DENTIN MICROSHEAR BOND STRENGTH OF VARIOUS LUTING AGENTS TO ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMICS

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A Thesis Submitted in Partial Fulfillment of the Requirements

for the Degree of Master of Science in Esthetic Restorative and Implant Dentistry

Common Course

Faculty of Dentistry

Chulalongkorn University

Academic Year 2018

การศึกษาแรงยึดติดแบบเฉือนระดับจุลภาคของเนื้อฟันด้วยสารยึดติดระบบต่าง ๆ ที่มีต่อวัสดุลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนีย

นายณัฐพงษ์ อิทธิพงศธร

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม ไม่สังกัดภาควิชา/เทียบเท่า
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2561
ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

Thesis Title	DENTIN MICROSHEAR BOND STRENGTH OF VARIOUS LUTING AGENTS TO ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMICS
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ณัฐพงษ์ อิทธิพงศธร : การศึกษาแรงยึดติดแบบเฉือนระดับจุลภาคของเนื้อฟันด้วยสารยึดติดระบบ ต่าง ๆ ที่มีต่อวัสดุลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนีย. (DENTIN MICROSHEAR BOND STRENGTH OF VARIOUS LUTING AGENTS TO ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMICS) อ.ที่ปรึกษาหลัก : รศ. ทญ. ดร.ศิริวิมล ศรีสวัสดิ์

วัตถุประสงค์: เพื่อทดสอบแรงยึดติดแบบเฉือนระดับจุลภาคของสารยึดติดระบบเอชแอนด์ริ้น ยูนิเวอ ซัล และเซลฟ์แอดฮีซีฟ ที่ใช้ยึดลิเทียมซิลิเกตที่เสริมความแข็งแรงด้วยเซอร์โคเนีย (ZLS) กับเนื้อฟัน

วิธีการศึกษา: ก้อนไวต้า สุพรินิตี้ [Vita Suprinity® (VS, Vita Zahnfabrik)] และเซลทรา ดูโอ [Celtra® Duo (CD, Dentsply Sirona)] ถูกตัดเป็นแท่งขนาด 1×1×3 ลูกบาศก์มิลลิเมตร จำนวน 36 และ 72 แท่ง ตามลำดับ โดย VS จะเข้าสู่กระบวนการตกผลึกทั้งหมด ขณะที่ครึ่งหนึ่งของ CD จะได้รับการเผาเพิ่มเติม และถูกเรียกว่า ไฟร์เซลทรา ดูโอ (fired-Celtra® Duo, FCD) และ CD ที่เหลือจะถูกเรียกว่า อันไฟร์เซลทรา ดูโอ (unfired-Celtra® Duo, UCD) จากนั้นแต่ละแท่งของ ZLS จะถูกยึดกับเนื้อฟันของฟันกรามเล็กมนุษย์ ด้วยสาร ยึดติด สกอตช์บอนด์ มัลติ-เพอโพส [Scotchbond™ Multi-purpose (SM, 3M ESPE]] ซิงเกิ้ล บอนด์ ยูนิเวอ ซัล [(Single Bond Universal (SU, 3M ESPE)] ร่วมกับ รีไลน์เอกซ์ อัลทิเมต [RelyX™ Ultimate (RXU, 3M ESPE)] และรีไลน์เอกซ์ ยูนิเซ็ม [RelyX™ Unicem (U2, 3M ESPE)] จำนวน 12 ชิ้นงานต่อกลุ่ม รวม 9 กลุ่ม การทดลอง จากนั้นทุกชิ้นงานจะถูกแช่ในน้ำที่อุณหภูมิ 37 องศาเซลเซียสเป็นเวลา 24 ชั่วโมง ก่อนนำไปวัด ค่าแรงยึดติดแบบเฉือนระดับจุลภาค และนำไปวิเคราะห์ทางสถิติด้วยการวิเคราะห์ความแปรปรวน 2 ทางและทูกี้ โพส-ฮอค เทสที่ระดับนัยสำคัญ .05 รวมถึงดูลักษณะของการแตกภายใต้กล้องจุลทรรศน์ที่กำลังขยาย 40 เท่า

ผลการศึกษา: การวิเคราะห์ความแปรปรวน 2 ทาง พบว่าชนิดของ ZLS ไม่มีผลต่อค่าแรงยึดติดแบบ เฉือนระดับจุลภาค (P=.699) ในขณะที่ชนิดของสารยึดติด และการมีปฏิสัมพันธ์ระหว่างกันของทั้ง 2 ปัจจัย มีผล ต่อค่าแรงยึดติดแบบเฉือนระดับจุลภาคอย่างมีนัยสำคัญ (P<.001 และ .002 ตามลำดับ) นอกจากนี้ทูกี้ โพส-ฮอค เทส พบว่า U2 ให้ค่าแรงยึดติดแบบเฉือนระดับจุลภาคน้อยกว่าสารยึดติดที่เหลืออีก 2 ระบบอย่างมีนัยสำคัญ ยกเว้นกลุ่ม UCDU2 ที่ไม่แตกต่างกับ UCDSU (P=.478)

สรุป: สารยึดติดระบบเอชแอนด์ริ้น และยูนิเวอซัล เหมาะสมต่อการใช้ยึด ZLS เข้ากับเนื้อฟัน

สาขาวิชา	ทันตกรรมบูรณะเพื่อความสวยงาม และทันตกรรมรากเทียม	ลายมือชื่อนิสิต ....
ปีการศึกษา	2561	ลายมือชื่อ อ.ที่ปรึกษาหลัก ....

5875811132 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY

KEYWORD: microshear bond strength; self-adhesive resin cement; three-step etch-and-rinse adhesive; universal adhesive; zirconia-reinforced lithium silicate ceramics

Natthapong Itthipongsatorn : DENTIN MICROSHEAR BOND STRENGTH OF VARIOUS LUTING AGENTS TO ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMICS. Advisor: Assoc. Prof. SIRIVIMOL SRISAWASDI, Ph.D.

Statement of problem. Performance of adhesive and resin luting cements used to bond zirconia-reinforced lithium silicate ceramics (ZLS) to dentin has not been well established. Purpose. To examine microshear bond strength (µSBS) of etch-and-rinse adhesive system, universal adhesive and self-adhesive resin cement that were used to bond ZLS to dentin. Material and Methods. Vita Suprinity® (VS, Vita Zahnfabrik) and Celtra® Duo (CD, Dentsply Sirona) blocks were sectioned into 36 and 72 microbars ($1 \times 1 \times 3$ mm^3) respectively. All VS were crystallized, while half of CD were additionally fired and defined as fired-Celtra® Duo (FCD). The others were defined as unfired-Celtra® Duo (UCD). Each microbar was cemented to each flat occlusal dentin surface of human premolar, following the adhesive luting systems: Scotchbond™ Multi-purpose (SM, 3M ESPE) and Single Bond Universal (SU, 3M ESPE) combined with RelyX™ Ultimate (RXU, 3M ESPE), and RelyX™ Unicem (U2, 3M ESPE) (n=12 per group). 24-hour µSBS was then determined, and data were analyzed using two-way ANOVA and a Tukey post-hoc test (α = .05). Failure modes were analyzed under a stereomicroscope at 40×. Results. Two-way ANOVA revealed that type of ZLS had no influence on µSBS (P=.699). In contrast, kind of adhesive luting cements and their interaction had a statistically significant effect on µSBS (P<.001 and .002 respectively). According to Tukey post-hoc test, U2 had a statistically significant lower mean µSBS regardless of the type ZLS, compared with SM and SU, while UCDU2 did not have a statistically significant difference in µSBS from UCDSU (P=.478).

Conclusions. Etch-and-rinse and universal adhesive resin luting systems may be suitable for cementation of ZLS to dentin.

Field of Study:	Esthetic Restorative and Implant Dentistry	Student's Signature	....
Academic Year:	2018	Advisor's Signature	....

ACKNOWLEDGEMENTS

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CHAPTER I INTRODUCTION

Rationale and Significance of the Problem

Today, all-ceramic restorations, such as veneers, crowns, inlays and onlays,

are widely used in an attempt to overcome the esthetic limitations of metal-ceramic

restorations. Recently, computer-aided-designing and computer-aided-manufacturing

(CAD/CAM) technology has become rapidly popular as an alternative to traditional

manufacturing processes, in combination with advances in dental ceramic materials

and adhesive technology, which provide more conservative and simplified restorative

procedures with sufficient physical properties to increase the longevity of restorations

(1, 2).

Currently, a novel material, zirconia-reinforced lithium silicate ceramic (ZLS),

has been launched and claimed that 10% by weight zirconia can reinforce the

material, thereby avoiding crack propagation (3). Vita Suprinity® (VS, Vita Zahnfabrik,

Germany) is a precrystallized ZLS. On the contrary, Celtra® Duo (CD, Dentsply Sirona,

Germany) being an already crystallized ceramic, can be delivered directly after

finishing and polishing. The milled restoration (unfired-Celtra® Duo, UCD) has a

flexural strength of 210 MPa. However, an additional firing (fired-Celtra® Duo, FCD)

increased the material's flexural strength to 370 MPa (4).

A new family of adhesive system, called a universal or multi-mode adhesive,

has been invented and claimed to use for direct and indirect restorations (5). For

versatility bonding with different substrates in adhesive cementation, some universal

adhesives contain silane and a specific functional monomer named 10-

methacryloyloxydecyl dihydrogen phosphate (10-MDP), which contribute to luting of

metal alloys, zirconia, glass ceramics, resin composite and other ceramics (6).

Moreover, for the purpose of simplicity of application during cementation

procedure and prevention of the collapse in demineralized dentin using phosphoric

acid conditioning, self-adhesive resin cement has been introduced (7, 8). The

multifunctional phosphoric acid methacrylates in organic matrix of this cement class

can demineralize and subsequently infiltrate to the tooth structure resulting in

micromechanical interlocking without additional tooth surface pre-treatment (9).

There have been studies investigating the efficacy of three-step etch-and-

rinse adhesive, universal adhesive and self-adhesive resin luting cement which were

used to bond zirconia or glass ceramic to dentin (10-12). However, the effectiveness

of these cementation systems on ZLS has not been thoroughly reported.

Research Questions

1. What are the efficacies of the three-step etch-and-rinse adhesive, universal

adhesive and self-adhesive resin luting cement in the viewpoint of microshear bond

strength (µSBS) on ZLS bonded to dentin?

2. Do the different forms of ZLS equally achieve µSBS when they are bonded

to dentin using various resin luting cements?

Research Objectives

1. To examine the performance of three adhesive luting systems used to

bond different forms of ZLS to dentin in the aspect of µSBS.

2. To investigate that the different forms of ZLS can affect µSBS or not when

they were bonded to dentin using various resin luting cements.

Hypotheses

Null hypotheses

1. There was no significant difference in µSBS of different resin luting systems used to cement ZLS to dentin.

2. There was no significant difference in µSBS of different forms of ZLS bonded to dentin using resin luting cements.

Alternative Hypothesis

1. There was at least one significant difference in µSBS of the three-step etch-and-rinse adhesive, universal adhesive and self-adhesive resin luting cement used to bond ZLS to dentin.

2. There was at least one significant difference in µSBS of different forms of ZLS bonded to dentin using various resin luting cements.

Conceptual Framework
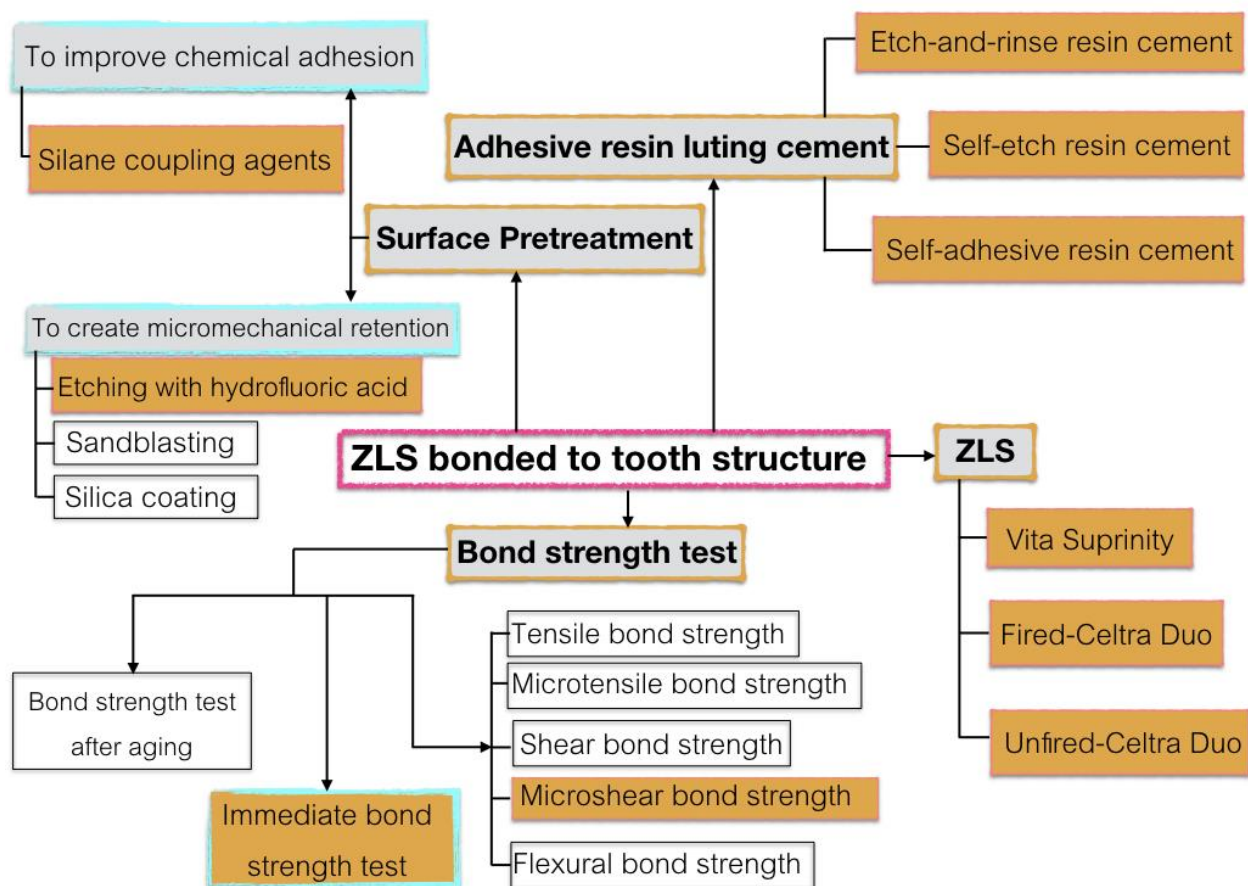
Figure 1. Diagram of Conceptual Framework

Keywords

microshear bond strength, self-adhesive resin cement, three-step etch-and-rinse adhesive, universal adhesive, zirconia-reinforced lithium silicate ceramics

Expected Benefit of the Study

Outcome of this present study may provide clinician useful information

regarding selection both adhesive resin luting agent for cementation and form of

zirconia-reinforced lithium silicate ceramic bonded to tooth structure.

CHAPTER II REVIEW OF THE LITERATURES

The literatures in these following topics have been reviews.

Dental ceramics

Surface treatment

Adhesive systems

Luting cements

Bond strength test

Dental ceramics

The traditional porcelain such as feldspatic, alumina-based ceramics has the

disadvantages of brittleness, crack propagation, low tensile strength, wear resistance,

and marginal inaccuracy (13). So the combination of predictable strength and

acceptable esthetics has continued to make traditional metal-ceramic restorations

popular (14). However, patient demand in high esthetics restoration has driven the

development of all ceramic for use with veneer, inlays, onlays, crowns, fix partial

denture prosthesis, and implant-supported restorations (15).

The improvement of all ceramic material systems which can offer good

esthetics and simplify fabrication procedures have been introduced (16). Heat-

pressing is a process which can solve the problems both in homogeneity and

porosity occurring during ceramming (17, 18). The first heat-press ceramic material,

IPS Empress (Ivoclar-Vivadent, Schaan, Liechtenstein), is a type of leucite-reinforced

glass ceramic and has a flexural strength of 182 megapascal (MPa) (18). The material

is designed for the fabrication of inlays, onlays and veneers (17). IPS Empress 2

(Ivoclar-Vivadent, Schaan, Liechtenstein) is a lithium disilicate ($2SiO_2$ -Li_2O) glass

ceramic has improved flexural strength by a factor of 3 over IPS Empress. The

fracture strength of this material was found to be 350 MPa (19, 20). Therefore, it has

been indicated for both anterior three-unit fixed partial dentures and restorations in

the posterior region extending to the second premolar (19, 20).

In 2005, an improved press ceramic material compared to IPS Empress 2

called IPS e.max Press (Ivoclar-Vivadent Schaan, Liechtenstein) was introduced. The

IPS e.max Press material consists of a lithium disilicate pressed glass ceramic. The

chemical basis of the material is the same as the chemical basis of IPS Empress 2

($2SiO_2$ -Li_2O), but its physical properties and translucency are improved by a different

firing process (21). So the framework can be veneered with a new type of sintered

fluoroapatite porcelain.

To create the restoration, the material is injected in a mold of coating

obtained by loss wax technic under high temperature and pressure. This system

reduced the problem of contraction during the burn of ceramic, common found in

feldspathic materials due to the high pressure of injection in high temperature mold.

Because of that, dimensional variation only occurs during the cooling, and it can be

controlled by adequate expansion of the investment material (22).

A characteristic of lithium disilicate glass ceramic is the quality to be acid

sensitive, in other words, it suffers morphological changes in front of acid treatment

with hydrofluoric acid in different concentrations. This phenomenon occurs due to

the micro-structural characteristics of the material. The main crystalline phase

consists of elongated lithium disilicate crystals. The second crystalline phase consists

of lithium orthophosphate. A glass matrix surrounds both crystalline phases.

Hydrofluoric acid in 10% of concentration is capable to remove the glass matrix and

the lithium orthophosphate crystalline phase exposing only lithium disilicate crystals

creating an irregular surface fundamental to a good adhesion (22).

Ceramics tend to be rigid and brittle, while resin composites are more

compliant, soft and experience high wear. The ideal goal for restorative dentistry

would be to replace lost tooth substance by a restorative material with tooth like

structure and matching physical properties. Toward this objective a novel material

that attempts to emulate the properties of natural teeth in its structure and physical

properties was developed and named polymer-infiltrated-ceramic-network material

(PICN). The goal is to achieve a material with enhanced mechanical characteristics,

compared to conventional restorative materials like ceramics and resin composites

(23).

A new ceramic material for dental restorations has been lately introduced.

ZLS is based on a lithium-metasilicate (Li_2SiO_3) glass ceramic, reinforced with about

10% of zirconium dioxide (ZrO_2) and crystallized by diphosphorus pentoxide (P_2O_5) as

nucleation agent of lithium-metasilicate. After final crystallization process, this

material leads to the formation of fine grained microstructure ($Li_2O-ZrO_2-SiO_2$),

resulting in four times smaller lithium silicate crystals. ZLS belongs to a new

generation of materials intended for CAD/CAM use. It combines the positive

mechanical characteristics of the zirconia with the aesthetic appearance of glass-

ceramic. Unlike the zirconia restorations, according to the manufacturer's

instructions, ZLS could be etched and cemented with adhesive systems (24, 25).

Currently, there are two brands of ZLS in the market. Suprinity (Vita

Zahnfabrik) and Celtra Duo (Dentsply Sirona) were launched for chairside as well as

lab site processing. ZLS Vita Suprinity® is a precrystallized ceramic material.

Accordingly, the CAM processing is comparable with lithium disilicate ceramic

materials in the aspect of crystallization firing after milling to achieve the final

density. However, the ZLS Celtra Duo (Dentsply Sirona) is a finally crystallized

ceramic. It is especially suitable for chairside application, as the final restoration is

available after a milling time of only 10 to 22 minutes. The milled restorations have

a flexural strength of 210 MPa. An additional stain and glaze firing increased the

material's flexural strength to 370 MPa (26). Thus, the final crystallized ZLS offered

combination of short processing times and high stability (27).

Guazzato et al. evaluated the fracture toughness of zirconia-based dental

materials found values ranging from 4.8±0.5 MPa m$^{1/2}$ to 7.4±0.6 MPa m$^{1/2}$ and

hardness values between 11±0.9 GPa and 13±0.3 GPa (28). While Traini et al. found

that fully crystalized state ZLS presented values of fracture toughness equal to

4.7±0.8 MPa m$^{1/2}$ and Vickers hardness equal to 7.6±0.7 GPa (24).

On the other hand, for pressable lithium-disilicate ceramics it was reported

fracture toughness of 1.13±0.02 MPa m$^{1/2}$, and hardness value of 5.38±0.28 GPa (29),

while the CAD/CAM lithium disilicate ceramic was reported to have values of fracture

toughness ranging from 2.27±0.16 MPa m$^{1/2}$ to 2.37±0.28 MPa m$^{1/2}$ and values of

Vickers hardness of 6.02±0.2 GPa (30). Otherwise, the fracture toughness of the

dental enamel was reported as ranging between 0.7±0.2 MPa m$^{1/2}$ and 1.77±0.2 MPa

m$^{1/2}$ while, the hardness values showed 4.7±0.3 GPa (31).

The collected data prove that ZLS exhibits superior mechanical properties

compared to lithium-disilicate glass ceramics and comparable to those of existing

zirconia-based ceramics. The comparison with enamel also showed that the material

is suitable for oral function, even in the posterior regions where the masticatory

forces ranged between 600 and 900 N (32).

Surface treatment

The other important requirement for success of ceramic restorations is the

achievement of adequate adhesion between the ceramic and tooth substrate. The

selection of an appropriate surface treatment and adhesive system plays an

important role in clinical success.

The extension of the etching time indicated by the manufacturer for an acid-

sensitive ceramic was analyzed by Zogheib and others, who concluded that lithium

disilicate ceramic required more than 60 seconds of hydrofluoric acid etching for the

creation of effective microretention (33). However, Menees and others found that

hydrofluoric acid etching for 20 seconds in concentrations varying from 5% and 9.5%

was enough to remove the glass matrix. Despite extensive removal for 120 seconds,

it was clear that the resulting etch pattern for these conditions was uniform and was

not enough to affect the bending strength of the lithium disilicate ceramic(34).

Traini et al. (24) found that zirconia reinforced lithium silicate ceramic, Vita

Suprinity® (Vita Zahnfabrik) which, was treated by hydrofluoric acid gel at 4.9% for 20

s showed the best result with preservation of microstructure. While increasing the

etching time to 40 s, the surface degradation of ZLS Vita Suprinity® (Vita Zahnfabrik)

microstructure appeared evident. At the same time, the increase of hydrofluoric acid

concentration to 9.5% either for 20 s and 40 s produced a progressive surface

degradation with a large destructuring of the ZLS material.

According to the manufacturer, ZLS was pre-treated with hydrofluoric and

silane coupling agent in order to improve bonding performance (35). The hydrofluoric

acid attacked the glassy phase of the ceramics, dissolving the surface in a few

micrometers depth, creating micro porosities which was required for

micromechanical retention, removing surface impurities, such as oxides and other

inorganic and organic debris, making the surface readily wettable for the

subsequently applied silane coupling agent and resin cement (36). This porous

surface not only provided more surface area for resin bonding, but also exposed and

generated hydroxyl groups on the ceramic surface that were responsible for chemical

bonding via silane coupling agents (26, 37).

After etching, the ceramic surface is treated with an activated silane coupling

agent to improve chemical adhesion (38, 39) and to provide reliable and durable

chemical bonding with adhesive resin cement (37). The specific silane used in

dentistry is 3-methacryloxypropyltri-methoxysilane. Silane coupling agent is hybrid

inorganic-organo-functional trialkoxysilane monomers and capable of unifying organic

and inorganic materials. In general, silane has non-hydrolysable groups (such as

methacrylate) and hydrolysable groups (such as ethoxy), which is why they are

chemically bifunctional (26). When reactive silane is applied over the etched ceramic

surface, the hydrolysable alkoxy groups react with exposed hydroxyl groups, and

non-hydrolyzable organic groups polymerize with unset resin cement (40). Hence, for

reliable and durable chemical bonding, it is necessary that the ceramic surface

should be conditioned before resin luting cements, including self-adhesive resin

cements, are used (41). However, such chemical reactions are not applicable for non-

silica containing zirconia-based ceramics.

However, the use of hydrofluoric acid requires careful attention due to its

potential risk for the degradation of organic matter (39). For this reason, other options

have been investigated for ceramic surface treatment, including air abrasion with

silica-coated aluminum oxide particles. During silica coating, the high energy of the

shock resulting from the aluminum oxide particles was responsible for the fusion of

these silica particles to the ceramic surface, making it chemically reactive to the resin

cement through the silane agent and also increasing bond strength to ceramics (42).

Conversely, silica coating treatment is controversial, because some authors

reported a decrease in mechanical strength of the material and the induction of

crack propagation (43, 44) whereas others have shown no deleterious effect on long-

term mechanical behavior (44, 45).

Valandro et al. studied about surface treatment by using 10% hydrofluoric

acid for 20 seconds and 40 seconds and using CoJet™ sandblasting apply to the ZLS

before cementation on composite blocks. They found that the lower bond strength

was obtained with acid etching for 40 seconds. It was explained by the removal of a

greater quantity of glass matrix and exposure of lithium silicate crystals and particles

of zirconia creating a surface with lower wettability. Moreover, surface modification

by the use of silica coating did not guarantee a stable bonding between ZLS and

resin cement in the long term (3).

Adhesive systems

Adhesive bonding procedure in dentistry is a process depending on various

factors, such as what kind of substrates (46), type of adhesive agents (47), humidity of

the substrates and surrounding condition (48), and practitioner's ability in performing

the bonding procedure (49). In the dental substrates aspect, adhesive procedures are

usually performed to bond to enamel and dentin. Enamel is a highly-mineralized

substrate composed of almost 100 wt% of hydroxyapatite crystals, which do not

need a wet surface during the procedures for proper bonding. It requires the

application of a hydrophobic material only (46). Consequently, bonding to enamel

has been demonstrated to be easy and durable (47). Whereas, dentin is a

complicated substrate constituted of both mineral and organic phases as well as

water. Therefore, bonding to dentin is challenging because an ideal moisture

condition has to be maintained to avoid collapse of the collagen matrix and cause

proper adhesive infiltration of the adhesive into the demineralized substrate (46).

Dental adhesive systems are generally characterized by the application of

three different substances, which fill three dissimilar clinical steps: etching, priming,

and bonding. Etching is the application of an acid agent to demineralize the dental

substrate surface; priming is the preparation of the etched surface before application

of the adhesive. Bonding is the application of the hydrophobic resin bond adhesive

over enamel and dentin.

According to the type of adhesive agents, available adhesives could be

classified by the bonding strategies, as etch-and-rinse (total etch) systems and self-

etching systems (47). The etch-and-rinse systems necessitate phosphoric acid etching

and rinsing of enamel and dentin prior to applying adhesives agents, whereas the

self-etching systems contain acid functional monomers which can condition both

enamel and dentin simultaneously, without rinsing. Dentin bonding mechanism is

based on the infiltration of resin monomers into the porosities created by removal of

mineral or inorganic material from the dental tissues. This exchange results in micro-

mechanical interlocking in the porosities formed. Successful dentin bonding could be

achieved through several routes. The etch-and-rinse technique is the conventional

three-step which use primer and adhesive separately or two-step which combines

primer and adhesive agent together. Differences in material composition and

adhesive application technique can affect many properties such as film thickness,

bond strength, radiopacity, adaptation and marginal seal (50-52). In this technique,

the tooth substrate is first etched with 30-40% phosphoric acid (H_3PO_4) and leaved it

for 15-30s depended on what kind of substrate. Then rinsed off. Following acid

etching, primer and adhesive agent is applied on the conditioned tooth surface either respectively or simultaneously. For dentin, the bonding mechanism of etch-and-rinse adhesives primarily depends on micro-mechanical retention of resin with the exposed collagen fibrils. For enamel, total etch technique is the most effective and reliable method for long-term clinical success (53).

In the self-etching approach, adhesives condition and prime dentin are applied at the same time, and no rinsing is required. In this procedure the clinical application time is shortened and technique sensitivity is significantly reduced. Self-etch adhesives can be categorized as mild and strong. Strong self-etch adhesives with functional monomers have low pH<1 and their bonding mechanism is reported to be similar to etch-and-rinse adhesives. Mild self-etch adhesives (pH≈2) selectively demineralize the dentin surface and are reported to form a shallow hybrid layer. Adhesion is ensured by chemical interaction between residual hydroxyapatite and functional monomers (54).

One of the most recent innovation in adhesive dentistry was the introduction of universal or multi-mode adhesive. These materials are simplified adhesives. They usually are containing all bonding components in single bottle. Universal adhesives may be applied either in etch-and-rinse or self-etching approach, according to manufacturers' claims. In addition, some universal adhesives may contain silane in their formulation, potentially eliminating the silanization step when bonding to glass ceramics or resin composites. Nevertheless, it is known that simplified materials are associated with lower bond strength in vitro study and poorer in vivo longevity of restorations. These findings are probably a result of the complex formulation of simplified adhesives and their high content of solvents, which may impair complete solvent volatilization and consequently lead to poorer adhesive polymerization (55, 56).

One of the components in some universal adhesives is the 10-MDP. First time, the patent of 10-MDP was owned by the Kuraray company for 10 years until 2011. After that, a lot of products have been incorporated 10-MDP into various bonding and luting agents, such as primers, adhesives, and resins-based luting systems. The

adhesion capability of these materials is due to this component which is the

hydrophilic phosphate monomer.

The dihydrogen phosphate group from the 10-MDP is responsible for priming

and bonding. It increases resin diffusion and adhesion by causing acidic

decalcification and forming strong ionic bonds either with calcium ions from

hydroxyapatite that forms calcium salts with low solubility, or amino groups of tooth

structure. These complex substances may be responsible for the good long-term

performance of MDP-containing adhesives (54, 57-59). While its long hydrophobic

carboxyl chain copolymerizes the resin monomers of the resin cement and provides

hydrolytic stability of acidic monomers (60).

The hydrophilicity of the ceramic is important in order to enable the universal

adhesive to spread across its entire surface and establish optimum adhesion. For

non-silica-based substrates, such as metal or zirconia, the hydrophilic phosphate

terminal end of 10-MDP interacts chemically with the oxides on the internal surface

of restorations (60). However, for silica-based indirect restorations such as feldspathic

porcelain, leucite-reinforced ceramic, or lithium disilicate glass ceramic, the reaction

between silane and 10-MDP promotes the bonding mechanism and improving

surface wettability. The free silanol groups form hydrogen bonds with the hydroxyl

groups of the indirect restoration. Then cross-linkages are formed between the

methacrylate groups of the resin cement with organofunctional groups from the

silane coupling agent, as well as between the siloxane bonds and the restoration

substrate (38). Due to, the versatility of the substrate application, 10-MDP-containing

adhesives may also be suitable for intraoral restoration repairs, since they could be a

practical alternative to bonding different fractured substrates at the same time (61).

Luting cements

Dental luting agents provide the essential functional link between a fixed

prosthesis and the supporting prepared tooth structure. In general, dental luting

cement's two main functions are to provide a seal and establish or increase

retention of the prosthesis to abutments and to maintain its integrity. To succeed in

both, an ideal material should fulfill specific biological, physio-mechanical, and

handling requirements (62).

Several studies have reported that high stresses can be imposed upon luting cements, most especially in the marginal areas (63), and laboratory fatigue studies have suggested that luting cement microfracture is the initial failure mode that enables the progression to catastrophic failure (64). Accordingly, luting agent failure can be predisposed by the initial setting contraction stresses generated by the cement's adhesion to the tooth structure; this is further amplified in a geometric configuration that provides few opportunities for stress relief by cement flow or cement flow and plastic deformation (65).

Presently, 5 categories of luting agents are commercially available for permanent cementation of fixed prostheses: zinc phosphate, polycarboxylate, glass ionomer, resin-modified glass ionomer (RMGI) andd resin cements. These different categories are largely physically and chemically unique, with no one luting agent being ideal for all situations (66).

Resin cements are composites composed of a resin matrix, for example, bis-GMA or urethane dimethacrylate, and a filler of fine inorganic particles. Resin luting

cements differ from restorative composites primarily in their lower filler content and

lower viscosity. Resin cements have not only excellent aesthetic shade matching

potential, but also better flexural and compressive strength compared with other

dental cements. In terms of shear and tensile bond strength, resin cements are

stronger than other types of cement; the adhesive nature of the resin cements

results in restorations with superior retention and fracture resistance. Moreover,

minimal microleakage and lower water solubility occur with adhesive cementation

(67).

The success of ceramic restorations depends on obtaining a strong, durable

bond between the resin cement and dentin/enamel (68). The magnitude of these

bonds is directly proportional to an adequate cement polymerization. Polymerization

is crucial for achieving optimal physical properties and satisfactory clinical

performance of resinous materials. Resin cements can be categorized according to

polymerization type: chemical-cured, light-cured, or dual-cured.

Chemical-cured resin cement, which is mostly used for metallic restoration, requires a long setting time and has an uncontrollable working time; it is cured evenly even in clinical situations that the light does not reach the cement material.

In contrast, light-cured cement presents easier removal of excess cement, and command setting requires no mixing, and therefore, the cement is more homogenous with reduced porosity. The lack of tertiary amines in the cement composition provides excellent color stability. However, the porcelain thickness could prevent complete photopolymerization (68).

About dual-cured resin cements, their polymerization is initiated by light and chemically and are therefore the materials of choice to lute indirect tooth-colored restorations with a thickness more than 3 mm (69). When these dual-cured resin cements are light polymerized, the highest conversion rate is reached (70), with a consequent increase in the physico-mechanical properties (71).

For cementation of all-ceramic restorations, multi-step systems were used, requiring etching, priming, bonding and the application of a composite cement. This

complicated procedure resulted in high technique sensitivity. As further

development, one or two bottle systems for dentin bonding were introduced by the

manufacturers. The application of a separate acid-etching step is unnecessary when

using self-etching resin luting agents. These materials have become popular for their

simplicity and because they require fewer procedural steps when compared with

previous systems that used separate acid conditioning and primer/adhesive steps

(67). Most recently the number of application steps was further reduced by the

development of self-adhesive resin cements. The benefit of these materials lies in

the ability to bond dentin without any type of pre-treatment (9).

The monomers in self-adhesive luting agents contain phosphorylated

methacrylates that have the ability to generate self-adhesion. Furthermore, the

presence of phosphoric acid groups within the material creates an acidic bonding

surface environment. The low pH environment that is created provides for

demineralization of the tooth surface, which, in turn, allows for subsequent

penetration of the resin cement into the demineralized bonding surface. Once the

resin cement polymerizes, micromechanical retention is achieved between the

cement and tooth (72).

A number of studies have evaluated the bond strength of self-adhesive resin

cements compared to conventional multi-step luting agents. The results showed

favorable bond strength behavior on dentin, while lower bond strengths were found

on enamel surfaces compared to those provided by multi-step luting agents (9, 73)

It was reported that the adhesive luting technique improved the fracture

resistance of glass ceramic crowns with lower strength values (e.g. feldspathic and

leucite-reinforced glass ceramics), while fracture loads of high-strength ceramics like

zirconia, alumina, lithium disilicate or ZLS were not significantly influenced by the

mode of cementation (4, 74, 75). In contrast, a study by Borges (2009) reported about

a significant increase in fracture load for different ceramic crowns (lithium disilicate,

leucite-reinforced, and glass-infiltrated alumina ceramics) when they were cemented

with a resin cement compared to a resin-modified glass-ionomer cement (76).

Bond strength test

The bond strength of ZLS bonded to tooth structure can be measured by a

great number of methods such as tensile bond strength test, flexural bond strength

test, or shear bond strength test.

Bond strength tests are the most frequently used tests to screen adhesives.

The rationale behind this testing method is that the stronger the adhesion between

tooth and biomaterial, the better it will resist stress imposed by resin polymerization

and oral function. Different bond strength tests have been developed (77). It is

important to note that a bond strength value cannot be considered as a material

property. Therefore, the absolute test values cannot be used to draw conclusions

from, or be compared with, data gathered in other studies. Only relative study

outcomes, in the sense of 'A is better than B', are a valid basis for further

interpretation of the results. Nevertheless, bond-strength testing can reveal valuable

clinical information, when gathered in a well-controlled design (55).

The bond strengths of restorative materials to dental hard tissues is usually

reported as the load at failure divided by the cross-sectional area of the bonded

interface (F/A). Strength values calculated in this way are referred to as the "nominal

strength" values, but this is valid only if the applied load is equally distributed

throughout the entire bonded interface. Therefore, a crucial factor in evaluation of

the usefulness of a specific bond strength test is a thorough awareness of the stress

patterns involved in bond failure (78).

Various methods are used to evaluate bond strength, including flexural,

tensile, shear, microtensile, and microshear tests (μSBS). The easiest to perform are

shear tests. According to the ISO/TS 11405 standard (2015), the load can be

distributed as in lap-shear or blunt-end shear bar or inter-facial (wire loop in shear).

There is a strong tendency to develop a bending moment in most shear tests (77).

Shear bond strength tests were performed in specimens with relatively large bonded

areas, usually 3-6 mm in diameter (~ 7-28 mm^2) (79), However, the validity of

expressing bond strength has been questioned due to the heterogeneity of the stress

distribution at the bonded interface, influence by variability in specimen geometry,

loading conditions and material properties. Thus, the microshear test is preferred and

is practical for testing the bond strength in case a small bonded surface can be

created. Furthermore, this test results in a more uniform stress distribution, resulting

in more reliable data and higher incidence of adhesive failure between the resin

adhesive and dentin interface compared with conventional shear tests (80).

Microshear test was used in the present study because the specimens were

prepared without trimming, thus reducing the formation of structural defects such as

microcracks, which might cause premature failure. Furthermore, the microshear test

was a better representation of the forces clinically experienced by a restoration (81,

82).

CHAPTER III MATERIALS AND METHODS

Research design

This study was an in vitro experimental study. The interventions of this study were kinds of adhesive agents following, three-step etch-and-rinse adhesive, universal adhesive and self-adhesive resin luting cement in bonding of ZLS (Vita Suprinity®, fired-Celtra® Duo and unfired-Celtra® Duo) to tooth structure. Dependent variable was the microshear bond strength measured in MPa, when the specimens was cracked or fractured.

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Research methodology
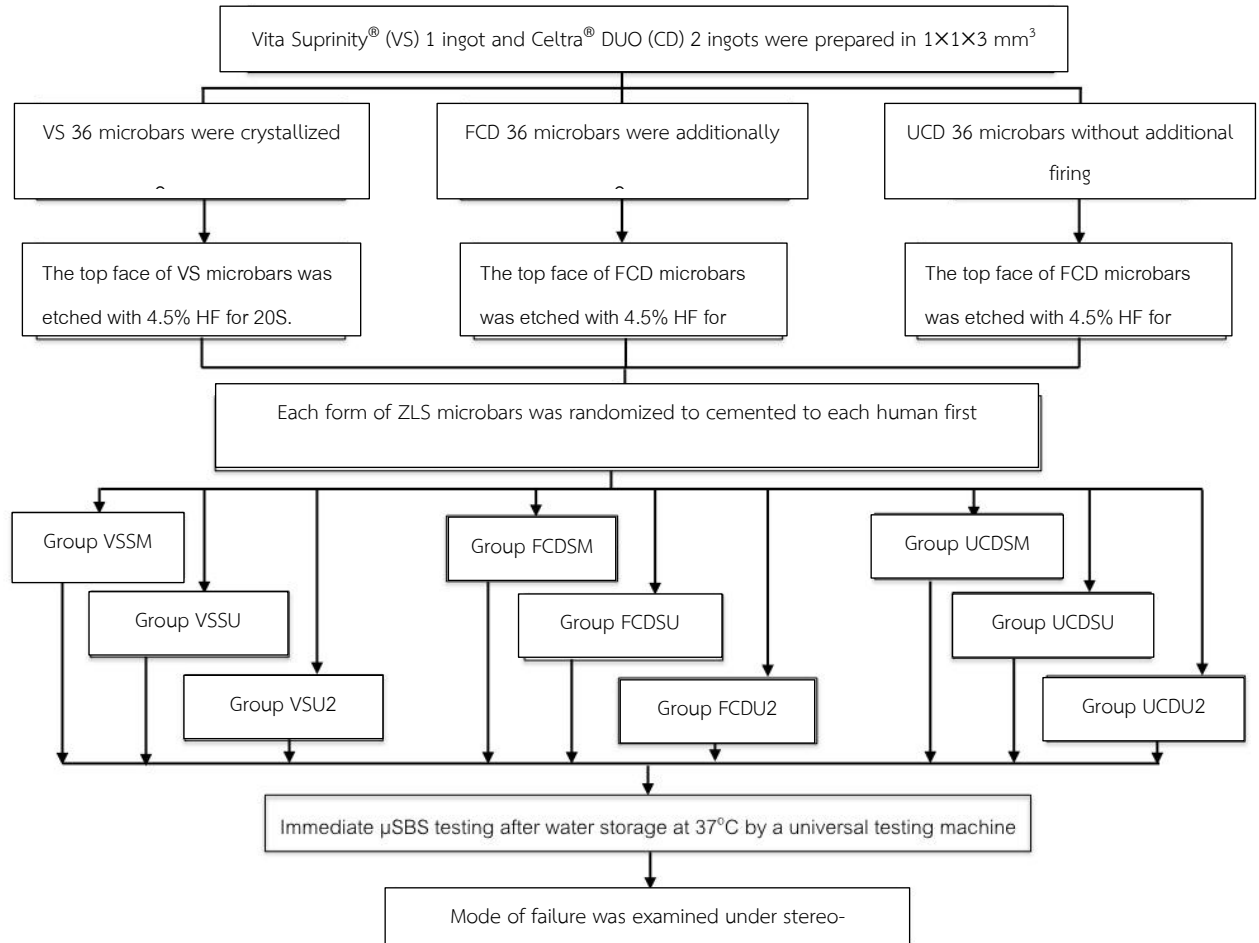
Figure 2. Diagram of study design

ZLS, zirconia-reinforced lithium silicate ceramics; VS, Vita Suprinity®; CD, Celtra® Duo;

FCD, Fired-Celtra® Duo; UCD, Unifired-Celtra® Duo; SM, Scotchbond™ Multi-purpose;

SU, Single Bond Universal; U2, RelyX™ Unicem; μSBS, microshear bond strength.

Sample size description

Sample size was calculated by using the formula for two independent groups

shown below;

$$n = \frac{(Z_{1-\frac{\alpha}{2}} + Z_{1-\beta})^2 [\sigma_1^2 + \frac{\sigma_2^2}{r}]}{(\mu_1 - \mu_2)^2}$$

$$r = \frac{n_2}{n_1}$$

n is sample size estimation (per group).

Z_α is the value of the standardized score cutting off α/2 proportion of each

tail of a standard normal distribution (for a two-tailed hypothesis test) (Z_α=1.96 for α

= 0.05).

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

= 0.84 for β = 0.2 = 80% power).

μ is mean of microshear bond strength in each group.

σ is standard deviation of microshear bond strength in each group.

The mean and standard deviation values for calculation were obtained from the results of previous published articles, which had experimental design using microshear bond strength testing of the adhesive luting agents to dentin (12, 83).

The highest number of specimen was calculated from values of microshear bond strength of Scotchbond[TM] Multipurpose adhesive system and Single Bond Universal adhesive system as shown in the equation below:

$$n_1 = \frac{(Z_{1-\frac{\alpha}{2}}+Z_{1-\beta})^2[\sigma_1^2+\frac{\sigma_2^2}{r}]}{(\mu_1-\mu_2)^2}$$

n_1 = (1.96 + 0.84)^2[9.92+5.90]/(24.9-24.82)^2

n_1 = (7.84)[15.82] /(0.0064)

n_1 = 19379.5

12 numbers of specimens in each group were selected for this study, due to the limitation of time, budget and the number of 19380 specimens was too high for this study. There were 9 experimental groups in this study so the total number of specimens was 108 specimens.

Table 1. Mean microshear bond strength and standard deviation from previous published articles and sample size calculation

Adhesive agents comparison		μ_1	σ_1	μ_2	σ_2	n
SM	SU	24.90	3.15	24.82	2.43	19379.56
SU	U2	24.82	2.43	20.18	2.01	3.62
U2	SM	20.18	2.01	24.90	3.15	4.91

SM, Scotchbond[TM] Multi-purpose; SU, Single Bond Universal; U2, RelyX[TM] Unicem; n, sample size estimation; μ, mean of microshear bond strength in each group; σ is standard deviation of microshear bond strength in each group.

Materials

Table 2. Datasheet of ceramics used

Material (manufacturer)	Composition	Crystallization/ additional firing process
Vita Suprinity® (Vita Zahnfabrick, Bad Säckingen)	SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments	Crystallization in furnace (Programat P700, Ivoclar Vivadent) at 840°C for 20 mins.
fired-Celtra® Duo (Dentsply Sirona)	SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments	Additionally fired in furnace (Programat P700, Ivoclar Vivadent) at 820°C for 8 mins.
unfired- Celtra® Duo (Dentsply Sirona)	SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments	No additional firing.

Table 3. Adhesive systems used in the study

Adhesive systems	Manufacturers/ Batch number	Composition
Scotchbond^{TM} Multi-purpose	3M ESPE/ N851438	Etchant: 35% phosphoric acid Primer: Polyalkenoic acid copolymer HEMA, water Adhesive: Bis-GMA, HEMA, tertiary amines, photo-initiator.
Single Bond Universal	3M ESPE/ 651936	10-MDP, Bis-GMA, phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane-treated silica
RelyX^{TM} Ultimate	3M ESPE/ 662726	Base paste: methacrylate monomers, radiopaque silanated fillers, initiator, stabilizer, rheological additives Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator, stabilizer, pigments, rheological additives, fluorescence dye, dark cure activator for Single Bond Universal
RelyX^{TM} Unicem	3M ESPE/ 652453	Powder: glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator

Apparatus

Table 4. Instrument used in this study

Instrument	Manufacturer
Low-Speed Cutting Machine (Isomet®1000)	Buehler Ltd., Lake Bluff, IL, USA
Ceramic furnace	Programat P700, Ivoclar Vivadent
Automatic temperature checking set (ATK2)	Ivoclar Vivadent, Schaan, Liechtenstein
Universal Testing Machine (EZ-S Shimadzu)	Shimadzu, Japan
Grinder-Polisher Machine (Automet® 250)	Buehler, USA
Durometer, ASTM D 2240 Type A	PTC Instrument, USA
Diamond Wafering Blade	Buehler, USA
Rotomix	3M ESPE, USA
LED Light-Curing System: Demi™ Plus	Kerr, USA
Radiometer: Model 100 Optilux	Kerr, USA
Stereomicroscope: ML 9300	MEIJI, Japan
Incubator: Contherm 160M	Contherm, New Zealand

Experimental groups and their details

Table 5. Description of groups according to variables

Group	Method
VSSM	VS + 4.5% HF (20 s) + ceramic primer + SM Adhesive + RXU
	Dentin + 35% H_3PO_4 + SM Primer/Adhesive
VSSU	VS + 4.5% HF (20 s) + SU + RXU
	Dentin + SU
VSU2	VS + 4.5% HF (20 s) + ceramic primer + U2
	Dentin without any adhesive agent application
FCDSM	FCD + 4.5% HF (30 s) + ceramic primer + SM Adhesive + RXU
	Dentin + 35% H_3PO_4 + SM Primer/Adhesive
FCDSU	FCD + 4.5% HF (30 s) + SU + RXU
	Dentin + SU
FCDU2	FCD + 4.5% HF (30 s) + ceramic primer + U2
	Dentin without any adhesive agent application
UCDSM	UCD + 4.5% HF (30 s) + ceramic primer + SM Adhesive + RXU
	Dentin + 35% H_3PO_4 + SM Primer/Adhesive
UCDSU	UCD + 4.5% HF (30 s) + SU + RXU
	Dentin + SU
UCDU2	UCD + 4.5% HF (30 s) + ceramic primer + U2
	Dentin without any adhesive agent application

VS, Vita Suprinity®; FCD, fired-Celtra® Duo; UCD, unfired-Celtra® Duo; SM, Scotchbond™ Multi-

purpose; SU, Single Bond Universal; U2, RelyX™ Unicem; RXU, RelyX™ Ultimate; HF, hydrofluoric

acid; H_3PO_4, phosphoric acid.

Definition of specimen groups

Group VSSM: The polished surface of Vita Suprinity® (VS, VITA Zahnfabrik)

microbars was etched with 4.5% hydrofluoric acid (HF, IPS ceramic etching; Ivoclar

Vivadent) for 20 s and rinsed with water for 60 s. Subsequently, etched ceramics

were cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-dried. After

that, RelyX™ Ceramic Primer (3M ESPE, USA) was applied at the etched surface, let

react for 60 s, and air dried for 5 s. Scotchbond™ Multi-purpose (SM) Adhesive (3M

ESPE, USA) was applied uniformly creating a thin coating for 15 s.

While, prepared dentin was applied with the 35% phosphoric acid etching gel

(Scotchbond™ Etchant, 3M ESPE, USA) and allowed to react for 15 s. Then rinsed

thoroughly with water for 15 s and blot dried with foam pellets. Scotchbond™ Multi-

purpose (SM) Primer (3M ESPE, USA) was applied at etched dentin with a light

scrubbing motion for 15 s. Then a gentle stream of air over the liquid until the

solvent had evaporated completely. SM Adhesive was applied uniformly creating a

thin coating for 15 s and light-curing for 20 s. The resin cement, RelyX™ Ultimate

(RXU, 3M ESPE, USA), was applied copiously to the ceramics using the auto-mix syringe.

Group VSSU: The polished surface of VS microbars was etched with 4.5% HF for 20 s and rinsed with water for 60 s. Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-dried. Single Bond Universal (SU, 3M ESPE, USA) was applied at both dentin which was moist and the etched surface for 20 s. Subsequently, a gentle stream of air over the liquid until the solvent had evaporated completely. Light-curing for 20 s to dentin. RXU was applied copiously to the ceramics.

Group VSU2: The polished surface of VS microbars was etched with 4.5% HF for 20 s and rinsed with water for 60 s. Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-dried. After that, RelyX™ Ceramic Primer was applied at the etched surface, let react for 60 s, and air dried for 5 s. RelyX™ Unicem (U2, 3M ESPE, USA) was dispensed directly on etched ceramics using the applicator.

Group FCDSM: The polished surface of fired-Celtra® Duo (FCD, Dentsply

Sirona) microbars was etched with 4.5% HF for 30 s and rinsed with water for 60 s.

Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-

dried. After that, RelyX™ Ceramic Primer was applied at the etched surface, let react

for 60 s, and air dried for 5 s. SM Adhesive was applied uniformly creating a thin

coating for 15 s.

While, prepared dentin was applied with the 35% phosphoric acid etching gel

and allowed to react for 15 s. Then rinsed thoroughly with water for 15 s and blot

dried. SM Primer was applied at etched dentin with a light scrubbing motion for 15

s. Then a gentle stream of air over the liquid until the solvent had evaporated

completely. SM Adhesive was applied uniformly creating a thin coating for 15 s and

light-curing for 20 s. RXU was applied copiously to the ceramics.

Group FCDSU: The polished surface of FCD microbars was etched with 4.5%

HF for 30 s and rinsed with water for 60 s. Subsequently, cleaned in the ultrasonic

bath with 98 % alcohol for 3 mins and air-dried. SU was applied at both dentin which

was moist and the etched surface for 20 s. Then a gentle stream of air over the

liquid until the solvent had evaporated completely. Light-curing for 20 s to dentin.

RXU was applied copiously to the ceramics.

Group FCDU2: The polished surface of FCD microbars was etched with 4.5%

HF for 30 s and rinsed with water for 60 s. Subsequently, cleaned in the ultrasonic

bath with 98 % alcohol for 3 mins and air-dried. After that, RelyX^{TM} Ceramic Primer

was applied at the etched surface, let react for 60 s, and air dried for 5 s. U2 was

applied copiously to the etched ceramics.

Group UCDSM: The polished surface of unfired-Celtra$^{®}$ Duo (UCD, Dentsply

Sirona) microbars was etched with 4.5% HF for 30 s and rinsed with water for 60 s.

Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-

dried. After that, RelyX^{TM} Ceramic Primer was applied at the etched surface, let react

for 60 s, and air dried for 5 s. SM Adhesive was applied uniformly creating a thin

coating for 15 s.

While, prepared dentin was applied with the 35% phosphoric acid etching gel and allowed to react for 15 s. Then rinsed thoroughly with water for 15 s and blot dry. SM Primer was applied at etched dentin with a light scrubbing motion for 15 s. Then a gentle stream of air over the liquid until it the solvent had evaporated completely. SM Adhesive was applied uniformly creating a thin coating for 15 s and light-curing for 20 s. RXU was applied copiously to the ceramics.

Group UCDSU: The polished surface of UCD microbars was etched with 4.5% HF for 30 s and rinsed with water for 60 s. Subsequently cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-dried. SU was applied at both dentin which was moist and the etched surface for 20 s. A gentle stream of air over the liquid until the solvent had evaporated completely. Light-curing for 20 s to dentin. RXU was applied copiously to the ceramics.

Group UCDU2: The polished surface of UCD microbars was etched with 4.5% HF for 30 s and rinsed with water for 60 s. Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins and air-dried. After that, RelyX™ Ceramic Primer

was applied at the etched surface, let react for 60 s, and air dried for 5 s. U2 was

applied copiously to the etched ceramics.

Possible Impediments and Solutions

In order to control the quality of bonding technique, one researcher

performed the whole procedure as listed cutting specimen, bonding procedure,

microshear bond strength test.

Methods

Tooth Selection

A total of 108 human first premolars, extracted for orthodontic purposes and

stored in 0.1% thymol solution at 4°C no longer than 2 months after extraction, were

selected (84). The teeth were analyzed using a stereomicroscope (ML 9300 MEIJI) at

4× magnification using the following selection criteria: no caries or previous

restorations, no cracks, and the presence of completely formed apexes. After the

selection process, residual soft tissue was removed by hand scaling.

Tooth preparation

Then, each tooth was embedded in a polyvinyl chloride tube, 2.2 cm in diameter and 2.2 cm in height, leaving the cemento-enamel junction at the top surface of acrylic resin base (Trey Resin II, Shofu). 2.0 mm thick occlusal portion underneath the central pit of all teeth was removed by means of a water-cooled precision diamond saw (Isomet 1000 Precision Saw, Buehler) to expose flat deep dentin surface. In the case of pulp exposure being detected, the tooth would be rejected.

Deep dentin surface then underwent grinding using a 600-grit silicon carbide paper at 100 rpm for 30 s to produce standard smear layer, which was comparable to bur-cut dentin surface (85-87). The grit silicon carbide paper was changed after grinding of 10 dentin specimens. Cementation area at the center of dentin specimen was defined and isolated to a 1×1 mm^2 by means of perforated Teflon tape. After that, all teeth were randomly divided into 9 groups (n=12 per group).

Figure 3. Grinder-Polisher Machine (AutoMet® 250)
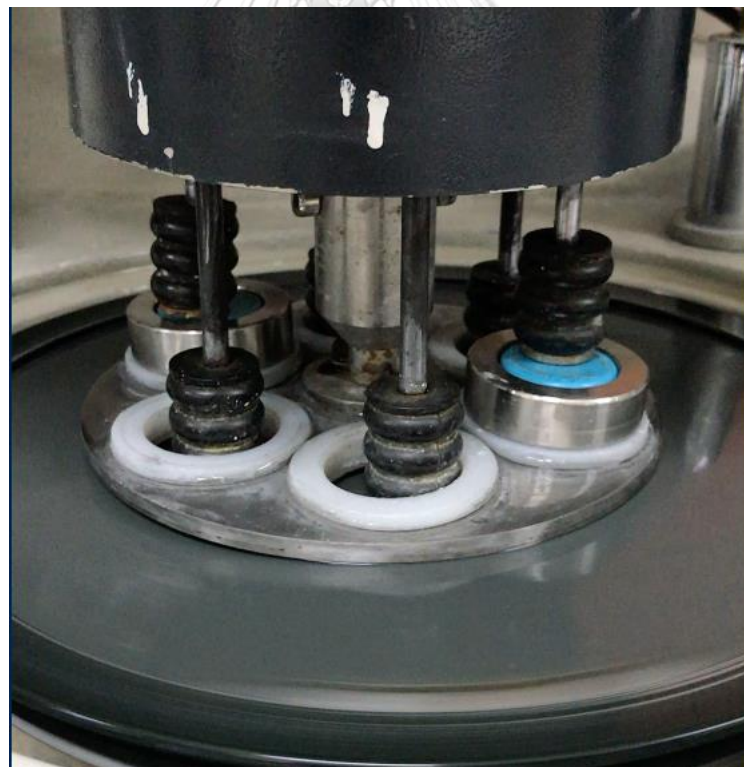
Figure 4. Deep dentin surface underwent grinding using a 600-grit silicon carbide
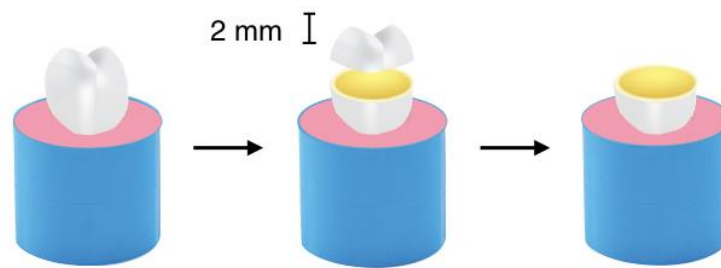
Figure 5. Preparation of dentin specimen
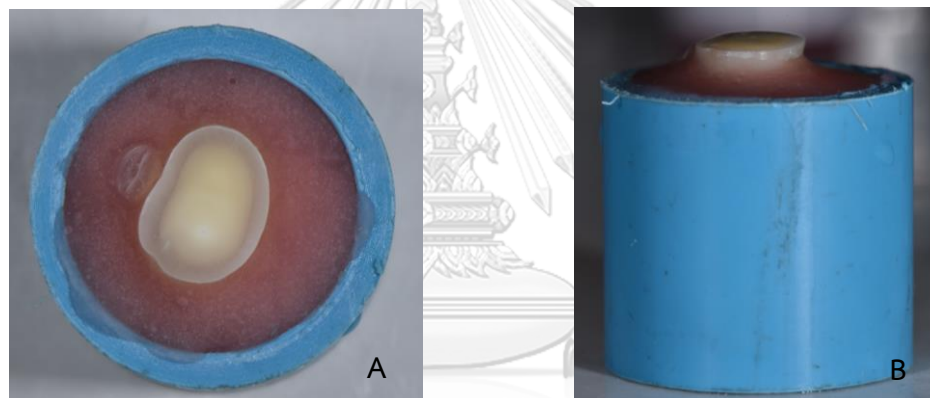
Figure 6. A) Prepared tooth after polishing in a polyvinyl chloride tube at top view

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

Ceramic microbar preparation

ZLS in the form of CAD/CAM ceramic ingots (shade A2) were cut into 36

microbars for VS and 72 microbars for CD in the dimensions of 1×1×3 mm^3, using a

diamond saw. The automatic temperature checking set (ATK2) was used to check

and adjust the firing temperatures in furnace with automatic calibration program for

the ATK2 system before firing the ceramics.

For VS, all the ceramic microbars were crystallized in a ceramic furnace

(Programat P700, Ivoclar Vivadent, Schaan, Liechtenstein) according to the

manufacturer's instruction. The starting temperature was 400°C and holding time at

the initial temperature was 8 minutes. The heating rate was 55°C/minute to reach

crystallization temperature, 840°C. After that, the temperature was held for 8

minutes. Finally, the ending temperature was 680°C.

For CD, 32 microbars were additionally fired in a ceramic furnace according to

the manufacturer's instruction to increase the material's flexural strength to 370

MPa. The starting temperature was 500°C and heating rate was 55°C/minute to reach

the final temperature, 820°C. After that the temperature was held for 1 minute and

30 second and cooled for 3 minutes. Later on, the bonding area of each microbar

was definitely measured using a stereomicroscope.

Top face of each ceramic microbar was polished with 120-, 240-, 400-, 600-

grit silicon carbide paper respectively at 100 rpm under running water for 10 s per

item. This step simulated the preparation of ceramic surface with a medium-coarse

diamond bur following with fine diamond (88). The grit silicon carbide paper was

changed after grinding of 10 ceramic microbars.

| 3 mm | 1 mm | 3 mm | 1 mm | 3 mm | 1 mm |
| 1 mm | | 1 mm | | 1 mm | |

36 microbars of VS   36 microbars of FCD   36 microbars of UCD
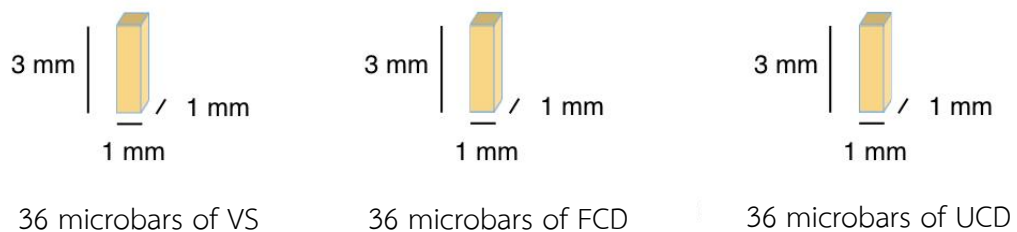
Figure 7. Preparation of ZLS microbars

Figure 8. Ceramic furnace (Programat P700, Ivoclar Vivadent)

Surface pre-treatment and cementation procedures

The polished surfaces of VS, FCD and UCD were etched with 4.5%

hydrofluoric acid (HF) (IPS ceramic etching; Ivoclar Vivadent) for 20, 30, and 30 s

respectively. The etched surfaces were then thoroughly rinsed with water for 60 s,

cleaned in an ultrasonic bath with 98 % alcohol for 3 mins and air-dried.

Then, the etched ZLS ceramics were randomly assigned into 9 groups (n=12

per group) following 3 kinds of adhesive resin luting cements: SM, SU and U2 (Table

5).

For groups VSSM, VSU2, FCDSM, FCDU2, UCDSM, and UCDU2, ceramic primer

(RelyX™ Ceramic Primer, 3M ESPE) was applied to the etched ceramic microbars,

allowed to react for 60 s, and air dried for 5 s. After that, only in groups VSSM,

FCDSM, and UCDSM, Scotchbond™ Multi-purpose (SM) Adhesive (3M ESPE) was

applied uniformly creating a thin coating. Meanwhile, 35% phosphoric acid etching

gel (Etchant, 3M ESPE) was applied to prepared dentin, and allowed to react for 15 s

before rinsing thoroughly with water for 15 s and blot drying with foam pellets. The

surface was shiny and did not have any puddles on it. After that, SM Primer (3M

ESPE) was applied at the etched dentin with a light scrubbing motion for 15 s. Then a

gentle stream of air was blown over the liquid for about 5 s until no further

movement was observed, and the solvent had evaporated completely. Then, SM

Adhesive was applied uniformly creating a thin coating with a brushing motion for 15

s and light-curing for 20 s.

For groups VSSU, FCDSU, and UCDSU, Single Bond Universal (SU, 3M ESPE)

was applied and rubbed to the moist prepared dentin, and etched ceramics for 20 s.

Then, a gentle stream of air was blown over the liquid for about 5 s until no further

movement could be observed, and the solvent had evaporated completely. Light-

curing for 20 s to the dentin followed. Then, for groups VSSM, VSSU, FCDSM, FCDSU,

UCDSM and UCDSU, the resin cement RelyX^{TM} Ultimate (RXU, 3M ESPE) was applied

copiously to etched ceramics using auto-mix syringe.

For the groups VSU2, FCDU2, and UCDU2, the RelyX™ Unicem (U2, 3M ESPE)

capsule was activated and mixed for 10 s (Rotomix, 3M ESPE). Then, cement was

dispensed directly on to the etched ceramics.

Subsequently, each ceramic microbar with the resin cement on top was

positioned on each prepared dentin surface under a constant load of 1 kg placed on

top using a custom-made loading device (Durometer, ASTM D 2240 Type A, PTC

Instrument) (89). Excess material was removed with a brush tip.

In this study, an LED light-curing system (Demi Plus, Kerr Coperation, Orange,

CA, USA) with 1,100 mW/cm^2 intensity was utilized. The light guide was held

perpendicularly and within 1 mm away from ceramic slabs for 20 s per surface. Then,

the load was removed and the specimen was additionally light-cured from the top

during 20 s (100 s light-curing in total). After that, the specimens were left for 10 mins

and the perforated Teflon tapes were removed (90).

The light output from the light-polymerizing unit was tested every 10

specimens to check for intensity of light output using a radiometer (Model 100

Optilux, Kerr Coperation, Orange, CA, USA) throughout the experiment.
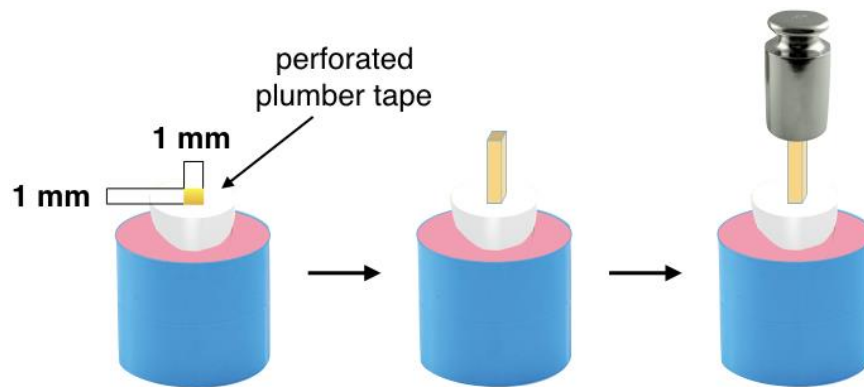
Figure 9. Cementation of the ceramic microbar to dentin specimen

Figure 10. ZLS microbar cemented to dentin specimen

Figure 11. Durometer (ASTM D 2240 Type A, PTC Instrument, USA)

All specimens were then examined under a stereomicroscope at 25×

magnification to verify that no bonding defects, air bubble inclusions, interfacial gaps,

or excess cement could be observed. If the excess cement was found, Blade No. 11

was used to remove it and changed every specimen. All specimens were

subsequently stored in distilled water at 37°C in an incubator (Contherm 160M,

Contherm Scientific Ltd., New Zealand) for 24 hours.

Microshear bond strength testing (µSBS)

All specimens were subjected to µSBS testing. Each polyvinyl chloride tube

with a ZLS microbar was placed horizontally on a support base so that the ceramic

microbar was unsupported. The adhesive interface was parallel to the shearing force.

Later on, the axial load with a 5-N load cell at a crosshead speed of 0.5 mm/min was

applied by a blunt blade at the dentin/adhesive interface, as close to the surface of

the tooth as possible, until fracture of the specimen occurred as shown in Figure 13

(91). The maximum force (Fmax (N)) was recorded. The µSBS values (MPa) were

calculated by Fmax (N) / bonding area (mm^2); bonding area measured by

stereomicroscope = width × length (mm^2) resulting in 12 µSBS values per group for

statistical analysis.

Figure 12. Universal testing machine (EZ-S Shimadzu, Japan)
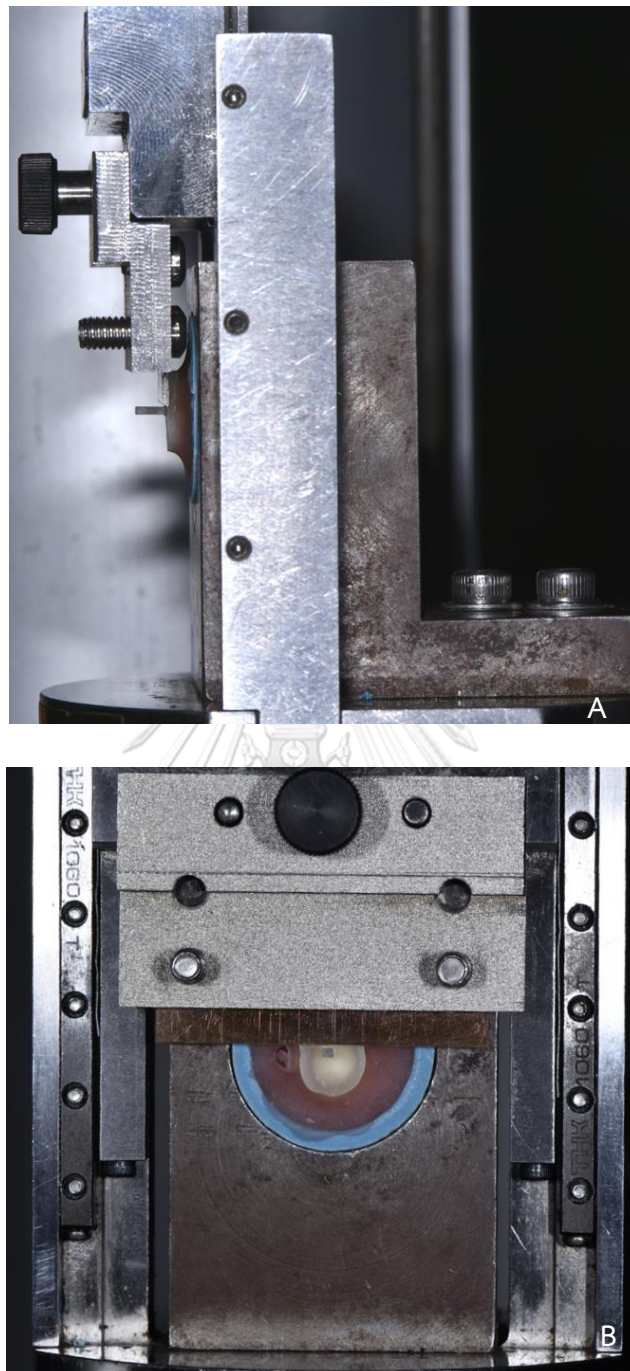
Figure 13. A) Microshear bond strength testing at proximal view

B) Microshear bond strength testing at frontal view

Failure Mode Analysis

After debonding, the specimens were examined under a stereomicroscope at a magnification of 40× to verify failure type. Failure types were classified as shown in Table 6 (92-94).

Table 6. Types of failure

Type	Character
Adhesive failure between cement and ceramic	Where resin cement completely remained on top of dentin surface
Adhesive failure between cement and dentin	Where resin cement completely remained on ceramic surface
Cohesive failure in luting cement	Where remnants of resin cement partially remained on both dentin and ceramic surface
Cohesive failure in dentin	The failure was within dentin
Cohesive failure in ceramic	The failure was within the ceramic
Mixed failure	Failure at the cement and adhesive interface including cohesive failure of the neighboring substrates

Data Collection and Analysis

All data were collected and analyzed using a statistical software (IBM$^{®}$ SPSS$^{®}$ 20, SPSS, Chicago, IL). The normality of the data was determined using the Kolmogorov-Smirnov test (K-S test). Two-way analysis of variance (two-way ANOVA) was used to statistically analyze the effects of ceramic materials, adhesive luting cements, and their interactions on the mean μSBS values. Moreover, a Tukey post-hoc multiple comparison test was performed to determine difference among means. Significance level was set at $P \le .05$.

CHAPTER IV RESULTS

There was no pre-test failure before or during the microshear bond strength testing. The K-S test indicated that the data were normally distributed. Mean and standard deviation of the tested groups were in the range of 7.63 ± 4.55 MPa (VSU2) to 38.02 ± 8.54 MPa (VSSM) as shown in Figure 14 and Table 8. Two-way ANOVA revealed that type of ZLS did not have a statistically significant influence on μSBS values of ZLS bonded to dentin (*P*=.699) (Table 7). On the other hand, kind of adhesive resin luting cements and their interaction did have a statistically significant effect on mean μSBS at *P*<.001 and .002 respectively (Table 7). According to Tukey post-hoc test, all ZLS bonded to dentin with SM and SU showed comparable mean μSBS (*P*=.066) (Table 8), although ZLS had the tendency to give the highest value when using SM. Meanwhile, ZLS cemented to dentin using U2 had a statistically significantly lower mean μSBS value compared to the ceramic bonded with SM and SU except in the case of UCDU2 which did not have a statistically significantly different μSBS value from UCDSU (*P*=.478) (Table 8).
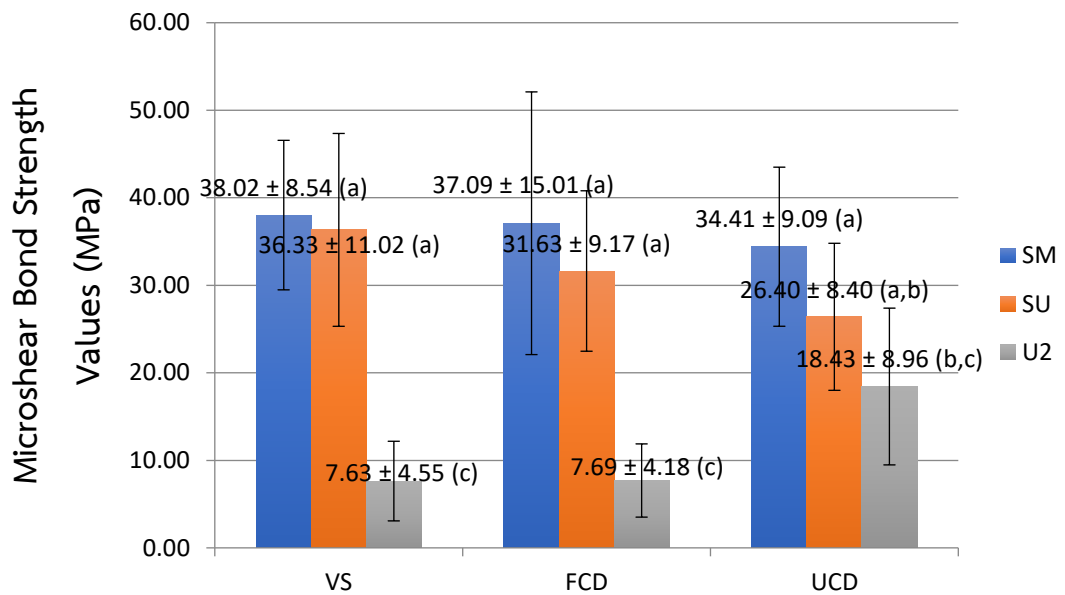
Figure 14. Microshear bond strength values (mean (MPa) ± SD) and statistical

comparison of different groups

Values with the same superscript letters indicate no significant differences between

groups. SD indicated by vertical bar. VS, Vita Suprinity®; FCD, fired-Celtra® Duo; UCD,

unfired-Celtra® Duo; SM, Scotchbond™ Multi-purpose; SU, Single Bond Universal; U2,

RelyX™ Unicem.

Table 7. Two-way ANOVA reveals the significant effects of adhesive luting and the interaction factor

Source of variation	df	Sum of squares	Mean square	F	P
ZLS factor	2	61.887	30.943	.359	.699
Adhesive factor	2	12856.577	6428.289	74.635	<.001
Interaction	4	1543.408	385.852	4.480	.002

ANOVA, Analysis of variance; ZLS, zirconia-reinforced lithium silicate ceramics.
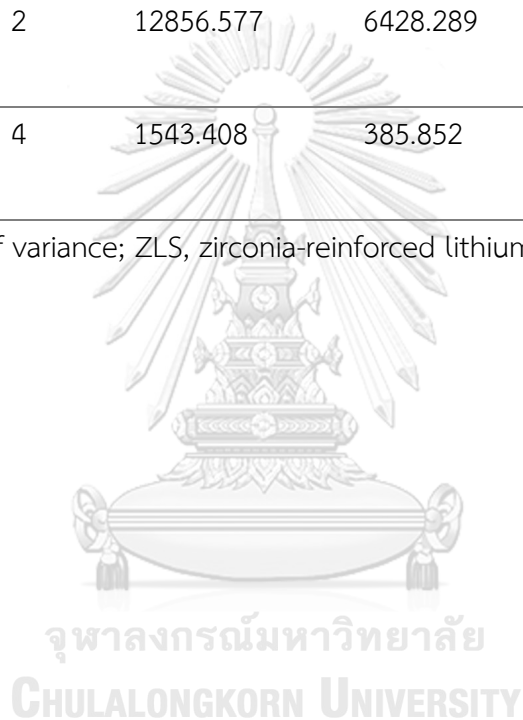
Table 8. Microshear bond strength values [mean (MPa) ± SD] and statistical

comparison of different groups

Group	Tukey HSD Subset		
	1	2	3
VSU2	7.63 ± 4.55		
FCDU2	7.69 ± 4.18		
UCDU2	18.43 ± 8.96	18.43 ± 8.96	
UCDSU		26.40 ± 8.40	26.40 ± 8.40
FCDSU			31.63 ± 9.17
UCDSM			34.41 ± 9.09
VSSU			36.33 ± 11.02
FCDSM			37.09 ± 15.01
VSSM			38.02 ± 8.54
Sig.	.114	.478	.066

VS, Vita Suprinity®; FCD, fired-Celtra® Duo; UCD, unfired-Celtra® Duo; SM,

Scotchbond™ Multi-purpose; SU, Single Bond Universal; U2, RelyX™ Unicem. Means

for groups in homogeneous subsets are displayed. The data are based on observed

means. The error term is mean square (error) = 86.130. The harmonic mean sample

size = 12.000. α=.05

For VSSM, FCDSM, and UCDSM, it was observed that failure mode was

predominantly cohesive failure in luting cement (58.3%) as well as for FCDSU

(66.7%). While, adhesive failure between cement and dentin was noticed to be a

major finding in VSSU and UCDSU (58.3%). In addition, in the others it was shown that

they all had only adhesive failure between cement and dentin (100%). Failure type

frequencies were given by group in Figure 15.
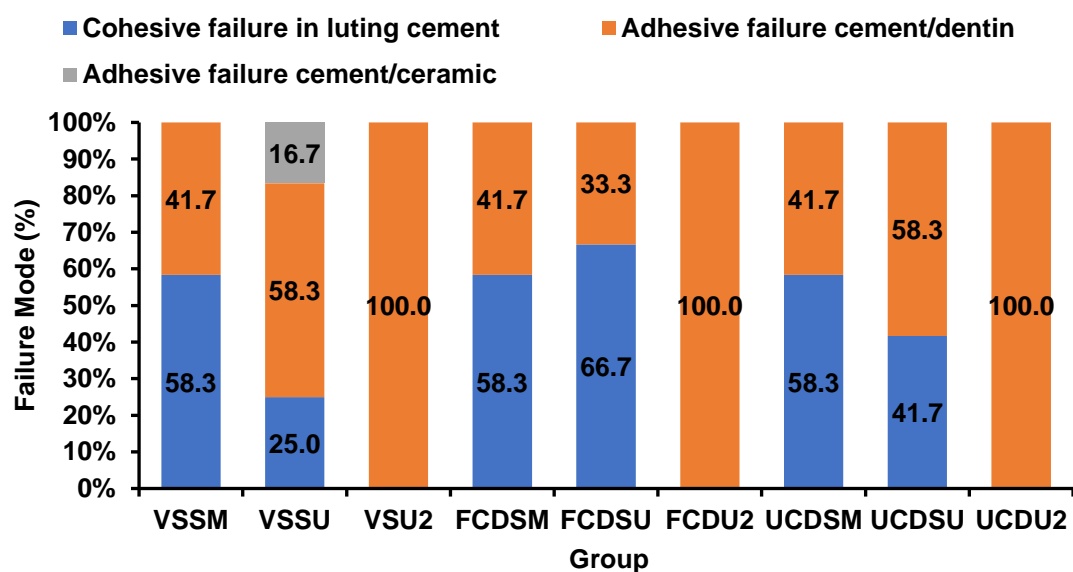
Figure 15. Failure mode percentages of all groups

VS, Vita Suprinity®; FCD, fired-Celtra® Duo; UCD, unfired-Celtra® Duo; SM,

Scotchbond™ Multi-purpose; SU, Single Bond Universal; U2, RelyX™ Unicem.
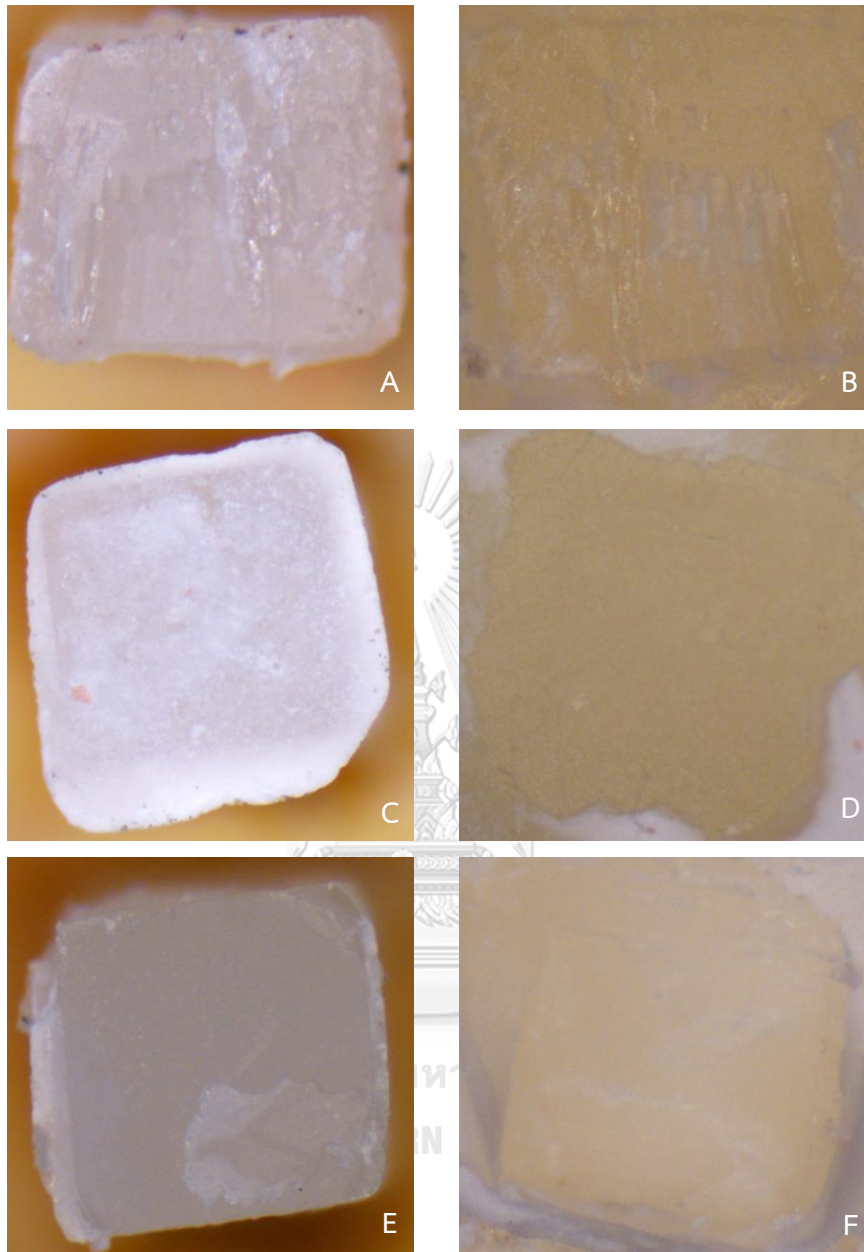
Figure 16. A), B) Cohesive failure in luting cement at ZLS and tooth-side

C), D) Adhesive failure between cement and dentin at ZLS and tooth-side

E), F) Adhesive failure between cement and ceramic at ZLS and tooth-side

CHAPTER V DISCUSSION AND CONCLUSIONS

Discussion

This study was conducted to investigate 24-hour μSBS of a three-step etch-and-rinse bonding system, a universal adhesive and a self-adhesive resin luting cement on ZLS bonded to dentin, and whether or not the distinct forms of ZLS could affect the bond strength. The first null hypothesis was rejected because different kinds of adhesive luting system had a statistically significant effect on mean μSBS whereas the other was accepted.

Mean μSBS values of Scotchbond™ Multi-purpose and Single Bond Universal were comparable regardless of kind of ZLS bonded to dentin (P=.066). The reason of high μSBS obtained from Scotchbond™ Multi-purpose, which is a three-step etch-and-rinse adhesive, may be due to the effect of etching dentin by phosphoric acid that removed smear layer and smear plug. This phenomenon resulted in a higher dentin demineralization effect, surface roughness, wettability, and ionization of the acidic monomers of the adhesive resin or the resin cement, thus the total etch

adhesive system probably created a suitable pattern of dentin hybridization (95-97).

However, the important issue, which was mostly related to the etching step, was that

the proper amount of moisture was required to prevent collagen collapse of

demineralized dentin before primer/adhesive application (46, 97).

Single Bond Universal comprised a various composition that mixed different

functional components, including water, ethanol and silane into the solution (98). In

a previous investigation, it was hypothesized that without removing the smear layer

and to incorporate it into the adhesive interface, penetration of resin monomers and

bonding effectiveness of this class of adhesive could be compromized (99). Previous

studies showed that adhesives which included silane in the composition were not

effective to bond lithium disilicate with resin composite (41, 100). In agreement with

earlier studies, it was reported that the presence of silane, which had an optimal

working pH range of about 4 to 5, in the solution of this adhesive, which had pH

value of 2.7 to 3.0, impaired the quality of the adhesive interface, and underwent

premature self-condensation reactions (38, 91). On the other hand, the dihydrogen

phosphate group of 10-MDP in this adhesive was responsible for reaction that led to

creation of ionic bond with calcium ions of hydroxyapatite, and formation a nano-

layering structure of MDP-Ca salt at the adhesive interface (58, 101). Meanwhile, the

long hydrophobic carboxyl chain of this functional monomer copolymerized with

resin monomers of the resin cement and provided hydrolytic stability of the bonding

interface (60). Moreover, 10-MDP was proven to offer a bond-mediating capacity to

zirconia (102, 103). Hydrophilic phosphate terminal end of this functional monomer

has been claimed to interact chemically with the oxide of zirconia in ZLS creating

high bond strength comparable to Scotchbond[TM] Multi-purpose in this study.

ZLS bonded to dentin using RelyX[TM] Unicem had a statistically significantly

lower mean µSBS than other adhesive luting agents in this study. This was in

agreement with the previous study by Rojpaibool, T. and Leevailoj, C., who

concluded that glass ceramics bonded to the tooth structure using etch-and-rinse

adhesive resin cements achieved higher significant fracture loads than self-adhesive

resin cements (104). Also, in the earlier study by Peumans, M. et al, it was found that

Celtra® Duo bonded to self-etch adhesive resin luting cement had higher µTBS than

self-adhesive resin cement (105). This phenomenon was explained by the fact that

high viscosity, a short interaction time at the cement/dentin interface before light

curing, and a lack of dentin demineralization caused incomplete resin infiltration

observed in both TEM and SEM (9, 106, 107). As seen in this study, most failure

modes occurred in groups of ZLS bonded with RelyX^{TM} Unicem with 100% adhesive

failure between cement and dentin.

In the present study, distinct forms of ZLS had no significant effect on µSBS

of these ceramics cemented to dentin using different resin luting agents. This

phenomenon can be explained by the fact that the manufacturers cooperated with

the Fraunhofer Institute for Silicate Research (ISC) in Würzburg, Germany to launch

Vita Suprinity$^®$ and Celtra$^®$ Duo for which the structure and chemical composition

were similar. The ceramics comprised round and slightly elongated lithium-

metasilicate (Li_2SiO_3) crystals, round lithium orthophosphate (Li_3PO_4) granules, and

were reinforced with about 10% zirconium dioxide (ZrO_2) so that the ceramics could

be pretreated by HF, silanized then bonded by adhesive resin luting cement (108-

110).

According to the finite element analysis, it was concluded that shear bond

strength (SBS) testing in large bonded areas had been associated with the

development of heterogeneity of stress distributed along tested interfaces, and had

a greater tendency to produce cohesive failures leading to misinterpretation of the

results (111, 112). In this present investigation, the µSBS test was used rather than

SBS to evaluate bond strength at dentin-ceramic interface because small-sized

cementation area created a more uniform distribution of loading stress, and lower

probability of encountering a flaw at the interface than large-sized bonded area (80,

81). Moreover, all of the failure modes were observed at the adhesive interface and

in luting cement, therefore, the value measured when specimen cracked represented

a more reliable µSBS. In previous study comparing wire loop and blade method for

µSBS testing, it was found that if the force was loaded at a contact point between

the blade and interface, it created a less even uniformity of shear force (111, 113).

That was the reason why cross-section of microbar of ZLS was prepared as a

rectangular area in this study. When the blade touches the interface, shear force is

loaded at the contact area, consequently creating more uniform loading stress.

Furthermore, no pretest failure was found because the microbars of ZLS were

prepared before cementation. Thus, the occurrence of structural defects from

trimming after the adhesive procedure in specimen preparation was reduced.

From the perspective of the clinician, this present study showed that

Scotchbond™ Multi-purpose with RelyX™ Ultimate utilizing etch-and-rinse adhesive

system, and Single Bond Universal with RelyX™ Ultimate utilizing self-etch adhesive

system significantly achieved greater μSBS than RelyX™ Unicem self-adhesive luting

system.

Limitations

1. This study investigated only one ceramic system (ZLS), 2 kinds of adhesive

   agents and luting cements. Therefore, the results from this study might not

   be inferred to other adhesive and ceramic systems.

2. This result might not be inferred to real clinical situation because only 24-

   hour μSBS test was performed.

Suggested further studies

This study investigated 24-hour bond strength of ZLS cemented to dentin with different adhesive luting systems. Further studies should be carried out to test the bond strength and fatigue failure load of ZLS under different aging conditions and adhesive situations.

Conclusions

Under the conditions of this *in vitro* study, the following conclusions can be drawn:

1. Etch-and-rinse and universal adhesive resin luting systems used to bond ZLS to dentin perform well rather than self-adhesive resin cement.

2. The different forms of ZLS comparably achieved microshear bond strength when they were bonded to dentin using resin luting cements.

Clinical implication

Resin cement used with etch-and-rinse and universal adhesive agents are recommended to be used for cementation of ZLS to dentin, rather than using self-adhesive resin cement.

Declaration of Conflicting Interest

The authors declare that there is no conflict of interest.

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APPENDICES

Appendix A. Microshear bond strength values of Vita Suprinity® cemented to dentin

using Scotchbond™ Multi-purpose with RelyX™ Ultimate utilizing etch-and-rinse

adhesive system.

Units	MPa
VSSM-1	46.2560
VSSM-2	44.7782
VSSM-3	48.9838
VSSM-4	36.8980
VSSM-5	39.5468
VSSM-6	48.5736
VSSM-7	29.8953
VSSM-8	22.6272
VSSM-9	39.3393
VSSM-10	36.1703
VSSM-11	37.2003
VSSM-12	25.9626
MEAN	38.0193
SD	8.5432

Appendix B. Microshear bond strength values of Vita Suprinity® cemented to dentin

using Single Bond Universal with RelyX™ Ultimate utilizing self-etch adhesive system.

Units	MPa
VSSU-1	31.3474
VSSU-2	27.4241
VSSU-3	54.5224
VSSU-4	38.4515
VSSU-5	51.9068
VSSU-6	39.5574
VSSU-7	36.8959
VSSU-8	36.7538
VSSU-9	45.1985
VSSU-10	22.9759
VSSU-11	16.8285
VSSU-12	34.1125
MEAN	36.3312
SD	11.0180

Appendix C. Microshear bond strength values of Vita Suprinity® cemented to dentin

using RelyX™ Unicem self-adhesive luting system.

Units	MPa
VSU2-1	3.0432
VSU2-2	13.4588
VSU2-3	14.2343
VSU2-4	3.5579
VSU2-5	2.5490
VSU2-6	9.4611
VSU2-7	10.3775
VSU2-8	2.3113
VSU2-9	10.9300
VSU2-10	11.8868
VSU2-11	6.1411
VSU2-12	3.5972
MEAN	7.6290
SD	4.5498

Appendix D. Microshear bond strength values of fired-Celtra® Duo cemented to

dentin using Scotchbond™ Multi-purpose with RelyX™ Ultimate utilizing etch-and-

rinse adhesive system.

Units	MPa
FCDSM-1	57.8736
FCDSM-2	55.1799
FCDSM-3	49.6085
FCDSM-4	10.9228
FCDSM-5	45.3492
FCDSM-6	38.9823
FCDSM-7	17.2974
FCDSM-8	43.5895
FCDSM-9	34.1044
FCDSM-10	18.2022
FCDSM-11	35.4461
FCDSM-12	38.5214
MEAN	37.0898
SD	15.0056

Appendix E. Microshear bond strength values of fired-Celtra® Duo cemented to

dentin using Single Bond Universal with RelyX™ Ultimate utilizing self-etch adhesive

system.

Units	MPa
FCDSU-1	26.2611
FCDSU-2	29.4582
FCDSU-3	44.5136
FCDSU-4	19.2647
FCDSU-5	23.9970
FCDSU-6	16.1633
FCDSU-7	37.5749
FCDSU-8	29.0358
FCDSU-9	33.3473
FCDSU-10	37.4287
FCDSU-11	43.4360
FCDSU-12	39.1191
MEAN	31.6333
SD	9.1653

Appendix F. Microshear bond strength values of fired-Celtra® Duo cemented to

dentin using RelyX™ Unicem self-adhesive luting system.

Units	MPa
FCDU2-1	5.6341
FCDU2-2	17.6221
FCDU2-3	4.9310
FCDU2-4	3.6261
FCDU2-5	8.0662
FCDU2-6	13.2334
FCDU2-7	5.8924
FCDU2-8	4.8240
FCDU2-9	3.1609
FCDU2-10	8.5990
FCDU2-11	7.7666
FCDU2-12	8.9716
MEAN	7.6940
SD	4.1812

Appendix G. Microshear bond strength values of unfired-Celtra® Duo cemented to

dentin using Scotchbond™ Multi-purpose with RelyX™ Ultimate utilizing etch-and-

rinse adhesive system.

Units	MPa
UCDSM-1	31.3196
UCDSM-2	36.9276
UCDSM-3	27.5701
UCDSM-4	26.3918
UCDSM-5	45.1383
UCDSM-6	53.2294
UCDSM-7	23.7966
UCDSM-8	35.7760
UCDSM-9	23.5110
UCDSM-10	34.7351
UCDSM-11	42.8622
UCDSM-12	31.6348
MEAN	34.4077
SD	9.0893

Appendix H. Microshear bond strength values of unfired-Celtra® Duo cemented to

dentin using Single Bond Universal with RelyX™ Ultimate utilizing self-etch adhesive

system.

Units	MPa
UCDSU-1	23.3156
UCDSU-2	21.0296
UCDSU-3	16.3791
UCDSU-4	13.9535
UCDSU-5	26.1370
UCDSU-6	28.7970
UCDSU-7	22.7619
UCDSU-8	40.0423
UCDSU-9	29.8975
UCDSU-10	40.2096
UCDSU-11	21.0638
UCDSU-12	33.1846
MEAN	26.3976
SD	8.4007

Appendix I. Microshear bond strength values of unfired-Celtra® Duo cemented to

dentin using RelyX™ Unicem self-adhesive luting system.

Units	MPa
UCDU2-1	12.5968
UCDU2-2	22.6683
UCDU2-3	11.6162
UCDU2-4	29.9708
UCDU2-5	18.3494
UCDU2-6	1.5674
UCDU2-7	12.4158
UCDU2-8	12.2462
UCDU2-9	18.4840
UCDU2-10	20.8844
UCDU2-11	32.3499
UCDU2-12	28.0485
MEAN	18.4331
SD	8.9579

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