Self-etch or etch-and-rinse mode did not affect the microshear bond strength of a universal adhesive to primary dentin

Benjaporn THANARATIKUL, Busayarat SANTIWONG and Choltacha HARNIRATTISAI

INTRODUCTION

Resin composites have become one of the most popular materials used to restore primary teeth because of their superior esthetics, durability, and minimal intervention required due to their adhesion to tooth structure. However, resin composites require the use of a dental adhesive to achieve a micro-mechanical bond to tooth structure.

Dental adhesives are classified into three major categories based on their clinical application mode. The first system is termed etch-and-rinse and is divided into two approaches: three-step and two-step. Three-step etch-and-rinse adhesive systems involve applying phosphoric acid to demineralize dental hard tissue, and then rinsed with water, followed by the use of a primer, which typically contains hydrophilic resin and solvent. The primer solvent is evaporated, and a bonding resin is applied and photo-polymerized. In the two-step etch-and-rinse systems, phosphoric acid is applied and washed out as in the three-step system. However, in the two-step system the primer and bonding resin are used in combination and light-cured before the resin composite is applied.

The second system is termed a self-etching primer system, in which the etchant is combined with the primer in a single application step. The solvent is allowed to evaporate, and the adhesive (bonding resin) is applied and light polymerized. In contrast, the third system contains etchant, primer, and bonding resin in a single application, and is known as an all-in-one or one-step self-etching system.

Several dental adhesives have been developed to simplify clinical procedures, reduce working time, and minimize technique sensitivity. A new type of adhesive described as a universal, multi-purpose, or multi-mode adhesive has been introduced that can be used as either a two-step etch-and-rinse or one-step self-etching system. These versatile materials give the clinician a choice of bonding strategies, depending on personal preference or different clinical situations.

The introduction of a new adhesive requires its laboratory and clinical evaluation. Laboratory determined bond strength is commonly used to assess a new dental adhesive. Perdigao et al. reported that the bond strengths of a universal adhesive, which is marketed in the USA and Europe as Scotchbond Universal Adhesive or Single Bond Universal (SBU) in other countries, to permanent dentin using different application modes were the same or higher than those of contemporary adhesives. In contrast, Munoz et al. found that the performance of an SBU adhesive used on permanent dentin in either etch-and-rinse or self-etch mode was inferior compared to a conventional etch-and-rinse adhesive (Adper Single Bond II, ASB) and a two-step self-etch adhesive (Clearfil SE Bond, CSE). Although the bond strength of SBU to permanent tooth dentin has been investigated, there is scant information about its use on primary teeth. Several studies have evaluated the differences between primary and permanent teeth, finding differences in their chemical composition, physical structure, and micromorphology, which can significantly affect the bond strengths of adhesive systems.

The aims of this study were: (1) to use a microshear test to compare the bond strength of SBU to primary tooth dentin when applied in either etch-and-rinse or self-etch mode, (2) to evaluate the fracture mode after debonding, and (3) to observe the resin-dentin interface using a scanning electron microscope (SEM).
## MATERIALS AND METHODS

### Tooth selection and preparation

The protocol for the present study was approved by the Human Ethics Committee of the Faculty of Dentistry, Chulalongkorn University. A power calculation based on the study of Soares et al.\(^{14}\) indicated that the minimum specimen size for each group was 10 (\(\alpha=0.05, \beta=0.10\)). Forty sound extracted or exfoliated human primary incisors were collected with informed consent. The teeth were disinfected in 0.1% thymol, stored in distilled water at 4°C, and used within 3 months. The crowns were decoronated 2–3 mm apical to the cementoenamel junction using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling. The labial dentin was exposed using 600-grit silicon carbide paper (Struers, Copenhagen, Denmark) under water cooling to create a standardized smear layer, and observed under a stereomicroscope at 40× magnification to determine that there was no remaining enamel and that the pulp was not exposed. The specimens were ultrasonically cleaned for 10 min to remove any debris. The specimens were stored in distilled water at 4°C until used.

### Specimen preparation

The specimens were randomly divided into four groups (\(n=10\): ASB, CSE, SBU etch-and-rinse (SBU-ER), and SBU self-etch (SBU-SE)). The adhesive systems were applied per their manufacturers’ instructions (Table 1). The adhesive was applied to the dentin surface of the specimen and a 1 mm length of polyethylene tygon tubing, 0.7 mm internal diameter (Micro-bore\(^{3}\) Tygon Medical Tubing, Saint Gobain Performance Plastics, Akron, OH, USA), was placed on the bonding area. The adhesive was cured with an LED light unit (Elipar\(^{TM}\) S10, 3M ESPE, St. Paul, MN, USA) at a light intensity of 1,200 mW/cm\(^2\). Resin composite (Filtek Z350, 3M ESPE) was packed into the polyethylene tubing, light cured for 40 s, and the specimen was stored in distilled water at 37°C for 24 h. The plastic cylinder was removed using a scalpel blade and each specimen was examined under a stereomicroscope at 25× magnification to verify that no bonding defects, air bubble inclusions, or interfacial

### Table 1  Adhesive materials, composition, and application method

<table>
<thead>
<tr>
<th>Adhesive materials (Batch number)</th>
<th>Composition</th>
<th>Self-etch technique</th>
<th>Etch-and-rinse technique</th>
</tr>
</thead>
</table>
| Adper Single Bond II (3M ESPE, St. Paul, MN, USA) (N 496548) | 1. Etchant: 35% phosphoric acid (Scotchbond Etchant)  
2. Adhesive: Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly(itaconic) acids, 10% by weight of 5 nm-diameter spherical silica particles | 1. Apply etchant for 15 s  
2. Rinse for 10 s  
3. Blot excess water  
4. Apply two consecutive coats of adhesive for 15 s with gentle agitation  
5. Gently air dry for 5 s  
6. Light cure for 10 s | N.A. |
| Clearfil SE Bond (Kuraray, Osaka, Japan) (081187) | 1. Primer: water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate  
2. Adhesive: MDP, Bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica | 1. Apply primer to tooth surface and leave in place for 20 s  
2. Dry with air stream to evaporate the volatile ingredients  
3. Apply adhesive to the tooth surface and then create a uniform film using a gentle air stream  
4. Light cure for 10 s | N.A. |
| Single Bond Universal (3M Deutschland, Neuss, Germany) (520206) | 1. Etchant: 35% phosphoric acid (Scotchbond Etchant)  
2. Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane | 1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 s. If necessary, rewet the disposable applicator during treatment  
2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent has evaporated completely  
3. Light cure for 10 s | 1. Apply etchant for 15 s  
2. Rinse for 10 s  
3. Air dry 2 s  
4. Apply adhesive as for the self-etch mode |

Bis-GMA: bisphenol A-glycidyl methacrylate, HEMA: hydroxyethyl methacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate
gaps were present. All experimental procedures were carried out at room temperature by a single operator.

**Microshear bond strength (µSBS) test**
Each specimen was attached to a universal testing machine (Shimadzu; EZ-S, Shimadzu, Kyoto, Japan) using cyanacrylate adhesive (Model Repair II Blue, Dentsply Sankin, Tokyo, Japan) and the µSBS test was performed as described by Shimada et al. A 0.2-mm diameter orthodontic wire was looped around the base of the resin composite cylinder, making contact around half of its circumference, and gently held flush against the resin-dentin interface. The wire loop and the center of the load cell were aligned as straight as possible to ensure the correct application of the shear force. A shear load was applied at a crosshead speed of 1.0 mm/min until fracture. Bond strength was calculated from the maximum load at failure and converted to megapascals (MPa).

**Failure mode evaluation**
After bond strength testing, the de-bonded specimens were observed using an SEM (JSM-5410 LV, JEOL, Tokyo, Japan) at 100× magnification to determine the mode of failure. The fracture modes were classified as adhesive failure at the resin-dentin interface, cohesive failure in dentin, cohesive failure in resin, or mixed failure.

**Resin-Dentin interface observation**
For each group, three teeth were prepared in the same manner as for the bond strength test, but without the plastic tubing. The specimens were longitudinally sectioned perpendicular to the bonded interface using a low-speed cutting machine and embedded in epoxy resin at room temperature. After 24 h, the cut surfaces were sequentially polished using 600-, 800-, 1000-, and 1200-grit abrasive paper and 0.5 μm diamond paste (DP-Paste, Struers). The specimens were etched with 10% phosphoric acid solution for 5 s, immersed in 5% sodium hypochlorite for 5 min, rinsed with distilled water, dried in an auto-desiccator cabinet for 3 days, sputter-coated with gold (SPI-Module Sputter Coater, SPI Supplies, West Chester, PA, USA) and analyzed under the SEM.

**Statistical analysis**
The data were analyzed using SPSS software (20.0, SPSS, Chicago, IL, USA). The normality of the data was determined using the Kolmogorov-Smirnov test (K-S test) and analyzed with one-way analysis of variance (ANOVA) followed by the Tukey HSD post hoc test for multiple comparisons. Statistical significance was established at the 0.05 significance level.

**RESULTS**
We compared the µSBS values between the groups (Table 2). The CSE group showed the highest mean µSBS value followed by the SBU-SE and SBU-ER groups (25.3±2.7, 25.1±2.4, and 24.3±2.7 MPa, respectively). The ASB group demonstrated the lowest mean µSBS value (19.1±3.4 MPa). The K-S test indicated that the data were normally distributed, and ANOVA demonstrated that significant differences existed between the groups (p<0.05). The Tukey test revealed that the ASB group had a significantly lower bond strength compared with the other groups (p<0.05).

Adhesive failures were the most frequently identified failure in each group (Fig. 1). The etch-and-rinse adhesive groups SEM images demonstrated thick hybrid layers with funnel-shaped resin tags, and lateral ramifications (Fig. 2). No morphological differences were found between the interfaces of the ASB and SBU-ER groups. In contrast, the SEM images of the self-etching adhesive groups showed a thin hybrid layer and cylindrical-shaped resin tags (Fig. 3). There were however, differences in the hybrid layer between the CSE and SBU-SE groups. The hybrid layer in the CSE

**Table 2** Microshear bond strength (µSBS) values of the adhesive systems to primary dentin

<table>
<thead>
<tr>
<th>Adhesive system</th>
<th>n</th>
<th>µSBS mean (SD) MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adper Single Bond II (ASB)</td>
<td>10</td>
<td>19.1 (3.4)</td>
</tr>
<tr>
<td>Clearfil SE Bond (CSE)</td>
<td>10</td>
<td>25.3 (2.7)</td>
</tr>
<tr>
<td>Single Bond Universal: Etch-and-rinse (SBU-ER)</td>
<td>10</td>
<td>24.3 (2.7)</td>
</tr>
<tr>
<td>Single Bond Universal: Self-etch (SBU-SE)</td>
<td>10</td>
<td>25.1 (2.4)</td>
</tr>
</tbody>
</table>

Values with the different superscript letter are significantly different (p<0.05)
DISCUSSION

Our study investigated the microshear bond strength (μSBS) when using a universal adhesive in either etch-and-rinse or self-etch mode compared with those of similarly used adhesives. We found that Single Bond Universal resulted in similar μSBSs in either mode that were higher or comparable to their counterpart adhesives.

In the present study, ASB was used as a representative etch-and-rinse adhesive. ASB contains polyalkenoic acid copolymer (PAAC), the so-called Vitrebond® copolymer, whereas SBU also includes 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP), which makes it acidic, rendering it self-etching. We used CSE to represent self-etching adhesives because it is considered a prototype self-etching adhesive and provides high dentin bond strength in primary teeth. The clinical success of these materials has been demonstrated by many studies. The results from our study showed that the CSE group had a higher bond strength to primary dentin than that of the ASB group, which corresponded to the results of Nakornchai et al. However, some studies reported no significant differences in bond strength between these two adhesives and primary dentin. MDP is a functional monomer contained in many adhesive products because MDP-based adhesives can chemically bind to hydroxyapatite to create MDP-Ca salts, providing higher mechanical
Our study focused on SBU, a multi-mode adhesive that can be used in either an etch-and-rinse or a self-etching mode. When SBU was used in self-etching mode, the results showed no difference in bond strength between CSE and SBU to primary dentin. The bond strength of SBU to primary dentin in this mode is difficult to compare with previous studies because those studies were performed on permanent dentin and the results were inconclusive. Perdigao et al. found that CSE demonstrated a lower bond strength to permanent dentin compared with SBU-SE, in contrast to Munoz et al. who showed the opposite result.

Perdigao et al. suggested that the higher microtensile bond strength (µTBS) between SBU and permanent dentin might be due to the presence of PAAC, rather than MDP, which plays a crucial role in bonding by chemically binding to the calcium in hydroxyapatite. Despite the lower amount of MDP in SBU, the dentin bond strength of SBU was higher than that of CSE. Alternatively, Munoz et al. proposed that the presence of PAAC in SBU could prevent monomer polymerization and compete with MDP in binding to calcium, leading to reduced chemical bond strength of MDP-Ca salts to hydroxyapatite. This reduced chemical bond strength may be why SBU in self-etching mode generated a lower µTBS compared with CSE when applied to permanent dentin.

Different dentitions were used in our study from that of Perdigao et al., which may explain the differing results. The lower amount of mineralization, i.e. calcium, in primary dentin may reduce the likelihood of PAAC chemically binding to calcium. This is likely why the bond strengths of the SBU-SE and CSE groups were not significantly different when applied to primary dentin in our study.

When used in etch-and-rinse mode, we found that the µSBS between primary dentin and SBU was significantly higher than that of ASB, which only contains PAAC. In contrast, Munoz et al. reported a higher bond strength value when ASB was applied to permanent dentin. The use of different dentitions might again be a reason for these contradictory results.

Our SEM observations of the resin-dentin interface after adhesive application indicated that pH and etching mode influence the morphology of interface. The etch-and-rinse adhesives produced a thicker hybrid layer with funnel-shaped resin tags due to phosphoric acid etching (pH=0.1), while the self-etch adhesives showed a continuous thin hybrid layer with cylindrical resin tags due to the mild pH of the SE adhesive (pH of CSE=2, pH of SBU=2.7). This finding corresponds with many previous studies showing that the thickness of the hybrid layer and the shape of the resin tags depended on the acidity of the conditioner in each mode, and that bond strength was not associated with the thickness of a hybrid layer but rather its quality.

The CSE SEM images exhibited a thin detectable hybrid layer and resin tags with a funnel-shaped upper portion because its higher acidity could demineralize the upper portion of the dentinal tubules. In contrast, the SBU images showed cylindrical resin tags with an extremely thin hybrid layer. Because of the relatively ultra-mild pH value (pH>2.5) of one-step self-etching adhesives, the dentin is poorly demineralized, resulting in the formation of an extremely thin mildly decalcified interface, called the “nanointeraction zone”.

Laboratory bond strength tests are performed to predict the clinical performance of newly developed bonding materials. Various methods are used to evaluate bond strength, including tensile, shear, microtensile, and µSBS tests. Because of the higher bonding areas of macro-bond tests, specimen defects are more prone to be present compared with micro-bond tests. The µSBS test was used in the present study because the specimens could be prepared without trimming, thus reducing the formation of structural defects such as microcracks, which may cause premature failure. Thus, the µSBS test is preferred and is practical for testing the bond strength between an adhesive and primary dentin where a small flat surface can be created. Furthermore, the shear test is a better representation of the forces clinically experienced by a restoration. In addition, the micro-bond test is often used because this test results in a more uniform stress distribution, resulting in more reliable data and higher incidence of adhesive failure between the resin and dentin interface compared with conventional macro-bond tests. The SEM images of the specimens after the µSBS test indicated that most failures occurred either partially or completely adhesive failure, suggesting their bond strength values are valid.

The new multi-mode adhesive was developed to reduce the number of clinical steps required and to decrease technique sensitivity and clinical chair time without compromising bonding effectiveness. Our study showed that the bond strength of an SBU adhesive to primary dentin was not affected by the application mode, which is in agreement with the study by Marchesi et al. and Wagner et al. who found no differences in bond strength values between either application mode of SBU and permanent dentin. This adhesive may be an alternative bonding resin for resin composite restorations in primary teeth because of its multiple application modes. Thus, clinicians can choose the proper mode depending on the prepared cavity, the child’s behavior, and operator skill. For instance, the etch-and-rinse mode is appropriate for a cavity with sufficient remaining enamel and a co-operative patient, whereas the one-step self-etching mode should always be used with uncooperative patients to reduce clinical chair time. Further investigation should be carried out to determine the long-term bonding ability of SBU and its clinical performance.

CONCLUSION

Single Bond Universal Adhesive can be used in either etch-and-rinse or self-etching mode when bonding to primary dentin. In addition, the bond strength of Single Bond Universal when applied in etch-and-rinse mode
was higher than the control etch-and-rinse adhesive. When use in self-etching mode, however, the bond strength to primary dentin was comparable to the control self-etching adhesive. In the present study, the ultra-morphology of the fracture of the resin-dentin interface depended on the pH and etching modes of the adhesives used.

ACKNOWLEDGMENT

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REFERENCES