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The effect of additional etching and curing mechanism of composite resin on the dentin bond strength

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PURPOSE. The aim of this study was to evaluate the effects of additional acid etching and curing mechanism (light-curing or self-curing) of a composite resin on the dentin bond strength and compatibility of one-step self-etching adhesives. **MATERIALS AND METHODS**. Sixteen human permanent molars were randomly divided into eight groups according to the adhesives used (All-Bond Universal: ABU, Clearfil S3 Bond: CS3), additional acid etching (additional acid etching performed: EO, no additional acid etching performed: EX), and composite resins (Filtek Z-250: Z250, Clearfil FII New Bond: CFNB). Group 1: ABU-EO-Z250, Group 2: ABU-EO-CFNB, Group 3: ABU-EX-Z250, Group 4: ABU-EX-CFNB, Group 5: CS3-EO-Z250, Group 6: CS3-EO-CFNB, Group 7: CS3-EX-Z250, Group 8: CS3-EX-CFNB. After bonding procedures, composite resins were built up on dentin surfaces. After 24-hour water storage, the teeth were sectioned to make 10 specimens for each group. The microtensile bond strength test was performed using a microtensile testing machine. The failure mode of the fractured specimens was examined by means of an optical microscope at ×20 magnification. The data was analyzed using a one-way ANOVA and Scheffe's post-hoc test (α=.05). **RESULTS.** Additional etching groups showed significantly higher values than the no additional etching group when using All-Bond Universal. The light-cured composite resin groups showed significantly higher values than the self-cured composite resin groups in the Clearfil S3 Bond. **CONCLUSION.** The additional acid etching is beneficial for the dentin bond strength when using low acidic one-step self-etch adhesives, and low acidic one-step self-etch adhesives are compatible with self-cured composite resin**.** The acidity of the one-step self-etch adhesives is an influencing factor in terms of the dentin bonding strength and incompatibility with a self-cured composite resin. ***[J Adv Prosthodont 2013;5:479-84]***

KEY WORDS: Acidity; Adhesive; Bond strength; Compatibility; Self-etch

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INTRODUCTION

Adhesive technology has developed rapidly since Buonocore introduced the acid etching technique in 1955.[1] The main challenge for dental adhesive is to provide effective bonding to enamel and dentin, which have different characteristics. Enamel bonding is based on micromechanical interlocking between the resin and etched enamel, which has been proven to be reliable and durable. On the other hand, bonding to dentin is far more complex due to the inherent characteristics of dentin, such as variable tubular structure, high organic content and positive dentinal fluid flow.[2-4]

Traditionally, complicated and time-consuming multi-step adhesive systems have been used to achieve successful dentin bonding. Although multi-step adhesives can provide high bond strength, they are considered to be highly technique-sensitive, and many procedural errors can occur. As a consequence, clinicians have demanded simpler, more user-friendly and less technique sensitive adhesives. According to the clinicians' demand, dental material manufacturers have developed new simple-step systems.

On the other hand, there have been two concerns regarding simple step adhesive systems. In many studies, several authors have reported that selective enamel etching is effective in terms of the bond strength when using self-etch adhesives, but additional acid etching has adverse effects on the dentin bond strength, resulting in decreased bond strength.[5-8] Therefore, they suggested that prior acid etching should be limited to the enamel when using self-etch adhesives.

Some of the one-step self-etch adhesives were found to be incompatible with self-cured and dual-cured composite resins.[9] These resins are still used frequently in core build up for prosthodontic foundation. An adverse chemical interaction between the unpolymerized acidic resin monomer in the adhesive and the basic tertiary amine catalyst in the composite resin is believed be responsible for the incompatibility.[10] This interaction results in the consumption of tertiary amines in acid-base reactions, depriving them of their capacity to generate free radicals in the subsequent redox reaction.[11]

All-Bond Universal (Bisco, Schaumburg, IL, USA) is a recently commercialized one-step self-etch adhesive. The manufacturer states that it is suitable for both the self-etch and total-etch approach, and that it guarantees compatibility with self-cured composite resins.

Therefore, this study examined the effects of additional acid etching on the dentin bond strength of All-Bond Universal and assessed its compatibility with a self-cured composite resin *in vitro* using a microtensile bond strength (μTBS) test.

MATERIALS AND METHODS

In this study, sixteen non-restored, caries free human permanent molars extracted due to periodontal problems were used within 3 months after extraction. This study was approved by the Ethics Committee and Research Office of the Pusan National University Dental Hospital (PNUDH-2013-014). The teeth were washed and stored in distilled water at room temperature until used. A plastic mold was filled with an autopolymerizing resin (Tokusocurefast, Tokuyama, Tokyo, Japan), and the root surface was embedded in acrylic resin, leaving 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) under constant water cooling. The sectioned dentin surfaces were then hand-polished with a 600-grit silicon carbide abrasive paper for 60 seconds under running water to create a uniform surface and smear layer. The surfaces were then rinsed with distilled water for 30 seconds before the adhesive and composite resin applications.

The teeth were divided randomly into 8 groups according to the etching technique used and the curing mechanism of the composite resin. Table 1 lists the general com-

Table 1. Compositions and application procedures of materials used in this study

Material	Lot number	Composition	Application procedure
Ultra-Etch (Ultradent, South Jordan, UT, USA)	B6CCH	35% Phosphoric acid, Cobalt aluminate blue spinel, Cobalt zinc aluminate blue spinel	Apply for 15 seconds and rinse thoroughly
All-Bond Universal (pH=3.2) (Bisco, Schaumburg, IL, USA)	1200003202	MDP, Bis-GMA, ethanol	Apply 2 coats for 10-15 seconds/coat with agitation Air dry Light cure for 10 seconds
Clearfil S3 Bond (pH=2.7) (Kuraray, Osaka, Japan)	00069A	MDP, HEMA, Bis-GMA, water, ethanol, photo-initiator, silanated colloidal silica	Apply for 20 seconds Air dry Light cure for 10 seconds
Filtek Z-250 (3M ESPE, St.Paul, MN, USA)	N290141	Bis-GMA, UDMA, Bis-EMA, zirconia, silica	Apply 1 mm increments to 5 mm height Light cure for 20 seconds for each increment
Clearfil F II New Bond (Kuraray, Osaka, Japan)	051196	Base: Bis-GMA, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silica, accelerators Catalyst: Bis-GMA, triethyleneglycoldimethacrylate, silica, catalyst, pigments	Mix base and catalyst pastes for 10-15 seconds Apply mixed composite resin in bulk and let it cured for 2.5 minutes

Bis-EMA: ethoxylatedbisphenol A dimethacrylate, Bis-GMA: bisphenol A glycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, MDP: methacryloyloxydecyldihydrogen phosphate, UDMA: urethane dimethacrylate.

positions and application procedures of the materials used in this study. Clearfil S3 Bond (Kuraray, Osaka, Japan), which has a similar composition to All-Bond Universal, was used as a control. Table 2 shows the etching technique along with the adhesive and composite resin used in each group.

For additional dentin acid etching groups, the specimens were etched with phosphoric acid (Ultra-Etch; Ultradent, South Jordan, UT, USA) for 15 seconds, followed by 15 seconds rinsing with a three-way syringe and air drying. The adhesives were applied to the dentin surface according to the manufacturer's instruction. After the bonding procedures, the light-cured composite resin (Filtek Z-250, 3M ESPE, St.Paul, MN, USA) was applied by approximately 2 mm thick incremental layering to make 5 mm high resin for the light-cured composite resin groups. Each increment was polymerized for 20 seconds using a LED visible light polymerizing unit (IQ2, Dentsply, Konstanz, Germany). For the self-cured composite resin (Clearfil F II New Bond, Kuraray, Osaka, Japan) groups, the base and catalyst were hand mixed for 15 seconds, and a mixed composite resin was applied in bulk to the bonded adhesive surfaces and polymerized for 2 minutes and 30 seconds. The height of the total resin build up was approximately 5 mm. The restored teeth were stored in distilled water at room temperature for 24 hours.

The restored teeth were sectioned longitudinally to make an approximately 1 × 1 mm long stick and 10 mm long specimens using a diamond-saw under copious amount of water. Among several specimens, 5 specimens which were appropriate for microtensile test from each tooth were collected, soeach group contained 10 specimens. Each specimen was mounted to the jig of the microtensile testing machine (Bisco, Schaumburg, IL, USA) 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 occurred. The maximum load at failure was recorded.

The failure mode of each fractured specimen was examined by optical operating microscopy (Leica M320, Leica Microsystems, Wetzlar, Germany) at a ×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.

SPSS 15.0 software (SPSS Inc., Chicago, IL, USA) was used for statistical analysis. The µTBS data from All-Bond Universal and Clearfil S3 Bond were subjected separately to one-way ANOVA and a Scheffe's post-hoc test to determine if there were significant differences among the groups. The significance level was set at $P<.05$.

RESULTS

Table 3 lists the mean µTBS values, standard deviations and significant differences among the groups. Group 1 and 2 showed significantly higher values than Group 3 and 4

Table 2. Groups used in this study according to additional etching and materials

Group	Adhesive	Additional acid etching	Composite resin
1	All-Bond Universal	acid etching	Filtek-Z250
2			Clearfil F II New Bond
3		no acid etching	Filtek-Z250
4			Clearfil F II New Bond
5	Clearfil S3 Bond	acid etching	Filtek-Z250
6			Clearfil F II New Bond
7		no acid etching	Filtek-Z250
8			Clearfil F II New Bond

Table 3. Mean microtensile bond strength (MPa) values and standard deviations (n=10)

Group	Mean ± SD	Group	Mean ± SD
1	21.1 (3.6)^{A}	5	24.7 (3.9)^{a}
2	20.6 (5.9)^{A}	6	6.8 (2.1)^{b}
3	8.1 (3.6)^{B}	7	27.9 (2.8)^{a}
4	6.6 (1.6)^{B}	8	9.4 (3.5)^{b}

Different superscript letters indicate statistically significantly different groups in colummn.

Table 4. Modes of failure after microtensile bond strength testing (n=10)

Group	Adhesive	Mixed	Cohesive
1	9	1	0
2	9	0	1
3	10	0	0
4	10	0	0
5	9	0	1
6	10	0	0
7	8	0	2
8	10	0	0

respectively. No significant differences were observed between Group 1 and 2 and between Group 3 and 4. Group 5 and 7 showed significantly higher values than Group 6 and 8, respectively. No significant differences were observed between Group 5 and 7, and between Group 6 and 8.

Table 4 summarizes the failure modes observed in the specimens. For all groups, adhesive failure was the most frequent pattern of failure.

DISCUSSION

The adhesives currently used are classified into either total-etch and self-etch systems according to the manner they deal with the smear layer. In contrast to the total-etch adhesives, self-etch adhesives do not require separate etching and rinsing steps to remove the smear layer. They incorporate the smear layer into the hybrid layer,[12] and contain acidic monomers that condition and prime the dental substrate simultaneously. Therefore, demineralization and monomer infiltration occur simultaneously, creating a hybrid layer.[12] Because there is no need for separately applied acid etching and priming, the self-etch approach has been claimed to be user-friendly, less technique-sensitive and results in reliable clinical performance.[5] Another clinical benefit of self-etch adhesives is the low incidence of post-operative sensitivity, which was attributed to the less aggressive acid etching and more superficial interaction with dentin, leaving the dentinal tubules largely obstructed with a smear layer.[13-15] All these favorable features have made self-etch adhesives popular as a new alternative in current dental practice. Nevertheless, some concerns still remain regarding the bonding effectiveness of self-etch adhesives.

Smear layers, reinforced with impregnated resin in self-etch approach, may be too weak to provide strong, durable mechanical properties, particularly on the enamel.[16] Several authors have reported a higher bond strength on the enamel with additional acid etching when using self-etch adhesives[5] and some manufacturers still recommend the use of phosphoric acid etching on the enamel prior to the application of self-etch adhesives. This additional acid etching technique on the enamel is called selective enamel etching and is considered as an effective way of achieving higher bond strength. The most plausible explanation for the increased bond strength is the increase in enamel porosity, resulting in increased resin interlocking and micro-mechanical retention.[17]

In contrast, many studies have reported the adverse effects of additional acid etching, particularly on dentin.[5,8] They explained that removal of the dentinal smear layer by additional acid application might hinder the buffering ability of the dentin matrix and result in an over-etched dentin surface with incomplete monomer infiltration into the demineralized collagen network. Incomplete penetration of resin monomers into the collagen network can reduce the quality of the hybrid layer, making it prone to nanoleakage and continuous degradation. Therefore, many authors suggest that additional acid etching might be too aggressive on the dentin and recommend that it be limited to the enamel.

Gokce *et al.*[7] revealed over etched dentin surface through which the monomers infiltrate incompletely when prior acid etching is performed in self-etch adhesives using scanning electron microscopy observations. The authors explained that over etching of the dentin surface resulted in a decreased bond strength. Van Landuyt *et al.*[5] showed inferior quality of the hybrid layer after acid etching using transmission electron microscopy images. In contrast, in the present study, the All-Bond Universal groups showed significantly higher bonding strength with additional etching. In contrast, Clearfil S3 bond showed a non-significant decrease in the values .

Both All-Bond Universal and Clearfil S3 bond are 'ultra-mild' self-etch adhesives. The pH of All-Bond Universal and Clearfil S3 bond is 3.2 and 2.7, respectively, according to the manufacturer. Van Landuyt *et al.*[18] used experimental self-etch adhesives, and their pH was approximately 2. In their studies, the authors showed that additional etching had adverse effects on the dentin bonding strength due to deeply exposed collagen network. In previously mentioned Gokce's study, the pH of the used adhesive was 1.9. The reason why the All-Bond Universal groups showed significantly higher values after acid etching was their lower acidity. The low acidity of All-Bond Universal was assumed to be insufficient to etch the dentin surface effectively into which monomer infiltration occurs. Therefore, prior acid etching does not result in an over-etched dentin surface, which has been reported by other papers that used more acidic adhesives.

On the other hand, Clearfil S3 bond has higher acidity than All-Bond Universal: it showed decreased bond strength with prior acid etching but this decrease was not statistically significant. Ikeda *et al.*[8] used three different commercialized self-etching adhesives, BeautiBond, Adper Easy Bond and G-Bond Plus, which have a pH ranging from 1.5 to 2.7. They revealed a decreasing bond strength with acid etching, but the statistical significance was different among the adhesives. One of the tested adhesives showed a significant decrease, whereas two showed no significant decrease. They concluded that additional acid etching might not be acceptable in self-etch adhesives, but the degree of the decrease in bond strength was product dependent according to their compositions. Therefore, the mechanisms by which additional etching affects bond strength in self-etch adhesives rely not only on the pH of the adhesives but also the compositions of the adhesives. Further studies on this point will be needed.

The use of a light-cured composite resin for a direct adhesive restoration has largely superseded the use of self-cured composite resin owing to the improved storage stability, the extended working time, increased degree of conversion, reduced air porosity, and enhanced physical properties.[19] On the other hand, both self-cured and dual-cured composite resins still have important applications in restorative dentistry, including core buildups, adhesive luting of indirect restorations and bonding of endodontic posts.

Many researchers have revealed the incompatibility associated with the use of self-cured composite resin with some of the one-step self-etch adhesives.[9, 20-23] This incompatibility can be explained by an interaction between the chemical redox-initiator-accelerator and adhesives.[20] The acid-base interaction between the acidic resin monomer and tertiary amine was pointed out as the main reason for the incompatibility.

In this study, significantly decreased bond strength values were observed in the Clearfil S3 bond groups bonded with a self-cured composite resin compared to the lighted-cured composite resin regardless of prior acid etching. This result agrees with those of other studies showing the incompatibility of adhesives with self-cured composite resin.[10,20,22-23] The acidic functional monomers in Clearfil S3 bond might adversely affect the polymerization of a Clearfil F II New Bond by the mechanism described previously.

On the other hand, these results with All-Bond Universal bonded with Clearfil F II New Bond did not show a significantly different strength compared to those bonded with Filtek Z-250. These similar bond strengths mean that All-Bond Universal is compatible with both self and light-cured composite resins. The decrease in the bond strength of self-cured composites to dentin was inversely proportional to the pH of adhesives by Sanares *et al.*[20] They reported a linear relationship between the pH of the adhesives and the microtensile bond strength when using chemical-cured composite resins. In the present study, the higher pH of All-Bond Universal might not bring about incompatibility with a self-cured composite resin. The manufacturer's guarantee on the compatibility with dual-cure and self-cure composite resins might be based on the high pH of its product.

In this study, the bonding performance of All-bond Universal was different from that of other one-step self-etch adhesives used in previous studies and Clearfil S3 bond. In summary, All-Bond Universal showed an increase in bond strength with additional acid etching and did not exhibit incompatibility with the self-cured composite resin due to the high pH. In clinical aspect, additional acid etching could be considered to enhance dentin bond strength when using low acidic one-step self-etch adhesives.

CONCLUSION

Within the limitation of this study, acidity of the one-step self-etch adhesives is an influencing factor in terms of the dentin bonding strength and incompatibility with self-cured composite resin. This study shows that additional acid etching is beneficial for the dentin bond strength when using All-Bond Universal, and that All-Bond Universal is compatible with self-cured composite resin.

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