

Effect of additional etching and ethanol-wet bonding on the dentin bond strength of one-step self-etch adhesives

Joonghee Ahn¹,
 Kyoung-Hwa Jung¹,
 Sung-Ae Son¹, Bock
 Hur¹, Yong-Hoon
 Kwon², Jeong-Kil
 Park^{1*}

¹Department of Conservative Dentistry, ²Department of Dental Materials, School of Dentistry, Pusan National University, Yangsan, Korea

Objectives: This study examined the effects of additional acid etching on the dentin bond strength of one-step self-etch adhesives with different compositions and pH. The effect of ethanol wetting on etched dentin bond strength of self-etch adhesives was also evaluated. **Materials and Methods:** Forty-two human permanent molars were classified into 21 groups according to the adhesive types (Clearfil SE Bond [SE, control]; G-aenial Bond [GB]; Xeno V [XV]; Beauti Bond [BB]; Adper Easy Bond [AE]; Single Bond Universal [SU]; All Bond Universal [AU]), and the dentin conditioning methods. Composite resins were placed on the dentin surfaces, and the teeth were sectioned. The microtensile bond strength was measured, and the failure mode of the fractured specimens was examined. The data were analyzed statistically using two-way ANOVA and Duncan's *post hoc* test. **Results:** In GB, XV and SE (pH ≤ 2), the bond strength was decreased significantly when the dentin was etched ($p < 0.05$). In BB, AE and SU (pH 2.4 - 2.7), additional etching did not affect the bond strength ($p > 0.05$). In AU (pH = 3.2), additional etching increased the bond strength significantly ($p < 0.05$). When adhesives were applied to the acid etched dentin with ethanol-wet bonding, the bond strength was significantly higher than that of the no ethanol-wet bonding groups, and the incidence of cohesive failure was increased. **Conclusions:** The effect of additional acid etching on the dentin bond strength was influenced by the pH of one-step self-etch adhesives. Ethanol wetting on etched dentin could create a stronger bonding performance of one-step self-etch adhesives for acid etched dentin. (*Restor Dent Endod* 2015;40(1):68-74)

Received July 23, 2014;
 Accepted October 11, 2014.

Key words: Ethanol-wet bonding; One-step self-etch adhesive; pH of adhesives

¹Ahn J; Jung KH; Son SA; Hur B;
 Park JK, Department of Conservative Dentistry, School of Dentistry, Pusan National University, Yangsan, Korea

²Kwon YH, Department of Dental Materials, School of Dentistry, Pusan National University, Yangsan, Korea

***Correspondence to**
 Jeong-Kil Park, DDS, MSD, PhD.
 Associate Professor, Department of Conservative Dentistry, School of Dentistry, Pusan National University, Beomeo-ri, Mulgeum-eup, Yangsan, Korea 626-787
 TEL, +82-55-360-5221; FAX, +82-55-360-5214; E-mail, jeongkil@pusan.ac.kr

Introduction

Contemporary adhesive systems can be classified into etch-and-rinse and self-etch adhesives based on the underlying adhesion strategy to interact with the enamel and dentin substrate. Although the etch-and-rinse procedure is characterized by the use of 35% phosphoric acid, which is rinsed off before applying the adhesives, the self-etch systems are acidic monomer formulations that simultaneously demineralize and infiltrate the substrate. Therefore self-etch adhesives do not require a separate etching step.¹

Self-etch adhesives can be obtained as 'two-step' and 'one-step' adhesives, depending on whether a self-etching primer and adhesive resin are provided separately or are

combined into a single solution. Consequently, this approach has been claimed to be more user-friendly (shorter application time, less steps) and less technique-sensitive (no wet-bonding, simple drying) with a lower incidence of post-operative sensitivity.²⁻⁸ On the other hand, the etching effect of mild self-etch adhesives has been reported to be less effective in interacting with a thicker smear layer-covered dentin because the residual smear layer inhibits monomer infiltration into the underlying dentin.⁹⁻¹¹ Therefore, the main challenge for current self-etch adhesives is to dissolve the smear layer without excessively demineralizing the tooth surface.

Several methods have been used to remove or minimize the smear layer. Acid etching with 35% phosphoric acid is used widely in the adhesive dentistry to remove the smear layer created by instrumentation. Although the selective etching of enamel with phosphoric acid is strongly recommended to provide better bonding of self-etch adhesives, many studies have reported a decrease in the bond strength to dentin when acid etching is used before applying two-step mild self-etch adhesives, such as Clearfil SE bond. The reason for the decrease in the dentin bond strength of self-etch adhesives with additional acid etching is explained mainly by incomplete monomer infiltration into the demineralized collagen network.¹²⁻¹⁴ This discrepancy between demineralization and resin infiltration, which occurs at the resin-dentin interface, leads to nanoleakage and contributes to bond instability over time as a result of the degradation of unprotected collagen fibrils within the hybrid layer.^{15,16}

For the one-step self etch adhesives, which comprise the latest generation of most simple-to-use adhesives, there is some controversy regarding the use of additional phosphoric acid etching on dentin with studies showing an improved bond strength, no effect or reduced bond strength.¹⁷⁻¹⁹ All Bond Universal and Single Bond Universal are recently commercialized one-step self-etch adhesives. The manufacturer claims that they are suitable for both self-etch and etch-and-rinse approach. Clinically used one-step self-etch adhesives have a wide pH range. Depending on the pH of the self-etch adhesives, the actual infiltration depth of the adhesives at the dentin varies. Therefore the effects of additional etching might be affected by the pH of one-step, self-etch adhesives.

Ethanol is known to be a much better solvent for resin monomers than water since it may promote the infiltration of monomers into the collagen fibrils, which can lead to an improvement of the bonding performance.²⁰ This concept, called 'ethanol-wet bonding technique', has been reported in many studies.²⁰⁻²³ These studies used etch-and-rinse adhesives (three-step or two-step) while no study has used self-etch adhesives, particularly one-step self-etch adhesives.

The present study examined the effects of additional acid

etching on the dentin bond strength of one-step self-etch adhesives with different compositions and pH. The effects of wetting the etched dentin with ethanol on the dentin bond strength of self-etch adhesives were also evaluated. The null hypotheses tested were that the dentin bond strength of additional acid etching is not affected by the pH of the adhesive, and that ethanol-wetting of the acid etched dentin does not improve the bonding performance of one-step self-etch adhesive.

Materials and Methods

Forty-two non-restored, caries free human permanent molars within 3 months after extraction were used, which was previously approved by the Institutional Review Board of Pusan National University Dental Hospital (IRB, PNUDH-2013-014). The teeth were washed and stored in distilled water at room temperature until use. A plastic mold was filled with an autopolymerizing resin (Tokuso Curefast, Tokuyama, Tokyo, Japan) and the root surface was embedded in acrylic resin, with the clinical crown exposed. After removing the plastic mold, the teeth were sectioned horizontally at the mid-coronal level to obtain flat, sound dentin surfaces using a diamond saw (Accutom-50, Struers, Rødovre, Denmark) with constant water cooling. The sectioned dentin surfaces were then hand-polished with 600 grit silicon carbide abrasive paper for 60 seconds under running water to create a uniform surface and smear layer. The surfaces were rinsed for 30 seconds with distilled water before applying the adhesive and composite resin.

The teeth were divided randomly into 21 groups according to the dentin conditioning methods (no etch, etch and no ethanol-wet bonding, etch and ethanol-wet bonding) and the type of self-etch adhesives. Following self-etch materials were used: one two-step self-etch adhesive as a control group, Clearfil SE Bond (SE, Kuraray, Osaka, Japan); six one-step self-etch adhesives as test groups, G-aenial Bond (GB, GC Corp., Tokyo, Japan); Xeno V (XV, Dentsply Detrey, Kostanz, Germany); BeautiBond (BB, Shofu Inc., Kyoto, Japan); Adper Easy Bond (AE, 3M ESPE, St. Paul, MN, USA); Single Bond Universal Adhesive (SU, 3M ESPE); and All-Bond Universal (AU, Bisco Inc., Schaumburg, IL, USA). Tables 1 and 2 list the general compositions and application procedures of the materials used in this study.

Bonding procedure

The no acid etching group (NOAE group) applied adhesives to the untreated dentin surfaces. In the acid etching without ethanol-wet bonding group (AESEW group), the dentin surfaces were etched with phosphoric acid (Ultra-Etch, Ultradent, South Jordan, Utah, USA) for 15 seconds, rinsed and air-dried before applying the adhesives (water-wet dentin surface). For the acid etching with ethanol-

Table 1. Compositions of materials used in this study

Material	Code	Composition
Ultra-Etch (Ultradent, South Jordan, UT, USA)		35% Phosphoric acid, Cobalt aluminate blue spinel, Cobalt zinc aluminate blue spinel
Clearfil SE Bond (Kuraray, Osaka, Japan)	SE	Primer: MDP, water, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-Diethanol p-toluidine Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N,N-Diethanol p-toluidine, silanated colloidal silica, initiator
G-aenial Bond (GC Corp., Tokyo, Japan)	GB	Acetone, distilled water, dimethacrylate, 4-MET, phosphoric acid ester monomer, silicon dioxide, photoinitiator
Xeno V (Dentsply DeTrey, Konstanz, Germany)	XV	Bifunctional acrylamides, acrylamido alkylsulfonic acid, inverse functional phosphoric acid ester, acrylic acid, butylated benzenediol, water, tert-butanol, photoinitiator
BeutiBond (Shofu Inc., Kyoto, Japan)	BB	Phosphoric acid monomer, Bis-GMA, TEGDMA, water, carboxylic acid monomer, solvent, acetone, initiator
Adper Easy Bond (3M ESPE, St.Paul, MN, USA)	AE	Methacrylate functionalized polyalkenoic acid, HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, Vitrebond copolymer, water, ethanol, silica filler, photoinitiator
Single Bond Universal (3M ESPE, St.Paul, MN, USA)	SU	10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, silane, ethanol, water, filler, photoinitiator
All-Bond Universal (Bisco, Schaumburg, IL, USA)	AU	MDP, Bis-GMA, ethanol
Filtek Z-250 (3M ESPE, St.Paul, MN, USA)		Bis-GMA, UDMA, Bis-EMA, zirconia, silica

Compositions of the materials are provided by the manufacturers.

4-MET, 4-methacryloyloxyethyl trimellitate; MDP, methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A dimethacrylate.

Table 2. Application procedures of self-etch systems

Adhesive	Lot No.	pH	Application procedure
SE	Primer: 01155A Bond: 01733A	2.0	Apply primer and leave for 20 sec. Dry with mild air flow. Apply bond and distribute evenly with flow. Light cure for 10 sec.
GB	1105051	1.5	Apply and leave for 10 sec. Air dry for 5 sec. Light cure for 10 sec.
XV	1108001677	1.8	Apply two coats with rubbing for 20 sec. Air dry for 5 sec. Light cure for 20 sec.
BB	041173	2.4	Apply to dried dentin for 10 sec. Air dry for 3 sec. Light curing for 10 sec.
AE	399989	2.7	Apply to dried dentin for 20 sec. Air dry for 5 sec. Light curing for 10 sec.
SU	472584	2.7	Apply with rubbing for 20 sec. Air Dry for 5 sec. Light cure for 10 sec.
AU	1200002722	3.2	Apply 2 coats for 10 - 15 sec/coat with agitation. Air dry for 10 - 15 sec. Light cure for 10 sec.

pH and application procedure of the materials are provided by the manufacturers.

SE, Clearfil SE Bond; GB, Gaenial bond; XV, Xeno V; BB, BeutiBond; AE, Adper Easy Bond; SU, Single Bond Universal; AU, All bond Universal.

wet bonding group (AEWEW group), the etched dentin surface was immersed into 100% ethanol for 1 minute after etching with phosphoric acid for 15 seconds, rinsed and air dried. The excess ethanol was removed by gentle blotting with filter paper to leave a visible moist, ethanol-wet dentin surface. The adhesives were applied according to the manufacturer's instructions, as listed in Table 2. After the bonding procedures, light-cured composite resin (Filtek Z-250, 3M ESPE) was applied via increment layering. Each increment was polymerized for 20 seconds using an LED visible light polymerizing unit (SmartLight iQ2, Dentsply Cualk, Milford, DE, USA). The height of the total resin build up was 5 mm. The restored teeth were stored in distilled water at room temperature for 24 hours.

Microtensile bond strength testing

The restored teeth were sectioned longitudinally to produce 1 × 1 × 10 mm specimens using diamond saw under copious amounts of water. Each group contained 11 specimens. Each specimen was mounted onto the jig of the microtensile testing machine (Bisco Inc.) using cyanoacrylate cement (Zapit, Dental Ventures of America, Corona, CA, USA). A tensile load was applied at a 1.0 mm/min cross-head speed until bonding failure. The maximum load at failure was recorded.

Failure mode examination

The failure mode of each fractured specimen was examined using an optical operating microscope (Leica M320, Leica Microsystems, Wetzlar, Germany) at ×20 magnification. The failure mode was designated as follows: adhesive, if the bonded interface failed between the dentin and composite resin; cohesive, if the failure was in the

dentin or composite resin; or mixed, a combination of adhesive and cohesive failure.

Statistical analysis

Two-way analysis of variance (ANOVA) followed by Duncan *post hoc* test was used to determine statistically significant differences in the microtensile bond strength (μ TBS) according to the adhesives (7 self-etch adhesives) and the conditioning methods (NOAE vs. AESEW vs. AEWEW). A *p* value < 0.05 was considered significant. The software used was SPSS 15.0 (SPSS Inc., Chicago, IL, USA).

Results

Microtensile bond strength

Two-way ANOVA revealed that there were significant differences in μ TBS according to the type of adhesives and the conditioning methods (*p* < 0.001). And there was significant interaction effect between the type of adhesives and the conditioning methods (*p* < 0.001). Table 3 lists μ TBS of NOAE group, AESEW group, and AEWEW group. Figure 1 compares the μ TBS of NOAE group and AESEW group. In GB (pH = 1.5) and SE (pH = 2.0), the dentin bond strength was decreased significantly by additional etching of the dentin surface (*p* < 0.05). In XV (pH = 1.8), BB (pH = 2.4), AE (pH = 2.7) and SU (pH = 2.7), the dentin bond strength was similar regardless of the additional etching (*p* > 0.05). In AU (pH = 3.2), the dentin bond strength was increased significantly with additional etching (*p* < 0.05). In NOAE group, SE, BB and SU showed higher dentin bond strength than GB, XV and AU. In AESEW group, SU and AU had the highest bond strength among the materials while GB and XV showed the lowest.

Table 3. Effect of additional acid etching and ethanol-wet bonding on the dentin bond strength (mean \pm SD in MPa)

Adhesive	pH	NOAE	AESEW	AEWEW
SE	2.0	29.8 \pm 3.0 ^{gh}	24.7 \pm 5.2 ^{ef}	36.6 \pm 5.1 ⁱ
GB	1.5	16.4 \pm 1.7 ^{bc}	11.3 \pm 2.3 ^a	17.6 \pm 3.1 ^{bc}
XV	1.8	17.7 \pm 2.8 ^{bc}	14.0 \pm 2.0 ^{ab}	19.2 \pm 6.1 ^{cd}
BB	2.4	27.0 \pm 4.2 ^{fgh}	24.2 \pm 3.9 ^{ef}	27.7 \pm 5.5 ^{fgh}
AE	2.7	22.7 \pm 3.1 ^{de}	20.2 \pm 4.3 ^{cd}	26.7 \pm 5.7 ^{fg}
SU	2.7	29.7 \pm 5.2 ^{gh}	30.8 \pm 4.0 ^h	36.3 \pm 4.5 ⁱ
AU	3.2	16.3 \pm 4.2 ^{bc}	29.1 \pm 3.1 ^{gh}	35.4 \pm 4.4 ⁱ

Values are expressed as means \pm SD (n = 11 for each groups).

Different superscript letters indicate a statistically significant difference among groups (*p* < 0.05).

Results of two-way ANOVA: adhesives, *p* < 0.001; conditioning methods, *p* < 0.001; adhesives \times conditioning methods, *p* < 0.001. NOAE, no acid etching; AESEW, acid etching without ethanol-wet bonding; AEWEW, acid etching with ethanol-wet bonding; SE, Clearfil SE Bond; GB, Gaenial bond; XV, Xeno V; BB, BeutiBond; AE, Adper Easy Bond; SU, Single Bond Universal; AU, All bond Universal; SD, standard deviation.

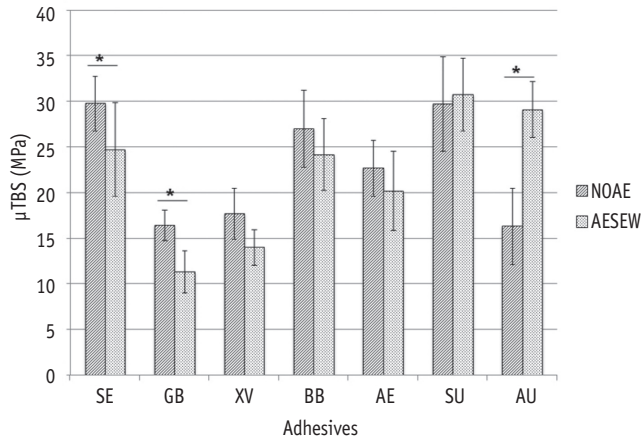


Figure 1. Effect of additional acid etching on the dentin bond strength (MPa) of adhesives. The asterisk indicates that there was significant difference. μ TBS, microtensile bond strength; NOAE, no acid etching; AESEW, acid etching without ethanol wet-bonding; SE, Clearfil SE Bond; GB, Gaenial bond; XV, Xeno V; BB, BeutiBond; AE, Adper Easy Bond; SU, Single Bond Universal; AU, All bond Universal.

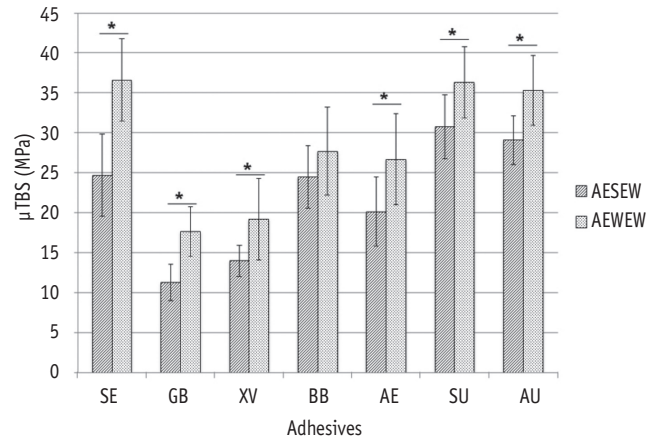


Figure 2. Effect of wetting etched dentin with ethanol on the dentin bond strength (MPa) of adhesives. The asterisk indicates that there was significant difference. μ TBS, microtensile bond strength; AESEW, acid etching without ethanol-wet bonding; AEWEW, acid etching with ethanol-wet bonding; SE, Clearfil SE Bond; GB, Gaenial bond; XV, Xeno V; BB, BeutiBond; AE, Adper Easy Bond; SU, Single Bond Universal; AU, All bond Universal.

Table 4. Modes of failure after microtensile bond strength testing

Adhesive	NOAE	AESEW	AEWEW
SE	0/11/0	0/11/0	0/5/6
GB	0/10/1	1/10/0	0/11/0
XV	0/11/0	0/11/0	0/11/0
BB	0/11/0	0/11/0	0/11/0
AE	0/9/2	0/11/0	0/11/0
SU	0/8/3	0/9/2	0/6/5
AU	0/11/0	0/10/1	0/7/4

Failure modes: mixed failure/adhesive failure/cohesive failure in dentin or composite.

NOAE, no acid etching; AESEW, acid etching without ethanol-wet bonding; AEWEW, acid etching with ethanol-wet bonding; SE, Clearfil SE Bond; GB, Gaenial bond; XV, Xeno V; BB, BeutiBond; AE, Adper Easy Bond; SU, Single Bond Universal; AU, All bond Universal.

The μ TBS of AESEW group and AEWEW group were compared in Figure 2. When the adhesives were applied to the acid etched dentin using the ethanol-wet bonding technique, the dentin bond strength was increased significantly compared to the acid etching with no ethanol-wet bonding for all adhesives except for BB.

Failure modes

The failure modes of the specimens were summarized in Table 4. For all groups, adhesive failure was the most frequent pattern of failure. On the other hand, the amount of cohesive failure increased after applying the ethanol-wet bonding technique for SE, SU and AU.

Discussion

Self-etch adhesives are promising developments in adhesive dentistry, particularly in terms of the reduction of the necessary application steps and the possibility of a chemical interaction with hydroxyapatite-coated collagen fibers.¹ On the other hand, bonding to dentin with additional phosphoric acid etching still remains controversial and has been discussed by a range of authors.¹⁷⁻¹⁹

In this study, the dentin bond strength of SE, which was used as the control group, was decreased when the dentin surfaces were treated previously with phosphoric acid. This result is in agreement with a previous study.¹² GB showed similar results. The pH of these adhesives was less than 2 (SE, 2.0; GB, 1.5). These results can be explained by incomplete adhesive infiltration into the demineralized collagen network due to the over-etching by phosphoric acid and acidic adhesive. Also, it may be from the removal of residual hydroxyapatite from the collagen mesh, which could reduce the potential for chemical adhesion.^{13,24,25} In XV (pH = 1.8), dentin bond strength was decreased by additional etching, but the difference was not significant. This discrepancy might be due to other factors, such as compositions.

In BB (pH = 2.4), AE (pH = 2.7), and SU (pH = 2.7), additional acid etching did not affect the dentin bond strength. This is in agreement with the manufacturer's description for SU. AE, which has the same pH as SU but different functional monomer, showed similar trend. This suggests that adhesives, which have a pH ranging from 2.4 to 2.7, do not over-etch the dentin surface and are appropriate for favorable resin monomer infiltration. In AU with pH 3.2, additional acid etching increased the dentin bond strength, which is different from the manufacturer's description. The low acidity of AU might not have been sufficient to etch the dentin surface effectively for resin monomer infiltration, and as a consequence, additional acid etching is necessary. These results suggest that the effect of additional acid etching on the dentin bond strength of one-step self-etch adhesives might be influenced by the pH of the adhesives. However, the dentin bond strength between the self-etch adhesives varied regardless of the pH. This suggests that the bond strength of adhesives might be dependent on both chemical composition and pH of adhesives. In this study, the pH of adhesives was taken from manufacturers' report. However, there is a possibility that the testing methods for pH might be different among manufacturers. Therefore, to obtain more scientific and accurate evidence, pH measurement may be necessary.

When one-step self-etch adhesives were applied to acid-etched dentin with ethanol-wet bonding (AEWEW group), the dentin bond strength was higher compared to no ethanol wet bonding (AESEW group) for all adhesives

except for BB. Moreover, AEWEW group showed larger percentage of cohesive failure, particularly for SE, SU and AU, compared to the other groups. This can be explained by the effect of ethanol-wet bonding. Ethanol is a much better solvent for resin monomers and has been shown to decrease the collagen fibrillar diameter and increase the interfibrillar space in the hybrid layers, thereby facilitate the infiltration of resin monomers.²⁰ This technique can provide better resin sealing of the collagen matrix and reduce the permeability of the resin-dentin bonding interface which would diminish the collagenolytic activities of endogenous enzymes and improve the bonding durability to dentin.^{26,27} Although ethanol-saturated dentin might be a better substrate for adhesive infiltration, the ethanol would evaporate rapidly because its vapor pressure is greater than that of water, thus compromising the wettability of etched dentin after a short period time. Note that ethanol-wet bonding is quite sensitive to the adhesive application time.

In summary, the effect of additional acid etching on the dentin bond strength of the self-etch adhesive was affected by the pH of adhesives. The ethanol saturation of the etched dentin surface enhanced the bonding performance. Therefore, the null hypotheses was rejected. Considering the applicability of results of this study to the clinical situations, additional acid etching should be performed with careful consideration of the pH of the one-step self-etch adhesives used. For further understanding of the factors that contribute to the bonding characteristics, more adhesives with different pH, especially the pH over 3.0, should be tested. Moreover long-term *in vitro* studies should investigate the stability of these bonds over time.

Conclusions

Within the limitation of the present study, the effects of additional acid etching on the dentin bond strength were influenced by the pH of one-step self-etch adhesives. Wetting etched dentin with ethanol could enhance the bonding performance of one-step self-etch adhesives on acid etched dentin.

Orcid numbers:

Jeong-Kil Park, 0000-0001-6333-8138

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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