

MICROTENSILE BOND STRENGTH OF REPAIRED CERAMIC USING RESIN
COMPOSITE WITH UNIVERSAL ADHESIVE SYSTEM COMPARED TO CONVENTIONAL
BONDING SYSTEM IN VITRO

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บทคัดย่อและแฟ้มข้อมูลฉบับเต็มของวิทยานิพนธ์ตั้งแต่ปีการศึกษา 2554 ที่ให้บริการในคลังปัญญาจุฬาฯ (CUIR)
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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Esthetic Restorative and Implant Dentistry
Faculty of Dentistry
Chulalongkorn University
Academic Year 2016

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การเปรียบเทียบกำลังแรงยึดแบบดึงระดับจุลภาคของการเชื่อมเซรามิกด้วยเรซินคอมโพสิต
ระหว่างการใช้สารยึดระบบยูนิเวอซัลกับสารยึดติดระบบธรรมดา



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาทันตกรรมบูรณะเพื่อความสวยงามและทันตกรรมรากเทียม

คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2559

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

ชัชฎา เลิศกุลวิไลจิร : การเปรียบเทียบกำลังแรงยึดแบบดึงระดับจุลภาคของการซ่อมแซมเซรามิกด้วยเรซินคอมโพสิตระหว่างการใช้สารยึดระบบยูนิเวอร์ซัลกับสารยึดติดระบบธรรมดา (MICROTENSILE BOND STRENGTH OF REPAIRED CERAMIC USING RESIN COMPOSITE WITH UNIVERSAL ADHESIVE SYSTEM COMPARED TO CONVENTIONAL BONDING SYSTEM IN VITRO) อ.ที่ปรึกษาวิทยานิพนธ์
หลัก: ผศ. ทพญ. ดร. ศิริวิมล ศรีสวัสดิ์, 92 หน้า.

วัตถุประสงค์: เพื่อเปรียบเทียบประสิทธิภาพของการซ่อมแซมวัสดุเซรามิกส์ 2 ชนิดด้วยวัสดุคอมโพสิต เรซินระหว่างสารยึดติดระบบยูนิเวอร์ซัลและสารยึดติดระบบดั้งเดิม

วิธีการศึกษา: ชิ้นงานอินกอต 16 ชิ้น จากแต่ละกลุ่มของเซรามิกส์ ไอพีเอสเอ็มเพรสเอสเทติก (IPS Empress® Esthetic; "EE", Ivoclar Vivadent) และ ไอพีเอสอีแมกซ์เพรส (IPS e.max® Press; "EM", Ivoclar Vivadent) จะได้รับการขึ้นรูปเป็นชิ้นงานเซรามิกส์ขนาด 8x8x4 ลูกบาศก์มิลลิเมตร ชิ้นงานทุกชิ้นจะถูกปรับสภาพผิวของเซรามิกส์ด้วยกรดไฮโดรฟลูออริกความเข้มข้น 9.5 เปอร์เซ็นต์ หลังจากนั้นชิ้นงานเซรามิกส์จะได้รับการบูรณะด้วยคอมโพสิต เรซินฟิลล์เทค ซีสามห้าศูนย์เอกซ์ที เจดดี เอโฟว์ (Filtek™ 350 XT; 3M ESPE, USA) โดยใช้สารยึดติด 2 ระบบ ครั้งแรกได้รับการใช้สารยึดติดระบบยูนิเวอร์ซัล (U) ซึ่งประกอบด้วยสารยึดติดซิงเกิลบอนด์ยูนิเวอร์ซัล (Single Bond™ Universal; 3M ESPE, USA) ต่างจากระบบสารยึดติดแบบดั้งเดิมในครั้งกลุ่มหลัง (C) ที่จะมีการทาไฮเลนวีไลน์เอกซ์เซรามิกส์ (RelyX™ Ceramic; 3M ESPE, USA) ร่วมกับการใช้สารยึดติด แอดเบ็ป อสกอตซ์บอนด์มัลติเพอโพลพลัส (Adper™ Scotchbond™ Multipurpose Plus; 3M ESPE, USA) ชิ้นงานหลังจากได้รับการบูรณะด้วยเรซิน คอมโพสิตแล้วจะถูกแบ่งออกเป็น 8 กลุ่มการทดลอง โดยมีชิ้นงาน 36 ชิ้นต่อกลุ่ม หลังจากนั้นชิ้นงานในกลุ่มที่ต้องการทดสอบการเอจจิ้งชิ้นงานจะถูกนำไปแช่ในน้ำที่อุณหภูมิ 37 องศาเซลเซียสเป็นเวลา 24 ชั่วโมง ต่อด้วยการทำเทอร์โมไซคลิก (TC301, KMITL) จำนวนหนึ่งหมื่นรอบด้วยระยะเวลา 60 วินาทีต่อรอบก่อนนำมาทดสอบค่าแรงยึดติดแบบดึงระดับจุลภาคในทุกกลุ่มการทดลอง ข้อมูลค่าเฉลี่ยการยึดติดแบบดึงระดับจุลภาคได้ถูกวิเคราะห์ทางสถิติด้วย การวิเคราะห์ความแปรปรวน 3 ทาง และ บอนเฟอรินีโพสฮอคเทส ที่ระดับนัยสำคัญ 0.05 ความล้มเหลวของชิ้นทดสอบ (mode of failure) จะถูกตรวจสอบด้วยกล้องจุลทรรศน์แบบสเตอริโอ (ML 9300; MEIJI)

ผลการศึกษา: ค่าเฉลี่ยการยึดติดแบบดึงระดับจุลภาคระหว่างกลุ่ม อีเอ็มดีเอด และกลุ่ม เอจจิ้ง นั้นมีค่าแตกต่างกันอย่างไม่มีนัยสำคัญทางสถิติ จากสถิติบอนเฟอโรนีแสดงให้เห็นว่าค่าเฉลี่ยการยึดติดแบบดึงระดับจุลภาคมีค่าสูงที่สุดในกลุ่ม อีเอ็มซี (EMC) ตามด้วยกลุ่ม อีอีซี (EEC) และ อีเอ็มยู (EMU) และกลุ่มที่น้อยที่สุดคือกลุ่ม อีอียู (EEU) ถึงแม้ว่าจะมีค่าการยึดติดที่แตกต่างอย่างไม่มีนัยสำคัญทางสถิติกับกลุ่ม อีเอ็มยู (EMU) ก็ตาม

สรุป: ค่าเฉลี่ยการยึดติดแบบดึงระดับจุลภาคระหว่างคอมโพสิตเรซิน และเซรามิกส์ที่ใช้สารยึดติดระบบดั้งเดิมมีค่าการยึดติดที่สูงกว่าสารยึดติดระบบยูนิเวอร์ซัล โดยเฉพาะอย่างยิ่งในกลุ่มเซรามิกส์ไอพีเอสอีแมกซ์เพรส

สาขาวิชา ทันตกรรมบูรณะเพื่อความสวยงามและทันตศัลยกรรมมือชื่อนิสิต

รรมรากเทียม ปลายมือชื่อ อ.ที่ปรึกษาหลัก

5675828732 : MAJOR ESTHETIC RESTORATIVE AND IMPLANT DENTISTRY

KEYWORDS: MICROTENSILE BOND STRENGTH, REPAIRED CERAMIC, THERMOCYCLING, UNIVERSAL BONDING

CHISANU LERTTHAWINCHIRA: MICROTENSILE BOND STRENGTH OF REPAIRED CERAMIC USING RESIN COMPOSITE WITH UNIVERSAL ADHESIVE SYSTEM COMPARED TO CONVENTIONAL BONDING SYSTEM IN VITRO. ADVISOR: ASST. PROF. SIRIVIMOL SRISAWASDI, Ph.D., 92 pp.

The purpose of this study was toThe effectiveness of two types of ceramic, repaired using a resin composite and a universal adhesive were compared to a conventional adhesive.

The effectiveness of two types of ceramic, repaired using a resin composite and a universal adhesive were compared to a conventional adhesive. Leucite-reinforced glass ceramic ingots (IPS Empress[®] Esthetic; "EE"; Ivoclar Vivadent, Germany) and lithium-disilicate glass ceramic ingots (IPS e.max[®] Press; "EM"; Ivoclar Vivadent, Germany) were fabricated into a ceramic block size 8x8x4 mm. The ceramic surfaces were wet polished with silicon carbide paper and then treated with 9.5 % hydrofluoric acid (Ultradent[®] Porcelain Etch; Ultradent, USA). Resin composite (Filtek[™] Z350 XT; 3M ESPE, USA), shade A4, was built up with two adhesive systems, one half ("U") using universal dental adhesive (Single Bond[™] Universal; 3M ESPE, USA) and the other ("C") using total etch dental adhesive (Adper[™] Scotchbond[™] Multipurpose Plus; 3M ESPE, USA) combined with ceramic primer (Relyx[™] Ceramic Primer; 3M ESPE, USA). The specimens were stored in water at 37°C for 24 hours and then subjected to thermocycling for 10,000 cycles prior to a microtensile bond strength (μ TBS) test. Modes of failure were analyzed using a stereomicroscope (ML 9300; MEIJI, Japan). Three-way ANOVA and a Bonferroni post-hoc test was used to analyze the data ($n = 36$, $\alpha = 0.05$). There was no significant difference between the immediate and aging groups ($p = 0.207$). However, a Bonferroni post-hoc test revealed significant differences among all tested groups. The highest μ TBS was recorded by the "EMC" group (36.3 ± 13.1), while the lowest was found in the "EEU" group (22.0 ± 7.9). The μ TBS between the resin composite and ceramic repaired using a conventional adhesive system was higher compared with a universal adhesive system, especially in the lithium disilicate type.

Field of Study: Esthetic Restorative and Implant
Dentistry

Student's Signature
Advisor's Signature

Academic Year: 2016

ACKNOWLEDGEMENTS

This project would not have been possible without the support of many people. The author would like to use this opportunity to express his gratitude to everyone who supported him throughout the course of this project.

The author wishes to express his gratitude to his supervisor, Assistant Professor Sirivimol Srisawasdi, Ph.D. who was extremely helpful and offered invaluable assistance, support and guidance.

Deepest gratitude are also due to the member of supervisory committee, Associate Professor Chalernpol Leevailoj and Assistant Professor Sirichan Chiraputt Ph.D. without whose knowledge and assistance this study would not have been successful.

The author would like to express his warm thanks to Associate Professor Chanchai Hosawuan who's statistical methods were extraordinarily helpful.

Also, thanks to staffs of Dental Research center from Chulalongkorn University and Srinakharinwirot University for providing me with the facilities being required for the project.

Special thanks also to all his graduate friends who always been there together and all their encouragement.

The author wishes to express his love and gratitude to his beloved families; for their understanding and endless love, through the duration of his studies.

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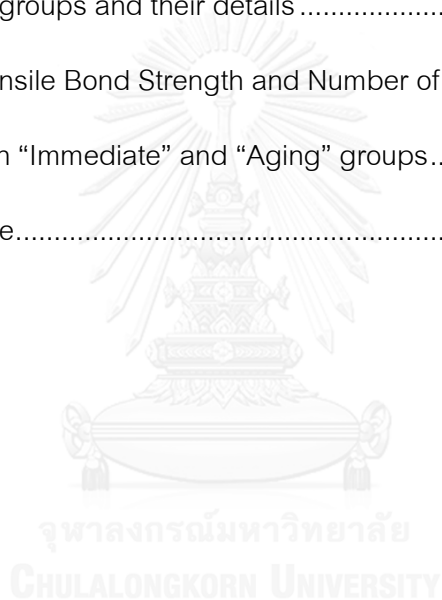
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Introduction

Rationale and Significance of the Problem

Esthetics is a major concern in many aspects of daily life, and possibly even more so in dentistry. Dental ceramics are restorative materials that fulfill esthetic requirements in both form and functional aspects. Due to their strength and esthetic properties, ceramics became popular for dental restorations including anterior crowns, veneers, and veneers on substructures.(1) Many classes of ceramic systems are available including feldspathic glass, leucite-reinforced glass, lithium-disilicate glass, and zirconia.

In the past, feldspathic glass ceramic was most commonly used for veneer restorations as its excellent optical properties, translucency, and color; resulted in a natural appearance. Later, leucite-reinforced glass ceramic was developed, composed of about 45% leucite by volume. IPS Empress[®]

Esthetic (Ivoclar Vivadent, Germany) is one example of commercialized leucite-reinforced ceramics that are most widely used nowadays. This material has flexural strength around 100-120 MPa, with such low strength, the clinical indication of leucite-reinforced ceramic is limited to anterior teeth restoration.(2) Another type of ceramics was developed, containing 65% of lithium disilicate by volume, lithium-disilicate glass ceramic has flexural strength around 350-450 MPa which is sufficient

for 3-unit fix partial denture prosthesis in anterior region.(3)

Despite both providing favorable esthetic result, ceramics have greater strength and durability compared to resin composites. Even so, fractures of ceramic restorations sometimes occur.(4-5)

Replacing the fractured ceramic restoration with a new one is the treatment of choice; however, in

most situations, patients decline this treatment, due to time constraints and/or financial limitations. In

these cases, repairing the ceramic with resin composite can be an attractive alternative treatment.(6)

The ceramic repair procedure begins with surface conditioning which can be performed using many techniques including etching the ceramic surface with 2.5%, 4.9%, 5%, 9.5%, 9.6%, 10%

hydrofluoric acid(7-10), sandblasting the ceramic surface with aluminum oxide particles(11), etching

the ceramic surface with 1.23% acidulated phosphate fluoride(12), or silica coating(11). Although 5%

hydrofluoric acid is particularly suggested as surface conditioning agent by the manufacturer, there

are many previous studies reporting that 9.5% hydrofluoric acid provided higher microtensile bond

strength than 5% hydrofluoric acid. Moreover, the gel-liked form of 9.5% hydrofluoric acid can be

easily controlled when applying intra-orally for repairing ceramic with resin composite. According to

these reasons, 9.5% hydrofluoric acid was chosen as surface conditioning agent in this study. After

surface conditioning, a silane coupling agent is applied in order to promote chemical bonds between

organic and inorganic components(13-14), accompanied by an adhesive agent and resin composite.

This procedure is known as conventional adhesive system. Disadvantages of this procedure include requiring various products, taking many steps and consuming chair-time, which make conventional adhesive system technique-sensitive. Hence, universal adhesive was developed to resolve these problems.

Many universal adhesives, commercially available now, have been claimed to be able to bond to many substrates such as enamel(15), dentin(15), glass ceramic(15-16) or even zirconia(17). In this study, Single Bond™ Universal was selected as a representative of universal adhesive system. Due to its composition that differs from the other adhesives, which is the incorporation of silane coupling agent in the bottle, Single Bond™ Universal is able to bond with glass ceramics.(16)

Even though many procedures have been developed for repairing ceramics with resin composites(18-20), with the performance of each technique intensively investigated, hydrofluoric acid in combination with a silane coupling agent was the most popular method of choice.(6, 18-19, 21) However, few studies have compared the microtensile bond strength (μ TBS) of the repair using the so-called 'universal' adhesives with the conventional method for two types of ceramic systems. This study compared the performance of a universal adhesive with a conventional adhesive for ceramic repair. Two types of ceramic systems; lithium-disilicate and leucite-reinforced glass ceramics, were chosen.

Research Question

Will the microtensile bond strength of a universal adhesive be comparable to conventional adhesive when used to repair leucite reinforced and lithium disilicate ceramic *in vitro*?

Research Objectives

The aim of this study was to determine microtensile bond strength of repaired leucite reinforced and lithium disilicate ceramics using resin composite with conventional bonding system compared to a universal adhesive, *in vitro*.

Hypotheses

Null hypotheses

1. There was no difference in μ TBS between repaired ceramic using resin composite with a universal adhesive or conventional adhesive system.
2. There was no difference in μ TBS between aged and non-aged groups of repaired ceramics using different adhesive systems.

Alternative Hypothesis

1. There was difference in μ TBS between repaired ceramic using resin composite with a universal adhesive or conventional adhesive system.
2. There was difference in μ TBS between aged and non-aged groups of repaired ceramics using different adhesive systems



Conceptual Framework

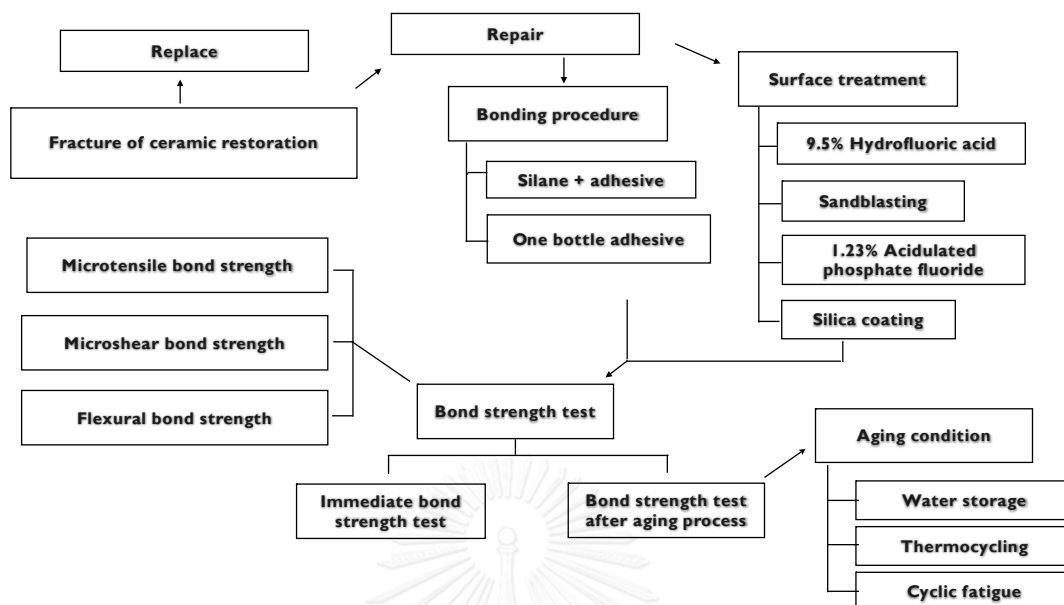


Figure 1: Diagram of conceptual framework

There were many kinds of ceramic utilizing for fabrication of restorations in dental treatment.

The present study focused on two types of ceramic systems; leucite reinforced and lithium disilicate systems. As previously mentioned, they were the most popular materials used to fabricate veneering

material or anterior full coverage restorations, due to their properties.

According to the results from previous studies, surface conditioning by 9.5% hydrofluoric acid followed by silane application was used as control materials.(22-24) In the experimental group, a universal adhesive was utilized to compare microtensile bond strength.

Limitations

1. This study investigated only 2 commercially available adhesive systems from 1 company.

Therefore, the results from this study might not be inferred to other adhesive systems.

2. This study used 2 types of ceramic system. Hence, the results found here might not be

extrapolated to other ceramic systems.

3. The results might not be inferred to real clinical situation, although the researcher tried to control

the confounding factors and simulated closely to clinical situation.

Keywords

Microtensile Bond Strength, Repaired Ceramic, Thermocycling, Universal Bonding

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Expected Benefit of the Study

The results from this study could facilitate dentist to decide whether to use the universal adhesive or the conventional adhesive for repairing ceramic with resin composite.

Review of the Literatures

The literatures in these following topics have been reviews.

- Dental ceramics
- Repairing procedures
- Bond strength test
- Aging process

Dental ceramics

Dental ceramics have been developed since 1962, due to their optical properties that make the restorations look like the natural teeth, they have become popular increasingly through the past 50 years.(25)

Currently, dental ceramics can be categorized by the main composition in the materials.

Different components make the materials different in mechanical properties. According to Conrad HJ et al., 2007, ceramics can be classified into 3 types; 1) glass ceramics, 2) alumina-based ceramics, and 3) zirconia-based ceramics.(1)

There are many types of glass ceramics that can be used to fabricate dental restorations. In order to be called glass ceramics, the first type of dental ceramic, the main component has to be SiO_2 . Examples are IPS Empress and IPS Empress 2 from Ivoclar Vivadent. IPS Empress is a leucite-reinforced glass ceramic ($\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O}$) that contains up to 45% by volume of leucite. Because of its low flexural strength (100-120 MPa)(26), the indication is limited to the single unit crown in the anterior region.(2) IPS e.max[®] Press was released as a successor of IPS Empress 2 in 2005.(1) The physical properties and translucency are developed due to the different of firing process.(1) IPS e.max[®] Press is also a lithium-disilicate glass ceramic ($\text{SiO}_2\text{-Li}_2\text{O}$) as the previous one, containing up to 65% by volume of lithium disilicate. The flexural strength of IPS e.max[®] Press is 3 times higher than that of IPS Empress[®] Esthetic (~350 MPa)(27) which leads to further application of 3 units fix partial denture prosthesis.(28) Both IPS Empress[®] Esthetic and IPS e.max[®] Press can be fabricated by heat-pressed techniques.(1) IPS ProCAD (Ivoclar Vivadent) is also a leucite-reinforced ceramic similar to IPS Empress[®] Esthetic, but was designed to use with the CEREC inLab system (Sirona Dental Systems, Germany). Moreover, IPS ProCAD is available in more numerous shades than IPS Empress[®] Esthetic.(29)

There are also other brands of glass ceramics from other manufacturers such as Vita Mark II and Vita TriLuxe Bloc from VITA Zahnfabrik. Vita Mark II (VITA Zahnfabrik, Germany) was introduced

in 1991 as a feldspathic porcelain for the CEREC 1 system (Siemens AG, Germany).(30) Vita Mark II contains SiO_2 about 60-64% by volume and Al_2O_3 about 20-23%. Its retention can be improved by etching with hydrofluoric acid, which will create the micromechanical retention for adhesive agent with composite resin cements.(29, 31) Another one is Vita TriLuxe Bloc (VITA Zahnfabrik, Germany), with a design of multichromatic ceramic block, to improve the optical properties of the restorations.(32)

Secondly, alumina-based ceramics, such as In-Ceram Alumina (VITA Zahnfabrik, Germany), was introduced in 1989, using slip-casting technique for fabrication.(33) Due to its high strength, In-Ceram Alumina can be fabricated for both single unit restorations and 3-unit anterior fix partial denture prostheses.(34) However, they have to be veneered with feldspathic porcelain to improve the esthetic result.

In 1994, In-Ceram Spinell (VITA Zahnfabrik, Germany) containing magnesia and alumina (MgAl_2O_3) was introduced as an option to avoid an opaque core of In-Ceram alumina. Nevertheless, flexural strength of In-Ceram Spinell is lower than that of In-Ceram Alumina, hence it can only be used for core material of anterior crowns, which will be later veneered with feldspathic porcelain.(35)

In-Ceram Zirconia (VITA Zahnfabrik, Germany) was modified from In-Ceram Alumina system, by adding 35% partially stabilized zirconia oxide to the slip part to improve the strength of the ceramic.(33) However, the core is opaque and lack of translucency, therefore its indication is located for posterior crown copings and fix partial denture prosthesis frameworks.(33)

Another Alumina-based ceramic was brought into the market by Nobel Biocare (Nobel Biocare AB, Sweden), Procera All Ceram, fabricating by densely sintered technique. Procera AllCeram contains 99.9% high purity aluminum oxide combined with low-fusing feldspathic porcelain. Procera AllCeram has the highest strength of all the alumina-based materials.(36)

The last type of dental ceramic, zirconia-based ceramics contain zirconia, a polymorphic material, which can transform into 3 phases, cubic phase, tetragonal phase, and monoclinic phase. At the melting point (2680°C), zirconia expressed as a cubic form. It changed into tetragonal phase when the temperature was below 2370°C.(37) The transformation from tetragonal phase to monoclinic phase occurred when the temperature was lower than 1170°C. In this altering phase, the volume of zirconia expanded 3-5% by volume creating high internal stresses.(37)

Yttrium-oxide (Y_2O_3 3% mol) played an important role, stabilizing zirconia in the tetragonal phase at the room temperature.(38) Under stress, defect or crack may be created in the zirconia,

then the tetragonal phase transformed into the monoclinic phase with an expansion of 3-5%.(39) This increase in volumes created the compressive stress, which stopped the crack propagation. This phenomenon has been known as transformation toughening.(39)

Zirconia has flexural strength of 900-1200 MPa, fracture toughness of 6-10 MPa/m^{1/2}, and compression resistance of 2000 MPa, which has been proven to be the highest among all-ceramic systems. From these high mechanical properties, zirconia could be fabricated into single crown and 3-4 units fix partial denture prostheses.(30, 40)

In the past, due to its appearance, IPS Empress[®] Esthetic was very popular for fabricated veneer or anterior crown restoration. Nowadays, IPS e.max[®] Press is a better choice for fabricated veneer, anterior crown restoration due to its strength and appearance. While IPS e.max[®] Press has superior strength than IPS Empress[®] Esthetic, the appearance of IPS e.max[®] Press can be comparabled to IPS Empress[®] Esthetic. In the case of using monolithic type, IPS e.max[®] Press can be used as posterior crown.(28)

Zirconia is an another material of choice for tooth color crown restoration. Zirconia has more flexural strength than those ceramics, though its downside is that the material is opaque and its surface can not be bonded with any kind of substrate.(30)

Thus, this research will focus on IPS Empress[®] Esthetic and IPS e.max[®] Press as these two systems are highly used in fabrication of veneer and anterior crown restoration. Moreover, both of them can be repaired with resin composite using proper surface conditioning and adhesive system.

Repairing procedure

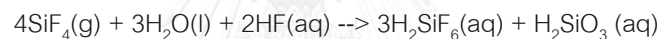
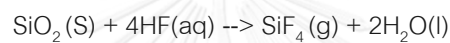
Previous literature from Sailer I et al., 2007 stated that fracture of porcelain is about 13.6% after 5 years of observation.(4) Sometimes fracture occurred within the first few months after permanent cementation of restoration. In order to avoid an expensive replacement, Intra-oral repair using light curing composite resins can be a reasonable alternative.

There are 3 factors leading to successful repairing procedure consisting of ceramic surface conditioning, silane coupling agent, and adhesive system.

Firstly, ceramic surface conditioning that has a great importance as it increases surface energy of the ceramic to exceed the critical surface energy. The concept of critical surface energy states that when the substrate surface energy is higher than the surface tension of the liquid, that liquid will spread spontaneously and readily onto that surface.(41) Moreover, this process also increases the surface roughness by creating micropores for infiltration of silanes and resin cements.

Surface conditioning can be done by many techniques. Sandblasting technique uses alumina particles with size around 50 μm with an air pressure of 380 kPa for 10-15 seconds in the perpendicular short distance (10 mm), resulting in attachment of particles on the surfaces. Then after silanization, $=\text{Al-O-Si}\equiv$ bonds will be formed. Nevertheless, these bonds are hydrolytically unstable.(21); Tribochemical silica coating uses high-energy coating to increase the surface energy of a ceramic surface. Example of device using in this method is Rocatec® system (3M ESPE, USA), which blasts silica-coated alumina particles. When these particles hit the surface, the temperature will increase up to 1,200°C due to the transfer of kinetic energy of the particles. Then the particles can be embedded on the surface, which will form covalent bond between silica-coated particle and resin composite after silanization. Moreover, this process also increases micromechanical retention for the resin.(13); For chemical treatment of ceramic materials in normal environment with humidity, oxide layers on the ceramic and metal surface will be mostly changed into the hydroxyl groups. Moreover, after surface conditioning with acid, density of the hydroxyl groups will be further increased, resulting in an increase of the linkage formed between ceramic surface and the silane coupling agent. These will improve the strength of adhesive layer between the silanized surface and the resin cements.(13)

Surface conditioning of dental ceramics with hydrofluoric acid gives higher bond strength when compared to the surface without acid conditioning. Tetrafluorosilane (SiF_4 (g)) will be formed when the porcelain surface is conditioned by hydrofluoric acid. Afterwards, tetrafluorosilane will be reacted with hydrofluoric acid forming a soluble hydrofluorosilicic acid (H_2SiF_6). This glassy matrix is easily dissolved and rinsed away. The chemical reactions of hydrofluoric acid with porcelain are as followed (13):



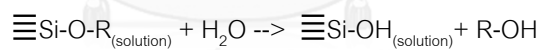
1.23% acidulated phosphate fluoride can also be used for etching ceramic surface.(12, 42)

Kukiattrakoon B. et al., 2007 found that shear bond strength between composite resin and ceramic after etching with 1.23% acidulated phosphate fluoride for 7-10 minutes was comparable to that obtained from 9.6% hydrofluoric acid for 4 minutes. SEM showed, however, that etching ceramic surface with 9.6% hydrofluoric acid for 4 minutes created greater surface roughness.(12)

The second factor of repairing procedure, silane coupling agents, played role as mediators raising the adhesion between organic and inorganic matrices through dual reactivity.

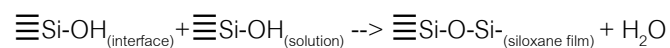
Organofunctional silanes contain two functional groups. The first one, $Y-(CH_2)_m-Si(OR)_3$, or OR, the hydrolysable functional group which reacted with the surface hydroxyl group of inorganic substrates (ceramic surface). The other side or Y was the non-hydrolysable organic group that had a carbon-carbon double bond, which reacted with the resin agents containing double bonds. Lastly, a linker, $-(CH_2)_m-$, is usually a propylene link.(21)

The silanization process started with the hydrolysable functional group that was activated by hydrolysis ($\equiv SiOR \rightarrow \equiv SiOH$) and, then they bonded with the substrate surface via -OH groups. Water was needed for the silanol conversion (hydrolysis).(21) The reaction mechanism is as presented:



$\equiv Si-OH$ will be embedded and polymerized on the substrate surface, hydrogen bonds and covalent

Si-O-Si bonds occurred during this process, called silanization.(21)



After silanization, a thin layer of hydrophobic and branched three-dimensional siloxane film will be created on a substrate surface. If it was too thick, it may cause a cohesive failure. On the other hand if this layer was too thin, it may lead to incomplete coverage of the silane layer on the ceramic

surface resulting in insufficient contact between the silanized inorganic surface and resin cement.(21)

The most widely used silane in dentistry is 3-methacryloxypropyltrimethoxy-silane (MTS). It can be in two forms either pre-hydrolysed (pre-activated) or two-bottle system, which will be activated by mixing for immediate hydrolysis in the clinical situations. In the past, pre-activated silanes had a short shelf life, however not in currently. Pre-activated silanes often diluted in ethanol-water solution, adjusting to pH 4-5 by acetic acid. Using low concentration of silane had been reported to increase the shelf life of the material to 2-3 years. Applying MTS after surface conditioning can improve the bond strength between many substrates such as ceramic-to-ceramic, metal to composite, composite-to-composite, and composite to ceramic. Reason for choosing MTS as dental silane is due to the compatibility of its functional methacrylate group with the dimetacrylates that used in the resin composites.(43)

The last factor, adhesive systems currently have been developed and can be bonded with all types of substrate, such as metal, resin composite, dentin, enamel, glass-ceramic, and zirconia. Ikemura et al., 2011 found that mixing of adhesive monomer with silane coupling agent had improved bond strength between glass-ceramic and resin adhesive. Moreover, adding 10-methacryloyloxydecyl 6,8 dithiooctanoate (10-MDDT) which is a one form of 10-MDP into the adhesive was also found to increase the bond strength with metal and zirconia.(44)

Chen L. et al., 2013 discovered that adding BisGMA into silane primer had caused the decreasing in shear bond strength between resin cement and lithium disilicate glass ceramic. This phenomenon might be the result from the low contact angle. Due to the need of water in order to form a stable siloxane (Si-O-Si) bond(45), bisGMA resin lowered the bond strength by retarding the water evaporation causing the inhibition of condensation reaction between silanol (Si-OH) of silane primer and the substrate (OH) of ceramic.(45)

Nowadays, universal adhesive from many manufacturers have been released into the market, for example, All-Bond Universal (Bisco, USA), Peak Universal Adhesive System (Ultradent, USA), and Single Bond™ Universal Adhesive (3M ESPE, USA). The manufacturers claimed that these adhesives could be used to bond many types of substrate, and also could be used as either etch-and-rinse approach or self-etching approach. According to table 1, due to the inclusion of silane and the MDP in the bottle, the manufacturer claimed that Single Bond™ Universal Adhesive is the only product that could bond glass-ceramic and zirconia without using any silane or zirconia primer.

Table 1: Products of universal adhesive

Adhesive system	Composition
1. All-Bond Universal (Bisco, USA)	1. Etchant Uni-Etch: 32% phosphoric acid, benzalkonium Chloride 2. Adhesive: MDP, bis-GMA, HEMA, ethanol, water, initiators
2. Peak Universal Adhesive System (Ultradent, USA)	1. Etchant: 35% phosphoric acid 2. Peak SE Primer: ethyl alcohol methacrylic acid, 2-hydroxyethyl methacrylate 3. Peak LC Bond resin: ethyl alcohol, 2-hydroxyethyl methacrylate
3. Single Bond™ Universal Adhesive (3M ESPE, USA)	1. Single Bond™ Universal Etchant: Etchant: 34% phosphoric acid, water, synthetic amorphous silica polyethylene glycol aluminium oxide. 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, filler, ethanol, water, initiators, silane

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; MDP, methacryloyloxydecyl

dihydrogenphosphate

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Bond strength test

The resin-ceramic bond strength can be measured by many methods such as tensile bond strength test, flexural bond strength test, or shear bond strength test, with the shear bond strength test being the most popular.(46)

Until the mid-nineties, tensile and shear bond strength tests were experimented in the specimens with a large bonded area, usually 3-6 mm in diameter ($\sim 7-28 \text{ mm}^2$). However, Sano et al. in 1994 found that specimens with large bonded area mostly had cohesive failure in dentin substrate at less than 5 MPa, which was significantly lower than tensile bond strength of the dentin that was previously reported to be in the range of 52-104 MPa.⁴⁷ The result might be due to the heterogeneity of the stress distribution at the bonded area. For the better result, using the specimens with smaller bonded area approximately $1.6-1.8 \text{ mm}^2$ was proposed. By using this smaller bonded area, failure of the specimens usually occurred at the adhesive interface instead. These new methods were called microtensile and microshear tests.⁽⁴⁸⁾

The shear bond strength test is a common laboratory technique for measuring the bond strength between resin-bonded ceramic restorations and ceramic repair systems. However, this technique is very sensitive to the method of application of the adhesive and design of testing technique. From these reasons they can lead to the results misinterpretation.⁽⁴⁶⁾

Della Bona and Van Noort found that when apply the force near the adhesive interface, the tensile stress would be initiated and caused crack or fracture at base of the specimen. These can cause the false interpretation as a cohesive failure. After test with FEA analysis, they also discovered uneven distribution of the force while testing shear bond strength test.⁽⁴⁶⁾

Preparation of specimens for microtensile testing is very technique sensitive requiring an experienced investigator. Specimens have to be cut into a number of slabs and further sectioned into a stick with approximately 0.5-1.5 mm thickness. Each stick composes of two substrates such as resin composite-dentin or resin composite-ceramic, which are bonded together and can be tested at the interface.(49) The shape of specimens can be prepared either in non-trimming bar-shape or trimmed with bur at the bonding site to create an hourglass profile which will reduce the bonding area and make the stress more concentrated at the bonding site.(49)

Aging process

Many techniques for aging the specimens before testing the bond strength were proposed. Examples are, storing in boiling water for 8 hours, soaking in citric acid, storing in water with room temperature for a period of time, and thermocycling. Among these techniques, the most popular are storing in water and thermocycling technique.(50)

Aging by storing in water, the specimens mostly stored in pure water at 37°C were utilized. The time can be varied from a few months up to 4-5 years (51), or may be longer. According to the ISO TR 11450 standard (1994), after 6 months storage in water at 37°C can cause a significant decrease in bond strength. Many studies showed significant decreasing in bond strength even with a

short storage period of 3 months.(52-53) This technique causes degradation of the interface from the hydrolysis process. Moreover, water can infiltrate into the polymer matrix leading to the swelling and breaking down between the polymer chains. This process is called plasticization.(54) In order to eliminate the effect of water, many chemicals were suggested to acidify in the storage solution, such as sodium azide(52), and chloramine.(55-56) Moreover, antibiotics were also proposed to prevent bacterial contamination.(53)

Thermocycling is another method used for aging the specimen. According to the ISO TR 11450 standard (1994), thermocycling term comprising of 500 cycles in water with temperature around 5 to 55°C is a suitable condition for aging test. Gale and Darvell, (1999) stated that 10,000 cycles of thermocycling could be compared to 1 year of function *in vivo*.(57) Degradation process from thermocycling can be caused in 2 ways, which are high temperature of water will increase the hydrolysis of the adhesive layer, by infiltrating and breaking down the polymer chains or poorly polymerized resin oligomers.(58), Due to the higher coefficient of thermal expansion of the restorative material when compared with the tooth tissue, repetition of cycles initiates the contraction/expansion stresses at the interface. The cracks along adhesive layer occurred due to these stresses.

Furthermore, gap can be created. Changing gap dimensions caused in- and outflow of oral fluids, a process known as percolation.(57)

A recent meta-analysis from Leloup et al., 2001 reviewing the literatures published between 1992 and 1996 showed that thermocycling method had no significant effect on the bond strength.(59) The results might be the consequences from using the ISO standard of 500 cycles by most of the studies in this meta-analysis, which was stated by Gale and Darvell to be too low for an aging effect to be obtained.(57)

Nikaido et al., 2002 showed that the thermocycling did not decrease the bond strength of flat resin-dentin μ TBS sticks. However, a significant decrease in bond strength was found when using similar method in the restored cavities.(60) On contrary, Xie et al., 2002 stated that thermocycling of resin-dentin μ TBS sticks with diffusion path less than 1 mm resulted in a significant decrease in bond strength.(61) Wolf D.M., et al., 1992 found that thermocycling for 1000 cycles decreased the tensile bond strength of composite bonded with ceramic disks.(62) Kato H et al., 1996 also found that shear bond strength of ceramic-resin based luting agent disks had been decreased after aging with thermocycling for 20,000 cycles.(63)

When the techniques of storing in water and thermocycling were compared, a study found that the decreasing in microtensile bond strength of ceramic-composite specimens storing in water for 150 days at 37°C has not been as much as the group aging with thermocycling for 37,500 cycles.(50)

MATERIALS AND METHODS

Operational Definition

1. IPS Empress[®] Esthetic (Ivoclar Vivadent, Germany) – Leucite reinforce glass ceramic
2. IPS e.max[®] Press (Ivoclar Vivadent, Germany) – Lithium disilicate glass ceramic
3. Filtek[™] Z350 XT (3M ESPE, USA) – Resin composite
4. RelyX[™] Ceramic Primer (3M ESPE, USA) – Silane
5. Single Bond[™] Universal Adhesive (3M ESPE, USA) – Dental Adhesive
6. Adper[™] Scotchbond[™] Multi-Purpose Adhesive (3M ESPE, USA) – Dental Adhesive
7. Model Repair II Blue (Dentsply, Japan) – Dental glue

Assumption

All specimens prepared in this study were fabricated according to the manufacturer's instruction. Only one researcher did the bonding procedure. Accordingly, the microtensile bond strength was depended only on the difference in technique of the adhesive systems and types of ceramic.

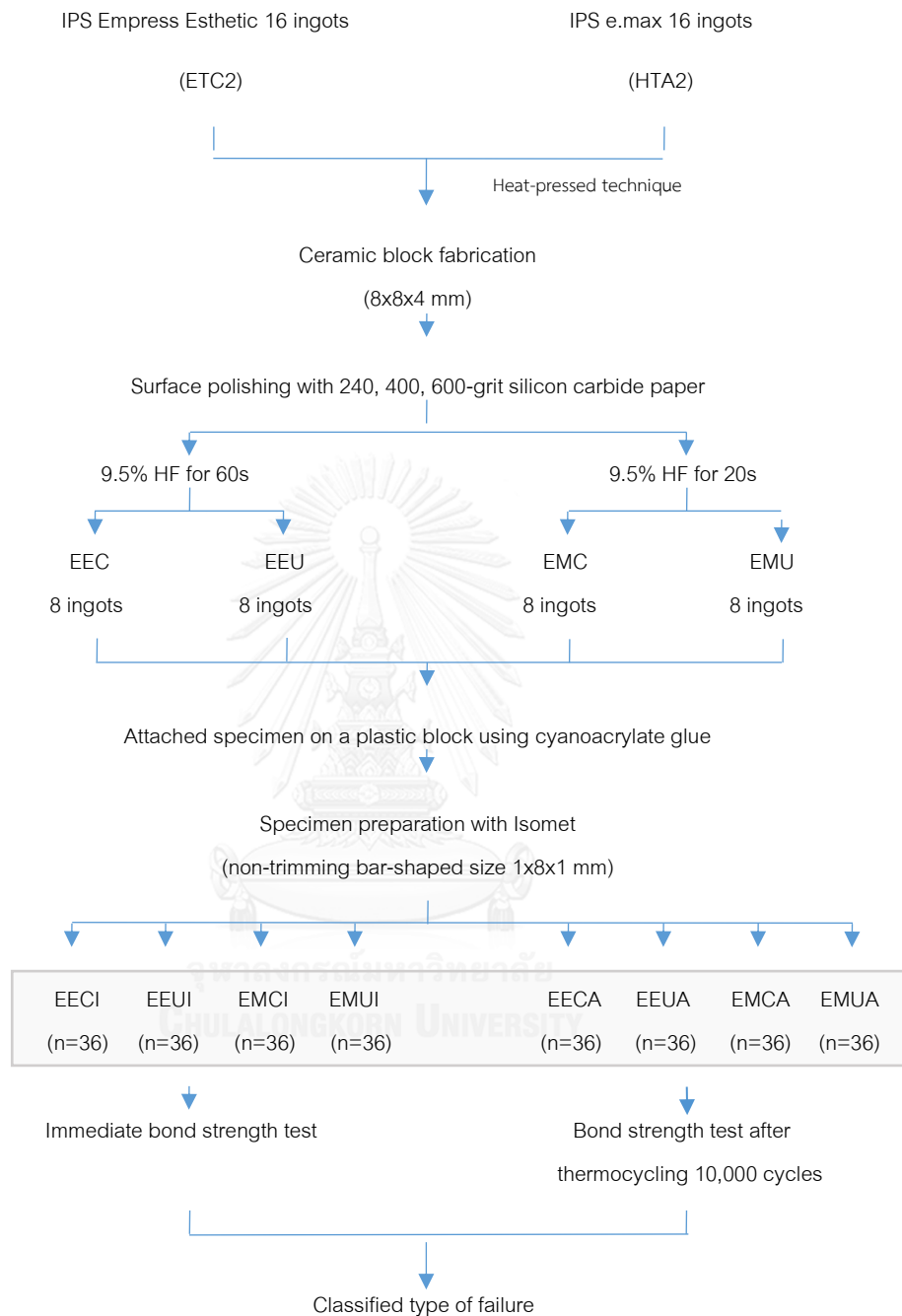
Research Design

This is an *in vitro* experimental study.

An intervention of this study was a universal adhesive, which contained adhesive and silane in one bottle. Dependent variable is microtensile bond strength tested until the specimens is cracked or fractured, measured in MPa.



Research Methodology



Abbreviations: EE, IPS e.max[®] press; EM, IPS empres[®] esthetic; C, conventional adhesive system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling 10,000 cycles before microtensile bond strength test

Figure 2: Diagram of study design

Subjects

Sample size calculation was done as the equation shown below;

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

The α and β values utilizing were 0.05 and 0.20 respectively.

The values of $\mu_1, \mu_2, \sigma_1, \sigma_2$ were selected from the result from pilot study as shown in table 2

Table 2: Mean microtensile bond strength and standard deviation from pilot study

Group	Mean microtensile bond strength (μ)	Standard diviation (σ)	Number of specimen
EECI	33.5	4.2	3
EEUI	25.3	5.7	6
EMCI	32.6	11.5	5
EMUI	34.3	5.2	12

Abbreviations: EE, IPS e.max[®] Press; EM, IPS Empress[®] Esthetic; C, conventional adhesive

system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling

10,000 cycles before microtensile bond strength test

The highest numbers of specimen were calculated from values of EECI and EMCI groups as shown in the equation below:

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

$$n_1 = \frac{(1.96 + 0.84)^2 [17.796 + 131.272 / (5/3)]}{(33.5 - 32.6)^2}$$

$$n_1 = \frac{(7.84) [17.796 + 78.606]}{(0.707)}$$

$$n_1 = 1069$$

36 numbers of specimens were selected for this study, due to the limitation of time and budget, the number of 1069 specimens is too high for this study. From pilot study, 9 specimens could be prepared from 1 ingot, thus, 4 ingots were used in each group.

Materials

Table 3: Materials used in this study

Materials	Manufacturer
IPS Empress [®] Esthetic ingots	Ivoclar Vivadent, Germany
IPS e.max [®] Press ingots	Ivoclar Vivadent, Germany
9.5% hydrofluoric acid	Ultradent, USA
Single Bond [™] Universal Adhesive	3M ESPE, USA
Adper [™] Scotchbond [™] Multi-Purpose Adhesive	3M ESPE, USA
Filtek [™] Z350 XT	3M ESPE, USA
RelyX [™] Ceramic Primer	3M ESPE, USA
A 240-, 400-, 600-grit silicon carbide paper	TOA, Germany
Model Repair II Blue	Dentsply, Japan

Apparatus

Table 4: Apparatus used in this study

Instrument	Manufacturer
Low-Speed Cutting Machine (Isomet [®] 1000)	Buehler, USA
Universal Testing Machine (EZ-S Shimadzu)	Shimadzu, Japan
Thermocycling Machine (TC 301)	Kmitl, Thailand
Grinder-Polisher Machine (Automet [®] 250)	Buehler, USA
Digital Vernier Caliper	Mitutoyo, Japan
Diamond Wafering Blade	Buehler, 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

Bonding technique

Table 5: Materials showing manufacturer, composition, and instructions for use

Material / Manufacturer	Composition	Procedure following manufacturer's instruction
RelyX™ Ceramic Primer (lot no. N636821, 3M ESPE, USA)	Methacryloxypropyl trimethoxysilane, ethanol, water	1) Apply 0.04 microliter of primer, measured by micropipette, on the ceramic surface in one direction 2) Allow it to react for 3 mins 3) Blow gently for 10 s, with 2-bar pressure, from 10-mm distance
Adper™ Scotchbond™ Multi-Purpose Adhesive (lot no. 596612, 3M ESPE, USA)	Adhesive: Bis-GMA, HEMA, EMAB, dimethacrylate, initiators	1) Apply 0.04 microliter of adhesive, measured by micropipette, on the ceramic surface in one direction 2) Light-cure for 10 s
Single Bond™ Universal Adhesive (lot no. N553960, 3M ESPE, USA)	Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1) Apply 0.04 microliter of adhesive, measured by micropipette, in one direction and rub it for 20 s on the ceramic surface 2) Blow gently until no movement of liquid with 2-bar pressure, from 10-mm distance 3) Light-cure for 10 s

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate

Experimental groups

Table 6: Experimental groups and their details

Group	Method	Aging process
Group 1 EECI	IPS Empress [®] Esthetic, 9.5% HF + RelyX [™] Ceramic Primer + Adper [™] Scotchbond [™] Multi-Purpose Adhesive + Filtek [™] Z350 XT	Immediate bond strength (24 hours after water storage at 37°C)
Group 2 EEUI	IPS Empress [®] Esthetic, 9.5% HF + Single Bond [™] Universal Adhesive + Filtek [™] Z350 XT	Immediate bond strength (24 hours after water storage at 37°C)
Group 3 EMCI	IPS e.max [®] Press, 9.5% HF + RelyX [™] Ceramic Primer + Adper [™] Scotchbond [™] Multi-Purpose Adhesive + Filtek [™] Z350 XT	Immediate bond strength (24 hours after water storage at 37°C)
Group 4 EMUI	IPS e.max [®] Press, 9.5% HF + Single Bond [™] Universal Adhesive + Filtek [™] Z350 XT	Immediate bond strength (24 hours after water storage at 37°C)
Group 5 EECA	IPS Empress [®] Esthetic, 9.5% HF + RelyX [™] Ceramic Primer + Adper [™] Scotchbond [™] Multi-Purpose Adhesive + Filtek [™] Z350 XT	Bond strength test after thermocycling for 10,000 cycles
Group 6 EEUA	IPS Empress [®] Esthetic, 9.5% HF + Single Bond [™] Universal Adhesive + Filtek [™] Z350 XT	Bond strength test after thermocycling for 10,000 cycles
Group 7 EMCA	IPS e.max [®] Press, 9.5% HF + RelyX [™] Ceramic Primer + Adper [™] Scotchbond [™] Multi-Purpose Adhesive + Filtek [™] Z350 XT	Bond strength test after thermocycling for 10,000 cycles
Group 8 EMUA	IPS e.max [®] Press, 9.5% HF + Single Bond [™] Universal Adhesive + Filtek [™] Z350 XT	Bond strength test after thermocycling for 10,000 cycles

Abbreviations: EE, IPS e.max[®] Press; EM, IPS Empress[®] Esthetic; C, conventional adhesive

system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling

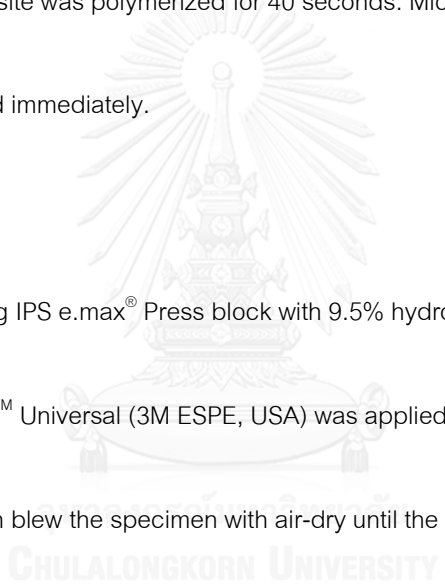
10,000 cycles before microtensile bond strength test

Definition of specimen group

Group EECl: After etching IPS Empress® Esthetic block with 9.5% hydrofluoric acid (Ultradent, USA) for 60 seconds, ceramic primer (3M ESPE, USA) was applied on the specimen surface and blown with air-dry until dry. Then, Adper™ Scotchbond™ Multi-Purpose Adhesive (3M ESPE, USA) was applied in a thin layer and light-cured for 10 seconds before building up resin composite Filtek™ Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was then tested immediately.

Group EEUI: After etching IPS Empress® Esthetic block with 9.5% hydrofluoric acid (Ultradent, USA) for 60 seconds, Single Bond™ Universal (3M ESPE, USA) was applied in a thin layer and allowed to react for 20 seconds, then blew the specimen with air-dry until the liquid is no longer moved. Each specimen was light-cured for 10 seconds before building up resin composite Filtek™ Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was then tested immediately.

Group EMCI: After etching IPS e.max[®] Press block with 9.5% hydrofluoric acid (Ultradent, USA) for 20 seconds, ceramic primer (3M ESPE, USA) was applied on the specimen surface and blown with air-dry until dry. Then, Adper[™] Scotchbond[™] Multi-Purpose Adhesive (3M ESPE, USA) was applied in a thin layer and light-cured for 10 seconds before building up resin composite Filtek[™] Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was then tested immediately.



Group EMUI: After etching IPS e.max[®] Press block with 9.5% hydrofluoric acid (Ultradent, USA) for 20 seconds, Single Bond[™] Universal (3M ESPE, USA) was applied in a thin layer and allowed to react for 20 seconds, then blew the specimen with air-dry until the liquid is no longer moved. Each specimen was light-cured for 10 seconds before building up resin composite Filtek[™] Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was then tested immediately.

Group EECA: After etching IPS Empress® Esthetic block with 9.5% hydrofluoric acid (Ultradent, USA) for 60 seconds, ceramic primer (3M ESPE, USA) was applied on the specimen surface and blown with air-dry until dry. Then, Adper™ Scotchbond™ Multi-Purpose Adhesive (3M ESPE, USA) was applied in a thin layer and light-cured for 10 seconds before building up resin composite Filtek™ Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was measured after aging with thermocycling for 10,000 cycles

Group EEUA: After etching IPS Empress® Esthetic block with 9.5% hydrofluoric acid (Ultradent, USA) for 60 seconds, Single Bond™ Universal (3M ESPE, USA) was applied in a thin layer and allowed to react for 20 seconds, then blew the specimen with air-dry until the liquid is no longer moved. Each specimen was light-cured for 10 seconds before building up resin composite Filtek™ Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was measured after aging with thermocycling for 10,000 cycles

Group EMCA: After etching IPS e.max[®] Press block with 9.5% hydrofluoric acid (Ultradent, USA) for 20 seconds, ceramic primer (3M ESPE, USA) was applied on the specimen surface and blown with air-dry until dry. Then, Adper[™] Scotchbond[™] Multi-Purpose Adhesive (3M ESPE, USA) was applied in a thin layer and light-cured for 10 seconds before building up resin composite Filtek[™] Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was measured after aging with thermocycling for 10,000 cycles

Group EMUA: After etching IPS e.max[®] Press block with 9.5% hydrofluoric acid (Ultradent, USA) for 20 seconds, Single Bond[™] Universal (3M ESPE, USA) was applied in a thin layer and allowed to react for 20 seconds, then blew the specimen with air-dry until the liquid is no longer moved. Each specimen was light-cured for 10 seconds before building up resin composite Filtek[™] Z350XT shade A4 (3M ESPE, USA) into dimensions of 8x8x4 mm using silicone mold, which each 2-mm increment of resin composite was polymerized for 40 seconds. Microtensile bond strength of each specimen was measured after aging with thermocycling for 10,000 cycles

Methods

16 ingots of leucite-reinforced glass ceramic (IPS Empress[®] Esthetic, ETC2; EE; Ivoclar Vivadent, Germany) and 16 ingots of lithium-disilicate (IPS e.max[®] Press, HTA2; EM; Ivoclar Vivadent, Germany) were fabricated into ceramic blocks with dimensions of 8x8x4 mm (Fig. 3A) using a heat-pressed technique according to manufacturer's instructions. The ceramic blocks were placed in stainless steel holder and passively polished with 240-, 400-, and 600-grit silicon-carbide paper (TOA, Germany) through running water for 2 minutes each, respectively, by grinder-polisher machine (Automet[®] 250; Buehler, USA). All the ceramic surfaces were treated with 9.5% hydrofluoric acid (Ultradent[®] Porcelain Etch; Ultradent, USA), 60 seconds for EE and 20 seconds for EM. Resin composite (Filtek[™] Z350 XT, shade A4; 3M ESPE, USA) was used as a build-up material using a silicone mold with dimensions 8x8x4 mm (Fig. 3B), then bonded to the treated ceramic surface according to the manufacturer's instructions. The dimensions of the final specimens of ceramic bonded with resin composite were 8x8x8 mm. Each 2-mm increment was polymerized using a LED light-curing system (Demi[™] Plus; Kerr, USA) with 1,100 mW/cm² intensity for 40 seconds (Fig. 3D). The light guide was held perpendicularly 1 mm above the silicone mold. Light output from the light-polymerizing unit was checked by a radiometer (Model 100 Optilux; Kerr, USA) throughout the experiment.

Following the bonding procedures in Table 5, the specimens were stored in water at 37°C for 24 hours. Thereafter, the specimens were sectioned into slabs with dimensions 1x8x8 mm (Fig. 3E). The specimen was prepared into 1x1x8 mm non-trimming bar-shaped beams. (Fig. 3F), using a low-speed cutting machine at a speed of 350 rpm and loading of 150 g (Isomet[®] 1000, Buehler, USA) with constant water spray. The bonded specimens were then divided into 8 groups, with 36 specimens in each group according to ceramic type, bonding technique and storage condition. Details of all the groups are shown in table 6.

Specimens were then stuck on an experimental jig for microtensile testing using cyanoacrylate glue (Model Repair II Blue; Dentsply, USA) (Figure 3G). The μ TBS test was performed using a universal testing machine (Shimadzu, Japan) with cross-head speed 1 mm/min and data were recorded in MPa.

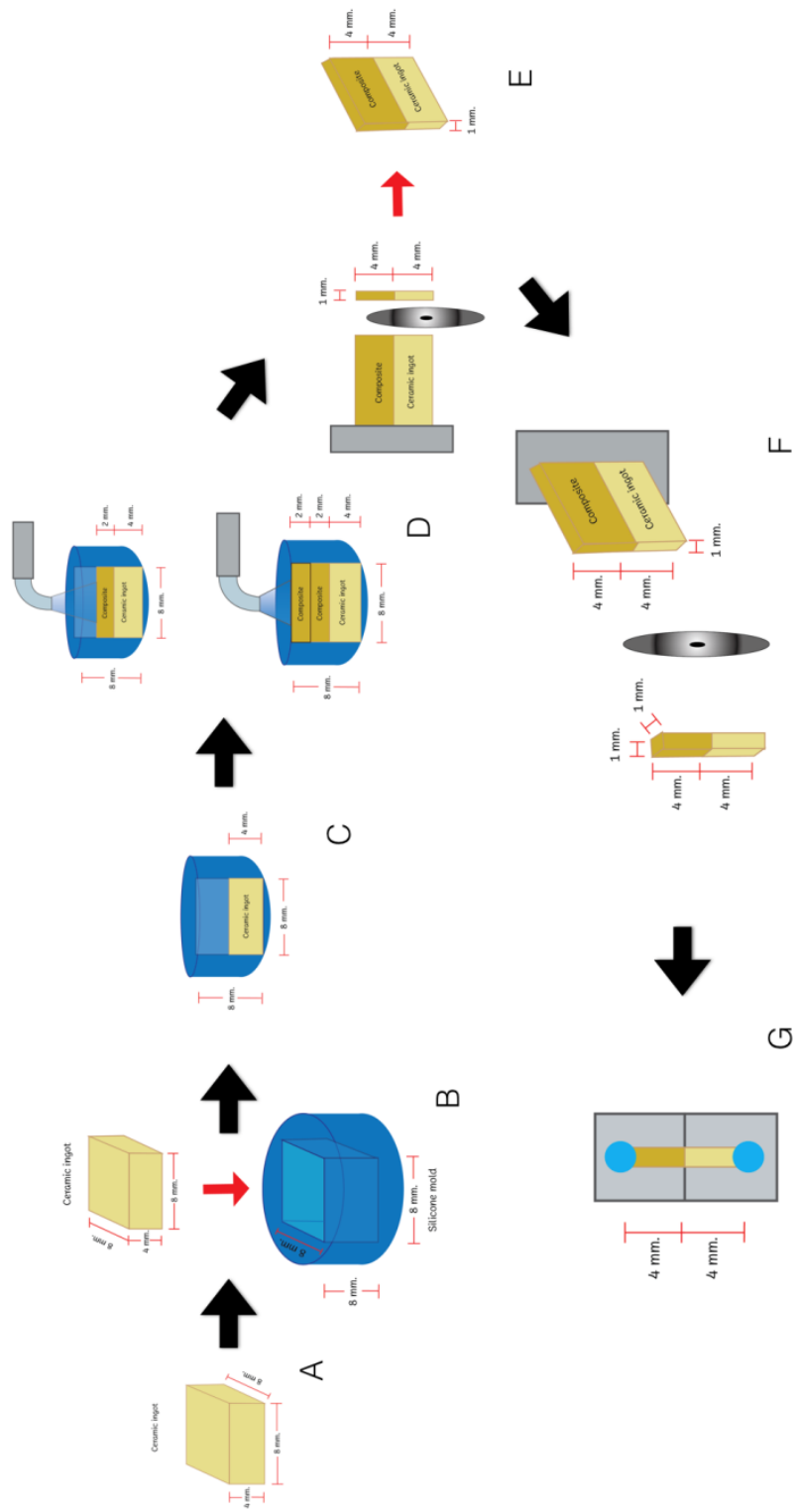


Figure 3A-G: *Diagram of sample preparation*

3A) Surfaces of the ceramic ingot with dimensions of 8x8x4 mm prepared by silicon carbide paper of various grit, 9.5% hydrofluoric acid, followed by the adhesive procedure according to the manufacturer's instructions.

3B, C) The ceramic ingot was seated inside the silicone mold with 4 mm space for further resin composite build up.

3D) Each 2 mm-layer of resin composite was cured by a light-curing unit for 40 seconds.

3E) The specimen was then stuck to a plastic block and cut into a slab with dimensions of 1x8x8 mm by a diamond wafering blade.

3F) The slab was cut into non-trimming bar-shapes with dimensions of 1x1x8 mm using a diamond wafering blade.

3G) The bar-shape specimen was stuck to an experimental jig for microtensile testing using cyanoacrylate glue.

The mode of failure was determined using a stereomicroscope (ML 9300; MEIJI, Japan) at a magnification of 40x, and classified into one of four categories as follows:

Type I: Adhesive failure – fracture occurred at the resin-ceramic interface (>50% of failure between resin-ceramic interface)

Type II: Cohesive failure in resin composite – fracture occurred within the resin composite layer (>50% of failure within the resin composite)

Type III: Cohesive failure in ceramic – fracture occurred within the ceramic layer (>50% of failure within the ceramic)

Type IV: Mixed failure – fracture occurred involving both the resin-ceramic interfaces and the neighboring substrates

If any of the specimens were broken prior to test, the bond strength value was recorded as a minimum μ TBS of each group.(64-65)

Possible Impediments and Solutions

In order to control the quality of bonding technique, one researcher performed the whole procedure of sample preparation: cutting specimen, bonding procedure, microtensile bond strength testing.

Data Collection and Statistical Analysis

Data were analyzed using statistical software (IBM SPSS Statistics 20, SPSS). Mean μ TBS values were collected and analyzed by three-way ANOVA followed by a Bonferroni post hoc test.

Results with p-value < 0.05 were considered statistically significant.



Present Study



Figure 4



Figure 5

Figure 4: IPS Empress[®] Esthetic using ingot shade ETC2 dimension of 8x8x4 mm

Figure 5: IPS e.max[®] Press using ingot shade HTA2 dimension of 8x8x4 mm



Figure 6



Figure 7

Figure 6: Dimension of ceramic ingots was confirmed by digital vernier caliper (Mitutoyo, Japan).

Figure 7: Light-curing unit with 1,100 mW/cm² intensity was checked with Radiometer (Kerr, USA).



Figure 8: Grinder-Polisher Machine (AutoMet[®] 250)



Figure 9: Polishing ceramic block with a 240, 400, 600-grit silicon carbide paper

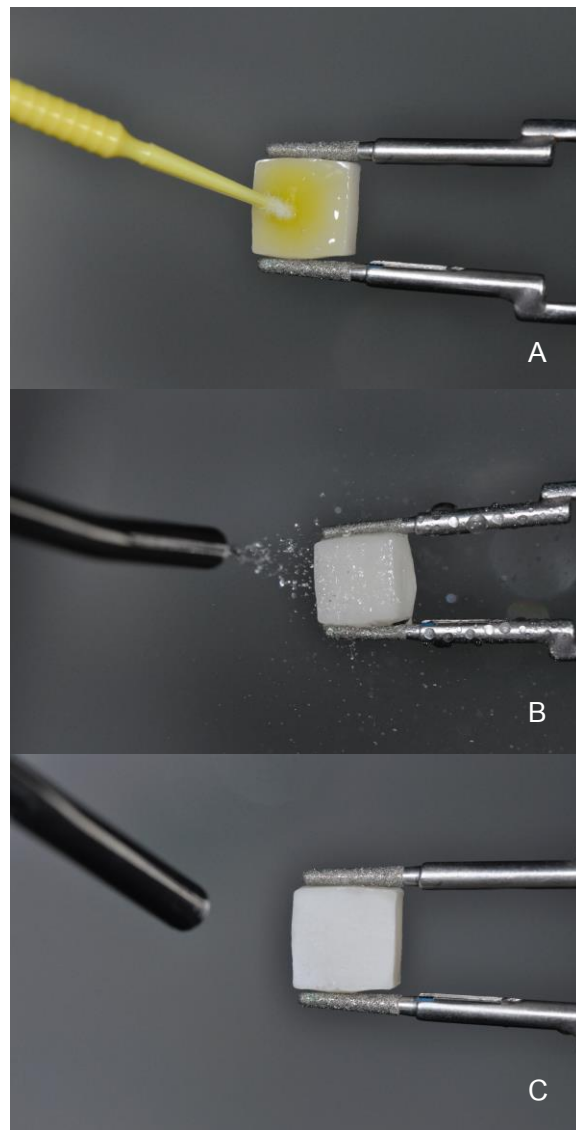


Figure 10

Figure 10: A) 9.5% hydrofluoric acid was applied on the ceramic surface (20 seconds for IPS e.max[®]

Press, 60 seconds for IPS Empress[®] Esthetic).

B) Ceramic surface was thoroughly rinsed with water spray with 10 mm distance from the surface for 10 seconds.

C) Dry ceramic surface with air blow until the surface showed chalky appearance.

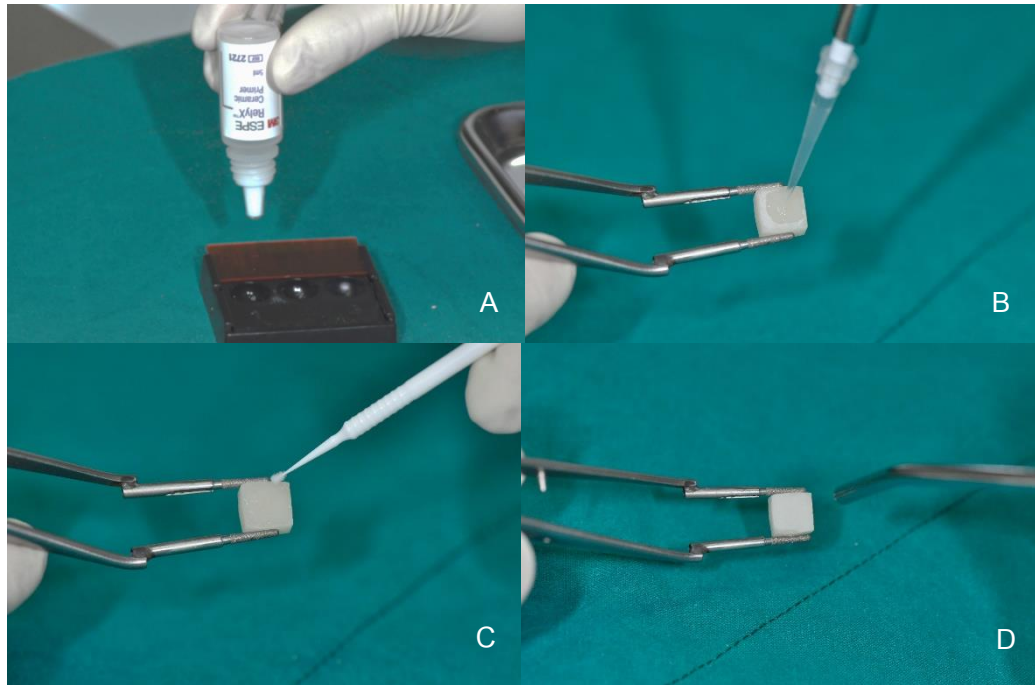


Figure 11

Figure 11: A) One drop of silane was prepared while the bottle perpendicular with the 3-Well Mixing.

B) Silane primer was dropped on the ceramic surface using micropipette.

C) Silane primer was applied using microbrush in one direction.

D) Gently air blow until the ceramic surface was dry.



Figure 12: The amount of silane primer and adhesive agent was controlled about 0.04 microliter using micropipette.

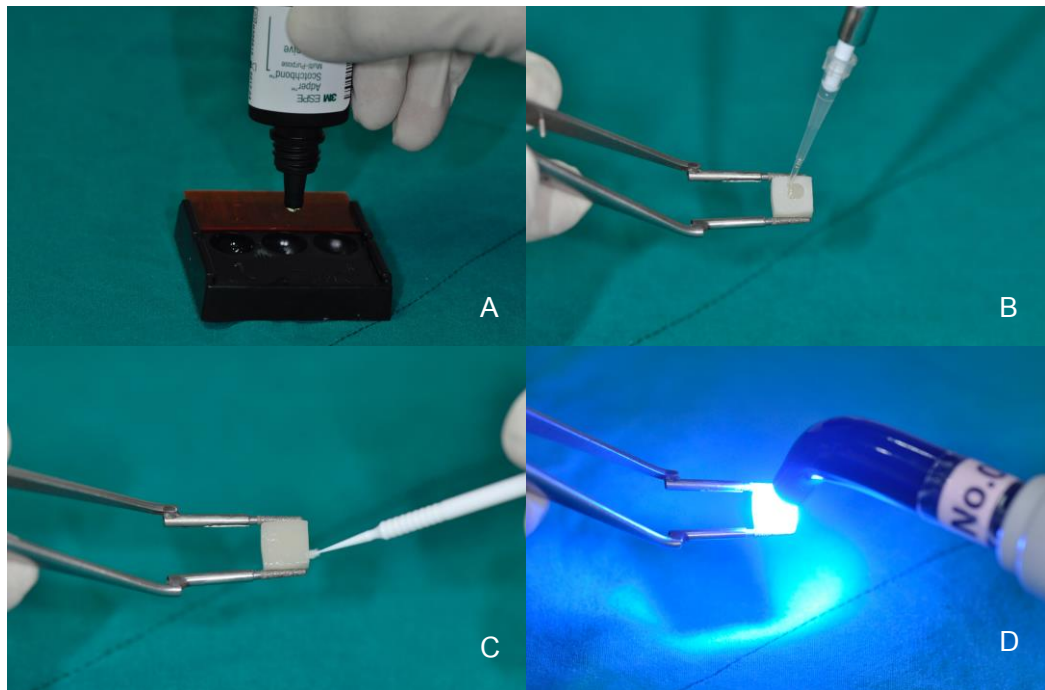


Figure 13

Figure 13: A) One drop of conventional adhesive was prepared while the bottle perpendicular with the 3-Well Mixing.

B) Conventional adhesive was dropped on the ceramic surface using micropipette.

C) Adhesive agent was applied using microbrush in one direction.

D) Light-cured for 10 seconds according to manufacturer's instruction.



Figure 14: One drop of universal adhesive was prepared while the bottle perpendicular with the 3-Well Mixing.

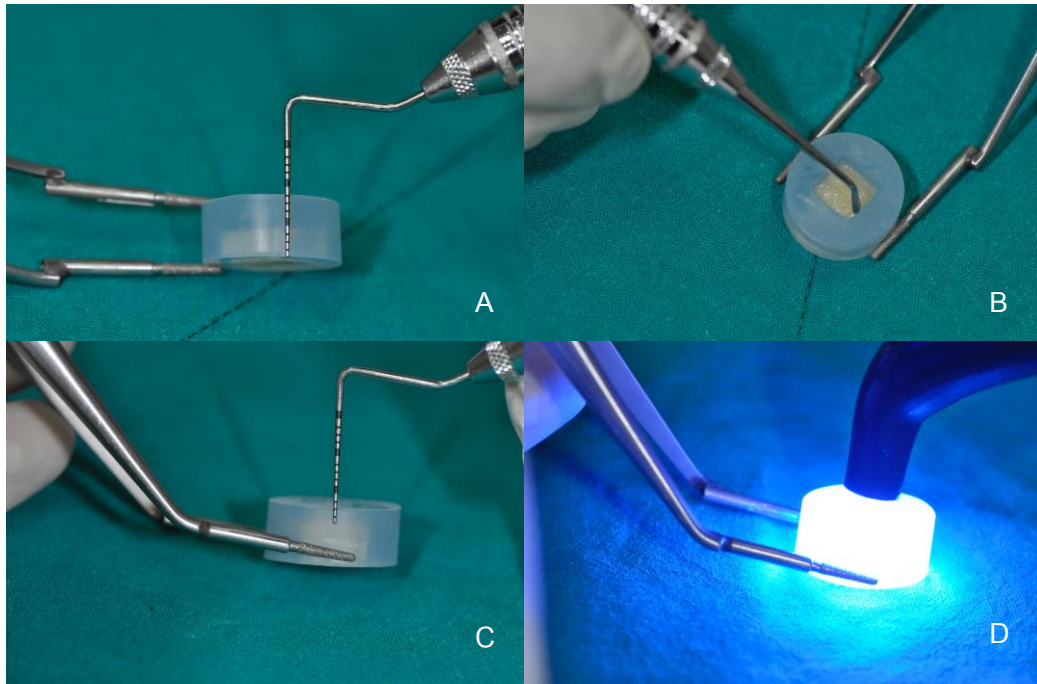


Figure 15

Figure 15: A) Periodontal probe showed 4 mm left as a space for resin composite build up.

B) Resin composite was then filled and plugged by hand instrument.

C) After filled the first layer, periodontal probe showed 2 mm left as a space for second layer.

D) The first layer of resin composite was light-cured for 40 seconds.

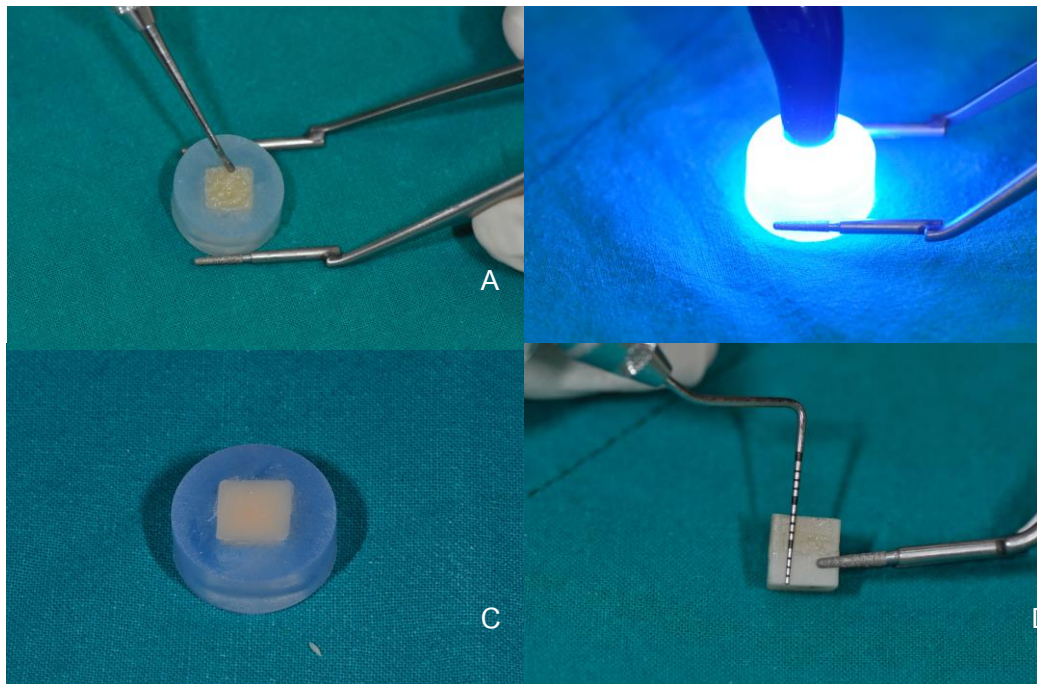


Figure 16

Figure 16: A) Resin composite was then filled and plugged by hand instrument.

B) The second layer of resin composite was light-cured for 40 seconds.

C) The resin-ceramic specimen with dimensions of 8x8x8 mm was cured with light-curing unit in silicone mold.

D) From periodontal probe showed the resin-ceramic specimen with dimensions of 8x8x8 mm

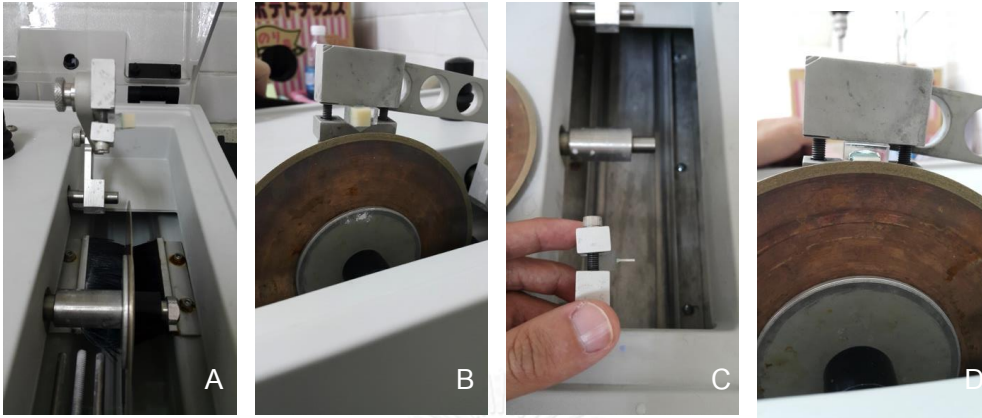


Figure 17

Figure 17: A) Resin-ceramic specimen was attached with plastic block using cyanoacrylate glue

(Model repair II blue, Dentsply, USA).

B) Specimen block size 8x8x8 mm was cut into slab size 8x8x1 mm by low-speed cutting machine (ISOMET[®] 1000, Buehler, USA).

C) Resin-ceramic slab using resin-ceramic side was attached to the plastic block.

D) Specimen slab size 8x8x1 mm was cut into slab size 1x8x1 mm by low-speed cutting machine (ISOMET[®] 1000, Buehler, USA).



Figure 18: Stereomicroscope ML 9300 (MEIJI, Japan)



Figure 19: Bonded area was measured by stereomicroscope (MEIJI, Japan)



Figure 20

Figure 20: A) Figure showed specimen dimension 1x8x1 mm for microtensile bond strength test.

B) Microtensile bond strength was tested using universal testing machine (EZ-S Shimadzu, Japan).

C) Specimen was broken at resin-ceramic interface.



Figure 21: Thermocycling Machine TC 301 (Kmitl, Thailand)

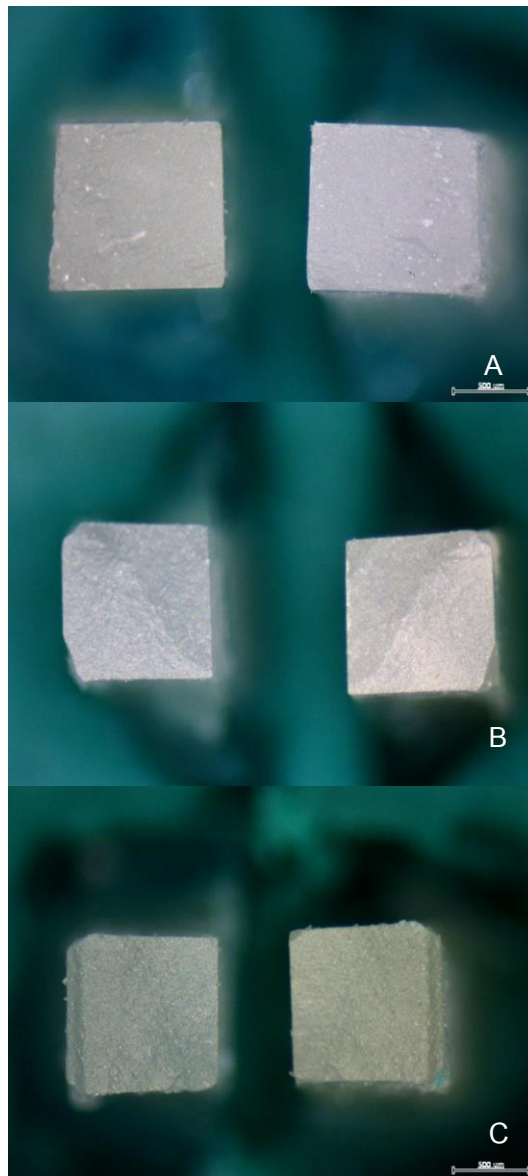


Figure 22

Figure 22: A) The fracture occurred at the resin-ceramic interface (adhesive failure).

B) The fracture occurred within the resin composite layer (cohesive failure).

C) The fracture occurs involving both of resin-ceramic interfaces and also neighboring substrates.

RESULTS

A Shapiro-Wilk's test ($p > 0.05$) and a visual inspection of their histograms, normal Q-Q plots, and box plots showed that the mean μ TBS in all tested groups was approximately normally distributed.

There were no pre-test failures in any group. Mean values of the microtensile bond strength of each group are shown in Table 7. The highest mean microtensile bond strength was recorded in the EMCI group and the lowest in the EEUA group.

Table 7: Mean Microtensile Bond Strength and Number of Specimens

Group	Mean Microtensile Bond Strength (MPa)	Standard Deviation	Number of Specimens
EECI	28.2 ^{B,C}	10.5	36
EECA	26.7 ^C	8.9	36
EEUI	23.6 ^{C,D}	8.6	36
EEUA	20.4 ^D	7.0	36
EMCI	38.3 ^A	13.9	36
EMCA	34.3 ^{A,B}	12.2	36
EMUI	25.3 ^C	6.6	36
EMUA	24.9 ^C	8.1	36

Abbreviations: EE, IPS e.max[®] Press; EM, IPS Empress[®] Esthetic; C, conventional adhesive system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling 10,000 cycles before microtensile bond strength test

The overall values of μ TBS are shown in Table 7. EMCI and EMCA groups showed significantly highest mean μ TBS among the tested group ($P < 0.05$), following with EECI, EECA, EMUI, EMUA and EEUI groups respectively ($P < 0.05$). EECI group was not significant different from EMCA group ($P > 0.05$), and the lowest μ TBS was found in EEUA group ($P < 0.05$).

Three-way ANOVA results indicated a significant interaction between “bonding” and “ceramic type” ($p = 0.013$) (Fig. 24). Thus, the main effect of the two factors could not be tested. For bonding and ceramic type, EM and EE groups with conventional bonding gave significantly higher μ TBS than groups using universal bonding ($p < 0.05$). The effectiveness of the conventional adhesive system was higher when using EM ceramic type than EE ceramic type; however, there was no such correlation between the universal bond groups (Fig. 24).

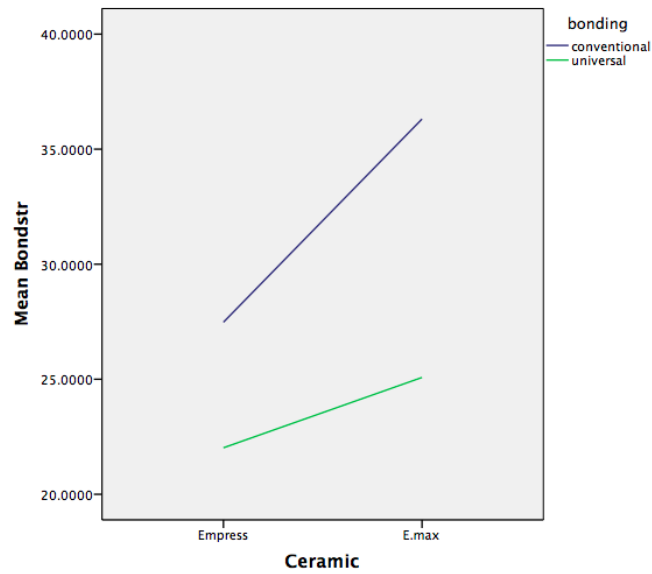


Figure 23: There was an interaction between the factors “bonding” and “ceramic type” (P-value = 0.013).

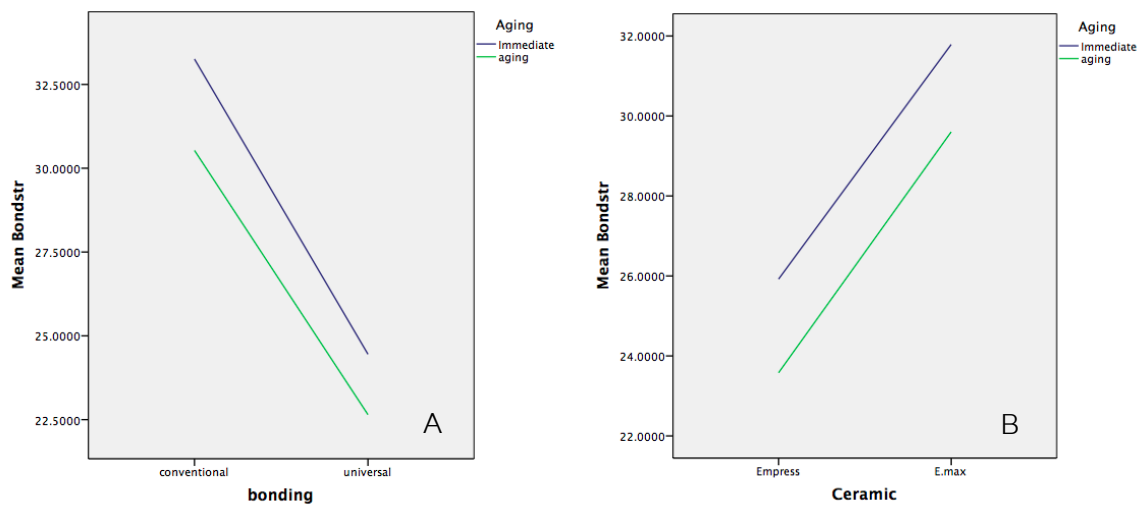


Figure 24A-B: A showed no interaction between “aging” factor and “bonding” (P-value = 0.689).

B showed no interaction between “aging” factor and “ceramic type” (P-value = 0.946).

“Aging” was the only factor that did not show any interaction with the others (Fig. 25A-B).

Comparing the μ TBS between the “immediate” and “aging” groups (Table 8), No significant difference of μ TBS was observed between the groups.

Table 8: μ TBS between “Immediate” and “Aging” groups

Group	N	Mean (MPa)	Standard Deviation
Immediate	144	28.9	11.6
Aging (Thermocycling 10,000 cycles)	144	26.6	10.4

The percentages of failure modes were investigated using a stereomicroscope at 40X magnification. The majority of the failures were adhesive failure between the resin composite and the ceramic (83.34%), followed by cohesive failure in the resin composite (9.02%), and mixed failure between the resin composite and the adhesive layer (7.64%) (Table 9).

Table 9: Mode of failure

Group	Mode of failure			
	Adhesive	Cohesive (composite)	Cohesive (ceramic)	Mixed failure
EECI	30	3	0	3
EECA	26	5	0	5
EEUI	22	6	0	8
EEUA	35	1	0	0
EMCI	28	5	0	3
EMCA	27	6	0	3
EMUI	36	0	0	0
EMUA	36	0	0	0
Total	240 (83.34%)	26 (9.02%)	0 (0%)	22 (7.64%)

Abbreviations: EE, IPS e.max[®] Press; EM, IPS Empress[®] Esthetic; C, conventional adhesive

system; U, universal adhesive system; I, immediate microtensile bond strength test; A, thermocycling

10,000 cycles before microtensile bond strength test

DISCUSSION AND CONCLUSIONS

Discussion

Results indicated that the mean μ TBS from the universal adhesive group (Single Bond™ Universal) was low compared with the conventional adhesive system; thus, the first hypothesis, presented that there was no difference in μ TBS between repaired ceramic using resin composite with a universal adhesive and conventional adhesive system, was rejected. Silane is known to promote wettability and form flexible siloxane bonds; with one side, the non-hydrolysable group reacting with the carbon-carbon double bond in the resin composite, and the other side, the hydrolysable group reacting with the hydroxyl group on the ceramic surface.(21, 66) The incorporation of silane is found in Single Bond™ Universal as claimed by the manufacturer. However, there are some studies reported that universal bonding systems containing water and acidic agent caused dehydration condensation of silane (16, 66-67), which did not bond with the glass phase of the ceramic surface, causing bond strength reduction.(13, 21) Similarly to the study from Kim R. *et al.* (2015) which found that the microshear bond strength of Single Bond™ Universal was not significantly different from that of All-Bond Universal (Bisco, USA), despite the fact that the latter does not contain silane. The microshear bond strength of the two universal adhesives was also lower when compared with conventional adhesive.(16) It was also supported by Yoshihara *et al.* (2016),

reporting that when using universal adhesive, the silica glass plate showed lower shear bond strength compared to the fresh silane and bonding agent group.(66) They noted that a suitable pH for silane was 4.6, but the pH of the universal bonding agent was acidic at 2.7 and possibly caused the silane solution to become unstable and inactive faster with a correspondingly shorter shelf life.(66) A stable siloxane bond requires water for condensation between silanol and the OH group.(21) However, Chen *et al.* (2013) determined that the condensation reaction was inhibited by Bis-GMA in the universal adhesive which slowed down water evaporation. Thus, bond strength and stability of the universal adhesive were inferior compared to conventional adhesive.(45)

It was well-established in many previous studies that surface treatment with 9.5% hydrofluoric acid following by applying silane when repairing ceramic with resin composite provided the most effective result. The interest of this study was focused on the performance of Single Bond™ Universal, which claimed to have included silane in one bottle, compared with gold standard technique using separated silane and hydrophobic adhesive. Applying only 9.5% hydrofluoric acid or only silane were not included as negative control groups since the effect of hydrofluoric acid or silane alone was not considered in this study.

An aging process utilizing thermocycling affected the μ TBS of ceramics repaired by resin composite.(22, 63, 68-69) Some studies indicated no significant difference of μ TBS between testing

groups aged using thermocycling.(70-71) ISO TR 11450 standard (1994) states 500 cycles of thermocycling in water between 5°C and 55°C as a suitable condition for the aging test. Gale and Darvell (1999) found that 10,000 cycles of thermocycling were comparable to one year of function *in vivo*.(57) In this study, 10,000 cycles of thermocycling with dwell time of 60 seconds (5°C, 35°C, 55°C, and 35°C for 5, 25, 5, and 25 seconds, respectively) were used to test the performance of the two adhesive systems. Results showed no significant differences from thermocycling on μ TBS between the “immediate” and “aging” groups ($p = 0.083$). Therefore, the second hypothesis stating there was no difference in μ TBS between aged and non-aged groups of repaired ceramics using different adhesive systems was accepted. Moreover, Foxton *et al.* (2002) stated that hydrolytic degradation weakened the bonding interface after water storage for six weeks(72); therefore, the aging process used here may not be adequate since the actual storage time was only 10 days.(73)

The EMC group recorded a statistically significant higher μ TBS than the EEC group ($p < 0.001$) for repaired ceramics using conventional adhesive; however, this trend was not found in the universal adhesive system. Della Bona *et al.* (2003) reported that the μ TBS of lithium disilicate treated with 9.6% hydrofluoric acid followed with silane was higher compared with leucite-reinforced ceramic under the same conditions as HF did more damage to leucite-reinforced ceramic surface than that of lithium disilicate. As hydrofluoric acid targeted more at the interface between leucite and glass

phase, leaving the remaining glass phase impaired, causing μ TBS of leucite-reinforced ceramic to be lower.(74)

Shear and μ TBS tests are common techniques used for measuring adhesive materials. But the advantages of microtensile bond strength test over shear bond strength test is that stress distribution is more focused in adhesive interface, causing adhesive failure, due to its smaller bonding area(49) which is also supported by Della Bona and Van Noort (1995). Their study found that tensile stress occurring near the adhesive interface initiated cracks or fractures at the base of the specimen when conducting the shear bond strength test and these caused misinterpretation as a cohesive failure. Moreover, finite element analysis (FEA) revealed an uneven distribution of force applied by the shear bond strength test.(46) The preparation of specimens for microtensile testing is very technique-sensitive requiring an experienced investigator, meaning they need to be cut into slabs with thicknesses ranging 0.5-1.5 mm in order to have small bonding area. Shape also affected the testing results which hourglass shapes provide more accurate μ TBS values than those with non-trimmed bar-shaped but require a more complex preparation method.(49)

Results showed that the most common modes of failure of both ceramics repaired by conventional and universal adhesive systems were adhesive failures (83.34%). Thus, the mean values of μ TBS for ceramics repaired by both adhesive systems were lower than the tensile strengths

of the ceramics and resin composites. Specimens of ceramics repaired by the conventional adhesive system showed cohesive and mixed failure in greater numbers than those repaired by the universal adhesive system. The mean values of μ TBS for the conventional adhesive system were higher than those of the universal adhesive system. Therefore, repairing ceramics using the former was considered to be more effective than using the latter.

Aging processes had no effect on mode of failure in the experimental groups, except for the EEU group. Before aging, the EEUI group showed dominant cohesive and mixed failure of the specimens. After aging, specimens in the EEUA group broke at the adhesive layer, indicating that the immediate bond strength of repaired EE ceramics using universal adhesive systems was effective; however, the thermocycling process reduced the bonding ability resulting in adhesive failure.

Pretest failures could be dealt with in many ways. When pretest failures were excluded from sum of bond strength value, the mean μ TBS would be overvalued.(75) On the other hand, assigning them the value of zero would minimize the mean μ TBS.(76-77) In this study, they were assigned as minimum μ TBS of each group, so the mean μ TBS stayed in normal level.(64-65)

Suggested further studies

Since 10,000 cycles of thermocycling could not produce any difference in repairing performance between 2 adhesive systems, alternative aging process is suggested. Further study could be done with increased cycles of thermocycling and/or including water storage. Moreover, study investigating the repaired ability of both adhesive systems on recently launched ceramic should be conducted.

Conclusion

Within the limitations of this study, the following conclusions can be drawn. Higher microtensile bond strength between a resin composite and ceramic was achieved using a conventional adhesive system compared to a universal adhesive system. This advantage was distinct in the lithium disilicate group. The aging process utilizing 10,000 cycles of thermocycling did not affect the microtensile bond strength of repaired ceramic using resin composites and adhesive systems

Clinical implication

After 10,000 thermal cycles, both universal and conventional adhesive systems demonstrated acceptable reparability. However, repairing ceramic with resin composite by treating ceramic surface with 9.5% hydrofluoric acid and using conventional adhesive technique may provide favorable results, especially with lithium-disilicate glass ceramic.

Declaration of Conflicting Interest

The authors declare that there is no conflict of interest.



REFERENCES



REFERENCES

1. Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. *J Prosthet Dent* 2007;98:389-404.
2. Fradeani M, Redemagni M. An 11-year clinical evaluation of leucite-reinforced glass-ceramic crowns: a retrospective study. *Quintessence Int* 2002;33:503-10.
3. Wolfart S, Eschbach S, Scherrer S, Kern M. Clinical outcome of three-unit lithium-disilicate glass-ceramic fixed dental prostheses: up to 8 years results. *Dent Mater* 2009;25:e63-71.
4. Sailer I, Pjetursson BE, Zwahlen M, Hammerle CH. A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part II: Fixed dental prostheses. *Clin Oral Implants Res* 2007;18 Suppl 3:86-96.
5. Sailer I, Feher A, Filser F, Luthy H, Gauckler LJ, Scharer P, et al. Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow-up. *Quintessence Int* 2006;37:685-93.
6. Kimmich M, Stappert CF. Intraoral treatment of veneering porcelain chipping of fixed dental restorations: a review and clinical application. *J Am Dent Assoc* 2013;144:31-44.
7. Saracoglu A, Cura C, Cotert HS. Effect of various surface treatment methods on the bond strength of the heat-pressed ceramic samples. *J Oral Rehabil* 2004;31:790-7.
8. Lee HY, Han GJ, Chang J, Son HH. Bonding of the silane containing multi-mode universal adhesive for lithium disilicate ceramics. *Restor Dent Endod* 2017;42:95-104.
9. Chen JH, Matsumura H, Atsuta M. Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin. *Oper Dent* 1998;23:250-7.
10. Kukiattrakoon B, Thammasitboon K. Optimal acidulated phosphate fluoride gel etching time for surface treatment of feldspathic porcelain: on shear bond strength to resin composite. *Eur J Dent* 2012;6:63-9.
11. Blum IR, Nikolinakos N, Lynch CD, Wilson NH, Millar BJ, Jagger DC. An in vitro comparison of four intra-oral ceramic repair systems. *J Dent* 2012;40:906-12.
12. Kukiattrakoon B, Thammasitboon K. The effect of different etching times of acidulated phosphate fluoride gel on the shear bond strength of high-leucite ceramics bonded to composite resin. *J Prosthet Dent* 2007;98:17-23.
13. Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. *Dent Mater* 2012;28:467-77.
14. Newburg R, Pameijer CH. Composite resins bonded to porcelain with silane solution. *J Am Dent Assoc* 1978;96:288-91.

15. Isolan CP, Valente LL, Münchow EA, Basso GR, Pimentel AH, Schwantz JK, et al. Bond strength of a universal bonding agent and other contemporary dental adhesives applied on enamel, dentin, composite, and porcelain. *Applied Adhesion Science* 2014;2:25.
16. Kim RJ, Woo JS, Lee IB, Yi YA, Hwang JY, Seo DG. Performance of universal adhesives on bonding to leucite-reinforced ceramic. *Biomater Res* 2015;19:11.
17. Kim JH, Chae SY, Lee Y, Han GJ, Cho BH. Effects of multipurpose, universal adhesives on resin bonding to zirconia ceramic. *Oper Dent* 2015;40:55-62.
18. Spohr AM, Sobrinho LC, Consani S, Sinhoreti MA, Knowles JC. Influence of surface conditions and silane agent on the bond of resin to IPS Empress 2 ceramic. *Int J Prosthodont* 2003;16:277-82.
19. Filho AM, Vieira LC, Araujo E, Monteiro Junior S. Effect of different ceramic surface treatments on resin microtensile bond strength. *J Prosthodont* 2004;13:28-35.
20. Hayakawa T, Horie K, Aida M, Kanaya H, Kobayashi T, Murata Y. The influence of surface conditions and silane agents on the bond of resin to dental porcelain. *Dent Mater* 1992;8:238-40.
21. Matinlinna JP, Vallittu PK. Bonding of resin composites to etchable ceramic surfaces - an insight review of the chemical aspects on surface conditioning. *J Oral Rehabil* 2007;34:622-30.
22. Roulet JF, Soderholm KJ, Longmate J. Effects of treatment and storage conditions on ceramic/composite bond strength. *J Dent Res* 1995;74:381-7.
23. Kumbuloglu O, Lassila LV, User A, Toksavul S, Vallittu PK. Shear bond strength of composite resin cements to lithium disilicate ceramics. *J Oral Rehabil* 2005;32:128-33.
24. Nagai T, Kawamoto Y, Kakehashi Y, Matsumura H. Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents. *J Oral Rehabil* 2005;32:598-605.
25. Kelly JR, Benetti P. Ceramic materials in dentistry: historical evolution and current practice. *Aust Dent J* 2011;56 Suppl 1:84-96.
26. Yilmaz H, Aydin C, Gul BE. Flexural strength and fracture toughness of dental core ceramics. *J Prosthet Dent* 2007;98:120-8.
27. Chen YM, Smales RJ, Yip KH, Sung WJ. Translucency and biaxial flexural strength of four ceramic core materials. *Dent Mater* 2008;24:1506-11.
28. Holand W, Schweiger M, Frank M, Rheinberger V. A comparison of the microstructure and properties of the IPS Empress 2 and the IPS Empress glass-ceramics. *J Biomed Mater Res* 2000;53:297-303.
29. Fasbinder DJ. Restorative material options for CAD/CAM restorations. *Compend Contin Educ Dent* 2002;23:911-6, 18, 20 passim; quiz 24.

30. Denry I, Kelly JR. State of the art of zirconia for dental applications. *Dent Mater* 2008;24:299-307.
31. Otto T. Computer-aided direct all-ceramic crowns: preliminary 1-year results of a prospective clinical study. *Int J Periodontics Restorative Dent* 2004;24:446-55.
32. Kurbad A, Reichel K. Multicolored ceramic blocks as an esthetic solution for anterior restorations. *Int J Comput Dent* 2006;9:69-82.
33. Sundh A, Sjogren G. A comparison of fracture strength of yttrium-oxide- partially-stabilized zirconia ceramic crowns with varying core thickness, shapes and veneer ceramics. *J Oral Rehabil* 2004;31:682-8.
34. Haselton DR, Diaz-Arnold AM, Hillis SL. Clinical assessment of high-strength all-ceramic crowns. *J Prosthet Dent* 2000;83:396-401.
35. Magne P, Belser U. Esthetic improvements and in vitro testing of In-Ceram Alumina and Spinell ceramic. *Int J Prosthodont* 1997;10:459-66.
36. Oden A, Andersson M, Krystek-Ondracek I, Magnusson D. Five-year clinical evaluation of Procera AllCeram crowns. *J Prosthet Dent* 1998;80:450-6.
37. Garvie RC NP. Phase analysis in zirconia systems. *J Am Ceram Soc* 1972;55:303-5.
38. Luthardt RG, Sandkuhl O, Reitz B. Zirconia-TZP and alumina--advanced technologies for the manufacturing of single crowns. *Eur J Prosthodont Restor Dent* 1999;7:113-9.
39. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dent Mater* 1999;15:426-33.
40. Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: an overview. *Dent Mater* 2008;24:289-98.
41. Van Noort R. An introduction to dental materials. 2002:68-78.
42. Sposetti VJ, Shen C, Levin AC. The effect of topical fluoride application on porcelain restorations. *J Prosthet Dent* 1986;55:677-82.
43. Soderholm KJ, Shang SW. Molecular orientation of silane at the surface of colloidal silica. *J Dent Res* 1993;72:1050-4.
44. Ikemura K, Tanaka H, Fujii T, Deguchi M, Endo T, Kadoma Y. Development of a new single-bottle multi-purpose primer for bonding to dental porcelain, alumina, zirconia, and dental gold alloy. *Dent Mater J* 2011;30:478-84.
45. Chen L, Shen H, Suh BI. Effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers. *J Prosthet Dent* 2013;110:402-7.

46. Della Bona A, van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. *J Dent Res* 1995;74:1591-6.
47. Sano H, Ciucchi B, Matthews WG, Pashley DH. Tensile properties of mineralized and demineralized human and bovine dentin. *J Dent Res* 1994;73:1205-11.
48. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. *Dent Mater* 1994;10:236-40.
49. Ferrari M, Goracci C, Sadek F, Eduardo P, Cardoso C. Microtensile bond strength tests: scanning electron microscopy evaluation of sample integrity before testing. *Eur J Oral Sci* 2002;110:385-91.
50. Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. *Int J Prosthodont* 2002;15:267-72.
51. Fukushima T, Inoue Y, Miyazaki K, Itoh T. Effect of primers containing N-methylolacrylamide or N-methylolmethacrylamide on dentin bond durability of a resin composite after 5 years. *J Dent* 2001;29:227-34.
52. Burrow MF, Satoh M, Tagami J. Dentin bond durability after three years using a dentin bonding agent with and without priming. *Dent Mater* 1996;12:302-7.
53. Shono Y, Terashita M, Shimada J, Kozono Y, Carvalho RM, Russell CM, et al. Durability of resin-dentin bonds. *J Adhes Dent* 1999;1:211-8.
54. Ferracane JL, Berge HX, Condon JR. In vitro aging of dental composites in water--effect of degree of conversion, filler volume, and filler/matrix coupling. *J Biomed Mater Res* 1998;42:465-72.
55. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, et al. Four-year water degradation of total-etch adhesives bonded to dentin. *J Dent Res* 2003;82:136-40.
56. Armstrong SR, Keller JC, Boyer DB. Mode of failure in the dentin-adhesive resin-resin composite bonded joint as determined by strength-based (μ TBS) and fracture-based (CNSB) mechanical testing. *Dent Mater* 2001;17:201-10.
57. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999;27:89-99.
58. Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. In vivo degradation of resin-dentin bonds in humans over 1 to 3 years. *J Dent Res* 2000;79:1385-91.
59. Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. *J Dent Res* 2001;80:1605-14.

60. Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, et al. Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin. *Dent Mater* 2002;18:269-75.
61. Xie B, Dickens SH, Giuseppetti AA. Microtensile bond strength of thermally stressed composite-dentin bonds mediated by one-bottle adhesives. *Am J Dent* 2002;15:177-84.
62. Wolf DM, Powers JM, O'Keefe KL. Bond strength of composite to porcelain treated with new porcelain repair agents. *Dent Mater* 1992;8:158-61.
63. Kato H, Matsumura H, Tanaka T, Atsuta M. Bond strength and durability of porcelain bonding systems. *J Prosthet Dent* 1996;75:163-8.
64. Kumari RV, Poluri RK, Nagaraj H, Siddaraju K. Comparative Evaluation of Bond Strength of Dual-Cured Resin Cements: An In-Vitro Study. *J Int Oral Health* 2015;7:43-7.
65. Bravis T, Pilecki P, Wilson RF, Fenlon M, Watson TF, Foxton RM. Effect of loading on the microtensile bond strength and microleakage of a self-etching and etch-and-rinse adhesive in direct Class II MOD composite restorations in vitro. *Dent Mater J* 2012;31:924-32.
66. Yoshihara K, Nagaoka N, Sonoda A, Maruo Y, Makita Y, Okihara T, et al. Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dent Mater* 2016;32:1218-25.
67. Kitahara N, Itoh K, Kusunoki M, Oikawa M, Miyazaki T. One-bottle silane coupling agent containing 4-META. *Dent Mater J* 2013;32:409-12.
68. Matsumura H, Kato H, Atsuta M. Shear bond strength to feldspathic porcelain of two luting cements in combination with three surface treatments. *J Prosthet Dent* 1997;78:511-7.
69. Appeldoorn RE, Wilwerding TM, Barkmeier WW. Bond strength of composite resin to porcelain with newer generation porcelain repair systems. *J Prosthet Dent* 1993;70:6-11.
70. Brendeke J, Ozcan M. Effect of physicochemical aging conditions on the composite-composite repair bond strength. *J Adhes Dent* 2007;9:399-406.
71. Wagner A, Wendler M, Petschelt A, Belli R, Lohbauer U. Bonding performance of universal adhesives in different etching modes. *J Dent* 2014;42:800-7.
72. Foxton RM, Pereira PN, Masatoshi N, Tagami J, Miura H. Long-term durability of the dual-cure resin cement/silicon oxide ceramic bond. *J Adhes Dent* 2002;4:125-35.
73. Peumans M, Hikita K, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, et al. Bond durability of composite luting agents to ceramic when exposed to long-term thermocycling. *Oper Dent* 2007;32:372-9.
74. Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. *J Adhes Dent* 2000;2:305-13.

75. Armstrong SR, Vargas MA, Fang Q, Laffoon JE. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage. *J Adhes Dent* 2003;5:47-56.
76. Pashley EL, Agee KA, Pashley DH, Tay FR. Effects of one versus two applications of an unfilled, all-in-one adhesive on dentine bonding. *J Dent* 2002;30:83-90.
77. Tay FR, Pashley DH, Yiu CK, Sanares AM, Wei SH. Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part I. Single-step self-etching adhesive. *J Adhes Dent* 2003;5:27-40.





Appendix A. Immediate microtensile bond strength of repaired IPS Empress[®] Esthetic with resin composite by conventional adhesive system

Units	MPa	Units	MPa
EECI-1	19.8212	EECI-20	36.6649
EECI-2	31.9505	EECI-21	29.3536
EECI-3	19.8527	EECI-22	24.1434
EECI-4	35.7689	EECI-23	29.3432
EECI-5	31.3399	EECI-24	26.5535
EECI-6	30.6521	EECI-25	40.4632
EECI-7	14.1904	EECI-26	43.4763
EECI-8	15.1100	EECI-27	16.4182
EECI-9	20.7975	EECI-28	55.9258
EECI-10	16.6477	EECI-29	40.0179
EECI-11	36.2338	EECI-30	29.9768
EECI-12	40.0632	EECI-31	46.5541
EECI-13	25.6769	EECI-32	13.0652
EECI-14	36.2166	EECI-33	16.8685
EECI-15	13.7701	EECI-34	39.0609
EECI-16	24.5891	EECI-35	24.3358
EECI-17	27.2861	EECI-36	20.5798
EECI-18	26.3299	Mean	28.2193
EECI-19	16.7973	SD	10.4572

Appendix B. Microtensile bond strength of repaired IPS Empress[®] Esthetic with resin composite by conventional adhesive system after thermocycling for 10,000 cycles

Units	MPa	Units	MPa
EECA-1	6.9915	EECA-20	33.0741
EECA-2	25.7964	EECA-21	31.5285
EECA-3	30.5010	EECA-22	16.0224
EECA-4	26.8069	EECA-23	22.9123
EECA-5	28.2941	EECA-24	10.7438
EECA-6	29.6171	EECA-25	22.4043
EECA-7	34.1687	EECA-26	50.1103
EECA-8	19.2148	EECA-27	18.7384
EECA-9	39.1859	EECA-28	33.7735
EECA-10	23.1749	EECA-29	23.6579
EECA-11	36.6287	EECA-30	32.0549
EECA-12	34.0580	EECA-31	16.5406
EECA-13	22.9253	EECA-32	17.6185
EECA-14	23.6679	EECA-33	13.8634
EECA-15	35.8022	EECA-34	23.4962
EECA-16	23.5698	EECA-35	38.9847
EECA-17	33.0692	EECA-36	22.4315
EECA-18	35.3634	Mean	26.7376
EECA-19	25.7634	SD	8.9039

Appendix C. Immediate microtensile bond strength of repaired IPS Empress® Esthetic with resin composite by universal adhesive system

Units	MPa	Units	MPa
EEUI-1	38.5706	EEUI-20	27.2161
EEUI-2	13.1938	EEUI-21	27.6066
EEUI-3	14.6055	EEUI-22	18.9544
EEUI-4	20.6613	EEUI-23	26.0508
EEUI-5	24.4697	EEUI-24	25.6153
EEUI-6	21.9952	EEUI-25	18.7538
EEUI-7	8.8499	EEUI-26	30.0670
EEUI-8	28.0396	EEUI-27	27.6029
EEUI-9	26.6260	EEUI-28	19.5047
EEUI-10	29.1123	EEUI-29	13.2972
EEUI-11	34.7612	EEUI-30	12.1139
EEUI-12	20.7095	EEUI-31	22.5692
EEUI-13	14.4771	EEUI-32	19.1890
EEUI-14	40.0400	EEUI-33	30.0325
EEUI-15	19.8493	EEUI-34	14.8054
EEUI-16	24.8253	EEUI-35	20.2949
EEUI-17	18.3958	EEUI-36	44.4133
EEUI-18	13.1819	Mean	23.6211
EEUI-19	39.9068	SD	8.5955

Appendix D. Microtensile bond strength of repaired IPS Empress® Esthetic with resin composite by universal adhesive system after thermocycling for 10,000 cycles

Units	MPa	Units	MPa
EEUA-1	9.3446	EEUA-20	31.9577
EEUA-2	14.9511	EEUA-21	23.5593
EEUA-3	31.3062	EEUA-22	19.2416
EEUA-4	22.4150	EEUA-23	19.3209
EEUA-5	14.4883	EEUA-24	14.9856
EEUA-6	19.6214	EEUA-25	16.4379
EEUA-7	26.1415	EEUA-26	24.5625
EEUA-8	6.4268	EEUA-27	34.4062
EEUA-9	22.6773	EEUA-28	27.3285
EEUA-10	20.7018	EEUA-29	16.5217
EEUA-11	18.7190	EEUA-30	24.7998
EEUA-12	14.2760	EEUA-31	31.2441
EEUA-13	16.6145	EEUA-32	24.7314
EEUA-14	16.6255	EEUA-33	19.2490
EEUA-15	8.5386	EEUA-34	9.8305
EEUA-16	18.9913	EEUA-35	15.9588
EEUA-17	33.3976	EEUA-36	19.5219
EEUA-18	26.3350	Mean	20.4202
EEUA-19	19.9036	SD	7.0037

Appendix E. Immediate microtensile bond strength of repaired IPS e.max[®] Press with resin composite by conventional adhesive system

Units	MPa	Units	MPa
EMCI-1	44.7786	EMCI-20	21.1766
EMCI-2	32.4662	EMCI-21	46.0516
EMCI-3	42.2133	EMCI-22	23.6549
EMCI-4	50.0156	EMCI-23	12.3232
EMCI-5	45.8610	EMCI-24	17.0839
EMCI-6	33.1642	EMCI-25	41.3618
EMCI-7	46.9706	EMCI-26	32.1609
EMCI-8	51.2430	EMCI-27	27.0741
EMCI-9	53.6200	EMCI-28	63.8994
EMCI-10	69.3895	EMCI-29	36.3096
EMCI-11	48.5766	EMCI-30	45.9245
EMCI-12	27.6905	EMCI-31	58.4602
EMCI-13	23.7726	EMCI-32	50.0087
EMCI-14	21.8363	EMCI-33	48.5506
EMCI-15	42.8680	EMCI-34	20.3097
EMCI-16	29.8349	EMCI-35	54.0595
EMCI-17	30.7981	EMCI-36	29.0482
EMCI-18	33.7454	Mean	38.2935
EMCI-19	22.2633	SD	13.9104

Appendix F. Microtensile bond strength of repaired IPS e.max[®] Press with resin composite by conventional adhesive system after thermocycling for 10,000 cycles

Units	MPa	Units	MPa
EMCA-1	31.0256	EMCA-20	30.6185
EMCA-2	23.6945	EMCA-21	30.0076
EMCA-3	19.3872	EMCA-22	35.1954
EMCA-4	36.6831	EMCA-23	54.4786
EMCA-5	11.1979	EMCA-24	40.7173
EMCA-6	25.0559	EMCA-25	34.8268
EMCA-7	19.9451	EMCA-26	20.4470
EMCA-8	24.0342	EMCA-27	50.3336
EMCA-9	36.9070	EMCA-28	20.4470
EMCA-10	28.2434	EMCA-29	22.6455
EMCA-11	27.5420	EMCA-30	38.3533
EMCA-12	32.2713	EMCA-31	56.7662
EMCA-13	24.5625	EMCA-32	35.1712
EMCA-14	45.0764	EMCA-33	55.3074
EMCA-15	40.9491	EMCA-34	45.3034
EMCA-16	40.6656	EMCA-35	42.4460
EMCA-17	42.0894	EMCA-36	63.098
EMCA-18	29.7526	Mean	34.3269
EMCA-19	20.5235	SD	12.1656

Appendix G. Immediate microtensile bond strength of repaired IPS e.max[®] Press with resin composite by universal adhesive system

Units	MPa	Units	MPa
EMUI-1	34.7050	EMUI-20	25.6471
EMUI-2	12.5926	EMUI-21	19.1159
EMUI-3	25.4810	EMUI-22	16.6475
EMUI-4	35.2308	EMUI-23	22.0823
EMUI-5	21.4203	EMUI-24	20.2963
EMUI-6	24.5280	EMUI-25	22.9864
EMUI-7	23.3540	EMUI-26	30.7442
EMUI-8	26.4962	EMUI-27	30.0069
EMUI-9	35.9188	EMUI-28	28.0481
EMUI-10	18.1749	EMUI-29	25.1536
EMUI-11	34.0890	EMUI-30	30.1625
EMUI-12	28.5631	EMUI-31	33.9611
EMUI-13	21.5113	EMUI-32	25.4702
EMUI-14	33.3684	EMUI-33	20.1501
EMUI-15	20.0967	EMUI-34	35.6979
EMUI-16	19.9962	EMUI-35	21.7172
EMUI-17	22.4029	EMUI-36	12.5927
EMUI-18	17.9179	Mean	25.2806
EMUI-19	33.7749	SD	6.5709

Appendix H. Microtensile bond strength of repaired IPS e.max[®] Press with resin composite by universal adhesive system after thermocycling for 10,000 cycles

Units	MPa	Units	MPa
EMUA-1	38.175	EMUA-20	23.3234
EMUA-2	21.2529	EMUA-21	11.7876
EMUA-3	30.6708	EMUA-22	24.7020
EMUA-4	28.7246	EMUA-23	23.4551
EMUA-5	21.1024	EMUA-24	26.9268
EMUA-6	17.8177	EMUA-25	23.4221
EMUA-7	18.9790	EMUA-26	23.6264
EMUA-8	17.8325	EMUA-27	25.6558
EMUA-9	30.5665	EMUA-28	41.3593
EMUA-10	29.6076	EMUA-29	16.8588
EMUA-11	29.0441	EMUA-30	17.8433
EMUA-12	17.4648	EMUA-31	19.1494
EMUA-13	19.4119	EMUA-32	41.4499
EMUA-14	21.5718	EMUA-33	28.9158
EMUA-15	27.7011	EMUA-34	16.4263
EMUA-16	5.4356	EMUA-35	41.3619
EMUA-17	26.8950	EMUA-36	33.6623
EMUA-18	22.8577	Mean	24.8772
EMUA-19	30.5428	SD	8.0971

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