	1.000	1.000
Group L4 and group L6	1.000	1.000
Group L5 and group L6	1.000	1.000
*Significance was considered when *P* value was ≤ 0.05		

SEM analysis

From SEM analysis, some gaps were found between tooth structure and restorations related to the dye leakage scores. The gingival margin showed more gaps than the occlusal margin. Several cracks were seen propagating within the both CGICs and RMGIC. Examples of SEMs at the gingival (dentin) margin are shown in Figure 5.

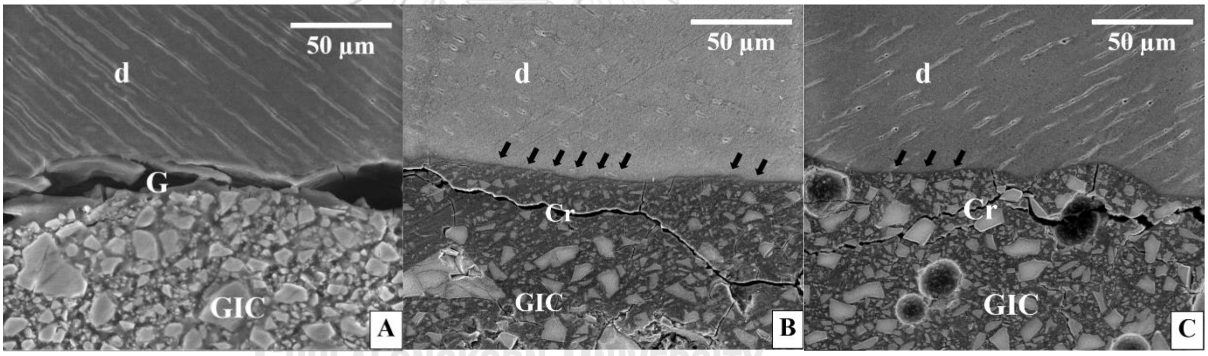


***Figure 5**: Scanning electron microscopic photomicrographs of the tooth-restoration interface (A B, C: Fuji II LC, Equia Forte Fil and Fuji BULK respectively)*

A shows large gap (G) between GIC and dentin. B and C show the gap-free junction (black arrow) and cracks (Cr) within the GIC. Bar = 50 µm.; Dentin (d); Glass ionomer cement (GIC).

CHAPTER V

DISCUSSION

In this study, RMGIC (Fuji II LC) was used as a comparison material because its clinical performance is acceptable.(38) The tested materials that were used were Equia Forte Fil and Fuji BULK which were improved from traditional CGIC.

According to the results of the present study, there was no significant difference between aged and young dentin in μSBS test. Thereby, the first null hypothesis was accepted. A possible explanation for this might be that the bonding area located in the middle dentine region was not altered by age change.(3)

Fuji II LC had the highest μSBS value which was consistent with earlier studies.(37, 39) The first reason for these findings might be due to the different rate of adsorption on dentin surface and polymerization reaction in RMGIC. Light curing initially increases ion exchange by the photochemical initiator, and this results in a strong adsorbed layer on dentin. Meanwhile, the Internal cross-linking reactions in material also create the free radical polymerization process. Therefore, RMGIC will have secondary bonding between the polymers that can prevent the cations cross-linking to the carboxyl groups. On the other hand, CGIC has the low number of carboxyl groups which can decrease the rate of adsorption on dentin surface.(39, 40) The second reason is that the higher amount of resin monomer (HEMA) in RMGIC can provide superior wetting ability and greater fracture strength than CGIC.(37)

Analysis of the failure mode showed that Fuji II LC and Fuji BULK presented high percentage of interfacial failures while Equia Forte Fil presented high percentage of mixed failures, in both young dentin and aged dentin. These results were inconsistent with the previous study which stated that CGIC had the highest percentage of cohesive failure when compared with RMGI and nanofilled RMGIC.(37) This may be because of the different loading methods used. El Wakeel et al.(37) used a wire loop while this study used knife-edge chisel which was more sensitive to detect subtle differences among materials and could produce higher bond strength values than a wire loop. The disadvantage of a wire loop is that when the loop is stretched during load application, it may dislodge from the bonded interface and develop a bending moment at the interface. The second reason may be because of the crosshead speed used in this study, 0.5 mm/min, resulted in more interfacial failures than cohesive failures, in agreement with the study by Munoz et al.(23) No direct relationship between the μSBS values and fracture mode was observed in this study, supported by that of Wakeel et al.(37) and Almuammar et al.,(41) who both found that the high bond strength values were not related to cohesive failure.

Microleakage evaluation was made by dye penetration using 0.5% basic fuchsin as a tracer because it is the most commonly used tracer, easy to manipulate and inexpensive.(32, 34) Moreover, basic fuchsin showed an acceptable correlation with SEM analysis especially at dentin margin while methylene blue showed no correlation at either margin.(33) From the result, there was significantly higher dye penetration at the gingival margins than the occlusal margins for all three materials, which is in agreement with previous results.(42) This may be because enamel has high percentage of inorganic material, while dentin is more complex organization and has high percentage of organic material.(15)

Among the three materials evaluated, RMGIC (Fuji II LC) showed significantly more dye penetration at the occlusal margin than the other materials, which is similar to the study by Gopinath.(28) The possible reason for the greater microleakage scores of Fuji II LC may be due to the resin component in RMGIC that has polymerization shrinkage during the light curing.(43) RMGIC tends to present a higher shrinkage than CGIC.(44) Other reasons were that RMGIC showed low percentage mass change when compare to CGIC at first 24 h,(45) and RMGIC would expand to offset the shrinkage after immersed in water for 1 week.(46) However, these results are rather contradictory to the previous studies.(43, 47, 48) This may be due to the different commercial products and different study protocols (e.g., tooth storage, different times in different dyes, immersion temperature, etc.) compared to the present study.

Fuji II LC showed significantly different microleakage scores between young and aged dentin at both margins. The microleakage scores in aged dentin were also less than young dentin in Equia Forte Fil and Fuji BULK, although not statistically different. Thus, the second null hypothesis is rejected. Fuji II LC had less leakage in aged dentin than young dentin. When the age increases, dentin becomes more sclerotic, harder and has more mineral content. Inorganic material in aged dentin is greater than in young dentin.(3) A carboxyl group in GIC ionically bonds to calcium ion in dentin,(40) therefore, more mineral content in aged dentin may explain why the seal of both CGIC and RMGIC to aged dentin is better than to young dentin.

In this microleakage study, because of the brittle nature of GIC and because dehydration in a high vacuum SEM may desiccate the GIC and create more cracks, the specimens were examined under low vacuum using a scanning electron microscope without gold sputtering. The six specimens showed quite similar appearance of some gaps between the GIC and dentin, and some cracks propagating within the GIC, which is consistent with the study by Eronat et al.(49) Microstructural porosity or voids within GIC, which occurred from specimen preparation and dehydration, can typically produce cracks.(50) It seemed that this SEM appearance did not conform to the dye penetration scores because although the stereomicroscopic images showed no leakage, the SEM photomicrographs showed the bonded area with some cracks within the GIC and gaps between the GIC and tooth substrate. Moreover, a good adaptation as seen in SEM may not mean that no dye penetration occurred.(33)

Fuji II LC showed the highest μSBS value but showed more leakage than the other GICs. Conversely, Equia Forte Fil and Fuji BULK showed lower μSBS values but tended to have lower leakage. This is probably because bond strength may be not correlated with microleakage, as reported in previous studies.(22, 30, 51) Microleakage specimen preparations such as cavity size and shape, location of bonding area and test procedures are different from the μSBS test, and thus these two tests should be discussed separately.(51) Microshear bond strengths and microleakage tests are some of the laboratory methods used to evaluate marginal adaptation, but may not predict the clinical performance: μSBS test results do not correlate with the clinical retention of cervical restorations; and microleakage does not correlate with the occurrence of hypersensitivity and secondary caries.(52)

From the results of this study, we assumed that the materials used in this study may be suitable to use in elderly people because they have less leakage in aged dentin. However, Equia Forte Fil and Fuji BULK may not be used in stress-bearing areas because they have low bond strength. In area that receives more stress, Fuji II LC may be more suitable than others. Further studies and long-term clinical data of GICs should be evaluated and monitored in elderly patients.

CHAPTER VI

CONCLUSION

Under the conditions of the present study, the following conclusions could be derived:

1. For the GICs used in this study, there was no significant difference in μSBS when bonded to aged and young dentin. RMGIC had the highest bond strength among three materials.
2. Aged dentin had less microleakage than young dentin.

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APPENDICES

1. Raw data of each test

1.1. Microshear bond strength test

Table showed raw data of age and microshear bond strength value of each specimen in young group.

Young group							
Teeth number	age	Microshear bond strength value					
		Group S1	Fracture mode	Group S2	Fracture mode	Group S3	Fracture mode
Y1	22	8.92435	2	4.36744	1	1.84968	1
Y2	22	8.69999	1	1.63779	2	2.16378	1
Y3	23	6.28194	2	2.95152	2	7.46354	1
Y4	24	3.23071	2	2.79197	2	4.69401	2
Y5	24	8.23134	1	6.41655	1	2.06407	1
Y6	25	8.79721	1	2.09897	2	1.86464	2
Y7	26	5.09538	2	1.09435	2	3.24068	1
Y8	28	7.18435	1	8.1391	2	7.42864	2
Y9	29	9.13374	1	1.39848	2	1.42092	1

Table showed raw data of age and microshear bond strength value of each specimen in aged group.

Aged group							
Teeth Number	age	Microshear bond strength value					
		Group S4	Fracture mode	Group S5	Fracture mode	Group S6	Fracture mode
O1	67	4.4859	1	3.5198	1	3.53733	1
O2	67	13.4538	1	1.92447	2	1.62034	2
O3	68	3.63206	1	1.83722	2	1.43837	1
O4	69	8.27372	1	1.37604	2	1.27384	2
O5	70	9.6373	1	1.48822	2	5.36209	1
O6	71	7.8541	1	1.1118	2	5.8083	2
O7	74	11.1953	2	0.79771	1	1.83722	1
O8	80	10.2755	2	5.61386	2	1.72255	2
O9	80	8.50804	1	4.173	2	5.38203	1

1.2. Microleakage test

Table showed raw data of microleakage score.

group	number	age	occlusal	gingival
L1	1	26	0	1
	2	24	1	3
	3	22	1	3
	4	24	1	3
	5	21	1	3
	6	24	0	3
	7	24	0	3
	8	21	0	3
	9	22	2	3
	10	23	3	3
L2	1	27	0	3
	2	24	0	3
	3	25	0	3
	4	22	0	3
	5	22	0	3
	6	22	0	3
	7	21	0	3
	8	26	0	3
	9	21	0	3
	10	19	0	3
L3	1	21	0	1
	2	23	0	1
	3	23	0	0
	4	24	0	3
	5	18	0	2
	6	20	0	1
	7	20	0	3
	8	27	0	3
	9	21	0	1
	10	25	0	0

L4	1	69	0	0
	2	75	0	0
	3	77	0	3
	4	67	0	1
	5	65	0	0
	6	65	0	0
	7	71	1	1
	8	67	0	1
	9	70	0	3
	10	80	0	0
L5	1	67	0	0
	2	71	0	0
	3	77	0	2
	4	70	0	1
	5	68	0	0
	6	66	0	1
	7	65	0	1
	8	69	0	3
	9	76	0	0
	10	78	0	3
L6	1	71	0	1
	2	67	0	0
	3	69	0	0
	4	70	0	3
	5	70	0	1
	6	67	0	0
	7	73	0	3
	8	78	0	0
	9	65	0	3
	10	67	0	3

2. SPSS statistic tables

2.1. Microshear bond strength

Descriptive Statistics

Dependent Variable: Max stress

age in group	restoration	Mean	Std. Deviation	N
Young	Fuji II LC	7.2865567	2.04749189	9
	Equia Forte	3.4329078	2.42836979	9
	Fuji Bulk	3.5766622	2.40186868	9
	Total	4.7653756	2.86066953	27
Old	Fuji II LC	8.5906356	3.09141933	9
	Equia Forte	2.4269022	1.63437079	9
	Fuji Bulk	3.1091189	1.92442720	9
	Total	4.7088856	3.57858938	27
Total	Fuji II LC	7.9385961	2.63065156	18
	Equia Forte	2.9299050	2.07363554	18
	Fuji Bulk	3.3428906	2.12496133	18
	Total	4.7371306	3.20899712	54

Levene's Test of Equality of Error Variances[a]

Dependent Variable: Max stress

F	df1	df2	Sig.
.595	5	48	.704

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + Age_gr + restoration + Age_gr * restoration

Tests of Between-Subjects Effects

Dependent Variable: Max stress

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	291.459[a]	5	58.292	11.002	.000	.534
Intercept	1211.782	1	1211.782	228.713	.000	.827
Age_gr	.043	1	.043	.008	.929	.000
restoration	278.268	2	139.134	26.260	.000	.522
Age_gr * restoration	13.148	2	6.574	1.241	.298	.049
Error	254.317	48	5.298			
Total	1757.558	54				
Corrected Total	545.776	53				

a. R Squared = .534 (Adjusted R Squared = .485)

A. Comparison between age groups.

Estimates

Dependent Variable: Max stress

age in group	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
Young	4.765	.443	3.875	5.656
Old	4.709	.443	3.818	5.600

Pairwise Comparisons

Dependent Variable: Max stress

(I) age in group	(J) age in group	Mean Difference (I-J)	Std. Error	Sig.[a]	95% Confidence Interval for Difference[a]	
					Lower Bound	Upper Bound
Young	Old	.056	.626	.929	-1.203	1.316
Old	Young	-.056	.626	.929	-1.316	1.203

Based on estimated marginal means

a. Adjustment for multiple comparisons: Bonferroni.

Univariate Tests

Dependent Variable: Max stress

	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Contrast	.043	1	.043	.008	.929	.000
Error	254.317	48	5.298			

The F tests the effect of age in group. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

B. Comparison between restoration.

ANOVA

Max stress

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	205.512	2	102.756	19.350	.000
Within Groups	127.452	24	5.310		
Total	332.964	26			

Robust Tests of Equality of Means

Max stress

	Statistic[a]	df1	df2	Sig.
Welch	13.724	2	15.246	.000

a. Asymptotically F distributed.

Post Hoc Tests

Multiple Comparisons

Dependent Variable: Max stress

	(I) restoration	(J) restoration	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
Tukey HSD	Fuji II LC	Equia Forte	6.16373333*	1.08632787	.000	3.4508607	8.8766059
		Fuji Bulk	5.48151667*	1.08632787	.000	2.7686441	8.1943893
	Equia Forte	Fuji II LC	-6.16373333*	1.08632787	.000	-8.8766059	-3.4508607
		Fuji Bulk	-.68221667	1.08632787	.806	-3.3950893	2.0306559
	Fuji Bulk	Fuji II LC	-5.48151667*	1.08632787	.000	-8.1943893	-2.7686441
		Equia Forte	.68221667	1.08632787	.806	-2.0306559	3.3950893
Games-Howell	Fuji II LC	Equia Forte	6.16373333*	1.16562055	.001	3.0591142	9.2683525
		Fuji Bulk	5.48151667*	1.21382286	.001	2.2880229	8.6750105
	Equia Forte	Fuji II LC	-6.16373333*	1.16562055	.001	-9.2683525	-3.0591142
		Fuji Bulk	-.68221667	.84159821	.702	-2.8594740	1.4950407
	Fuji Bulk	Fuji II LC	-5.48151667*	1.21382286	.001	-8.6750105	-2.2880229
		Equia Forte	.68221667	.84159821	.702	-1.4950407	2.8594740

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

C. Comparison of shear bond strength in fracture mode

Mann-Whitney Test

Ranks

	fracture_mode	N	Mean Rank	Sum of Ranks
Max stress	interface failure	27	31.69	855.50
	Mixed failure	27	23.31	629.50
	Total	54		

Test Statistics[a]

	Max stress
Mann-Whitney U	251.500
Wilcoxon W	629.500
Z	-1.955
Asymp. Sig. (2-tailed)	.051

a. Grouping Variable: fracture_mode

2.2. Microleakage test

A. Descriptive data of age in each group.

Descriptives

	group			Statistic	Std. Error
age	Fuji II LC-young	Mean		23.10	.504
		95% Confidence Interval for Mean	Lower Bound	21.96	
			Upper Bound	24.24	
		5% Trimmed Mean		23.06	
		Median		23.50	
		Variance		2.544	
		Std. Deviation		1.595	
		Minimum		21	
		Maximum		26	
		Range		5	
		Interquartile Range		2	
		Skewness		.209	.687
		Kurtosis		-.457	1.334
	Equia forte-young	Mean		22.90	.795
		95% Confidence Interval for Mean	Lower Bound	21.10	
			Upper Bound	24.70	
		5% Trimmed Mean		22.89	
		Median		22.00	
		Variance		6.322	
		Std. Deviation		2.514	
		Minimum		19	
		Maximum		27	
		Range		8	
		Interquartile Range		4	
		Skewness		.298	.687
		Kurtosis		-.762	1.334
	Fuji Bulk-young	Mean		22.20	.854
		95% Confidence Interval for Mean	Lower Bound	20.27	
			Upper Bound	24.13	
		5% Trimmed Mean		22.17	
		Median		22.00	

	Variance		7.289	
	Std. Deviation		2.700	
	Minimum		18	
	Maximum		27	
	Range		9	
	Interquartile Range		4	
	Skewness		.286	.687
	Kurtosis		-.372	1.334
Fuji II LC-old	Mean		70.60	1.634
	95% Confidence Interval for Mean	Lower Bound	66.90	
		Upper Bound	74.30	
	5% Trimmed Mean		70.39	
	Median		69.50	
	Variance		26.711	
	Std. Deviation		5.168	
	Minimum		65	
	Maximum		80	
	Range		15	
	Interquartile Range		9	
	Skewness		.734	.687
	Kurtosis		-.617	1.334
Equia forte-old	Mean		70.70	1.491
	95% Confidence Interval for Mean	Lower Bound	67.33	
		Upper Bound	74.07	
	5% Trimmed Mean		70.61	
	Median		69.50	
	Variance		22.233	
	Std. Deviation		4.715	
	Minimum		65	
	Maximum		78	
	Range		13	
	Interquartile Range		10	
	Skewness		.561	.687
	Kurtosis		-1.250	1.334
Fuji Bulk-old	Mean		69.70	1.184
	95% Confidence Interval for	Lower Bound	67.02	

Mean	Upper Bound	72.38	
5% Trimmed Mean		69.50	
Median		69.50	
Variance		14.011	
Std. Deviation		3.743	
Minimum		65	
Maximum		78	
Range		13	
Interquartile Range		5	
Skewness		1.183	.687
Kurtosis		1.792	1.334

B. Correlation between occlusal score and gingival score.

Nonparametric Correlations

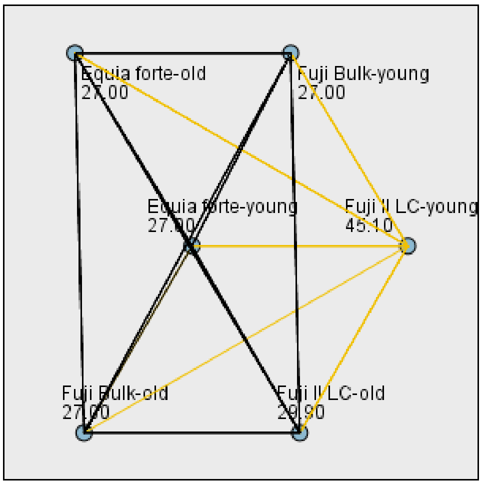
Correlations

			occlusal	gingival
Spearman's rho	occlusal	Correlation Coefficient	1.000	.267*
		Sig. (2-tailed)	.	.039
		N	60	60
	gingival	Correlation Coefficient	.267*	1.000
		Sig. (2-tailed)	.039	.
		N	60	60

*. Correlation is significant at the 0.05 level (2-tailed).

C. Occlusal margin

Pairwise Comparisons of group



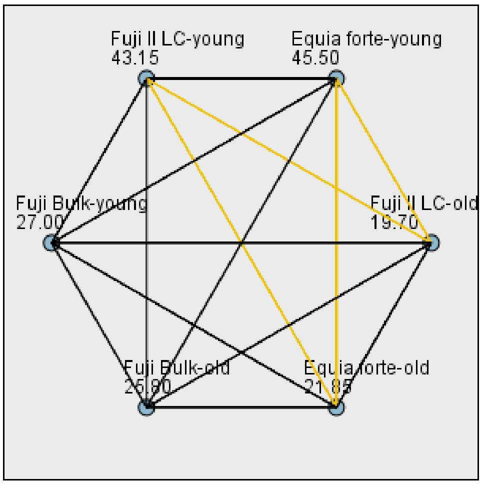
Each node shows the sample average rank of group.

Sample1-Sample2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj.Sig.
Equia forte-young-Fuji Bulk-young	.000	4.350	.000	1.000	1.000
Equia forte-young-Equia forte-old	.000	4.350	.000	1.000	1.000
Equia forte-young-Fuji Bulk-old	.000	4.350	.000	1.000	1.000
Equia forte-young-Fuji II LC-old	-2.900	4.350	-.667	.505	1.000
Equia forte-young-Fuji II LC-young	18.100	4.350	4.161	.000	.000
Fuji Bulk-young-Equia forte-old	.000	4.350	.000	1.000	1.000
Fuji Bulk-young-Fuji Bulk-old	.000	4.350	.000	1.000	1.000
Fuji Bulk-young-Fuji II LC-old	-2.900	4.350	-.667	.505	1.000
Fuji Bulk-young-Fuji II LC-young	18.100	4.350	4.161	.000	.000
Equia forte-old-Fuji Bulk-old	.000	4.350	.000	1.000	1.000
Equia forte-old-Fuji II LC-old	2.900	4.350	.667	.505	1.000
Equia forte-old-Fuji II LC-young	18.100	4.350	4.161	.000	.000
Fuji Bulk-old-Fuji II LC-old	2.900	4.350	.667	.505	1.000
Fuji Bulk-old-Fuji II LC-young	18.100	4.350	4.161	.000	.000
Fuji II LC-old-Fuji II LC-young	15.200	4.350	3.494	.000	.007

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same.
Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

D. Gingival margin

Pairwise Comparisons of group



Each node shows the sample average rank of group.

Sample1-Sample2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj.Sig.
Fuji II LC-old-Equia forte-old	-2.150	7.198	-.299	.765	1.000
Fuji II LC-old-Fuji Bulk-old	-6.100	7.198	-.847	.397	1.000
Fuji II LC-old-Fuji Bulk-young	7.300	7.198	1.014	.311	1.000
Fuji II LC-old-Fuji II LC-young	23.450	7.198	3.258	.001	.017
Fuji II LC-old-Equia forte-young	25.800	7.198	3.584	.000	.005
Equia forte-old-Fuji Bulk-old	-3.950	7.198	-.549	.583	1.000
Equia forte-old-Fuji Bulk-young	5.150	7.198	.715	.474	1.000
Equia forte-old-Fuji II LC-young	21.300	7.198	2.959	.003	.046
Equia forte-old-Equia forte-young	23.650	7.198	3.286	.001	.015
Fuji Bulk-old-Fuji Bulk-young	1.200	7.198	.167	.868	1.000
Fuji Bulk-old-Fuji II LC-young	17.350	7.198	2.410	.016	.239
Fuji Bulk-old-Equia forte-young	19.700	7.198	2.737	.006	.093
Fuji Bulk-young-Fuji II LC-young	16.150	7.198	2.244	.025	.373
Fuji Bulk-young-Equia forte-young	18.500	7.198	2.570	.010	.153
Fuji II LC-young-Equia forte-young	-2.350	7.198	-.326	.744	1.000

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same.
Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

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