Bonding effect of silane in universal adhesive on lithium disilicate glass ceramic

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Bonding effect of silane in universal adhesive on lithium disilicate glass ceramic

Directed by Professor Kwang-mahn Kim

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To *Byoung I Suh*,
who introduce me new scientific world,

to my *family*
who always have loved me unconditionally
and gave me the reason that I have to live,

to my friends and colleagues
who have supported and encouraged me

to Dr. Liang Chen
who provide me academic support

and

to Prof. *Kyoung-Nam Kim and Kwang-Mahn Kim*
who introduced me to dental biomaterials

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ABSTRACT

Bonding effect of silane in universal adhesive on lithium disilicate glass ceramic

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(Directed by Professor Kwang-Mahn Kim, D.D.S., Ph.D.)

The purpose of this study is to evaluate the effectiveness of silane on the surface treatment of silica based ceramic when silane is incorporated into the universal adhesive versus using silane separately from the adhesive.

The materials used in this study are as follows: only All-Bond Universal, Silane mixed with All-Bond Universal, only Porcelain Primer (which is a regular silane primer), and SBU (which is a commercial silane-containing universal adhesive). IPS e-Max press, a lithium disilicate, was used as the silica-based ceramic. In terms of shade matching, IPS e-max press has more aesthetic characteristics compared to any other CAD-CAM ceramic block. Duolink resin cement was used as a composite applied to the surface to test for shear bond strength.

Contact angle test is a measurement of hydrophobicity on a silica-based ceramic surface. The more hydrophobic the surface of the ceramic is, the better bonding strength to the adhesive. Shear bond strength was tested by universal testing machine (Instron 4466 crosshead speed=1mm/min). The surfaces of the specimen were polished in order to eliminate any bias from micro mechanical retention due to an uneven surface. Only silane applied to the silica based ceramic surface, result in highest contact angle and shear bond strength.

Silane should be used separately from a hydrophobic resin monomer in order to maximize its porcelain surface treatment function. Using silane separately and using silane incorporated into the universal adhesive (contains hydrophobic resin) do not perform equally on silica-based ceramic.

Key words: Silane, Universal adhesive, One bottle adhesive, Surface treatment, Silica based ceramic, Lithium disilicate,

Bonding effect of silane in universal adhesive on lithium disilicate glass ceramic

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I. INTRODUCTION

3-Methacryloxyproyltrimethoxysilane(MPS) is the commonly used clinical commercial silane primers (Zhang et al., 2014). There are two types of silane, one is a pre-hydrolyzed one-bottle system and the other one is a two-bottle system. Pre-hydrolyzed silane results in a self-condensation reaction over time, and thus cannot be used for the clinical purpose. This also results in a shorter shelf life compared to the two-bottled one. The two-bottle system contains un-hydrolyzed silane in ethanol in one bottle and an aqueous acetic acid solution in the other. Keeping them in separate bottles increases the shelf life of silane (Lung and Matinlinna, 2012). The combined use of silane with a resin adhesive improves the bond strength of the composite to the glass ceramic (Taira et al., 2012).

1. Condensation reaction

Condensation reaction, referred to as dehydration synthesis, is a chemical reaction in which two molecules combine to form a larger molecule with the loss of a small molecule. The most common small molecule is water. The condensation reaction of silane also produces a by-product, which is water. The larger molecule formed as a result of this condensation reaction loses the water molecule and has a greater density than the reactants (McNaught et al., 1997).

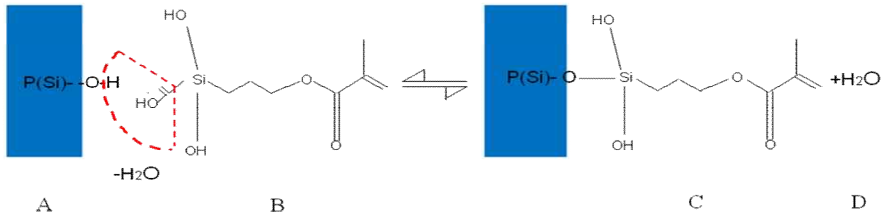

Figure 1. Reaction between the silica based ceramic surface and silane.

The condensation reaction between two hydroxyl groups (A and B) results in a covalent bond (chemical bond), where the silane molecule firmly attached to the porcelain surface (C). A by-product is the loss of a water molecule (D) (Matinlinna et al., 2004).

2. Le Chatelier’s principle

Le Chatelier’s principle, also called “The Equilibrium Law”, can be stated as “When a system at equilibrium is subjected to change in concentration, temperature, volume, or pressure, then the system readjusts itself to counteract the effect of the applied change and a new equilibrium is established”(Gall, 2002).

The condensation reaction of silane generates water, and this water changes the equilibrium in the system. The water must be eliminated in order to promote condensation reaction. This is the reason why warm air-drying is recommended after you apply silane (Papacchini et al., 2007).

3. Silane and hydrophobic monomer.

The chemical reaction between silane and a silica-based porcelain surface produces water as a by-product. This chemical reaction is called condensation reaction, and in order for this reaction to be continuous, the resulting by-product of water must be continuously removed. Equilibrium, according to “Le Chatelier’s” principle, can be achieved by water removal for further chemical reaction.

The dental adhesive has to contain a hydrophobic resin monomer in order to increase bond strength and prevent any hydrolytic degradation. If a hydrophobic resin monomer and silane are combined into a one-bottle adhesive system, then the hydrophobic resin monomer disturbs the process of by-product elimination, which is water evaporation.

The trend these days is to use a hydrophobic resin with higher hydrophobic properties in order to increase the longevity of the bonding layer. SBU is one adhesive system which contains this type of hydrophobic monomer, MDP.

There are several adhesives which include silane in the same bottle, including SBU, the new universal bonding system from 3M. It is convenient to use the same bottle for the adhesive as well as to treat the surface of the silica based ceramic. However, it is possible that the effectiveness of the resin and silane may be reduced because of the change in properties when they are combined in one bottle.

4. Hypothesis

Null hypothesis is that there is no signicant difference between a) the adhesive is applied after silane group and 2) silane and adhesive combined in one bottle group on silica-based porcelain in terms of suface hydrophobicity and shear bond strength.

II. MATERIALS AND METHODS

1. Material

Table 1. Materials used in this study.

	Ingredient	Manufacturer
ABU[a]	Bis-GMA, MDP	Bisco
ABU & Silane[b]	Bis-GMA,MDP, Silane	Experimental mixture
SBU[c]	Bis-GMA, MDP,Vitrebond copolymer, Silane	3M ESPE
Porcelain primer	Silane	Bisco
IPS e.max	Lithium disilicate	Ivoclar Vivadent
Duolink	Bis-GMA, Glass fiber	Bisco

a.;ABU; All bond universal
b;ABU & Silane; Premixed with ABU and 4 wt% of MPS (3-Methacryloxyproyltrimethoxysilane)
c. SBU; Scotchbond universal contains silane as an ingredient

A silane monomer, MPS, which is the active ingredient for most dental silane primers, was added into a universal adhesive (All-Bond Universal), resulting in an experimental silane-containing adhesive (Sil-ABU) which contains 4 wt% of the silane monomer.

The materials used in this study are as follows: only All-Bond Universal (ABU), Silane mixed with ABU, only Porcelain Primer (which is a regular silane primer), and Scotchbond Universal (SBU) which is a commercial silane-containing universal adhesive. IPS e-Max press, a lithium disilicate, was used as the silica-based ceramic. In terms of shade matching, IPS e-max press has more aesthetic characteristics compared to any other CAD-CAM ceramic block. Duolink resin cement was used as a composite applied to the surface to test for shear bond strength (Table 1).

2. Method

2.1 Contact angle

Contact angle test is a measurement of hydrophobicity on a silica-based ceramic surface. The more hydrophobic the surface of the ceramic is the better bonding strength to the adhesive.

For the contact angle test, lithium disilicate (IPS e.max, Ivoclar Vivadent) disc was etched with hydrofluoric acid (Bisco Porcelain Etchant, 4%HF, Lot # 1200010466) for 25 sec., rinsed with water and dried (control). The etched lithium disilicate disc was then treated with ABU (group 2), ABU&Silane (group 3), SBU (group 4), porcelain primer (group 5) and left undisturbed for 5min. (Table.2). And then it was cleaned by ultrasonication for 2min. in ethanol, and dried. Contact angles were measured with contact angle meter (NRL-CA Goniometer, NJ) (Figure2).

Table 2. Control and experimental group for measuring contact angle

Group	Procedure	Remark
1	HF etch-rinse-dry	Control
2	HF etch-rinse-dry-ABU	Experimental Group
3	HF etch -rinse-dry-Sil&ABU	Experimental Group
4	HF etch-rinse-dry-SBU	Experimental Group
5	HF etch-rinse-dry-Porcelain Primer	Experimental Group

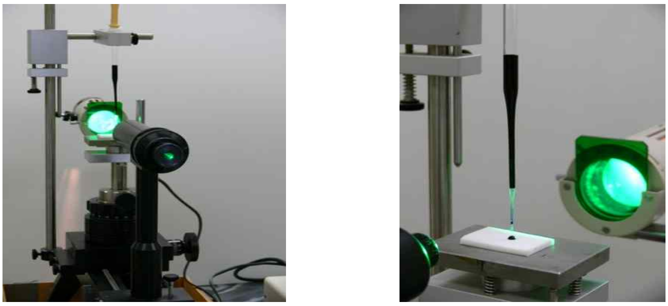

Figure 2. Contact angle tester.

2.2 Shear bond strength

Lithium disilicate (IPS e.max, Ivoclar Vivadent) was wet-polished with

320-grit SiC sand paper, rinsed with water and dried. The polished lithium disilicate disc was then applied dual cured resin cement Duolink(Bisco; Lot #1300004782), then light cured 40 sce/500mW/cm2 (Control). The polished lithium disilicate treated with ABU (group 2), ABU-Sil (group 3), and SBU (group4) left undisturbed (1 min), then light light-cured for 10 sec. Polished disc left undisturbed for 1 min after treated Porcelain Primer, then applied ABU separately, light cured 10 sec after (group 5). Duolink was used to fabricate a composite post using ultradent jig mold (bonding area=4.5mm^2). Duolink was light-cured for 40sec using light curing unit at energy density of 500mW/cm^2 from the top (Table3). The polymerized specimens were stored in de-ionized water for 24hr at 37 ℃, then tested until failure using universal testing machine (Instron 4466) at a cross head speed of 1 mm/min. (Figure 3).

Table 3. Control and experimental group for shear bond strength test

Group	Procedure	Remark
1	Polish-No primer-Duolink	Control
2	Polish-ABU-Duolink	Experimental Group
3	Polish-Sil&ABU-Duolink	Experimental Group
4	Polish-SBU-Duolink	Experimental Group
5	Polish-Silane-ABU-Duolink	Experimental Group

*SBS; Shear bond strength US; Ultrasonication

Figure 3. Universal testing machine.

2.3 Statistical analysis

The statistical significances of the resulting data were analyzed using one-way ANOVA. The statistical significance was accepted at confidence level of 95 % ($p < 0.05$) by Tukey′s test for a multiple comparison procedure. The SPSS PASW 18.0 program (SPSS Inc., IL, U.S.A.) was used for the statistical analysis.

III. RESULTS

1. Contact angle

The results of the contact angle test showed that the experimental groups (Groups 2-4) and the control group (Group 1) are not statistically different ($p>0.05$), except for group 5. Group 5, the group using purely silane, which is Porcelain primer, showed the highest contact angle compared to any other groups ($p<0.05$) (Figure.4).

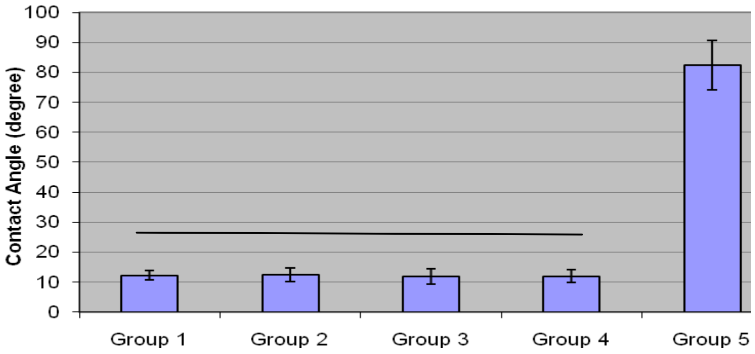

Figure 4. Contact angle on primed etched lithium disilicate.
Horizontal bar means that there are no significantly different ($p>0.05$).

2. Shear bond strength

In the shear bond strength test, all experimental groups are statistically different from the control group ($p<0.05$). However, there is a significant difference between Group 5 and the other experimental groups (Groups 2-4) and the control group, (Group 1) ($p<0.05$). This indicates that all of the experimental groups show better bonding strength than the control group ($p<0.05$). Group 5, which used purely silane, resulted in the significantly higher bonding strength.

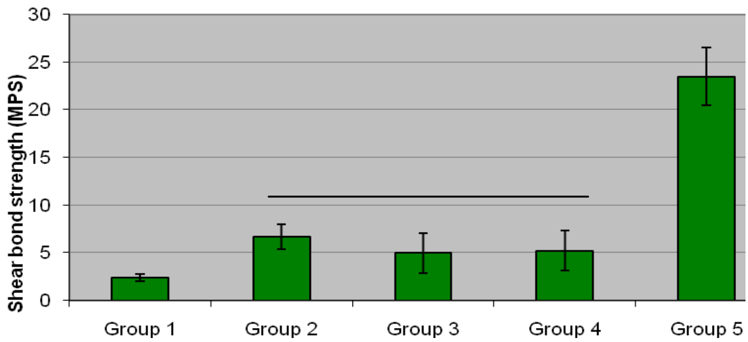

Figure 5. Shear bond strength on polished lithium disilicate.
Horizontal bar means that there are no significantly different ($p>0.05$).

IV. DISCUSSION

Silica-based ceramics, such as feldspathic porcelain and glass ceramics, are frequently used to veneer metal frameworks or high-strength ceramic coping for all-ceramic restorations. Availability of improved dental ceramic materials such as lithium disilicate, alumina, and zirconia as core materials has to led to a widespread use of all-ceramic restorations over the past decade (Ikemura et al., 2012).

Due to the development of new restorative material in today's esthetic dentistry, there emerges a clinical demand for the development of multi-purpose primers or adhesives that can deliver strong and durable adhesion to surfaces such as dental hard tissues, dental ceramics, dental precious metal alloys and dental non-precious metals (Ikemura et al., 2011).

Universal bonding adhesives are the most recent bonding agent in dental area. Universal bonding adhesive refers to a dental adhesive that can be used for both total etch and self etch techniques. These universal bonding adhesives are not only technically universal, but also functionally universal. They can be used as a multi-purpose surface treatment for silica-based ceramic, zirconia and dentin. Before this generation of one-bottle adhesives, MDP was and still is used separately to chemically treat the surface of zirconia (Inokoshi et al., 2013) and silane was and still is commonly used for treatment of silica-based ceramic surfaces. Now, the so-called universal bonding adhesives incorporate these ingredients into one bottle to treat different surfaces as well as to act as an adhesive.

Even though mechanical strength is an important factor that controls the clinical success of dental restoration (Hooshmand et al., 2008), the surfaces of the specimens were polished in order to eliminate any bias from micro mechanical retention due to an uneven surface. Two major factors of porcelain bonding strength are how to treat the porcelain surface both physically and chemically. Hydrofluoric acid etching helps to expand the surface (Nagai et al., 2005) and enhances bonding strength(Kimmich and Stappert, 2013). Silane primer increases chemical bonding strength through condensation reaction with the ceramic surface. Thus, in this study, all of the surfaces of the specimens were polished by sand paper to evaluate only the chemical function of silane.

Adhesion requires intimate contact of the materials to be joined. Surface has to be more hydrophobic for better adhesion, such as universal adhesive which hydrophobic resin as a ingredient (Marshall et al., 2010). Most manufacturers use Bis GMA or MDP as the hydrophobic monomer in their universal adhesives. Some of these adhesives combine both silane and a hydrophobic functional monomer, such as BisGMA or MDP.

However, this hydrophobic resin interferes with the condensation reaction of the pre-hydrolyzed silane inside the one-bottle universal adhesives (Chen et al., 2013). This resin interferes with the evaporation process of the water by-product of the condensation reaction and prevents equilibrium for further chemical reaction. There is a study that shows that warm air drying significantly increases the coupling potential of silane-based primers used as intermediate agents (Papacchini et al., 2007).

The result of this study shows that combining silane with ABU or SBU produces a relatively low contact angle. This means the surface is still hydrophilic and resin-unfriendly after applying the material (Figure.2). Silane does not work on a porcelain surface if it is mixed with a hydrophobic resin monomer. Applying silane to the porcelain surface generates water as a by-product. Water must be removed in order to accelerate the chemical reaction, but due to the hydrophobic resin inside the one bottle adhesive, water will be captured inside the resin matrix, and the chemical reaction cannot continue.

In terms of shear bond strengths, silane mixed with a hydrophobic resin monomer (Bis-GMA or MDP) showed relatively lower data than applying only silane. Silane in a hydrophobic resin monomer cannot effectively react with the porcelain surface, and results in a relatively low contact angle and poor bond strength (Figure.3).

Combining silane with the hydrophobic resin monomer hinders the silane from properly treating the porcelain surface. This is proven by the results of the contact angle tests and shear bond strength tests comparing one-bottle adhesives that include silane to using silane separately from the adhesive.

V. CONCLUSION

Silane should be used separately from a hydrophobic resin monomer in order to maximize its porcelain surface treatment function. Using silane separately and using silane incorporated into the universal adhesive (contains hydrophobic resin) do not perform equally on silica-based ceramic.

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ABSTRACT (IN KOREAN)

Porcelain 표면 처리제로서의 Silane이 함유된 One=bottle Adhesive

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서 우 경

본 연구의 목적은 “Universal adhesive”에 포함되어 있는 Silane의 Silica based ceramic의 표면처리제로서 기능을 평가하는 데에 있다.

All-bond Universal (ABU), Silane을 실험목적으로 ABU와 섞은 물질, Porcelain Primer, Silane을 성분으로 함유하고있는 Scotchbond Universal이 본 연구의 재료로 사용되어, IPS e-Max press, lithium disilicate, silica-based ceramic에 대한 표면처리제로서의 각각의 효과를 평가하였다..

Contact angle test 와 Shear bond strength 가 실험 방법으로 사용되었으며 Shear bond strength test에서는 시편을 polish한 상태로 사용함으로써 기계적인 결합력을 배제하였다.

실험 결과 Silane을 따로 사용한 시편에서 높은 Contact angle과 Shear bond strength가 측정되었고, Silane 은 소수성의 Resin과 분리 되어야 사용되는 것이 표면처리제로서의 기능

이 더 효과적인 것으로 나타났다.

결론적으로 Silane은 소수성의 Resin과 섞어서 사용 된다면 순수한 Silane만큼의 표면처리 효과를 기대하기 어렵다. 따라서, Silane의 임상적 사용에 있어서 순수 Silane의 사용이 효과적인 표면 처리제로서의 작용을 하므로 접착제와 분리된 제품을 사용하는 것을 추천한다.

핵심되는 말: Silane, Universal adhesive, One bottle adhesive, Surface treatment, Silica based ceramic, Lithium disilicate