

Effect of Hydrophobic, Neutral Adhesive on the Dentin Bond Strength of Self-etching Adhesive

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• Abstract

Objectives : This study investigated the hypothesis that the dentin bond strength of self-etching adhesives (SEAs) may be improved by applying a coat of hydrophobic, neutral adhesive resin in addition to SEA.

Method and Materials : The bond strengths of two SEAs - Experimental SEA (EX) and Adper Prompt (AP) - were measured with three bonding protocols. The D/E resin of All-Bond 2 was applied as the hydrophobic, neutral adhesive. Clearfil SE Bond (SE, self-etching primer system) and All-Bond 2 (AB, total etching system) were used as references. The following protocols were used: (1) EX1 (EX 1 coat); (2) EX2 (EX 2 coats); (3) EX+ (EX 1 coat + D/E resin); (4) AP1 (AP 1 coat); (5) AP2 (AP 2 coats); (6) AP+ (AP 1 coat + D/E resin); (7) SE (SE primer + SE bond); (8) SE+ (SE primer + D/E resin); (9) AB (etching + AB primer + D/E resin). Filtek Z250 composite resin was built up and the microtensile bond strength (MTBS) values of the specimens were compared. The fractured surfaces were observed using SEM.

Results : When SEA was used as self-etching primer and hydrophobic, neutral adhesive was applied as well, MTBS was significantly higher than that when either one coat or two coats of SEA only were used ($p < 0.05$).

Conclusion : The hydrophobic, neutral adhesive improved the integrity of the bonded interface obtained with SEA.

• Key word : self-etching adhesive, microtensile bond strength, neutral pH, hydrophobic adhesive, acidic monomer, SEM

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Introduction

Recent studies of dental adhesive systems have focused on the simplification of the clinical bonding procedure. Also termed “all in one” adhesive systems, self-etching adhesives (SEAs) have recently been introduced in the market¹. SEAs are convenient because priming and bonding are done simultaneously with one application. They have the same bonding mechanism as self-etching primer systems wherein functional acidic monomers in the primer perform simultaneous etching and priming upon application to the smear layer-covered enamel and dentin surface²⁻⁴. The collapse of the three-dimensional structure of the collagen network caused by air-drying is avoided because demineralization and monomer impregnation depths are the same². Maintaining the collagen network may reduce post-operative sensitivity⁵. Since there is no rinsing-off procedure, the formation of an incomplete hybrid layer by over-drying and over-wetting is avoided^{6,7}. As a result, SEAs and self-etching primer systems are less technique-sensitive than systems using separate acid etching and rinsing procedures^{1,5}.

Although they are convenient and less technique-sensitive, SEAs have exhibited low bond strength compared with two-step or three-step adhesive systems in many studies⁸⁻¹⁰. The thin adhesive layer of SEAs is deemed to be the cause their relatively low bond strength, but studies using multiple coats of SEAs also showed lower bond strength than that of earlier-generation adhesives^{1,9,11}.

Compared with these earlier adhesives, SEAs have higher concentration of hydrophilic acidic functional monomers that hasten the hydrolytic degradation of resin in the hybrid layer^{5,12-15}. Transmission electron micrographs (TEMs) of SEAs show water channels spreading from the hybrid layer through the adhesive layer to the composite resin¹⁵. Voids that might have been water blisters were also found on the scanning electron micrographic (SEM) images of fracture surfaces taken shortly after bonding^{12, 16}. The low pH (1.0 to 3.0) of SEAs might interfere with their copolymerization with the restorative composite resin¹⁶⁻¹⁸.

This study examined the hypothesis that the dentin bond strength of SEAs might be improved by applying a coat of hydrophobic, neutral adhesive resin, which could strengthen the adhesive layer and alleviate the pH difference between SEAs and restorative composite resin. SEAs were used as primer, with adhesive resin applied as the second coat. The

microtensile bond strength (MTBS) of two SEAs was evaluated.

Materials and Methods

Freshly extracted non-carious human molars were cleaned and immersed in 0.5% chloramine-T solution (Carl Roth GmbH+ Co., KG, Karlsruhe, Germany) for 24 h and stored in distilled water at 4°C. Each tooth was embedded in a cubic steel mold with self-curing acrylic resin. The occlusal enamel was removed perpendicularly to the long axis of the tooth using a low-speed diamond saw (Isomet; Buehler Ltd., Lake Bluff, IL., USA) to align the adhesive interface perpendicularly to the applied tensile load. The exposed dentin surface was polished with P500 SiC paper under running water using an automatic polishing machine (Rotopol-V, Struers Ltd., Glasgow G60 5EU, UK).

The bond strengths of two SEAs - Experimental SEA (EX) and Adper Prompt (AP, 3M ESPE, St. Paul, MN., USA) - were measured with three bonding protocols. The D/E resin of All-Bond 2 (pH 6.9, Bisco, Inc., Itasca, IL., USA) was additionally applied as the hydrophobic, neutral adhesive. Clearfil SE Bond (SE, self-etching primer system, Kuraray, Osaka, Japan) and All-Bond 2 (AB, total etching system) were used as references because they have higher bond strength than the 6th adhesive system. The following protocols were used: (1) EX1 (EX 1 coat); (2) EX2 (EX 2 coats); (3) EX+ (EX 1 coat + D/E resin); (4) AP1 (AP 1 coat); (5) AP2 (AP 2 coats); (6) AP+ (AP 1 coat + D/E resin); (7) SE (SE primer + SE bond); (8) SE+ (SE primer + D/E resin); (9) AB (etching + AB primer + D/E resin). The abbreviations of experimental groups and their bonding procedures are presented in Table 1. Filtek Z250 composite resin (A2 shade, 3M ESPE, St. Paul, MN., USA) was built up to approximately 3.5mm. Each 1.2mm-thick layer was polymerized for 20 s using a dental light curing unit (Hilux Ultra Plus, Benlioglu Dental, Inc., Ankara, Turkey; Light intensity: 600mW/cm²) according to the manufacturer's instructions.

After storing in distilled water for 24 h, each specimen was trimmed to a rectangular shape using an Isomet low-speed diamond saw. To shape each specimen as an hourglass, two notches were made along the dentin-adhesive interface line on opposite sides with a diamond bur mounted in a low-speed press drill (Pressdrill, Samchully Machinery Co. Ltd.,

Table 1. Bonding procedures, composition, and pH of each adhesive.

Group	Bonding procedure	Adhesive	Composition	pH
EX1	a(20s), c(20s), d, i(10s)	Experimental adhesive	EGMP, MEP, UDMA, HEMA, CQ, 4E, ethanol	1.96
EX2	a(20s), a(20s), c(20s), d, i(10s)			
EX+	a(20s), c(20s), d, h, i(20s)			
AP1	a(20s), c(20s), d, i(10s)	Adper Prompt (3M ESPE, St. Paul, MN, USA)	Liquid 1 : Methacrylated phosphoric esters, bis-GMA, initiators, stabilizers Liquid 2 : Water, HEMA, polyalkenoic acid, stabilizers	1.06
AP2	a(20s), a(20s), c(20s), d, i(10s)			
AP+	a(20s), c(20s), d, h, i(20s)			
SE	b(20s), c(20s), g, i(10s)	Clearfil SE Bond (Kuraray Co., Osaka, Japan)	Primer : HEMA, MDP, hydrophilic dimethacrylate, N,N-diethanol-p-toluidine, CQ, water Bond : MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, N,N-diethanol-p-toluidine, CQ, silanated colloidal silica	Primer: 1.9 Bond: 2.8
SE+	b(20s), c(20s), h, i(20s)			
AB	e(15s), f(5 times), d, h, i(20s)	All-Bond 2 (Bisco, Itasca, IL., USA)	Etchant : 32% H3PO4 Primer A : Acetone, ethanol, Na-N-tolyglycine glycidylmethacrylate Primer B : Acetone, ethanol, biphenyl dimethacrylate D/E resin : UDMA, bis-GMA, HEMA	Etchant: 0 Primer A: 9 Primer B: 5 D/E resin: 6.9

Procedures : a. apply self-etching adhesive with agitation; b. apply self-etching primer with agitation; c. waiting period; d. air-dry gently; e. etch and rinse; f. mix and apply primer; g. apply the bond of Clearfil SE bond; h. apply the D/E resin of All-Bond 2; i. light-cure

Abbreviations : EGMP - ethylene glycol methacrylate phosphate; MEP - MONO-2-(methacryloyloxy) ethyl phthalate; UDMA - urethane dimethacrylate; HEMA - 2-hydroxyethyl methacrylate; CQ - camphoroquinone; 4E - ethyl 4-dimethylaminobenzoate; bis-GMA - bisphenol A diglycidyl methacrylate; MDP - 10-methacryloyloxydecyl dihydrogen phosphate

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Shiheung City, Korea) as the procedure for the microtensile bond strength test. The specimen was serially sectioned with the low-speed saw so that the dimensions of the bonded surface area of the hourglass-shaped slab specimens were 1.05 ± 0.06 mm wide and 0.65 ± 0.07 mm thick (Figure 1). Copious water irrigation was done throughout the sectioning procedures. After the thickness and width of the narrowest neck of a specimen were measured with a digital micrometer, the specimen was attached to an Instron measuring apparatus that had two parallel pins using cyanoacrylate cement (Super Glue Gel, 3M, St. Paul, MN., USA) to guide the tensile load at a right angle to the bonded interface. The whole assembly was set up in a universal

testing machine (UTM, Instron model 4466, Instron Corp., Canton, MA., USA), and the strength value at breakage was measured at a crosshead speed of 1mm/min.

The fractured surfaces were examined using scanning electron microscopy (SEM, JSM-840A, JEOL Ltd, Tokyo, Japan).

The data were analyzed using two-way ANOVA, and a t-test with Bonferroni correction was performed post hoc at 5% level of significance. The data from each experimental group were compared with those of the reference groups using a t-test. Statistical analysis was performed using SigmaStat (ver. 3.11; Systat Software, Inc., Chicago, IL., USA).

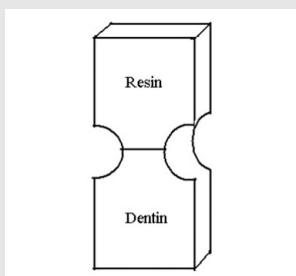


Figure 1. Diagram of microtensile bond strength test specimen.

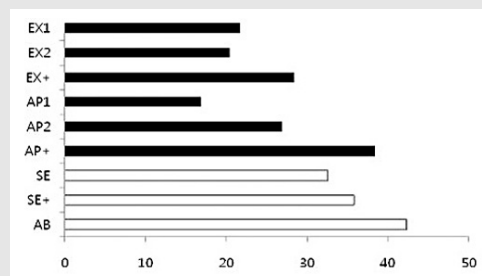


Figure 2. Microtensile bond strength (MTBS) of self-etching adhesives.

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Table 2. Microtensile bond strength (MTBS) of self-etching adhesives evaluated according to the bonding protocols (MTBS of self-etching primer system and that of total etch adhesive system was also measured for reference; units: MPa, mean \pm standard deviation)

Adhesives	One coat	Two coats	D/E resin coat added	Two-way interaction effect	
EX	EX1* 21.7 \pm 9.5 (18) c	EX2 20.4 \pm 8.6 (21) c	EX+ 28.4 \pm 11.7 (20) b	EX < AP	p = 0.031
AP	AP1 16.8 \pm 4.3 (19) c	AP2 26.9 \pm 10.4 (23) b	AP+ 38.4 \pm 11.5 (21) a	one coat = two coats < D/E resin	p < 0.001
SE	-	SE 32.6 \pm 10.4 (22)	SE+ 35.8 \pm 14.0 (18)	adhesive x bonding protocol	p = 0.003
AB	-	-	AB 42.3 \pm 12.6 (34)		

* Abbreviations are group names.

The numbers in parentheses denote the values of the samples.

Groups with the same superscript letters are not statistically different (two-way ANOVA, $p > 0.05$).

T-tests compared the groups bonded with a coat of D/E resin and each reference group. Statistically significant differences were observed between EX+ and AB ($p < 0.001$) and between SE and AB ($p = 0.004$). There was no significant difference among AP+, SE, SE+, and AB ($p > 0.05$).

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Results

The MTBSs of the groups bonded with one coat of SEA plus a coat of D/E resin (EX+ and AP+) were significantly higher than those of the groups bonded with two coats of SEAs (EX2 and AP2, $p < 0.05$). There was no statistically significant difference between the MTBS of EX1 and that of EX2 ($p > 0.05$). Note, however, that the MTBS of AP2 was significantly higher than that of AP1 ($p < 0.05$). AP+ did not show statistically different bond strength than the reference groups, SE, SE+, and AB (t-test, $p < 0.05$). Although it also had statistically similar bond strength as SE and SE+ ($p > 0.05$), EX+ showed significantly lower bond strength than AB ($p < 0.001$). SE's bond strength was not statistically different from that of SE+ but was significantly different from that of AB ($p = 0.004$) (Table 2 and Figure 2).

In the scanning electron micrographs of the fractured dentin surfaces of EX1, a fish scale-like pattern was observed; this

suggests that their relatively easy detachment from the overlying composite resin is due to the failure to copolymerize (Figure 3). With two coats of AP (AP2), a honeycomb appearance was observed (Figure 4). The fractured surfaces in the adhesive layer of specimens bonded with AB showed features typical of failure in brittle materials (Figure 5).

Discussion

SEAs simplify the clinical bonding procedure and reduce technique sensitivity but show lower bond strengths than those of the earlier generation adhesives^{1,8-10}. In this study, the bond strengths of the self-etching adhesives (EX and AP) were lower than those of the self-etching primer system (SE) and the total etching system (AB) when the materials were applied according to the manufacturers'

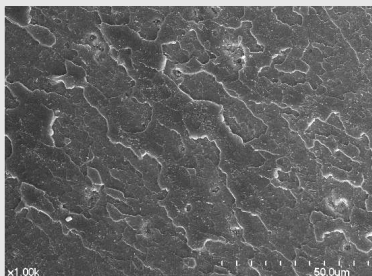


Figure 3. Scanning electron micrograph of the fractured dentin surface of the specimen bonded with one coat of experimental self-etching adhesive (EX1, $\times 1,000$). A fish scale pattern was observed with $\times 1,000$ high magnification, suggesting easy detachment from the overlying composite resin due to failure of copolymerization.

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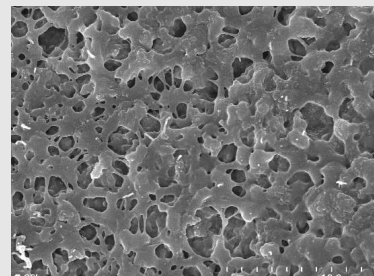


Figure 4. Scanning electron micrograph of the fractured dentin surface of the specimen bonded with two coats of Adper Prompt (AP2, $\times 5,000$). The honeycomb appearance was observed with $\times 5,000$ high magnification.

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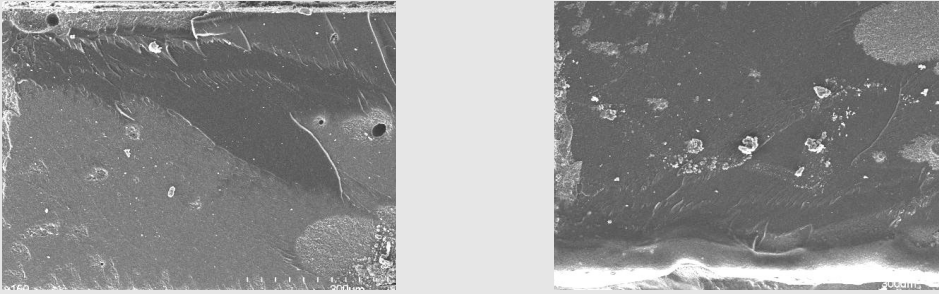


Figure 5. Scanning electron micrograph of the fractured surface of the specimen bonded with All?Bond 2 (AB, $\times 150$; a: resin side; b: dentin side). The fractured surface showed failure in the adhesive layer. The fracture occurring within the adhesive layer showed the typical fracture pattern of brittle materials.

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recommendations. SEAs are more hydrophilic than earlier generation adhesives because of the increased concentration of acidic functional monomers. Other studies suggest that such hydrophilicity causes the weaker bonding of SEAs¹²⁻¹⁵. Some of these studies suggest that polymerized SEAs act as permeable membranes that permit water to diffuse across the adhesive layer^{12,13}. Water-filled channels within the adhesive layer have been described as water blisters, honeycomb structures, microvoids, and water trees and have been considered the sites of incomplete water removal, phase separation and sub-optimal resin polymerization, and long-term hydrolytic degradation^{12,15}. In the brittle fracture of dentin bonding, defects such as the honeycomb appearance and interfacial gaps between the adhesive layer and the composite resin may cause the crack front to spread along the bonded complex and reduce the bond strengths¹⁹. In this study, there was no difference between the bond strength of EX1 and that of EX2 ($p > 0.05$). Note, however, that the bond strength of AP increased significantly when two successive coats were applied ($p < 0.05$). Contemporary SEAs are made very thin to penetrate beyond the dentin smear layer and demineralize the underlying dentin structure. Since the oxygen inhibition of the thin, unfilled adhesive layer results in defective bond formation, multiple coating has been recommended for SEAs^{1,11}. Although there is still controversy regarding the relationship between the adhesive layer thickness and bond strength^{18,20}, multiple coating may make the adhesive layer thick, distribute stresses well, and increase bond strength²¹. Considering the crack mechanism, shrinkage stresses of the polymerizing composite resin generally concentrate on the bonding interface. If these stresses exceed the mechanical

strength of the adhesive, ruptures within the bonded complex may occur²². In this study, among the groups with a coat of hydrophobic, neutral adhesive resin, only the EX+ group showed statistically low MTBS ($p < 0.05$). The low bond strength of EX+ may suggest its inability to develop the hybrid layer required for bonding. Once a hybrid layer is adequately made, bond strength may be affected by the mechanical properties of the adhesive layer²³. “All-in-one” SEAs are complex mixtures of acidic functional monomers, cross-linking monomers, solvent, inhibitors, and activators. The concentration of hydrophobic monomers is relatively low²⁴. Since the mechanical strength of the adhesive primarily comes from the polymerization of cross-linking monomers, the low concentration of monomers may impair bond strength^{25,26}. The application of the hydrophobic adhesive resin may have improved the mechanical properties of the adhesive layer, which explains the measured increase in bond strength^{24, 27, 28}. In this study, the light-curing time for D/E resin of AB was 20 s, and that for EX, AP, and SE was 10 s in keeping with the manufacturers’ recommendations. Although curing time may affect bond strength, strong adhesives showed high bond strength.

In this study, the pHs of EX, AP, primer of SE, and bond of SE were 1.96, 1.0, 1.9, and 2.8, respectively. Another study showed that the high acidity of SEAs hindered the polymerization reaction; hence the very low initial degree of conversion²⁹. If this is the case, the adhesive layer may not hold under the shrinkage stress of the polymerizing composite resin, and cracks may occur within the adhesives during the polymerization of the composite resin³⁰. This may reduce the strength of the SEA bond to dentin. An

inverse relationship between the acidity of the adhesive and MTBS of the chemically cured composite to dentin has been reported³¹. The high concentration of acidic monomer in the oxygen-inhibited layer has been found to interfere with the tertiary amine of light-cured composite resin, delaying polymerization⁶. In other words, the high acidity of SEA interferes not only with SEA polymerization but also with the copolymerization of the adhesive and the composite resin.

In this study, there was no significant difference between the MTBS of SE and that of SE+ ($p > 0.05$). Clearfil SE Bond showed relatively high bond strength when it was used according to the manufacturer's recommendation; such strength is attributed to functional monomer 10-MDP, which can interact chemically with hydroxyapatite³². The bond of Clearfil SE Bond is based on the filled adhesive resin composed of hydrophobic monomers, and it has no

solvent. Even though the bond of Clearfil SE Bond has a low pH, it showed a high degree of conversion with its specific initiator system compared with SEAs^{29, 33}. Therefore, SE can create a sufficiently strong adhesive layer with few voids using its own bond.

Conclusion

This study confirmed our hypothesis, i.e., applying a hydrophobic, neutral bonding resin as a second coat will improve the bond strength of SEAs. The hydrophobic, neutral adhesive resin improved the integrity of the bonding complex obtained with SEA. Therefore, we recommend that SEA be used as a self-etching primer with an additional coat of hydrophobic, neutral adhesive resin to increase its bond strength and bonding integrity.

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