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Effect of adhesive hydrophobicity on microtensile bond strength of low-shrinkage silorane resin to dentin

So-Yeun Cho, Hyun-Young Kang, Kyoung-A Kim, Mi-Kyung Yu, Kwang-Won Lee
Department of Conservative Dentistry, Jeonbuk National University School of Dentistry, Jeonju, Korea

ABSTRACT

Objectives: The purpose of this study was to evaluate μTBS (microtensile bond strength) of current dentin bonding adhesives which have different hydrophobicity with low-shrinkage silorane resin.

Materials and Methods: Thirty-six human third molars were used. Middle dentin was exposed. The teeth were randomly assigned to nine experimental groups: Silorane self-etch adhesives (SS), SS + phosphoric acid etching (SS + pa), Adper easy bond (AE), AE + Silorane system bonding (AE + SSb), Clearfil SE bond (CSE), CSE + SSb, All-Bond 2 (AB2), AB2 + SSb, All-Bond 3 (AB3). After adhesive's were applied, the clinical crowns were restored with Filtek LS (3M ESPE). The 0.8 mm × 0.8 mm sticks were submitted to a tensile load using a Micro Tensile Tester (Bisco Inc.). Water sorption was measured to estimate hydrophobicity adhesives.

Results: μTBS of silorane resin to 5 adhesives: SS, 23.2 MPa; CSE, 19.4 MPa; AB3, 30.3 MPa; AB2 and AE, no bond. Additional layering of SSb: CSE + SSb, 26.2 MPa; AB2 + SSb, 33.9 MPa; AE + SSb, no bond. High value of μTBS was related to cohesive failure. SS showed the lowest water sorption. AE showed the highest solubility.

Conclusions: The hydrophobicity of adhesive increased, and silorane resin bond-strength was also increased. Additional hydrophobic adhesive layer did not increase the bond-strength to silorane resin except AB2 + SSb. All-Bond 3 showed similar μTBS & water sorption with SS. By these facts, we could reach a conclusion that All-Bond 3 is a competitive adhesive which can replace the Silorane adhesive system. [J Kor Acad Cons Dent 2011;36(4):280-289.]

Key words: Adhesive hydrophobicity; Dentin bonding system; Microtensile bond strength; Silorane resin; Water sorption test

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INTRODUCTION

Recently, a new class of low-shrinking composites based on silorane technology (Filtek Silorane, 3M ESPE, Seefeld, Germany) was introduced. The silorane resin replaces the conventionally used methacrylate resin matrix within conventional dental composites, thereby providing lower polymerization shrinkage[1-3] as well as better hydrolytic stability.[4,5]

As the resin matrix of the silorane composite significantly differs from that of conventional methacrylate-based composites, a new adhesive is needed to be designed and developed to enable bonding of the silorane composite to tooth enamel and dentin. Filtek

Cho SY, *DDS, Resident;* Kang HY, *DDS, Resident;* Kim KA, *DDS, Graduate student;* Yu MK, *DDS, PhD, Professor;* Lee KW, *DDS, PhD, Professor,* Department of Conservative Dentistry, Jeonbuk National University School of Dentistry, Jeonju, Korea

***Correspondence to Kwang-Won Lee, DDS, PhD.**

Professor, Department of Conservative Dentistry, Jeonbuk National University School of Dentistry, Geumam-dong, Deokjin-gu, Jeonju, Jeonbuk, Korea 561-756

TEL, +82-63-250-2016; FAX, +82-63-250-2129; E-mail, lkw@jbnu.ac.kr

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Silorane therefore comes with a two-step self-etch adhesive, which is called Silorane System Adhesive (SSA, 3M ESPE). It still possesses features of conventional methacrylate adhesives, especially with regard to its bonding mechanism to tooth tissue. But, adaptation was needed, especially to make it compatible with the highly hydrophobic silorane matrix. The adhesive somewhat differs from a typical two-step self-etch adhesive since it involves the application of two resin solutions. The first one (SSA-Primer) is rather hydrophilic to bond to tooth tissue. The second solution (SSA-Bond) is on the contrary quite hydrophobic in order to adequately bridge the hydrophilic tooth substrate with the hydrophobic silorane composite. For this reason, each resin solution needs to be light-cured separately.[6,7]

It consists of a phosphate based functional monomer, dimethacrylates (HEMA, Bis-GMA, etc.), a copolymer of acrylic and itaconic acid, silica, and camphorquinone. All dissolved in a water-ethanol solvent (technical data as mentioned in the Material Safety Data Sheet provided by 3M ESPE, Table 1). The relatively high amount of HEMA keeps this resin solution homogeneous, preventing phase-separation effects like they have been typically documented for HEMA-poor/free one-step adhesives.[8] The secondly applied 'SSA-Bond' is methacrylate-based. Because it contains a high concentration of substituted dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), silica, a rather low concentration of functional monomer and camphorquinone, its nature is hydrophobic. Further details on how this methacrylate-based SSA-Bond links to the silorane composite are currently not known. According to the technical information provided by 3M ESPE, however, SSA-Bond contains hydrophobic bifunctional monomers to match the hydrophobic silorane resin. This second hydrophobic adhesive layer is indispensable as a clear incompatibility exists[9] with the more hydrophilic, one-step experimental precursor of SSA.

Some questions remain about the bonding abilities of this dedicated adhesive. Although this new composite can form a strong bond with identical material, its capacity to form bonds with dissimilar materials is still open to question. If the silorane composite resin can adhere to methacrylate-based adhesive, it raises the subject as about how to improve or at least to maintain acceptable bond strengths and levels of nanoleakage.[10]

The purpose of this study is to evaluate microtensile bond strength of current dentin bonding adhesives which have different hyprophobicity with low-shrinkage silorane resin. The null hypothesis is two-fold: (1) There is no difference in microtensile bond strength of silorane resin to dentin/enamel although adhesive system's hydrophobicity is increased. (2) Additional hydrophobic adhesive layering over current adhesive system does not affect microtensile bond strength of silorane resin to dentin/enamel.

MATERIALS AND METHODS

Thirty-six freshly extracted caries-free human third molars were selected for the study and stored in 0.5% Sodium Azide solution (Duksan Pure Chemical Co., Ansan, Korea) at 4℃ for up to one month after extractions. The teeth were scaled, cleaned, and stored in distilled water for 24 hours. The teeth were randomly assigned to nine experimental groups like below.

Middle dentin was exposed by sectioning the crowns parallel to the occlusal surface with a precision low-speed diamond saw (Isomet 1,000, Buehler, Lake Buff, IL, USA), under distilled water cooling. A dentin standard smear layer was created by polishing the occlusal surface with 1,000-grit Silicon Carbide sandpaper for 60 seconds. The bonded interface was prepared according to the experimental groups (Table 1).

After adhesive's are applied, the clinical crowns were restored with low-shrinkage composite Filtek LS (Lot number: 7BB, 3M ESPE, St. Paul, MN, USA) in 3 increments of 2.0 mm each. Each increment was light-cured for 40 seconds (Demetron/Kerr, Danbury, CT, USA) at 0.5 mm curing distance and light intensity of 800 mW/cm^2 constantly monitored with a radiometer.

Microtensile bond strengths (μTBS)

Specimens were sectioned parallel to the adhesive interface to obtain 0.8 ± 0.1 mm thick slabs. Samples were measured with a caliper. A digital

Table 1. Materials used

Group	Materials	Composition	Application
SS	Silorane System Adhesive (SSA) 3M ESPE, Seefeld, Germany	Self-etch primer: Phosphorylated methacrylates, vitrebond copolymer, bisphenol A diglycidylmethacrylate (Bis-GMA), 2-hydroxyethylmethacrylate (HEMA), water, ethanol, silane-treated silica filler, initiators, stabilizers (Lot number: 7AD) LS Bond: Hydrophobic methacrylates, phosphorylated methacrylates, triethylene glycol dimethacrylates (TEGDMA), silane-treated silica filler, initiators, stabilizers (Lot number:7AC)	1. Apply 1 coat of the self-etch primer for 15 sec with gentle agitation using fully saturated applicator. Gently air thin to evaporate solvent and obtain an even film. Light-polymerize for 10 sec 2. Apply the Bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 sec
SS+pa			1. Apply All Bond Etchant (32%) to dentin. Wait 15 sec and rinse for 10 sec. Blot excess water using cotton pellet. 2. Apply 1 coat of the self-etch primer for 15 sec with gentle agitation using fully saturated applicator. Gently air thin to evaporate solvent and obtain an even film. Light-polymerize for 10 sec 3. Apply the Bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 sec
AE	Adper easy bond 3M ESPE, St. Paul, MN, USA	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylatefunctionalised polyalkenoic acid (Vitrebond Copolymer), finely dispersed bonded silica filler with 7 nm primary particle size, ethanol, water, initiators based on comphorquinone, stabilizers	1. Apply adhesive to tooth surface for 20 sec 2. Dry the adhesive for 5 sec 3. Light cure for 10 sec
AE+SSb			1. Apply adhesive to tooth surface for 20 sec 2. Dry the adhesive for 5 sec 3. Light cure for 10 sec 4. Apply the Silorane system Bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 sec
CSE	Clearfil SE bond Kuraray, Osaka, Japan	Self-etching/primer: 2-Hydroxyethyl methacrylate (HEMA), 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone Water, Accelerators Dyes and others Bond: 2-Hydroxyethyl methacrylate (HEMA), 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bisphenol A diglycidylmethacrylate (Bis-GMA), 2-Hydroxyethyl methacrylate (HEMA), Hydrophobic dimethacrylate dl-Camphorquinone N,N-diethanol-p-toluidine Silanated colloidal silica	1. Apply self-etch primer on tooth surface and leave it for at least 20 sec. Evaporate the volatile ingredients with a mild air stream. 2. Apply bond on tooth surface. Expose to a gentle air stream Cure 10 sec
CSE+SSb			1. Apply on tooth surface and leave it for at least 20 sec. Evaporate the volatile ingredients with a mild air stream 2. Apply on tooth surface. Expose to a gentle air stream. Cure 10 sec 3. Apply the Silorane system Bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 sec

AB2			1. Apply All Bond Etchant (32%) to dentin. Wait 15 sec and rinse for 10 sec. Blot excess water using cotton pellet. 2. Mixing Primer A and B in equal amount and apply on tooth surface. expose to a gentle air stream. 3. Apply bond to tooth surface. cure 10 sec
AB2+SSb	All-Bond 2 Bisco. Itasca, IL, USA	primer: aceton, Ethanol, NTG-GMA, water, BPDM, photoinitiator Bond: Bis-GMA, HEMA, Camphorquinone, amin activator	1. Apply All Bond Etchant (32%) to dentin. Wait 15 sec and rinse for 10 sec. Blot excess water using cotton pellet. 2. Mixing Primer A and B in equal amount and apply on tooth surface. expose to a gentle air stream. 3. Apply bond to tooth surface. cure 10 sec 4. Apply the Silorane system Bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 sec
AB3	All-Bond 3 Bisco. Itasca, IL, USA	primer: Ethanol, NTG-GMA, Bis-GMA, HEMA, BPDM Bond: Bis-GMA, Urethane, TEGDMA, Dimethacrylate,	1. Etch the preparation for 15 sec and rinse thoroughly. Remove excess water using a foam pellet, leaving the preparation visibly moist. 2. Dispense an equal number of drops of All-Bond 3 Parts A and B (1 : 1) into a mixing well. Using a brush, mix well for 5 sec 3. Apply 1-2 coats onto the tooth preparation. 4. Gently but thoroughly air dry until there is no visible movement of the material. The surface should appear shiny; otherwise, apply additional coats of All-Bond 3 and repeat Step 4. 5. Light cure for 10 sec at 500 mW/cm^2. 6. Apply one thin coat of All-Bond 3 RESIN. Air thin if necessary. 7. Light cure for 10 sec at 500 mW/cm^2.

SS, Silorane self-etch adhesives; SS + pa, Silorane self-etch adhesives + phosphoric acid etching; AE, Adper easy bond; AE + SSb, Adper easy bond + Silorane system bonding; CSE , Clearfil SE bond; CSE + SSb, Clearfil SE bond + Silorane system bonding; AB2, All-Bond 2; AB2 + SSb, All-Bond 2 + Silorane system bonding; AB3, All-Bond 3.

caliper (Mitutoyo digital calipers, Mitutoyo Corp., Kanogawa, Japan) with an accuracy of 0.01 mm was used to measure the sizes of the bonding interface and to calculate the bonding area in square millimeters. The specimens were tested individually by attaching them to a microtensile jig using cyanoacrylate glue (ZapIt, DVA, Corona, CA, USA). The 0.8 mm × 0.8 mm sticks were then submitted to a tensile load using a Micro Tensile Tester (Bisco Inc., Schaumburg, IL, USA) at 1.0 mm/min cross-head speed (Figure 1). The load in Kg and the bonding

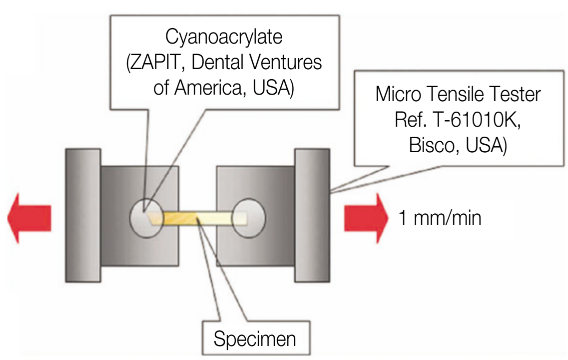

Figure 1. Diagram of measuring microtensile bond strength.

surface area of the specimen were registered and microtensile bond strengths calculated in MPa. Pretesting failures or spontaneous debonding were counted as 0 MPa.

Statistical analysis was performed with statistical software (SPSS 15, SPSS Inc., Chicago, IL, USA). A one-way analysis of variance (ANOVA) for dentin treatment was computed, followed by a Duncan's post hoc test ($p < 0.05$).

Failure mode analysis

Fracture surfaces were examined using optical microscopy (Zeiss, Carl Zeiss, oberkochen, Germany) to determine the mode of failure based on the fracture origin.[11] If it was not clear with optical microscopy, we confirmed the failure mode with scanning electron microscopy (SEM). Failures for each adhesive system were categorized as either adhesive (joint or mixed) or cohesive (dentin or composite).

Water sorption & solubility test

Five commercially available dental adhesives were chosen according to their different solvent-monomer combinations and their water sorption & solubility were tested: Adper easy bond (AE), Clearfil SE bond (CSE), All-Bond 2 (AB2), All-Bond 3 (AB3) and Silorane self-etch adhesives (SS). We expected to know the adhesive's hydrophobic feature by water sorption test.

Ten resin disks of each material were produced in a polymer mould (1.0 mm × 1.0 mm × 1.0 mm). The liquid adhesive was directly dispensed to completely fill the mould. The surface of the solvated, one-bottle system (Adper Easy bond) was gently blown with an oil/water-free compressed air for 90 seconds to facilitate solvent evaporation. All visible air bubbles trapped in the adhesives were carefully removed prior to photo-activation. A glass cover slip was placed on top of the adhesive, which was light-cured for 40 seconds at 800 mW/cm^2 (Demetron/Kerr). After removing the specimen from the mould, photoactivation was repeated on its opposite surface for another 40 seconds.

Immediately after polymerization, the specimens were placed in a desiccator and transferred to a preconditioning oven at 37℃. The specimens were repeatedly weighed after 24 hours intervals until a constant mass (m_1) was obtained. Thickness and diameter of the specimens were measured using a digital caliper (Mitutoyo digital calipers, Mitutoyo Corp., Kanogawa, Japan) and these measurements were used to calculate the volume (V) of each specimen. They were then individually placed in glass vials containing 10 mL of distilled water (pH 7.2) at 37℃. After fixed time intervals of 1, 2, 3, 4, 5, 6 and 7 days of storage, the specimens were washed in running water, gently wiped with a soft absorbent paper, weighed in an analytical balance (m_2) and returned to the vials containing 10 mL of fresh distilled water. Following the 7 days of storage, the specimens were dried inside a desiccator and weighed daily until a constant mass (m_3) was obtained (as previously described). The initial mass determined after the first desiccation process (m_1) was used to calculate the change in mass after each fixed time interval, during the 7 days of storage in water. Water sorption (WS) and solubility (SL) over the 7 days of water storage were calculated using the following formulae:

$$WS = (m_2 - m_3) / V$$

$$SL = (m_1 - m_3) / V$$

The data was statistically analyzed (Kruskal-Wallis Test).

RESULTS

Microtensile bond strengths (μTBS)

All-Bond 2 (AB2), Adper Easy bond (AE) and Adper Easy bond (AE)+Silorane System bonding (SSb) were unable to produce sufficient bond strength to hold the silorane composite resin in place. Most of the specimens spontaneously debonded during preparing 0.8 mm × 0.8 mm slab by Isomet. Thus, these groups were removed from the statistical analysis since there was no adhesion of Filtek silorane resin with AB2, AE and AE + SSb (Table 2).

Adper Easy bond did not bond to silorane resin. Additional layering with silorane system bond over Adper Easy bond could not make it bond to dentin/enamel.

Table 2. Mean microtensile bond strengths (MPa), standard deviation, and number of specimens

	Dentin treatment	Mean (SD)	N
Without additional layering	SS	23.2 (6.9)	15
	SS + pa	30.1 (4.4)	23
	AE	0	
	CSE	19.4 (4.4)	7
	AB2	0	
	AB3	30.3 (4.0)	20
Additional Silorane system bond layering	AE + SSb	0	
	CSE + SSb	26.2 (10.3)	12
	AB2 + SSb	33.9 (7.3)	15

SS, Silorane self-etch adhesives; SS + pa, Silorane self-etch adhesives + phosphoric acid etching; AE, Adper easy bond; AE + SSb, Adper easy bond + Silorane system bonding; CSE , Clearfil SE bond; CSE + SSb, Clearfil SE bond + Silorane system bonding; AB2, All-Bond 2; AB2 + SSb, All-Bond 2 + Silorane system bonding; AB3, All-Bond 3.

Clearfil SE bond and silorane system adhesive showed similar microtensile bond. When additional layering of silorane bonding agent was applied, statistically different improvement was not revealed (p = 0.399).

All-Bond 2 did not bond to silorane resin, but additional layering of silorane bonding agent increased microtensile bond strength (0 MPa vs 33.9 MPa). One-way ANOVA performed for AB2 + SSb and SS showed statistical difference (p = 0.001).

All-Bond 3 showed high microtensile bond strength (30.3 MPa), but it had no statistical difference compare to SS group (p = 0.063).

Failure mode analysis

Spontaneous debonding of Adper Easy bond, All-Bond 2, Adper Easy bond + Silorane system bonding was categorized to adhesive failure (100%). High value of μTBS was related to cohesive failure; AB2 + SSb (33.9 Mpa, 40%), AB3 (30.3 Mpa, 25%), SS + pa (30.1 Mpa, 35%), CSE + SSb (26.2 Mpa, 33%), SS (23.2 Mpa, 16%) (Table 3).

Table 3. Failure mode analysis

Group	Adhesive failure(%)	Cohesive failure		
		Dentine	Resin	Total
SS	21 (84%)	1 (4%)	3 (12%)	4 (16%)
SS + pa	29 (64%)	2 (4%)	14 (31%)	16 (35%)
AE	- 100%			
CSE	6 (86%)	0	1 (14%)	1 (14%)
AB2	- 100%			
AB3	15 (75%)	0 (0%)	5 (25%)	5 (25%)
AE + SSb	- 100%			
CSE + SSb	8 (66%)	0	4 (33%)	4 (33%)
AB2 + SSb	9 (60%)	2 (13%)	4 (27%)	6 (40%)

SS, Silorane self-etch adhesives; SS + pa, Silorane self-etch adhesives + phosphoric acid etching; AE, Adper easy bond; AE + SSb, Adper easy bond + Silorane system bonding; CSE , Clearfil SE bond; CSE + SSb, Clearfil SE bond + Silorane system bonding; AB2, All-Bond 2; AB2 + SSb, All-Bond 2 + Silorane system bonding; AB3, All-Bond 3.

Water sorption & solubility test

As Table 4 display, SS showed the lowest water sorption, followed by AB3, AE, CSE, AB2 (p = 0.008) and AE showed the highest solubility, followed by CSE, AB2 (p = 0.014). SS and AB3 showed no solubility in our study. Water sorption of AE was lower than we expected due to its high solubility. Solubility of SS and AB3 were zero. This is explained by their high hydrophobic feature (Figure 2).

Table 4. Water sorption and solubility (μm/mm³) of five adhesive systems

Adhesives	Water sorption	Solubility
CSE	62.05	16.06
SS	12.41	0
AB2	78.60	3.9
AB3	33.10	0
AE	41.64	165.48

CSE, Clearfil SE bond; SS, Silorane self-etch adhesives; AB2, All-Bond 2; AB3, All-Bond 3; AE, Adper easy bond.

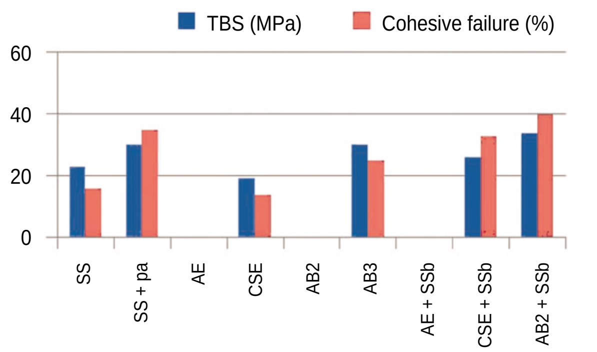

Figure 2. Failure mode and μTBS analysis. High value of μTBS was related to cohesive failure Water sorption & solubility.

SS, Silorane self-etch adhesives; SS + pa, Silorane self-etch adhesives + phosphoric acid etching; AE, Adper easy bond; CSE, Clearfil SE bond; AB2, All-Bond 2; AB3, All-Bond 3; AE + SSb, Adper easy bond + Silorane system bonding; CSE + SSb, Clearfil SE bond + Silorane system bonding; AB2 + SSb, All-Bond 2 + Silorane system bonding.

DISCUSSION

The null hypothesis rejected because there is a difference in microtensile bond strength of silorane resin to dentin/enamel although adhesive system's hydrophobicity is increased As the adhesive's hydrophobicity (lower water sorption) increased, bond strength with silorane resin to dentin/enamel also increased. It has been revealed that water sorption into adhesive polymers is related to the hydrophilicity of adhesives in several studies.[12,13] According to these studies a strong correlation between the mean of water sorption and the degree of hydrophilicity was determined.[14] In other words, the more hydrophilic the adhesives are, the more water their polymers absorb. It has also been reported that water sorption by hydrophilic resins contributes to the commonly observed decrease in their mechanical properties.[15]

In large part the hydrophilic nature of adhesive is a function of the chemistry of its monomers and its polymerization linkages. The extent and rate of water uptake into polymer networks are predominantly controlled by two main factors: resin polarity, dictated by the concentration of polar sites available to form hydrogen bonds with water[16,17] and network topology, which is related to the cohesive energy density of the polymer network.[16,18,19] The polymer polarity (water affinity for hydrophilic polar groups in the polymer) is a major determinant of water uptake into polymers.[14]

The presence of hydroxyl, carboxyl and phosphate groups in monomers and their resultant polymers make them more hydrophilic and more prone to water sorption. It is well known that hydrophilic constituents such as 2-hydroxyethyl methacrylate (HEMA) increase water sorption.[10] The hydrophobic nature of constituent monomer in adhesives, such as bis-GMA, MMA, would also be a major factor in decreasing water sorption.[20] Braden and Clarke and Mese *et al.* reported that filler volume is also related to water sorption, which means that resins contains higher filler volumes absorb less water.[21,22]

If an adhesive monomer has a polarity and a solubility which are similar to those of a polymer substrate, the monomer may act as a solvent for the polymer and may infiltrate it. If both parameters are sufficiently different, the monomer and polymer are immiscible.[23] In this regard, to make a comparable bond with silorane resin which is very hydrophobic, it is crucial to use a hydrophobic adhesive system.

In this study, Adper Easy bond shows the highest water sorption. Adper Easy bond is an one-step self-etching system. Adper Easy bond is water-based (28%) and contains a relatively small amount of ethanol (18%). Adper Easy bond has a minority of hydrophobic methylene groups and this justifies the presence of HEMA to prevent organic phase separations from the water based compositions.[24] One-step self-etching systems, however, are composed of high concentration of hydrophilic resin monomers, ionic resin monomers or both, creating thin coatings that may inhibit oxygen and may result in a poorly polymerized adhesive layer. The monomers are prone to phase separation because they behave like a permeable membrane after polymerization as the solvent evaporated from the solution. This is due to the lack of a nonsolvent hydrophobic adhesive layer, which allows for rapid dentinal fluid transudation across the polymerized adhesives.[25] These properties are not matched with hydrophobicity of Silorane System Adhesive. To overcome this gap, we applied additional layering of an SSA-bonding agent. This is supported by several studies. The results of some laboratory studies have indicated that treating an one-step self-etch system as a primer and covering it with a more hydrophobic adhesive layer could overcome the one-step self-etch systems' drawbacks and improve the immediate resin-dentin efficacy.[26] In this study, however, there is no increase in microtensile bond strength when hydrophobic adhesive layer was applied to Adper Easy bond. This indicates that there is considerable difference between polarity of Adper Easy bond and Silorane System Adhesive. Water sorption of Adper Easy bond is much lower than we expected. This is because Adper Easy bond shows not only high water sorption but also high solubility. Thus, water sorption test and microtensile bond strength does not yield consistent result on Adper Easy bond.

Clearfil SE bond shows silmilar bond strength to Silrorane adhesive system. Additional layering of SS-bond does not improve bond strength. However, water sorption and solubility of Clearfil SE bond is much higher when comparing to Silorane adhesive System. This fact may means quality and durability of bond could be different although bond strength is similar. According to Avishai *et al*, Clearfil SE bond reveals more nanoleakage comparing to Silorane adhesive System.[10]

All-Bond 2 does not bond to silorane resin. All-Bond 3 shows higher bond strength compared to Silorane adhesive System, but there is no significant difference. All-Bond 2 contains a tertiary aromatic amine in primer A which may be the sodium or magnesium salt of NTG-GMA, and a sparingly water-soluble carboxylic acid monomer in primer B, which is dissolved in acetone.[27,28] Up to date, to overcome the aceton-based adhesive's defect, ethanol-based adhesives are developed, including All-Bond 3. It is possible to coax comparatively hydrophobic monomers to acid-etched dentin with an ethanol-wet bonding protocol. The rationale behind this technique is that ethanol dehydration renders acid-etched dentin less hydrophilic, allowing the use of relatively hydrophobic monomers for infiltrating shrunken but non-collapsed demineralized collagen network that is suspended in ethanol. Theoretically, this would improve resin-dentin bond durability by minimizing water sorption through polymerized hydrophobic adhesive.[29]

All-Bond 3 shows similar bond strength compared to Silorane adhesive System. And water sorption was higher than Silorane adhesive system but much lower than other adhesives. By these facts, we could reach a conclusion that All-Bond 3 is competitive adhesive which can replace the Silrane adhesive system.

Manufacturers recommended applying dedicated adhesives when using Filtek silorane resin. But, within the limit of this study, hydrophobic adhesive system such as All-Bond 3 is compatible when combined with silorane resin. However, further research on the quality and durability of these Silorane bonds still needed to be conducted.

CONCLUSION

Within the limit of this study, we could reach the conclusion like below.

(1) The more hydrophobic the adhesive are, the higher their bond strength with silorane resin will be.
(2) Additional hydrophobic adhesive layer over non-dedicated adhesive system does not increase the bond-strength to silorane resin except AB2 + SSb.
(3) All-Bond 3 is competitive adhesive which can replace the Silrane adhesive system.

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국문초록

접착시스템의 소수성이 Low-shrinkage silorane resin과 상아질의 미세인장강도에 미치는 영향

조소연 · 강현영 · 김경아 · 유미경 · 이광원*

전북대학교 치의학전문대학원 치과보존학교실

연구목적: 본 연구의 목적은 다양한 소수성을 지닌 최신 상아질 접착시스템과 저수축 silorane 레진의 미세인장결합강도를 평가하는 것이다.

연구 재료 및 방법: 36개의 갓 발치된 제3대구치를 이용했다. Low-speed diamond saw를 사용하여 교합면에 평행하게 치관을 잘라 middle dentin을 노출시켰다. 치아를 무작위로 9 group으로 나눴다. Silorane self-etch adhesives (SS), SS + phosphoric acid etching (SS + pa), Adper Easy bond (AE), AE + Silorane system bonding (AE + SSb), Clearfil SE bond (CSE), CSE + SSb, All-Bond 2 (AB2), AB2 + SSb, All-Bond 3 (AB3). 접착제를 적용한 후에 Filtek LS (3M ESPE)를 2 mm씩 3회 적층충전하였다. 각 층은 40s씩 광중합하였다. 0.8 mm × 0.8 mm stick을 Micro Tensile Tester로 1 mm/min cross-head speed의 인장력을 가하였다. 파절양상를 관찰하기 위해 광학현미경을 이용하였다. 5가지 접착제의 소수성정도를 결정하기위해 water sorption test하였다.

결과: silorane 레진과 5가지 접착제의 μTBS: SS, 23.2 ± 6.9 MPa; CSE, 19.4 ± 4.4 MPa; AB3, 30.3 ± 4.0 MPa; AB2와 AE, no bond. Additional layering of SSb: CSE + SSb, 26.2 ± 10.3 MPa; AB2 + SSb, 33.9 ± 7.3 MPa; AE + SSb, no bond. 높은 μTBS는 cohesive failure와 관련있었다. SS는 낮은 가장 낮은 water sorption을 보였고 다음으로 AB3, AE, CSE, AB2 순서였다. AE는 가장 높은 용해도를 나타냈고 다음으로 CSE, AB2였다.

결론: 접착제의 소수성이 증가할수록, silorane 레진의 접착강도도 증가하였다. 비전용접착제 위에 silorane adhesive bonding을 layering하는 것은 AB2 + SSb 그룹에서만 결합강도를 유의하게 증가시켰다. AB3는 SS와 유사한 μTBS & water sorption을 나타냈다. 따라서 AB3는 siloran resin을 접착시키는데 SS를 대체할만한 경쟁력있는 접착제이다.

주요단어: 미세인장강도; 상아질접착제; 수분흡착검사; 접착제의 소수성; Silorane resin