“EFFECTS OF RESTORATIVE PRIMERS ON BOND STRENGTH OF POLYMER- INFILTRATED”

By

Dr. SPUN LENGLERDPHOL

Funded by
Research Institute of Rangsit University
2562

Title: Effect of The Different Types of Ceramic Primer On Bond Strength of Polymer Infiltrated Ceramic

Researcher: Dr. Spun Lenglerdphol
Institution: College of Dental Medicine, Rangsit University

Year of Publication: 2020
Publisher: Rangsit University
Sources: Rangsit University
No. of pages: 50 pages
Keywords: Polymer-infiltrated ceramic restoration, Surface treatment, Bonding strength, Universal ceramic primer.

Abstract

The polymer-infiltrated ceramic (VITA Enamic®) which contained hybrid materials, is recommended to use as single-unit fixed prosthesis. Treating surface with restorative primer is important procedure for increasing bond strength which affected to durability of the restoration. Nowaday, restorative primers from many manufacturer's, with different components, are available in market. Thus, this study was observed on different restorative primers on hybrid ceramic.

The impact of different restorative primer with resin composite cements (Panavia V5.0) on shear bond strength (SBS) to polymer infiltrated ceramic (VITA Enamic) was tested, subject to conditioning: Group 1C : Control group without restorative primer (n=10), Group 2 MN: Monobond N (n=10), Group 3 AP: Alloy Primer (n=10), Group 4 SB: Super-Bond Universal Ceramic Primer (n=10) Group 5 CF : Clearfil Ceramic Primer Plus (n=10). The SBS was measured; data were analyzed using Kolmogorov Smirnov test for normal distribution, Levene's test for homogeneity of variances. One-way ANOVA and post hoc Tamhane's T2 used for parametric analysis. Mode of failure was evaluated under stereomicroscope.

The SBS value the MN group showed the highest SBS values with no significant different from CF group but significantly higher than SB group. While, the CF groups showed not significant different from SB group. Only AP group was lower than control group. There were damages in ceramic in every group except control group. Specimens were mostly found to be adhesive failure, control group has the highest adhesive failures following by the AP group, CF group, SB group and MN group respectively.

Conclusion, primers containing silane promote the higher SBS between VITA Enamic and Panavia V5. Otherwise, primers that contain VBATDT can decreased. Primer containing 10-MDP in restorative primers or resin cement may not increase the SBS. Restorative primer may affect, both positive and negative, to bond strength. Thus, dentist should properly select restorative primer to enhance the bond strength of restoration.

Acknowledgement

Firstly, I am thankful to Rangsit University for the grant.

I also thank to the dean of College of Dental Medicine, Rangsit University, Rear Admiral Suchada Vuddhakanok RTN. for all the supports.

Lastly, I would like to thank to The Research Laboratory of Dental Materials of Faculty of Dentistry, Chulalongkorn University for providing us a place and the instrument in this research.

Contents

Chapter 1 Introduction

Chapter 2 Literature Reviews

Chapter 3 Materials and Methods

Chapter 4 Results

Chapter 5 Discussion and Conclusion

List of tables

Table **Page**

List of illustration

CHAPTER 1

INTRODUCTION

1.1 Background

The fixed partial prosthesis is an option for saving the tooth when the tooth has broken and unable to function normally and came with many materials of choice. Ceramic is one the material which widely used for esthetic reason. It is admittedly to be used for veneers, and anterior crown, since its advantages are high flexural strength and a great color stability with natural-looking, (Kramer & Frankenberger, 2005) while disadvantages of conventional ceramics are high antagonistic tooth wear and loss of tooth structure due to its minimum thickness of 1.5 to 2.0 mm. (Dirxen, Blunck, & Preissner, 2013) Nowadays, the ceramic has been improved its properties to fix the disadvantage and become more flexible to use. One of that is polymer infiltrated ceramics which has hybrid material between ceramic matrix and polymer matrix. It composed of ceramic matrix (86% in weight) and polymer matrix (14% in weight) making them become more flexible, kinder to opposite tooth, and more conservative. By having the interpenetrating polymer networking also reduced the fracture susceptibility of the restoration. (J. Schwenter, F. Schmidli, R. Weiger, & J. Fischer, 2016)

Becoming successful in restoring the tooth with fixed prosthesis, the key factor is the bonding strength between restoration and tooth. Bonding abilities between luting cement and both restoration and tooth surface can reduce the failure by prevent marginal leakage.(Noda et al., 2017) To achieve higher bonding strength in ceramic restoration, treating surface of the restoration is required, in both mechanical and chemical approach.(Mair & Padipatvuthikul, 2010) Roughening surface of restoration with grinding with diamond burs, etching with hydrofluoric acid, and sandblasting are the methods that improve the mechanical bond creating a micromechanical attachment, while silane coupling agent and primer provides a chemical bond.(Aida, Hayakawa, & Mizukawa, 1995; Matinlinna, Lassila, Ozcan, Yli-Urpo, & Vallittu, 2004)

Silane coupling agent can enhance the bond strength by promoting chemical bond between resin cement and ceramic restoration, its silanol group can form polysiloxane network and bond with silica surface. The monomeric end of silane molecules can react with methacrylate groups and bond with resin cement, (Moro, Ramos, Rocha, & Perez, 2017), moreover it can increase surface energy and wettability of the restoration's surface that also enhance effectiveness of the cementation.(Özcan, 2003)

Organo-phosphate monomers such as MDP, has been introduced to be the primer for treating the surface of oxide layer containing material, such as base metal and zirconia restoration. MDP exhibits a phosphate ester group that reacts with substrate and forms the covalent bond, moreover a hydrophobic alkylene group that prevents water penetration, and a terminal double bond group to copolymerize with resin. (Chuang et al., 2017; Murillo-Gómez, 2017)

Super-Bond Universal Ceramic Primer which contain both silane coupling agent and MDP, is claimed to be excellently performance with all types of ceramic and zirconia. However, there is no data available on using Super-Bond Universal Ceramic Primer comparing with other ceramic primers on polymer-infiltrated ceramic.

In the present study, ceramics found that mechanical and chemical surface treatment increased bond strength. Enamic treated with mechanical and chemical surface treatment can increase bond strength also. (Pnfd Silva, Martinelli-Lobo, Bottino, Melo, & Valandro, 2018) However, over the past 6 months, the bond strength has significantly decreased. We also need the role of the ceramic primers in many forms. Therefore, the aim of the study was to investigate the effects of different ceramic primers to bond strength of resin cement between polymer-infiltrated ceramic, to provide a recommendation for clinical procedure.

1.2 Research question

Does the different ceramic primers affect to bond strength of Panavia V5 when bonded polymer-infiltrated ceramic to composite resin.

1.3 Objective

To compare the effect of various ceramic primers on bond strength of polymer-infiltrated ceramic and composite resin.

1.4 Hypothesis

Various ceramic primers affect to bond strength of polymer-infiltrated ceramic and composite resin.

Hypothesis 1

H0 ; The bond strengths between polymer-infiltrated ceramic and composite resin using ceramic primer are not different to the control group, at 95% confidence level.

H1 ; The bond strengths between polymer-infiltrated ceramic and composite resin using ceramic primer are different to the control group, at 95% confidence level.

Hypothesis 2

H0 ; The bond strength between polymer-infiltrated ceramic and composite resin using different type of ceramic primers are indifferent, at 95% confidence level.

H1 ; The bond strength between polymer-infiltrated ceramic and composite resin using different type of ceramic primers are different, at 95% confidence level.

1.5 Scope of research

Sample : Enamic specimen bonded with resin composite block by using Panavia V5

Independent

Variable: Monobond N, Alloy Primer, Super-Bond Universal Ceramic Primer, Clearfil Ceramic Primer Plus, No primer

Dependent Variable: Shear bond strength

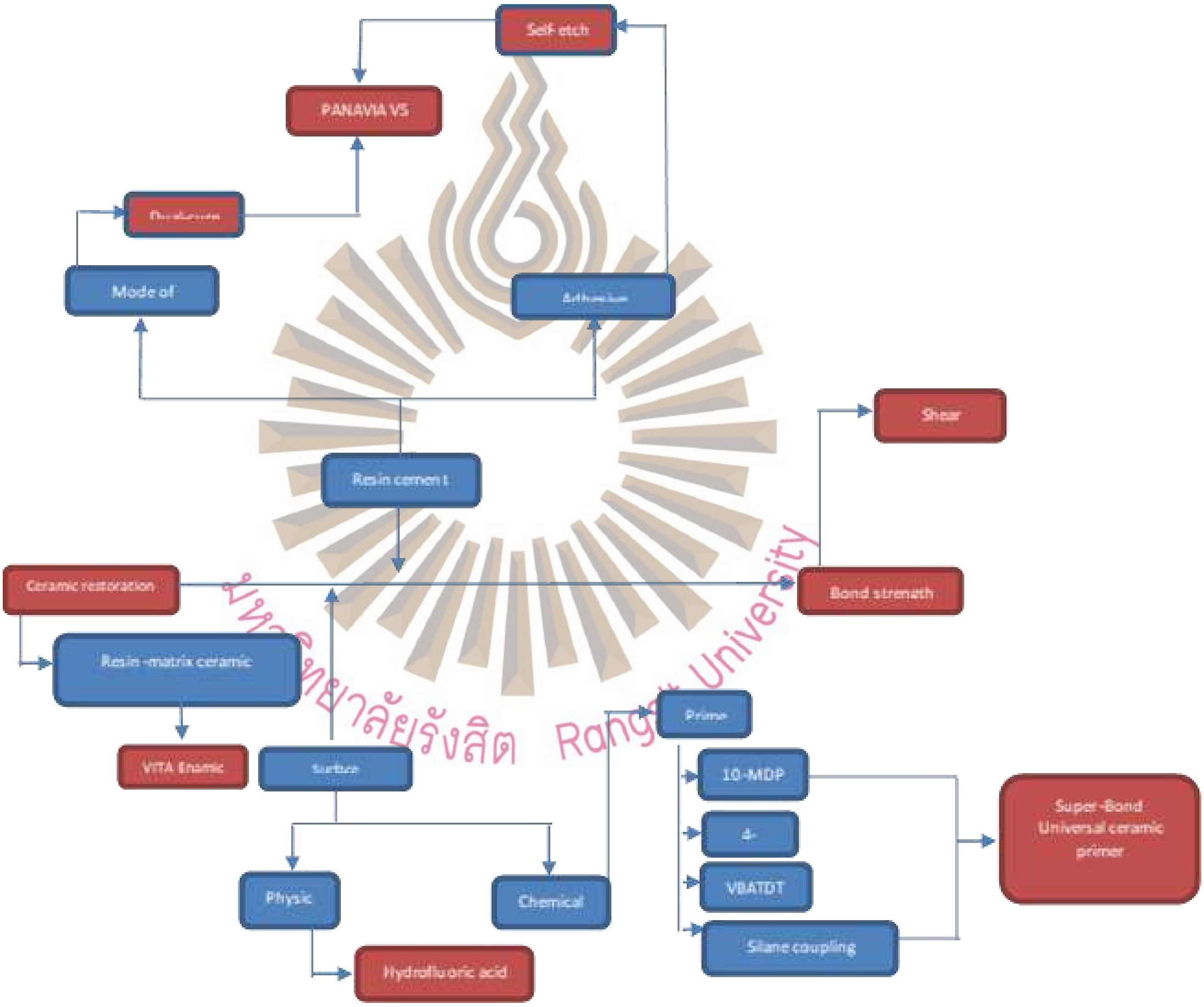
Interval of study: December, 2018 – September, 2019

Time schedule

Activity	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	June	July	Aug	Sep
1. Proposal development	←	—	→										
2. Data collection				←	—	—	—	—	—	→			

3. Data analysis										←	→		
4. Report preparation												←	→

1.6 Conceptual framework



1.7 Keywords

Polymer-infiltrated ceramic restoration

Surface treatment

Bonding strength

Universal ceramic primer

1.8 Expected benefit

To provide the proper of clinical procedure of choosing ceramic primers for ceramic surface preparation.

CHAPTER 2

REVIEW OF LITERATURE

2.1. Dental Ceramic Restoration

Ceramic is an inorganic compound of a metal, metalloid, or a non-metal atom which held by chemical bonds such as ionic bond and covalent bond. It has been used pervasively nowadays as people are pleasing of its esthetic, as a tooth-colored restorative material. It has been used for making crown, veneers, bridges, artificial denture teeth, and implants. In Dentistry, ceramics are usually composed of nonmetallic, inorganic structures containing oxygen and metallic elements like aluminum, calcium, magnesium, potassium, silicon, sodium, zirconium & titanium. (Anusavice, 2013; Rosenblum & Schulman, 1997)

Dental ceramics can be classified into 3 categories based on the presence of specific attributes in their formulation. (Gracis, Thompson, Ferencz, Silva, & Bonfante, 2015)

Category 1 Glass-matrix ceramic : Non-metallic inorganic ceramic materials that contain glass phase

Category 2 Polycrystalline ceramic : Non-metallic inorganic ceramic materials that do not contain any glass phase

Category 3 Resin-matrix ceramic (VITA Enamic, lava ultimate) : Polymer-matrices containing predominantly inorganic compound such as glass

Category 1 Glass-matrix ceramic

-Glass-based System which primarily silica-based

Glass-based systems are mainly containing silicon dioxide (silica), and which contain various amounts of potassium and sodium, are known as feldspars. ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$ or $Na_2O \cdot Al_2O_3 \cdot 6SiO_2$) Most of the world's feldspar is used in glass-making, alike in dentistry it has been used to create glass, feldspathic porcelain. This feldspar has a glassy-like surface, Leucite and a glass phase are formed when potassium feldspar is fired to high temperatures. This glass phase become soften and allowing condensation of the porcelain powder particles, this process is termed as liquid phase sintering. It has a low coefficient of thermal expansion (CTE) around $8 \, x \, 10^{-6}$/K, and a flexural strength at 60-70 MPa. The amount of leucite is adjusted to improve the CTE in the glass based. (A. Shenoy & N. Shenoy, 2010) It is high translucent and esthetic, but brittle. Thus it is popular for using as porcelain veneering for metal or ceramic substructure such as VITA VMK series, or as veneers by using in CAD/CAM system, such as Vitablocs Mark II.

Feldspathic Porcelain

It is composed of 75% feldspar, 22-25% quartz, 0-3% kaolin .Leucite has been added to increase the coefficient of thermal expansion (CTE) of the material so that they can be applied to metals and zirconia substructure. It is typical powder-liquid materials that used for veneering the core systems or using for porcelain veneers.

-Feldspar: Provides for transparency and influences the flow characteristics of the ceramics.

-Quartz (silicon oxide): Provides for adequate strength and also for transparency.

-Kaolin: Provides for the necessary opaque properties.

The original materials had a fairly random size and distribution of leucite crystals, with the average particle size being around 20 µm. This random distribution and large particle size contribute to the material's low fracture resistance and abrasive properties relative to enamel. (I., 2009) Newer generations of materials (eg, VM 13, VITA) have been developed with much finer leucite crystals and very even particle distribution throughout the glass. These materials are less abrasive and have much higher flexural strengths.(Giordano & McLaren, 2010)

-Glass-based systems (mainly silica) with fillers, usually crystalline

In this category of materials, the glassy matrix composition is basically the same as in the Category 1, but with adding of the crystalline fillers into it. The primary types of crystal that have been added are leucite, and lithium disilicate. Leucite is forming by increasing K_2O (potassium oxide) content of the alumino-silicate glass and Lithium-disilicate crystals are created by adding the Li_2O (lithium oxide).

Leucite-reinforce glass ceramic

A high leucite-containing glass, approximately of 50% leucite crystals, was added to improve CTE and strength in inhibit the crack propagation. The glassy phase is based on an alumino-silicate glass. It has been developed to be used in powder/liquid, machinable, and pressable forms. The processing techniques affect the fracture resistance, machinable and pressable systems have greater fracture resistance than powder/liquid systems, (A. Shenoy & N. Shenoy, 2010)and has given the excellently clinically result when used for posterior inlays and onlays, and anterior veneer and crown restorations. (Della Bona, Mecholsky, & Anusavice, 2004; Kramer & Frankenberger,

2005) Improving from the glass-based system the flexural strength is at 150-200 MPa making Leucite-reinforce glass ceramic available for making anterior crown restoration. The most widely used of this material is the IPS Empress® (Ivoclar Vivadent, Amherst, NY).

Lithium disilicate glass ceramic

Lithium disilicate's microstructure consists of many small interlocking plate-like crystals that are randomly oriented and are needle-like in shape, causing the cracks to deflect and arresting the propagation. The crystalline phase is contained about 70 percent of the volume of the glass ceramic, to providing a substantial increase in the flexural strength. The shape and volume of the crystals resulting in the greater flexural strength over 360 MPa and fracture toughness of this material.) Albakry, Guazzato, & Swain, 2003) Ivoclar vivadent has claimed that the strength of Lithium-disilicate glass ceramic is over twice that of other ceramic materials that do not require any layering material.(I., 2009) Even with the high ratio of crystalline component, but this material is still translucent due to the low refractive index of lithium-disilicate crystals. This material is translucent enough that it can be used for full contour restorations for the anterior teeth or for the highest aesthetics and can be veneered with porcelain. Lithium disilicate can be processed by using lost-wax-hot pressing technique or by using CAD/CAM. Ivoclar has also claimed that the restorations deliver 400 MPa of strength throughout pressing technique which is more than using the CAD systems which give out 360 MPa of strength through entire restoration, and less likely to wear the opposing tooth. (I., 2009)

Due to its esthetics, strength and versatility, this material can be utilized for Anterior/posterior crowns, Inlays/onlays, Veneers, Implant restorations, Anterior three-unit bridge

Glass infiltrated ; Alumina-based

In Ceram Spinell, In Ceram Alumina, In Ceram Zirconia

The first glass-infiltrate material, In-Ceram Alumina, is fabricated utilizing the slip-casting technique. A slurry of densely packed Al_2O_3 is sintered to a refractory die, and after a porous skeleton of alumina particles is formed, infiltration with lanthanum glass is performed in a second firing to infiltrate the porosity and increase strength. it composed of 2 phases are glassy phase and crystalline phase. The crystalline phases have 3 different Alumina 's particle size ,large elongated grain, faceted particle, spherical grains. Porcelain veneering is required because of it opacity . The use of this class of material is diminished due to the increased popularity of lithium disilicate and zirconia, particularly for CAD/CAM fabricate

Providing strength and fracture toughness, but have limited translucency.

-Crystalline- based systems with glass fillers (mainly alumina)

In-Ceram Alumina was the first glass-infiltrated ceramic that was developed by the slip- cast technique. We can called “alumina-reinforced ceramic” . It has been used as a core material for crowns and anterior three-unit fixed partial dentures)FPD) . This material is esthetic and biocompatibility.(Guazzato, Albakry, Swain, & Ironside, 2002) The slip cast technique is the method that the porous was produced in infrastructure and later filled with lanthanum glass. Hence, it composed of 2 phases are glassy phase and crystalline phase. The crystalline phases have 3 different Alumina ’s particle size ,larged elongated grain,faceted particle,spherical grains. Porcelain veneering is required because of it opacity. (Gracis et al., 2015; Isabelle, 2010)

It is densely composed of Alumina oxide and silica particles, which strengthen the restoration, and increased the flexural strength for five times comparing to Feldspatic porcelain. Examples of this ceramic categories is In-Ceram Alumina which has 80-82%wt Aluminum oxide and 5%wt silica. It can used as a core material for crown and bridge, but with its opacity making In-Ceram Alumina can not be used as an veneering structure. To solve this problem, the addition of Magnesium and Alumina becoming Spinell ($MgAl_2O_3$) has been used to increase its esthetics. In-Ceram Spinell is increased in its esthetic property but reduced in strength, so the its can only be used as the retorative material for anterior teeth. Moreover, adding zirconium insteadly, producing In-Ceram Zirconium has its indication for using as an core material in posterior teeth because of its advantage in strength, but low in esthetic.

Category 2 Polycrystalline ceramic

-Polycrystalline solids (alumina and zirconia)

Oxide ceramics be devided into Alumina(Al2O3) and Zirconia(ZrO2) . These 2 types of oxide ceramic are favored to use because of their high fracture resistance properties. (Jorquera Rivera, 2016) Oxide ceramic groups are cannot etching so they need to be sand-blasted and follow with primer that composed of MDP (Methacriloyloxydecyldihydrogen phosphate). And after that adhesive or resin cement and be applied. The flexural strength of Aluminium oxide ceramic is about 600 Mpa and 900-1100 Mpa in Zirconium oxide ceramic. (Arvind Shenoy & Nina Shenoy, 2010; M. A. Zakir, Usman & Tian, Tian & Han, Aifang & Qiao, Wei & Jin, Xiaozhuang & Zhang, M & Tsoi, James & Matinlinna, Jukka, 2016) Zirconium oxide has 3 phases depend on the temperature, “Monoclinic” is the phase at room temperature , “Tetragonal” at 1370°C and “Cubic” at 2320°C. Zirconium oxide is transformed each staged by increasing the temperature. In the late 1980s, ceramic engineers add the yttrium oxide as a stabilizer to stabilize the Tetragonal phase in room temperature . The reason for adding the stabilizer is about “Transformation toughening” that can trap energy within material and drive it back to monoclinic state so that, it can stop cracked .

Category 3 Resin matrix ceramic

suggest the wide range of indication for using these materials in restorative dentistry, due to its developed properties such as closely simulates the modulus of elasticity of dentin when comparing with traditional-ceramics, or the ability to facilitate repairing or modification with composite resin. Their composition varies but they can be processed by The Resin-matrix ceramics is the materials highly containing with ceramic particles in an organic matrix. The materials in the category are composed predominantly (>50% by weight) of the inorganic compounds and the other less predominant organic phase or polymer. The manufacturers CAD/CAM. Currently, the resin matrix ceramics can be divided into several subfamilies, according to their composition of organic materials.(Gracis et al., 2015)

3.1 Resin Nanoceramic

The resin nanoceramic is consisted with a highly cured resin matrix reinforced with the nanoceramic particles (approximately 80% by weight) including discrete silica nanoparticles (20 nm diameter), zirconia particle (4-11 nm diameter) and zirconia-silica nanoclusters. The example of this material in this category is Lava Ultimate, 3M ESPE

3.2 Glass ceramic in a resin interpenetrating matrix

This type of ceramic is composed of a dual network of an organic and inorganic substances: the polymer network consists of urethane dimethacrylate (UDMA) and triethylene glycerol dimethacrylate (TEGDMA) (14% by weight / 25 % by volume), feldspathic ceramic network (86% by weight / 75% by volume). The example of this hybrid ceramic is Enamic. (VITA, 2014)

VITA Enamic

Ceramics are high flexural strength and have good color stability. However ceramics can cause of excessive wear of opposite tooth because of its hardness. So that some material can decrease wear of tooth structure was combined. Composite resin is the material that proper to use due to of its properties that has lower hardness than enamel.(Dirxen et al., 2013) However, the polymer have some characteristic such as there curing shrinkage, low mechanical properties and poor wear resistance which worsen than ceramic.

The polymer infiltration was inspired from real teeth enamel, which consists of inorganic and organic components, by mixing of ceramic and polymer can be increase strength and elastic properties. This could can be called "Polymer-infiltrated ceramic network (PICN) or Hybrid ceramic".(Facenda, Borba, & Corazza, 2018) It is composing of ceramic matrix)86 %in weight/75% in volume (and polymer matrix)14 %in weight/25% by volume(, the polymer network are TEGDMA and UDMA .(Gracis et al., 2015) PICN showed similar flexural strength values to dentin tissue, but lower compressive strength values. The sample of this product is VITA ENAMIC(3M ESPE). (VITA, 2014)

When compared with normal dental composite, Enamic has a tree-dimensional framework of interconnected particle while the dental composite has a scattered distribution of particle. Moreover Enamic have more higher elastic modulus and hardness, more matching to natural tooth than dental composite material. Enamic is also more tolerance than glass ceramics.(Xu, Yu, Arola, Min, & Gao, 2017) On the other way, Enamic has high wear resistance from the ceramic composition and when testing with Vicker hardness test, by using SEM , the crack line is stopped by the interpenetrating polymer within the hybrid network.(Dirxen et al., 2013) From the study of Albero et al."Vita Enamic" has higher flexural strength when compared to Lava Ultimate .(Albero, Pascual, Camps, & Grau-Benitez, 2015) According to the manufacturer, Enamic has higher elasticity than Lava ultimate. Furthermore Vita Enamic are also improve its brittleness, decrease tooth wear ,high flexural strength.(Della Bona, Mecholsky, Barrett, & Griggs, 2008)

PICN have mechanical properties that closely natural teeth, Youngs modulus is close to dentin, Vickers hardness lies between enamel and dentin. So that this can be used with the patient who has bruxism, amelogenesis imperfecta and erosion.(Dirxen et al., 2013)

According to the manufacturer, Vita Enamic can used in anterior and posterior crown, inlays, onlays, crown on implant. Shouldn't been used as a bridge, free-end restoration, parafunctional habits.

2.2 Surface treatment

Surface Treatment were divided into 2 groups (Mechanical and Chemical Surface Treatment)

Chemical Surface Treatment

1. Functional monomer

The surface treatment with primers containing functional monomers such as MDP or other phosphoric acid acrylate monomer are often recommended to improve the bonding to oxide ceramic. Since results are not always significant, the combination of primers and air-abrasion methods tend to produce better bond strength, especially in long-term. (Takeuchi et al., 2010)

1.1 10-MDP

10-methacryloyloxy-decyl-dihydrogen-phosphate (10-MDP), commonly used in self-etching system as an etching monomer, due to the dihydrogenphosphate group, decalcifying dental hard tissue and binding to calcium ion. It is also a functional monomer use as the dentin adhesive monomer contained in a metal primer by its amphiphilic structure with the phosphate group and methacrylate group at both ends to form the chemical bond between oxide layers of metal restoration or zirconia restoration to resin cement , and increasing the diffusion and adhesion of resin cement to both dental tissue and restoration. (Sofan et al., 2017; Van Landuyt et al., 2007)

.

Figure 1 : methacryloyloxy-decyl-dihydrogen-phosphate

(http://www.dmhpi.com/adhesion-promoters.html)

10-MDP is a monomer that was originally synthesized by Kuraray co., Ltd. (Osaka, Japan) with the long carbonyl chain making it quite hydrophobic.

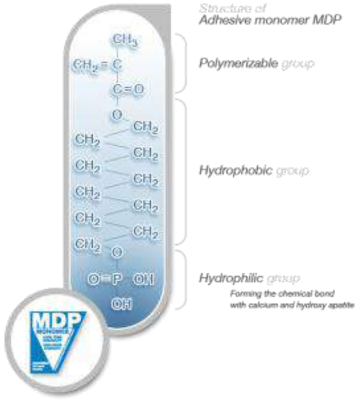


Figure 2 : methacryloyloxy-decyl-dihydrogen-phosphate

(https://kuraraydental.com/wp-content/uploads/2018/05/mdp.png)

Structure of adhesive monomer MDP

1. Polymerizable group (Methacryloyl group) : Necessary for copolymerization between the MDP monomer and matrix monomer in the resin cement

2. Hydrophobic group (Carbonyl chain) : Prevent penetration water into adhesive surface, which interfere the polymerization resulting in great hydrolytic stability.(Inoue et al., 2005)

3. Dihydrogen phosphate group : Forming chemical bond with tooth surface, Metal Oxides, and Zirconium oxide via Hydrogen bonding or Ionic interaction between PO- and positive ion (Al3+, Zr4+, etc.) (Inoue et al., 2005; Nagaoka et al., 2017)

1.2 4META

4-META is white crystalline structure, prepared by condensation of 2-hydroxyethyl methacrylate and trimellitic anhydride chloride. When it is added to the acrylic monomer, the molecule forms both a hydrophilic bond and a hydrophobic bond, which is responsible for the chemical bonding to certain dental alloys

Dental adhesive resin

The dental adhesive resins that contain 4-META are Orthomite Super-Bond, Super-Bond C&B, Meta-Dent, and Meta-Fast (Sun Medical Company, Kyoto,Japan); and Cover-Up, C&B Metabond, Amalgambond, Cover-Up II, Acrylic Solder, Etch-Free, Amalgambond Plus, EasyBond, and TotalBond(Chang, Hurst, Hart, & Estey, 2002)

1.3 VBATDT

VBATDT is a thione-thiol tautomer developed to improve the bonding between noble metal alloys and methacrylate-based resins. The coupling mechanism occurs by transforming thione in thiol and formation of the bond on the metal surface, and copolymerization of the vinyl groups with the resin cement. (Silikas, Wincott, Vaughan, Watts, & Eliades, 2007)

2. <u>Silane coupling agents</u>

Silane coupling agents (trialkoxysilanes) are hybrid inorganic-organic bifunctional molecules that can create a siloxane network with the hydroxyl of the silica in ceramic surface and copolymerize with the resin matrix of the composite, also, silane lower the surface tension and increase the surface energy. (Matinlinna et al., 2004)

Silanes are a broad category of chemical compounds of silicon, which is metalloid, semi-metal (a semi-conductor) and other atoms such as carbon, nitrogen and hydrogen. Silane can be used to wide range of applications to improve polymer crosslinking, dispersion, mechanical properties and increased adhesion between organic and inorganic phases. In dentistry, Silane is used to promote adhesion between resin composites and silica-based or silica-coated indirect restorative materials. Silane coupling agents contains trichlorosilane, such as 3-methacryloxyproyltrimethoxysilane (MPS), as the reactive key component. Silane is activated by dissolved in alcohol (ethanol) and mixed with aqueous acetic acid before immediate use to allow silane to form silanol (SiOH) before they can bond to the inorganic substrate.

Trichlorosilane contain two functional groups at the end of molecules, which connect unpolymerized resin matrix an inorganic substrate. The formula of silane is showed as follows :

$$\mathrm{L}\text{-}(\mathrm{CH}_2)_k\text{-}\mathrm{Si}\text{-}(OR)_3$$

- L is an organofunctional group (unpolymerized resin matrix) (e.g., methacrylate, acrylate, iso-cyanato, epoxy)
- $(\mathrm{CH}_2)_k$ is a linker (spacer) group that separates the organofunctional group
- The Si is silicon atom
- OR is a hydrolysable alkoxyl group (inorganic substrate) (methoxy, ethoxy)

Silane adhesion mechanism

Two steps of resin substrate bonding formation with silane coupling agents:

1. Substrate and silane bond formation activated by acid
2. Silane and resin bond formation activated by light curing

Silane is activated by acid to form silanol groups which react with the substrate surface hydroxyl (OH) groups by a condensation reaction (Si-OH + HO-substrate ⇒ Si-O-substrate) when it is applied onto a surface treated, *e.g.* a silica coated substrate.

The reaction between the organofunctional groups of silane (unpolymerized resin matrix with a C=C bond), and functional groups of the resin monomers containing C=C bond is induced by the reactive free radicals generated by photo-activation e.g. visible blue light (400–500 nm). Then polymerization occur to form new C-C bond.

Therefore, resin composite and substrate surface are connected by silane coupling agent.

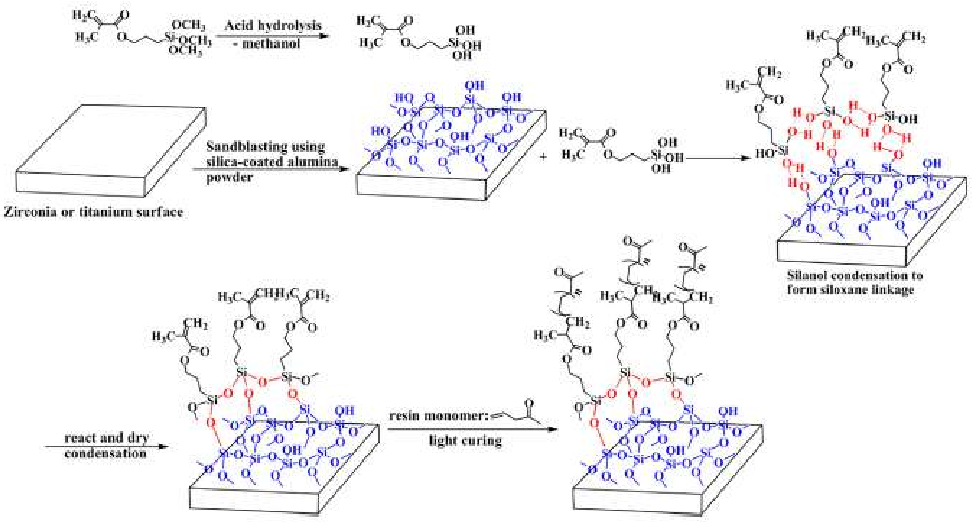


Figure 3 : Adhesion mechanism of resin bonding to silica coated substrates by an application of silane coupling agent using 3-methacryloxypropytrimethoxysilane as an example

(Matinlinna, Lung, & Tsoi, 2018)

Moreover, silanes still have portion that do not react with organic resin monomer. It is called Non-functional silanes. Non-functional silane may contain alkoxy(-OR) functional groups that form silanol groups after hydrolysis. Then silanol group will react with surface hydroxyl groups. For example, a cross-linking silane molecule which is also called a bis-functional silane. It has two silicon atoms, each one contains three hydrolysable alkoxy groups. Non-functional can form an extensive cross-linking network with functional silanes and stabilize the siloxane films for hydrolytic attacks. (Matinlinna et al., 2018)

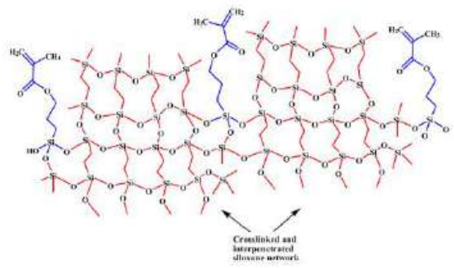


Figure 4 : Extensive siloxane network formed by cross-linking silane monomers, bis-1,2-(triethoxysilyl) ethane(BTSE), with functional silane, 3-acyloxypropyltrimethoxysilane

(Matinlinna et al., 2018)

Mechanical Surface Treatment

1. Hydrofluoric acid etching

Hydrofluoric acid etching will remove the glassy matrix of the glass ceramic which will create the high surface energy with microporosities for the resin composite to penetrate and polymerization, enabling the micromechanical interlocking. (Borges, Sophr, de Goes, Sobrinho, & Chan, 2003)

2. Air-abrasion with Aluminum oxide particle

   Air-abrasion with aluminum oxide particle is able to create the irregularities on the surface of glass ceramic. (Borges et al., 2003)

3. Si deposition methods

   Si deposition methods started in 1984 with the silicoater technology, (Torres et al., 2009) and then in 1989 it was developed into the Rocatec system, and later it was developed into CoJet system. (Kern & Thompson, 1995) These systems are based on the use of 110 μm (Rocatec) or 30 μm (CoJet) Si-coated alumina particles that are blasted onto the ceramic surface. Silica on the outer surface prone to silanization so it requires silane application before cementation. (C. Chen, Kleverlaan, & Feilzer, 2012)

4. Grinding

2.3 Cementation

Cement is a luting material that is created and adhere between the restoration and tooth. Dental cements can be classified into three categories according to setting reaction: the first category is acid-base reaction such as Zinc phosphate cement, polycarboxylate cements, glass-ionomer cements. The second category is polymerization such as resin composite cements and compomer. The third category is the hybrid between first and second categories such as resin-modified glass-ionomer cements.

Resin cement can be categorized by 2 methods. Firstly, it can be divided into three group according how the tooth's surface was prepared by the adhesive system: total-etch, self-etch, and self-adhesive. Example of Total-etch cement: RelyX ARC (3M ESPE), Variolink II (Ivoclar Vivadent), Choice 2 (BISCO) and Calibra®. Example of self-etching cement: Panavia 21 and Panavia F 2.0 (Kuraray Medical; Tokyo, Japan), Multilink N (Ivoclar Vivadent). Example of self-adhesive: RelyX Unicem, (3M ESPE). Self-adhesive cement may be referred to as "all-in-one" resin cements or universal cements. (Radovic, Monticelli, Goracci, Vulicevic, & Ferrari, 2008; Stamatacos & Simon, 2013)

Secondly, Resin composite cement can be divided into 3 polymerization mechanisms. First, chemically activated (Auto-polymerizing): Panavia™, C&B™ Cement. Second, photo activated (Light cure): RelyX™ Veneer Cement, Variolink®

Veneer, Choice™ 2 Light-Cured Veneer Cement. Last, chemical activated with photo activated (dual-activated): NX3 Nexus® Third Generation, RelyX™ ARC Adhesive Resin Cement (3M ESPE), Variolink® II (Ivoclar Vivadent Inc.) In additional, Resin-composite cements have composed of inorganic fillers embedded in organic matrix (i.e. Bis-GMA, TEGDMA, UDMA) like a conventional resin (Blatz, Sadan, & Kern, 2003; Stamatacos & Simon, 2013)

Total-Etch Resin Cements

Total-etch resin cements use a 30% to 40% phosphoric acid-etch to etch dentin and enamel. This etching procedure removes the smear layer, and dentinal tubules are opened. After etching, the adhesive is then applied to the preparation to bond the cement to the tooth. These cements and the adhesives used with them can be light- or dual-cured.

Total-etch resin cements have increased the bond strengths of resin-based cements to nearly that of enamel bonding and have significantly reduced microleakage.This category provides the highest cement-to-tooth bond but also requires the most steps to bond ceramic, composite resin, or metal to the tooth. This multi-step application technique is complex and consequently might compromise bonding effectiveness because each step represents a possible contamination

Self-Etch Resin Cements

Self-etch systems apply a self-etching primer to prepare the tooth surface, and mixed cement is applied over the primer. Bonds to tooth structure using this category of cements are almost as high as those of the total-etch cements. Self-etching systems are popular among dentists because they are easy to use, but as a general category they have demonstrated bond strength to enamel that is weaker than that of total-etch systems.

Therefore, the total-etch, three-step adhesive system of some 30 years ago still sets the standard in terms of versatility and long-term predictability. Resin cements that incorporate self-etching primers eliminate steps during application with the goal of reducing operator errors and technique sensitivity and increasing ease of use. However, it is imperative to follow the manufacturer’s instructions during adhesive cementation, including use of the manufacturer’s adhesive and resin cement combination, because investigators have found incompatibilities between some dual-cure resin cements and simplified adhesive systems.

Self-Adhesive Resin Cements

A number of resin cements have been introduced as onecomponent “universal adhesive cements”; they are said to have good bond strengths to dentin, enamel, and porcelains without the need for separate bonding agents.These self-adhesive cements can bond to an untreated tooth surface that has not been micro-abraded or pretreated with an etchant, primer, or bonding agent; thus, cementation is accomplished in a single step. These cements contain phosphoric acid, which is grafted into the resin. Once mixing is initiated, the phosphoric acid reacts with filler particles and dentin in the presence of water, forming a bond. The resin is polymerized into a cross-linked polymer, as is the case with composite resin bonding.

Light-Cure Resin Cements

Light-cure resin cements utilize photo-initiators, which are activated by light. The ability of light to penetrate all areas and activate the photo-initiators is important with this type of cement. An advantage of light-curing cements is that there can be an increased working time compared to the other cure types. The clinician has ability to remove excess cement before curing, and thus the finishing time required is decreased. Another advantage of light-cure cements is their color stability compared to dual-cure or chemical-cure resin cements. These cements are, therefore, suitable for esthetic restorations and metal-free restorations. Light-polymerized resins are recommended when cementing ceramic that is thin and fairly translucent, allowing the transmission of light through it to reach the resin cement.

Dual-Cure Resin Cements

Dual-cure resin cements are capable of being cured by means of both chemicals and light. Self-cure initiators that can cure the cement are present. In addition, a curing light can be used to activate the photo-initiators that are present in the cement. Dual polymerized resin cements are indicated when the ceramic is too thick or too opaque to allow transmission of light through it. Studies have shown that these dual-cure resin cements still require light-curing to reach a high degree of polymerization. These cements are used for metal-free restorations where light-curing may be performed to quickly seal margins.

Chemical-Cure Resin Cements

Chemical-cure resin cements polymerize with a chemical reaction and are referred to as “self-curing.” This means that two materials must be mixed together to initiate this reaction. These cements are especially useful in areas where light-curing is difficult. Some examples include metal restorations, endodontic posts, and ceramic restorations that prohibit the curing unit from adequately polymerizing the resin cement. Chemically polymerized resin cements do not offer much selection in terms of shade and translucency; therefore, dual-polymerized resin cements can be beneficial. Additionally, accessible areas benefit from light polymerization with dual-polymerized resin cements.

PANAVIA V5

Panavia V5 is the dental self-etching, fluoride-releasing and dual-cured adhesive resin cement. It contains phosphate monomer MDP which provides a strong bond to tooth structure, metal and zirconia. It uses an innovative “Ternary Catalytic system” that consisting of a highly stable peroxide, non-amine reducing agent and highly-active polymerization accelerator which will increase the

color stability of the resin cement and promotes polymerization more effectively. (Kuraray, 2015)

Features

1. Simple cementation procedure
2. Strong bond to tooth structure
3. Outstanding esthetics

2.4 Bond strength

Stress

The force per unit area acting on atoms or molecules in a given plane of material. Stress is the internal resistance of a material to an external load applied on that material. Stress can be classified as tensile, compressive, shear, flexural stress.

Tensile Stress

It is a stress occur when 2 forces are directed away from each other in the same straight line. Also, when one end is fixed and the other end is forced away from the fixed end.

Compressive Stress

It is a stress occur when 2 forces are directed towards each other in the same straight line. Also, when one end is fixed and the other end is forced towards the fixed end.

Shear Stress

It is a stress occur when 2 forces are directed parallel to each other but not in the same straight line.

Flexural Stress

It is forces that create flexural loading (Bending). A flexural force can produce all the types of the stress (Compressive, Tensile, Shear), but in the most cases fracture occurs due to the tensile component. (Anusavice, 2013)

Dislodging force that act to the tooth restored with crown is happened during mastication and parafunction.(Ghasemi, Rismanchian, & Haghayegh, 2018) The complex forces which are applied to the crown interfaces may be resolved into tensile and shear forces dependent on the surface of the tooth under investigation. (Mitchell, Abbariki, & Orr, 2000)

In normal situation, tensile force is rarely applied to the crown, except when masticating high viscosity foods that adhere to the occlusal surfaces of the opposing teeth.(Mitchell et al., 2000) In the C. A. Mitchell study, it was shown that specimens subjected to tensile test, failed in a same manner as in clinical situations. (Mitchell et al., 2000) Also, it was concluded that metal ceramic crowns rarely fail due to dentin fracture and most of the failures occur at cement-preparation interface. So, it is reasonable to use tensile test to evaluate the efficacy of different cements.(Ghasemi et al., 2018)

The importance reason of evaluating shear stresses in dentistry is mainly related to the fact that excessively high stress levels may lead to loss of adhesion at the interface of different materials with the consequent loss of structural integrity and eventually fracture of the restored tooth.(Amarante, Pereira, Darwish, & Camarão, 2008) Shear stress concentrations were also found to be present at core-crown interfaces, predisposing the structure to displacement of the crown during mastication.(Amarante et al., 2008)

2.5 Mode of failure

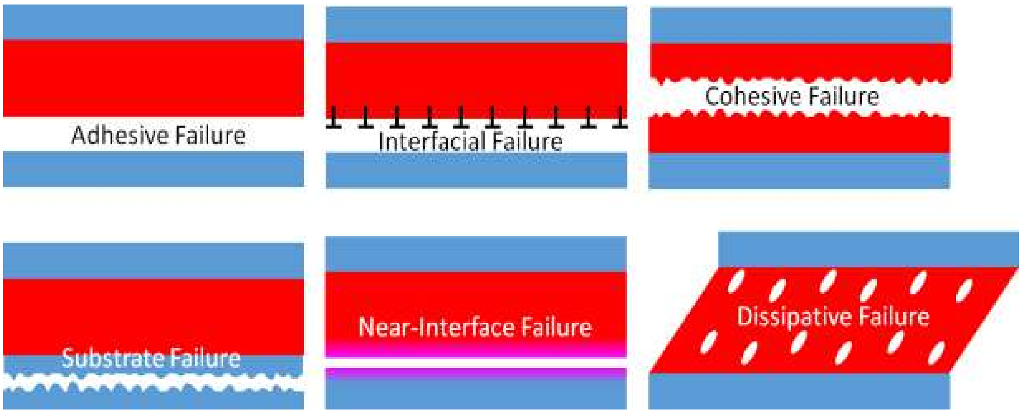


Figure 5 : Mode of failure

(Abbott, *2015*; J. Schwenter et al., *2016)*

Classification of failure type scanning electron microscopy was used to document the surface topography after polishing, etching and de-bonding and to identify the different types of failure: Adhesive failure is failure occurred in the interface. Interfacial failure is failure because of something specific (e.g. hydrolysis) at the interface. Cohesive failure is failure within the adhesive material, partially at the interface. Substrate failure is failure within the substrate. Near-interface is failure adherend and adhesive which are affecting each other locally. Dissipative failure is failure after (large) absorption of energy within the adhesive system.) Abbott, 2015; J. Schwenter et al., 2016)

CHAPTER 3

Materials and Methods

3.1 Materials

1. VITA Enamic (VITA Zahnfabrik, Bad Säckingen,Germany)
2. Super-Bond Universal Ceramic Primer (Sun medical, Japan)
3. Monobond N (Ivoclar Vivadent AG, Liechtenstein)
4. Alloy Primer (KURARAY CO., LTD. Japan)
5. Panavia V5 (KURARAY CO., LTD. Japan)
6. VITA Ceramics Etch) VITA Zahnfabrik(
7. Scotch blue Painter’s Tape) 3M, USA(
8. 3M Wetordry abrasive sheet(3M,USA)
9. Clearfil DC core plus)KURARAY CO., LTD. Japan)

Table 1 : Trade names, manufacturers and compositions of experimental materials

Trade name	Manufacturer	Composition	Lot No.
Monobond N	(Ivoclar Vivadent, Schaan, Liechtenstein)	Ethanol, water, silane methacrylate, phosphoric acid methacrylate, sulphide methacrylate, 10-MDDT: 10-methacryloyloxydecyl 6,8-dithiooctanoate	X17917
Clearfil Ceramic Primer Plus	(Kuraray Noritake dental)	silane coupling agent, MDP, MPTS, ethanol	8T0038
Alloy Primer	(Kuraray Medical inc japan)	MDP , VBATDT, Acetone	870094

Super-Bond Universal Ceramic Primer	(Sun medical, Japan)	Liquid A: MMA, 10- MDP Liquid B: MMA, Silane coupling	RR1
Clearfil DC core plus	(Kuraray Noritake dental)	A Paste Bisphenol A diglycidylmethacrylate (Bis-GMA) , Hydrophobic aliphatic dimethacrylate , Hydrophilic aliphatic dimethacrylate, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica , Colloidal silica, dl-Camphorquinone, Initiators, Pigments B Paste Triethyleneglycol dimethacrylate, Hydrophilic aliphatic dimethacrylate, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, Aluminum oxide filler, Accelerators	220278
Vita ceramic etch	(Vita Zahnfabrik; Bad Säckingen, Germany)	5% hydrofluoric acid	54110

Panavia V5	(Kuraray Noritake dental)	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, Initiators, Accelerators, Silanated barium glass filler, Silanated fluoroaluminosilicate glass filler, Colloidal silica, Silanated aluminium oxide filler, dl Camphorquinone, Pigments	AN0089
VITA Enamic	(VITA Zahnfabrik; Bad Säckingen, Germany)	SiO2, Al2O3, Na2O, K2O, B2O3, ZrO2, CaO, UDMA, TEGDMA	52460
K-etchant	(Kuraray Noritake dental)	35% phosphoric acid	AG0092

3.2 Specimen preparation

The 50 specimens of polymer infiltrated ceramic CAD/CAM materials (VITA® Enamic) is prepared to size 6x7x3 mm by using 4 inches of Low Speed Precision Cutting Machine - IsoMet™ (Buehler,USA). Fix the Enamic into the PVC mold. Metallographic paper P400, P600, P800 P1000 (3M Wetordry abrasive sheet, 3M,USA) are used respectively with pressure of 4 kg/cm^3 (Nano 2000 grinder-polisher with a FEMTO 1000 polishing head, Pace Technologies, USA) to polish the specimens by using clockwise and anticlockwise motion with 200 rpm for 15 minutes per specimen. Then clean all specimens for 2 minutes with deionized water in ultrasonic bath. Place the 80 micrometers thickness tape (Scotch blue Painter's Tape, 3M, USA) size 10x10 mm with the hole of 3 mm in diameter on to the specimen. Use VITA Ceramics Etch with specimen for 60 second and clean with deionized water for 60 second. Dry the specimens by using triple syringe with pressure of 40-50 pound/inches^2 from Mobile dental unit (Thai Dental Products (TDP), Thailand) for 10 second and the distances from triple syringe and specimens is 10 mm.

The specimens are randomly divided into five groups (n=15) with difference primers applied on them for Shear bond strength (SBS) test. Group 1 Control group without restorative primer (C), Group 2 Monobond N (MN), Group 3 Alloy Primer (AP), Group 4 Super-Bond Universal Ceramic Primer (SB) and Group 5 Clearfil Ceramic Primer Plus (CF). After applying the primers following manufacturer's instruction, Panavia V5 is applied on 3 mm in both diameter and height of cylindrical composite resin blocks (Clearfil DC core; Kuraray Medical Inc., Osaka, Japan) and cement the resin block to the specimen by using Durometer (ASTM D 2240TYPE A, D PTC Instruments, USA) with load size of 1000 g. After excess cement is removed, the specimens are light cured by using 1,000 mw/cm^2 (Elipar freelight 2 LED curing light, 3M ESPE, dental products, USA), in the condition, light curing unit perpendicular to the surface of specimens and distance between them is 2 mm. Light cure for 20 seconds in each positions, 12, 3, 6, 9 o'clock while the specimen is still in place for 10 minutes. Then the specimens are incubated in 37°C water for 24 +/- 2 hours. The SBS test is performed by using a universal testing machine (EZ-S 500 N; Shimadzu) with the cross-head speed of 0.5 mm/min. Mode of failure is performed with the stereomicroscope at 35x magnificent level and classified into 3 categories: 1. Adhesive failure 2. Cohesive failure 3. Mixed failure

Figure 6. To assess the potential of chemical bonding, shear bond strength tests were performed on polished surfaces (N=50)

Table 2 : manufacturer's instruction for each primer

Primer	Instruction
MN: Monobond N (Ivoclar Vivadent AG, Liechtenstein)	1. Preparing the specimens – Etch the specimens with 5% hydrofluoric acid for 60 seconds – Thoroughly rinse the specimens with water spray – Air dry 2. Apply a thin coat of Monobond N with a Microbrush to the pre-treated surfaces. Allow the material to react for 60 seconds. Subsequently, disperse any remaining excess with a strong stream of air.

AP: Alloy Primer (KURARAV CO.,LTD. Japan)	1. Preparing the specimens – Etch the specimens with 5% hydrofluoric acid for 60 seconds – Thoroughly rinse the specimens with water spray – Air dry 2. Apply the Alloy Primer to specimens with brush and leave it for drying.
SB: Super-Bond Universal Ceramic Primer (Sun medical, Japan)	1. Preparing the specimens – Etch the specimens with 5% hydrofluoric acid for 60 seconds – Thoroughly rinse the specimens with water spray – Air dry 2. Dispense an equal number of drops of UNIVERSAL PRIMER Parts A & B (1:1 ratio) into a clean mixing well. Replace the cap to avoid evaporation of solvent. 4. Apply UNIVERSAL PRIMER, scrubbing the specimens with a microbrush for 10-15 seconds. Do not light cure between coats. 5. Evaporate excess solvent by strongly air-drying with an air syringe for at least 10 seconds; there should be no visible movement of the adhesive. The surface should have a uniform glossy appearance; otherwise, apply an additional coat of UNIVERSAL PRIMER and repeat Step 5. 6. Light cure for 10 seconds.
CF : Clearfil Ceramic Primer Plus	1.If the adherent surface is composite resin, apply an etching agent (e.g. K-ETCHANT Syringe) onto the adherent surface and leave it for 5 seconds. Then thoroughly wash and dry. 2.Dispense the necessary amount of Clearfil Ceramic Primer Plus into a well of the mixing dish immediately before application. 3.Apply Clearfil Ceramic Primer Plus to the adherent surface of the restoration with an applicator brush. 4.Dry the entire adherent surface sufficiently using mild, oil-free air flow.

3.3 **Failure type**

After testing, failure types of all specimens were assessed using reflected-light microscope and categorized. For the adhesive type, the strength failure occurred in the interface between CAD/CAM. material and resin composite cement. For the cohesive type the failure was in the CAD/CAM material or in the resin composite cement. (Bahr et al., 2013)

3.4 Statistically analysis

The SBS data were analyzed by Kolmogorov Smirnov test for normal distribution in each group and Levene’s test for homogeneity of variances between groups. One-way ANOVA and post hoc Tamhane’s T2 used for parametric analysis.

Chapter 4

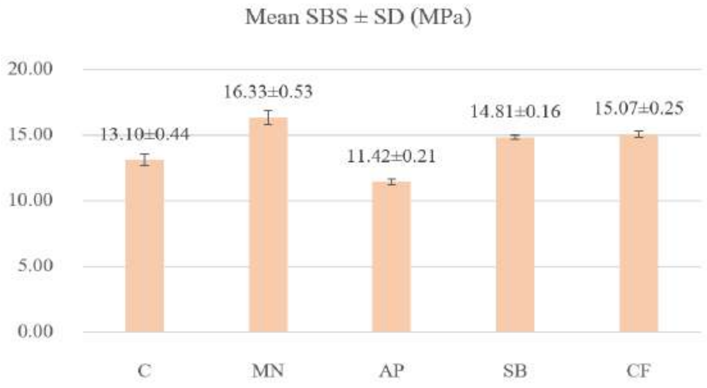
Results

The data was analyzed by Kolmogorov Smirnov test and presented normal distribution in all groups. However, the Levene’s test found that the homogeneity of variances are unequal. Thus, One-way ANOVA and post hoc Tamhane’s T2 tests were used for the comparison test.

The mean SBS values of all groups were summarized as shown in Table1. One-way ANOVA showed the significantly different between groups. From the Tamhane’s T2 test, the mean SBS value of MN group, CF group and SB group showed significantly higher than the control group ($p < 0.05$). Among these three groups, the MN group showed the highest SBS values with no significant different from CF group ($p>0.05$) but significantly higher than SB group ($p<0.05$). While the CF groups showed not significant different from SB group($p>0.05$). Only AP group was significantly lower than control group ($p < 0.05$).

After evaluating the specimens under the stereoscope, there were damages in VITA Enamic in every group except the control group. The specimens in all groups were mostly found to be adhesive failure, the control group has the highest number of adhesive failures following by the AP group, CF group, SB group and MN group respectively. The cohesive failure was only found in SB group with only 10% and the rest was found to be mixed failure in every group.

	Mean SBS
Control	13.10±0.44^{C}
MN	16.33±0.53^{A}
AP	11.42±0.21^{D}
SB	14.81±0.16^{B}
CF	15.07±0.25^{AB}



Different superscripts mean statistically significant differences between those mean values. ($p<0.05$)

Table 3 : Mean shear bond strength value (MPa) and standard deviation of each group (mean±SD).

	Adhesive Failure	Mixed Failure	Cohesive Failure	Crack in VITA Enamic
Control	10	-	-	-
AP	9	1	-	2
CF	7	3	-	4
SB	6	3	1	8
MN	5	5	-	8
Total	37	12	1	22

Figure 7 : Mode of failure

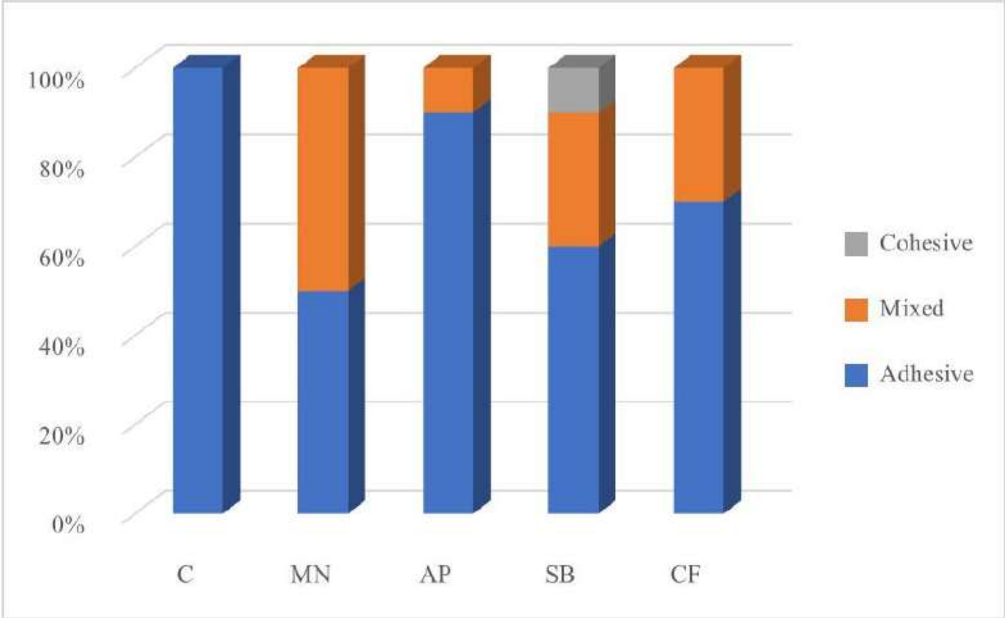
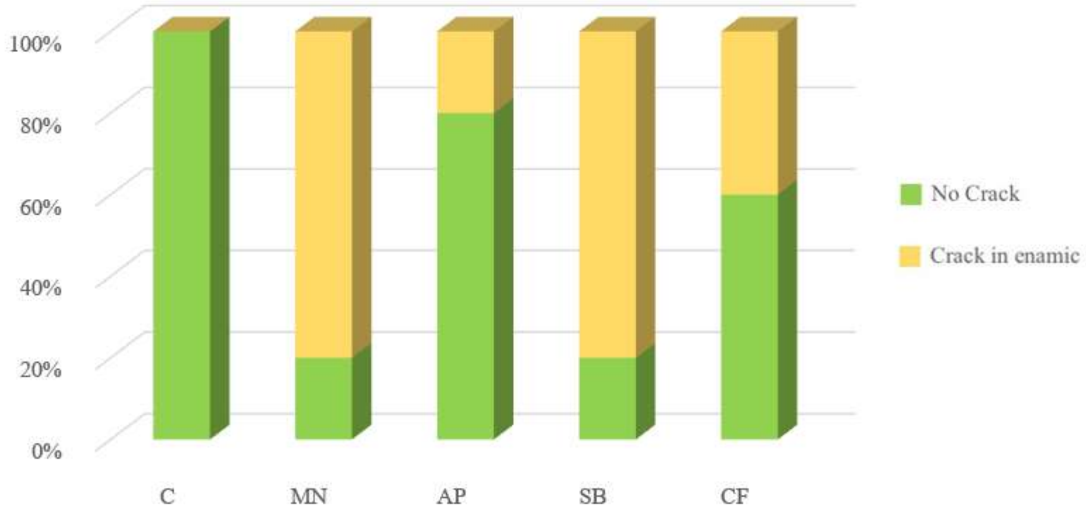


Figure 8 : Crack in Enamic



Chapter 5

Discussions and Conclusions

Discussion

To achieve higher bonding strength in ceramic restoration, treating surface of the restoration is required, both mechanical and chemical approach. Roughening surface by grinding, etching and sandblasting can improve the mechanical bond. While using various types of primer according to the chemical structure of restoration material can provide a chemical bond. (Chen, 2012; Mair & Padipatvuthikul, 2010)

VITA Enamic (Polymer-infiltrated ceramic network) were used in this study, composing of ceramic matrix (86% in weight/75% in volume) and polymer matrix (14% in weight/25% by volume). (VITA, 2014)The recommended surface treatment for VITA Enamic were application 5% hydrofluoric acid for 60 seconds, rinsed for 60 seconds and dry. Then, the silane coupling agent was applied for chemical surface treatment. Similar to the surface treatment method for the silica-based ceramics. (Domka, Krysztafkiewicz, & Kozak, 2002; Jayasuriya, 2017; Judith Schwenter, Fredy Schmidli, Roland Weiger, & Jens Fischer, 2016; Pollyanna Silva, Machado Martinelli-Lobo, Bottino, Melo, & Valandro, 2018; Thompson, Stoner, Piascik, & Smith, 2011) According to Cui et al, the degree of conversion of polymer matrix in VITA Enamic is 82.17%, hence the polymer matrix seems to be a minor part to create the chemical bond with resin cement. Further studies may focus on the role of polymer matrix in VITA Enamic on bonding ability, both mechanical and chemical, to resin cement. (Cui et al., 2017)

Four different primers used in this study were selected according to the different types of functional monomers and its composition. Alloy Primer is composed of VBATDT and MDP. Monobond N is composed of silane coupling agent, MDP and MDDT. Super-Bond Universal Ceramic Primer and Clearfil Ceramic Primer Plus is composed of MDP and silane coupling agent.

Silane is used for promoting an adhesion due to the promotion of the wettability of the surface restoration and the chemical bond between resin to silica-based or silica-coated indirect restorative materials. The siloxane group (inorganofunctional group) of the silane coupling agent can react with the silica or silica coating substrate (Matinlinna et al., 2018), while the organofunctional group such as methacrylate group can copolymerized with the resin matrix. (Moro et al., 2017) In 2019, Awad et al, studied the μTBS between VITA Enamic and Variolink esthetic DC, the results showed that the application of the silane-based primer was significantly higher than silane-containing Universal adhesive and silane-free Universal adhesive. (Awad et al., 2019) This confirms the important role of silane according to promoting the chemical bond of glass component in VITA Enamic and resin cements. From the results of our study, MN group, CF group, and SB group can enhance the SBS value compared to the control group, which is in agreement with the previous recent studies. (Awad et al., 2019),(Pnfd Silva et al., 2018)

Although MN group, CF group, and SB group are all contain silane coupling agent, but the difference in its composition and the system of using might affect the bond strength differently. Monobond N primer and Clearfil Ceramic Primer Plus are single bottle primer which is different from Super-Bond Universal Ceramic Primer, which is two bottle system as shown in Table 1 . The mixing ratio of liquid A and liquid B might affect the shear bond strength due to the among of the silane coupling agent may different in concentration. Thus, further studies may observe the effect of different ratio of liquid A and liquid B on bonding ability between glass containing restoration to resin cement.

In AP group, which contains 10-MDP and VBATDT, showed the lowest mean SBS values. The 10-MDP is used for forming the chemical bond between metal oxide layer or zirconia oxide layer by using phosphate group (PO_4^{3-}) and resin cement by using methacrylate group. (Chuang et al., 2017; Murillo-Gómez, 2017) The VITA Enamic contains the silica-based in the ceramic matrix, hence 10-MDP might not promote the chemical bond to the VITA Enamic. (Peumans, Bajraktarova Valjakova, Munck, Mishevska, & Meerbeek, 2016; Rohr, Brunner, Märtin, & Fischer, 2017) VBATDT is a primer that contains thiol group and vinyl group. The coupling mechanism occurs by transforming thione in thiol and formation of the bond on the precious metal surface, and copolymerization of the vinyl groups with the resin cement (Silikas, Wincott, Vaughan, Watts, & Eliades, 2007), these 2 functional monomers may not enhance the bond strength between resin cement and VITA Enamic.

Only AP group shown the lower mean SBS value than the control group, AP group can have a negative effect on bonding strength. According to the studies of Awad et al and Pnfd Silva et al, the excess unreacted thiol group in VBATDT can causes a chain transfer reaction during the propagation of vinyl or acrylic free radicals and that the monomer inhibits the polymerization of resin cements. (Awad et al., 2019; Pnfd Silva et al., 2018) Therefore, when resin cements use benzoyl peroxide (BPO)-Amine redox system, bond strength may deteriorate at the primer-applied surface. On the other hand, the functional group in Monobond N (10-MDDT) contains disulfide group which cannot be oxidized so it will not inhibit the polymerization of the resin cement.

Silane coupling agents improve the bond strength on VITA Enamic since silicate surfaces of VITA Enamic allow for covalent Si-O-Si bonds between ceramic and silane coupling molecules. As on silanized (MN, CF, and SB group) VITA Enamic shows more cohesive or mixed fractures compared to control group. While AP group and control group show mostly adhesive failure between resin cement and VITA Enamic. The failure of bond interface means that the bond in that area is weakest. Thus, it can be concluded that the bond strength between resin cement and VITA Enamic which established by silane coupling molecules, exceeds the fracture resistance of the resin cement. (Judith Schwenter et al., 2016) Moreover in MN, CF and SB group showed cracked in VITA Enamic more than SB and control group which in conform with the mode of failure that were observed.

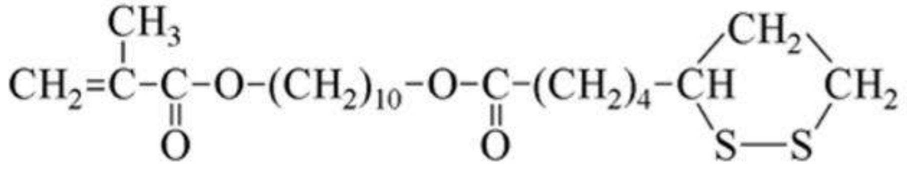


Figure 9 : 10-methacryloyloxydecyl 6,8-dithiooctanoate (10-MDDT)

Courtesy from : (Shimoe et al., 2010)

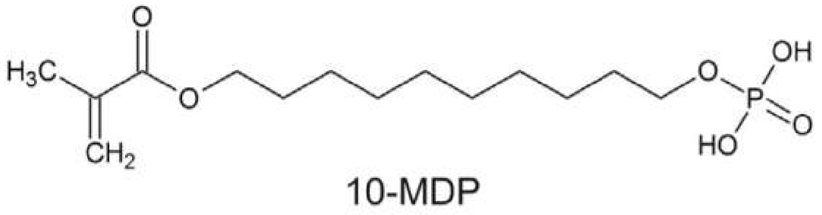


Figure 10 : 10-Methacryloyloxydecyl dihydrogen phosphate

Courtesy from : (Tian et al., 2016)

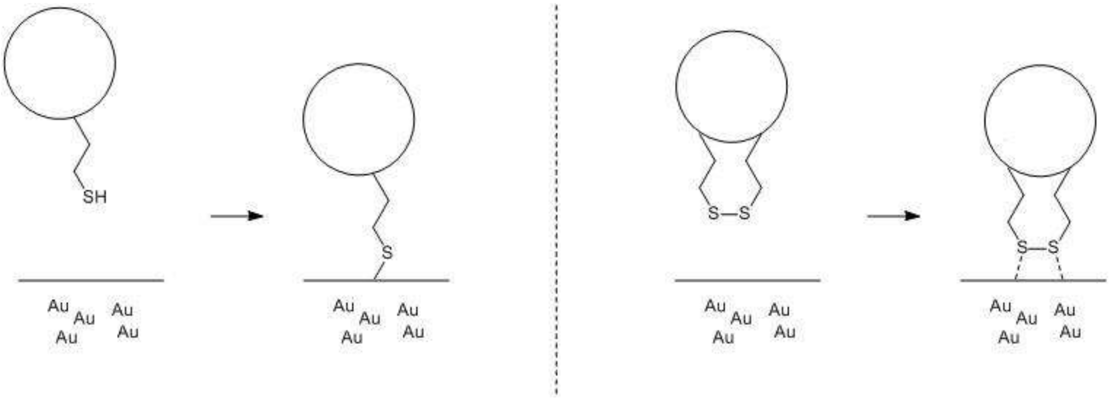


Figure 11 : Interaction of disulfide and mercaptan group to gold

Courtesy from : https://syntheticremarks.com/what-is-the-true-nature-of-gold-sulfur-bonds/

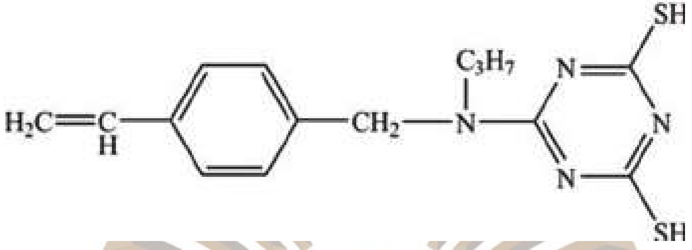


Figure 12 : 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4- dithione (VBATDT)

Courtesy from : (M. Zakir, Chu, Matinlinna, Tsoi, & Lung, 2014)

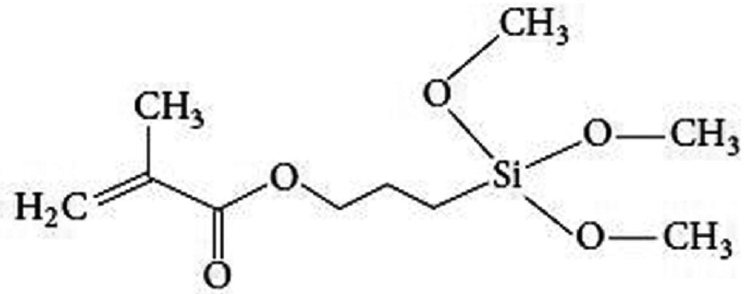


Figure 13 : 3 - Trimethoxysilyl propyl methacrylate (3-MPS)

Courtesy from : (Elshereksi, Ghazali, Muchtar, & Azhari, 2017)

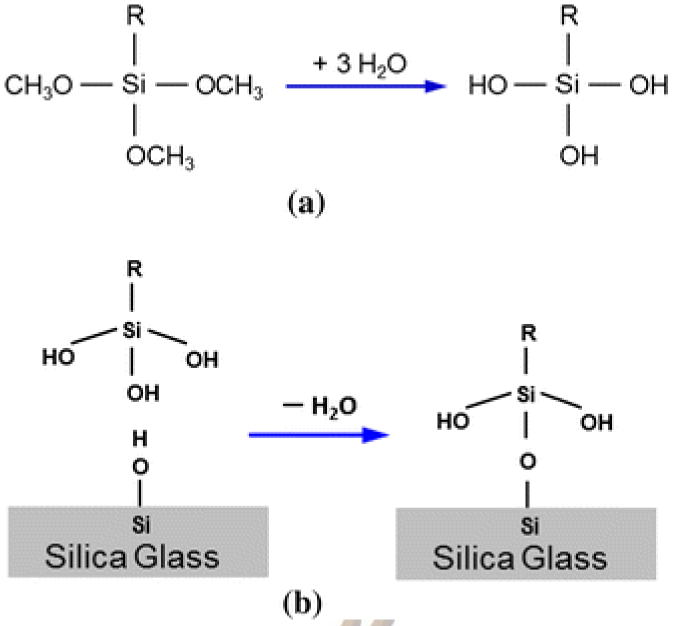


Figure 14 : Reactions showing a hydrolysis of Silane and coupling of silane with silica

Courtesy from : (Chowdhury & Gillespie Jr, 2017)

CONCLUSION

From the result of this study, it could be concluded as following:

1. Restorative primers that contain Silane coupling agent promote the higher SBS between VITA Enamic and resin composite via Panavia V5 than without using any primer. Due to the chemical reaction with Silica-component in VITA Enamic.
2. Restorative primers that contain VBATDT decreased in SBS between VITA Enamic and resin composite via Panavia V5 comparing to others.
3. 10-MDP in restorative primers or resin cement will not increase the SBS between VITA Enamic and composite resin.

The restorative primer may affect, both positive and negative, to the bond strength. Thus, dentist should properly select restorative primer to enhance the bond strength of the restoration.

References

Abbott, S. (2015). *Adhesion science: Principles and practice*: Lancaster, Pennyslvania : DEStech Publications, [2015] ©2015.

Aida, M., Hayakawa, T., & Mizukawa, K. (1995). Adhesion of composite to porcelain with various surface conditions. *J Prosthet Dent, 73*(5), 464-470.

Albakry, M., Guazzato, M., & Swain, M. V. (2003). Fracture toughness and hardness evaluation of three pressable all-ceramic dental materials. *J Dent, 31*(3), 181-188. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/12726702

Albero, A., Pascual, A., Camps, I., & Grau-Benitez, M. (2015). Comparative characterization of a novel cad-cam polymer-infiltrated-ceramic-network. *J Clin Exp Dent, 7*(4), e495-500. doi:10.4317/jced.52521

Amarante, M. V., Pereira, M. V. S., Darwish, F. A. I., & Camarão, A. F. (2008). Virtual analysis of stresses in human teeth restored with esthetic posts. *Materials Research, 11*, 459-463. Retrieved from http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-14392008000400014&nrm=iso

Anusavice, K. J., Phillips, R. W., Shen, C., & Rawls, H. R. (2013). Phillips' science of dental materials. *Elsevier/Saunders., 13*.

Awad, M. M., Albedaiwi, L., Almahdy, A., Khan, R., Silikas, N., Hatamleh, M. M., . . . Alrahlah, A. (2019). Effect of universal adhesives on microtensile bond strength to hybrid ceramic. *BMC Oral Health, 19*(1), 178. doi:10.1186/s12903-019-0865-7

Blatz, M. B., Sadan, A., & Kern, M. (2003). Resin-ceramic bonding: a review of the literature. *J Prosthet Dent, 89*(3), 268-274. doi:10.1067/mpr.2003.50

Borges, G. A., Sophr, A. M., de Goes, M. F., Sobrinho, L. C., & Chan, D. C. (2003). Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent, 89*(5), 479-488. doi:10.1016/S0022391302527049

Chang, J. C., Hurst, T. L., Hart, D. A., & Estey, A. W. (2002). 4-META use in dentistry: a literature review. *J Prosthet Dent, 87*(2), 216-224. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/11854680

Chen. (2012). Bonding of Resin Materials to All-Ceramics: A Review. *Current Research in Dentistry, 3*, 7-17. doi:10.3844/crdsp.2012.7.17

Chen, C., Kleverlaan, C. J., & Feilzer, A. J. (2012). Effect of an experimental zirconia-silica coating technique on micro tensile bond strength of zirconia in different priming conditions. *Dent Mater, 28*(8), e127-134. doi:10.1016/j.dental.2012.04.020

Chowdhury, S., & Gillespie Jr, J. (2017). Silica–silane coupling agent interphase properties using molecular dynamics simulations. *Journal of Materials Science, 52*, 1-18. doi:10.1007/s10853-017-1412-z

Chuang, S. F., Kang, L. L., Liu, Y. C., Lin, J. C., Wang, C. C., Chen, H. M., & Tai, C. K. (2017). Effects of silane- and MDP-based primers application orders on zirconia-resin adhesion-A ToF-SIMS study. *Dent Mater, 33*(8), 923-933. doi:10.1016/j.dental.2017.04.027

Cui, B., Li, J., Wang, H., Lin, Y., Shen, Y., Li, M., . . . Nan, C. (2017). Mechanical properties of polymer-infiltrated-ceramic (sodium aluminum silicate) composites for dental restoration. *J Dent, 62*, 91-97. doi:10.1016/j.jdent.2017.05.009

Della Bona, A., Mecholsky, J. J., Jr., & Anusavice, K. J. (2004). Fracture behavior of lithia disilicate- and leucite-based ceramics. *Dent Mater, 20*(10), 956-962. doi:10.1016/j.dental.2004.02.004

Della Bona, A., Mecholsky, J. J., Jr., Barrett, A. A., & Griggs, J. A. (2008). Characterization of glass-infiltrated alumina-based ceramics. *Dent Mater, 24*(11), 1568-1574. doi:10.1016/j.dental.2008.06.005

Dirxen, C., Blunck, U., & Preissner, S. (2013). Clinical Performance of a New Biomimetic Double Network Material. *The Open Dentistry Journal, 7*, 118-122. doi:10.2174/1874210620130904003

Domka, L., Krysztafkiewicz, A., & Kozak, M. (2002). Silane Modified Fillers for Reinforcing Polymers. *Polymers & Polymer Composites, 10*, 541-552. doi:10.1177/096739110201000706

Elshereksi, N. W., Ghazali, M., Muchtar, A., & Azhari, C. H. (2017). Review of titanate coupling agents and their application for dental composite fabrication. *Dent Mater J, 36*(5), 539-552. doi:10.4012/dmj.2016-014

Facenda, J. C., Borba, M., & Corazza, P. H. (2018). A literature review on the new polymer-infiltrated ceramic-network material (PICN). *J Esthet Restor Dent*. doi:10.1111/jerd.12370

Ghasemi, E., Rismanchian, M., & Haghayegh, N. (2018). Evaluation of Tensile Bond Strength between Dentin and Metallic Copings Using Different Resin Cements. *Journal of Dental Materials and Techniques, 7*(3), 135-140. doi:10.22038/jdmt.2018.11115

Giordano, R., & McLaren, E. A. (2010). Ceramics overview: classification by microstructure and processing methods. *Compend Contin Educ Dent, 31*(9), 682-684, 686, 688 passim; quiz 698, 700.

Gracis, S., Thompson, V. P., Ferencz, J. L., Silva, N. R., & Bonfante, E. A. (2015). A new classification system for all-ceramic and ceramic-like restorative materials. *Int J Prosthodont, 28*(3), 227-235. doi:10.11607/ijp.4244

Guazzato, M., Albakry, M., Swain, M. V., & Ironside, J. (2002). Mechanical properties of In-Ceram Alumina and In-Ceram Zirconia. *Int J Prosthodont, 15*(4), 339-346. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/12170847

I., V. (2009). E.max Lithium disilicate The Future of All-Ceramic Dentistry. *0*, 2-14.

Isabelle, D. H., Julie. (2010). Ceramics for Dental Applications: A Review. *Materials. 3. 10.3390/ma3010351*.

Jayasuriya, C. K. (2017). Interfacial Bonding in Polymer–Ceramic Nanocomposites. In.

Jorquera Rivera, G. M., Nicole & Walls, Stephanie & Mahn, Eduardo & Fernández, Eduardo. (2016). Simplified Classification for Dental Ceramics. *Journal of dental science and therapy, 1. 1. 10.24218/jdst.2016.09.* .

Kern, M., & Thompson, V. P. (1995). Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. *J Prosthet Dent, 73*(3), 240-249. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/7760272

Kramer, N., & Frankenberger, R. (2005). Clinical performance of bonded leucite-reinforced glass ceramic inlays and onlays after eight years. *Dent Mater, 21*(3), 262-271. doi:10.1016/j.dental.2004.03.009

Kuraray, A. (2015). Panavia V5. In.

Mair, L., & Padipatvuthikul, P. (2010). Variables related to materials and preparing for bond strength testing irrespective of the test protocol. *Dent Mater, 26*(2), e17-23. doi:10.1016/j.dental.2009.11.154

Matinlinna, J. P., Lassila, L. V., Ozcan, M., Yli-Urpo, A., & Vallittu, P. K. (2004). An introduction to silanes and their clinical applications in dentistry. *Int J Prosthodont, 17*(2), 155-164.

Matinlinna, J. P., Lung, C. Y. K., & Tsoi, J. K. H. (2018). Silane adhesion mechanism in dental applications and surface treatments: A review. *Dent Mater, 34*(1), 13-28. doi:10.1016/j.dental.2017.09.002

Mitchell, C. A., Abbariki, M., & Orr, J. F. (2000). The influence of luting cement on the probabilities of survival and modes of failure of cast full-coverage crowns. *Dent Mater, 16*(3), 198-206.

Moro, A. F. V., Ramos, A. B., Rocha, G. M., & Perez, C. D. R. (2017). Effect of prior silane application on the bond strength of a universal adhesive to a lithium disilicate ceramic. *J Prosthet Dent, 118*(5), 666-671. doi:10.1016/j.prosdent.2016.12.021

Murillo-Gómez, F. G., Mario. (2017). Effect of Different Silane Treatments on Long-Term Bonding Between Non-Etched Glass-Ceramic and Resin Cement. *Odovtos - International Journal of Dental Sciences*.

Noda, Y., Nakajima, M., Takahashi, M., Mamanee, T., Hosaka, K., Takagaki, T., . . . Tagami, J. (2017). The effect of five kinds of surface treatment agents on the bond strength to various ceramics with thermocycle aging. *Dent Mater J, 36*(6), 755-761. doi:10.4012/dmj.2016-383

Özcan, M. (2003). Adhesion of resin composites to biomaterials in dentistry: an evaluation of surface conditioning methods. *Groningen: s.n.*, 170 p.

Peumans, M., Bajraktarova Valjakova, E., Munck, J., Mishevska, C., & Meerbeek, B. (2016). Bonding Effectiveness of Luting Composites to Different CAD/CAM Materials. *The journal of adhesive dentistry, 18*. doi:10.3290/j.jad.a36155

Radovic, I., Monticelli, F., Goracci, C., Vulicevic, Z. R., & Ferrari, M. (2008). Self-adhesive resin cements: a literature review. *J Adhes Dent, 10*(4), 251-258. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/18792695

Rohr, N., Brunner, S., Märtin, S., & Fischer, J. (2017). Influence of cement type and ceramic primer on retention of polymer-infiltrated ceramic crowns to a one-piece zirconia implant. *The Journal of Prosthetic Dentistry, 119*. doi:10.1016/j.prosdent.2017.02.002

Rosenblum, M. A., & Schulman, A. (1997). A review of all-ceramic restorations. *J Am Dent Assoc, 128*(3), 297-307. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/9066214

Schwenter, J., Schmidli, F., Weiger, R., & Fischer, J. (2016). Adhesive bonding to polymer infiltrated ceramic. *Dent Mater J, 35*(5), 796-802. doi:10.4012/dmj.2015-271

Schwenter, J., Schmidli, F., Weiger, R., & Fischer, J. (2016). Adhesive bonding to polymer infiltrated ceramic. *Dental Materials Journal, 35*, 796-802. doi:10.4012/dmj.2015-271

Shenoy, A., & Shenoy, N. (2010). Dental ceramics: An update. *Journal of Conservative Dentistry, 13*(4), 195-203. doi:10.4103/0972-0707.73379

Shenoy, A., & Shenoy, N. (2010). Dental ceramics: An update. *J Conserv Dent, 13*(4), 195-203. doi:10.4103/0972-0707.73379

Shimoe, S., Tanoue, N., Satoda, T., Murayama, T., Nikawa, H., & Matsumura, H. (2010). Evaluation of single liquid primers with organic sulfur compound for bonding between indirect composite material and silver-palladium-copper-gold alloy. *Dent Mater J, 29*(1), 25-29. doi:10.4012/dmj.2009-039

Silikas, N., Wincott, P. L., Vaughan, D., Watts, D. C., & Eliades, G. (2007). Surface characterization of precious alloys treated with thione metal primers. *Dent Mater, 23*(6), 665-673. doi:10.1016/j.dental.2006.06.007

Silva, P., Machado Martinelli-Lobo, C., Bottino, M., Melo, R., & Valandro, L. (2018). Bond strength between a polymer-infiltrated ceramic network and a composite for repair: effect of several ceramic surface treatments. *Brazilian Oral Research, 32*. doi:10.1590/1807-3107bor-2018.vol32.0028

Silva, P., Martinelli-Lobo, C. M., Bottino, M. A., Melo, R. M., & Valandro, L. F. (2018). Bond strength between a polymer-infiltrated ceramic network and a composite for repair: effect of several ceramic surface treatments. *Braz Oral Res, 32*, e28. doi:10.1590/1807-3107bor-2018.vol32.0028

Stamatacos, C., & Simon, J. F. (2013). Cementation of indirect restorations: an overview of resin cements. *Compend Contin Educ Dent, 34*(1), 42-44, 46. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/23550330

Takeuchi, K., Fujishima, A., Manabe, A., Kuriyama, S., Hotta, Y., Tamaki, Y., & Miyazaki, T. (2010). Combination treatment of tribochemical treatment and phosphoric acid ester monomer of zirconia ceramics enhances the bonding durability of resin-based luting cements. *Dent Mater J, 29*(3), 316-323. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/20484831

Thompson, J. Y., Stoner, B. R., Piascik, J. R., & Smith, R. (2011). Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? *Dent Mater, 27*(1), 71-82. doi:10.1016/j.dental.2010.10.022

Tian, F. C., Wang, X. Y., Huang, Q., Niu, L. N., Mitchell, J., Zhang, Z. Y., . . . Tay, F. R. (2016). Effect of nanolayering of calcium salts of phosphoric acid ester monomers on the durability of resin-dentin bonds. *Acta Biomater, 38*, 190-200. doi:10.1016/j.actbio.2016.04.034

Torres, S. M., Borges, G. A., Spohr, A. M., Cury, A. A., Yadav, S., & Platt, J. A. (2009). The effect of surface treatments on the micro-shear bond strength of a resin luting agent and four all-ceramic systems. *Oper Dent, 34*(4), 399-407. doi:10.2341/08-87

VITA. (2014). VITA ENAMIC® Working Instructions (6). Retrieved from https://mam.vita-zahnfabrik.com/portal/ecms_mdb_download.php?id=31792&sprache=en&fallback=&cls_session_id=&neuste_version=1

Xu, Z., Yu, P., Arola, D. D., Min, J., & Gao, S. (2017). A comparative study on the wear behavior of a polymer infiltrated ceramic network (PICN) material and tooth enamel. *Dent Mater, 33*(12), 1351-1361. doi:10.1016/j.dental.2017.08.190

Zakir, M., Chu, C.-H., Matinlinna, J., Tsoi, J., & Lung, C. (2014). Bonding Dissimilar Materials in Dentistry: A Critical Review. *REVIEWS OF ADHESION AND ADHESIVES, 2*, 413-432. doi:10.7569/RAA.2014.097309

Zakir, M. A., Usman & Tian, Tian & Han, Aifang & Qiao, Wei & Jin, Xiaozhuang & Zhang, M & Tsoi, James & Matinlinna, Jukka. (2016). The Role of Silane Coupling Agents and Universal Primers in Durable Adhesion to Dental Restorative Materials - a Review. *Current Oral Health Reports, 10.1007/s40496-016-0108-9*.

ประวัติผู้วิจัย

ไฟล์รูปภาพ
นกวิจยขนำด
150*200

คำนำหน้า ☐ นาย ☐ นาง ☑ นางสาว
ตำแหน่งทางวิชาการ ☐ ศ . ☐ .รศ☐ ผศ ☑นๆ
ชื่อผู้วิจัย สปัน
นามสกุลผู้วิจัย เล็งเลิศผล
ชื่อภาษาอังกฤษ SPUN
นามสกุลภาษาอังกฤษ LENGLERDPHOL
วันปี เกิด/เดือน/ 3 กุมภาพันธ์ 2524
ที่อยู่(บ้าน) 52/501 หมู่บ้านเมืองเอก ตเมือง.หลักหก อ.
จังหวัด(บ้าน) ปทุมธานี
รหัสไปรษณีย์(บ้าน) 12000
โทรศัพท์(บ้าน) 082-416-9942
แฟ็กซ์(บ้าน) -
ที่อยู่(ที่ทำงาน) คณะทันตแพทยศาสตร์ มหาวิทยาลัยรังสิต
จังหวัด(ที่ทำงาน) ปทุมธานี
รหัสไปรษณีย์(ที่ทำงาน) 12000
โทรศัพท์(ที่ทำงาน) 02-7916000 ต่อ 4267
แฟ็กซ์(ที่ทำงาน)
E-mail Address : spun.l@rsu.ac.th

ปริญญาตรี
สาขา ทันตแพทยศาสตรบัณฑิต
ปีที่จบ 2546
สถาบัน มหาวิทยาลัยมหิดล
ประเทศ ไทย

ปริญญาโท
สาขา วิทยาศาสตร์มหาบัณฑิต (ทันตกรรรมประดิษฐ์(
ปีที่จบ 2553

สถาบัน จุฬาลงกรณ์มหาวิทยาลัย
ประเทศ ไทย

ผลงานวิจัยที่ได้นำเสนอในการประชุมทางวิชาการภายในประเทศโปรดระบุหัวข้อ)
(มนาและสถานที่สัม/ประชุม
“การรั่วซึมของฟันที่บูรณะด้วยเรซินคอมโพสิตอินเลย์โดยใช้สารยึดเรซินก่อนและหลังการฟอกสีฟัน”
(Leakage of Tooth restored with Resin Composite Inlay using Resin adhesive Before and After)
งานประชุมทางวิชาการเสนอผลงานวิจัยระดับบัณฑิตศึกษาครั้งที่ 11
มหาวิทยาลัยขอนแก่น

“เสถียรภาพเชิงมิติของวัสดุพิมพ์ปากชนิดซิลิโคนเมื่อเก็บไว้ในเวลาต่างกัน “
(Dimensional Stability of Silicone Impression Material at Different Time)
การประชุมวิชาการระดับชาติ ประจำปี ๒๕๕๗)National Research Conference 2014)
มหาวิทยาลัยรังสิต

ผลงานวิจัยที่ได้นำเสนอในการประชุมทางวิชาการในต่างประเทศโปรดระบุหัวข้อ)
(สัมมนาและสถานที่/ประชุม
Effect of tooth-bleaching with 38% hydrogen peroxide on marginal seal.
IADR 2009, Wuhan, China
Effects of surface coating agents to surface hardness of bis-acryl provisional restorations
IADR 2019, Vancouver, Canada
สาขาวิชาที่นักวิจัยเชี่ยวชาญ
ทันตกรรมประดิษฐ์ ทันตวัสดุศาสตร์