Shear Bond Strength of Different MDP-Containing Adhesive Systems on Enamel and Dentin from Primary Teeth

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Objective: *To compare the shear bond strength (SBS) after thermocycling of four universal adhesives applied in self-etch mode on dentin and etch-and-rinse mode on enamel.* ***Study design:*** *Flat 144 buccal or lingual dentin and enamel surfaces from 72 non-carious primary molars were prepared. Samples were segregated into 12 groups (n=12): Adper Single Bond 2 etch-and-rinse (SB_T) and Clearfil SE Bond self-etch (SE_S) applied to enamel and dentin served as controls. Scotch Bond Universal Adhesive (SBU), Clearfil S3 Bond Universal Adhesive (SEU), Tetric N-Bond Universal Adhesive (TEN), and All Bond Universal (BIS) were applied in etch-and-rinse mode to enamel and in self-etch mode to dentin. They were thermocycled for 5000 cycles. SBS testing and the evaluation of fracture mode were performed.* ***Results:*** *SB_T showed statistically higher SBS than other adhesive groups using etch-and-rinse mode on enamel. SE_S and BIS had statistically higher SBS than other adhesive groups using self-etch mode on dentin. Mixed failure was the most common failure mode in each group.* ***Conclusion:*** *The universal adhesives did not show higher SBS than SB_T when using etch-and-rinse on enamel. All universal adhesives showed higher SBS than SB_T and had SBS similar to SE_S, except SBU when using self-etch mode on dentin.*

Keywords: *Universal adhesive, Shear bond strength, Selective etching, Thermocycling, Primary Teeth*

INTRODUCTION

Traditionally, dentists have used adhesive systems following either an etch-and-rinse (ER) or self-etch (SE) approach.[1, 2] ER systems include phosphoric acid to etch the tooth before rinsing and subsequent application of an adhesive.[1] Acid etching of enamel produces an altered retentive surface highly suitable for adhesion of bonding resin.[3] ER systems used in enamel bonding showed higher,[4, 5] or similar [6, 7] bond strength to primary and permanent enamel when compared to SE systems. SE adhesives comprise acidic monomers, which etch and prime the dental hard tissues simultaneously.[1] SE systems applied to dentin revealed higher bond strength to primary teeth,[4, 7-9] or showed similar bond strength to permanent dentin,[8, 10] compared with ER systems. Thus, ER on enamel and SE on dentin may be considered as an optimal method to produce high bond strengths on both enamel and dentin. ER mode only on enamel prior to application of SE adhesives is commonly referred to as selective etch technique.[11]

The Clearfil SE adhesive system (Kuraray Noritake Dental, Okayama, Japan) has been considered as the gold-standard for SE systems because of its high bonding effectiveness to dentin.[12] It includes the functional monomer 10-methacryloyldecyl dihydrogen phosphate (10-MDP), which was reported to adhere to hydroxyapatite through ionic bonding[13] with its the resulting calcium salt appearing to be hydrolytically stable.[14] Self-assembled nano-layering was found at the interface of MDP and hydroxyapatite with this adhesive system, and this was suggested to provide a direct benefit to bond durability due to its hydrophobic nature.[15]

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Recently, new adhesives have been introduced to market, so called "Universal", "Multi-purpose", or "Multi-mode" adhesives because they may be used as SE mode, ER mode, or as ER adhesives on enamel and SE adhesives on dentin.[16, 17] A universal adhesive was reported to have higher enamel bond strength in ER mode than in SE mode,[17] and significant marginal staining was found 36 months after application of a universal adhesive in full SE mode, while the retention rate for the ER group was higher.[18] Though they have some differences in composition and pH, nearly all of the universal adhesives contain 10-MDP, and thus may bond chemically to hydroxyapatite through the same nano-layering that has been shown for Clearfil SE adhesive.[15, 17] Therefore, there may be differences in bond strength between the products, and they should be evaluated in comparison to the gold standards.

Thus, this study compared the shear bond strength (SBS) after thermocycling of four different universal adhesives applied to primary enamel in ER mode and primary dentin in SE mode. The two-step ER adhesive, Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA), and two-step SE adhesive, Clearfil SE Bond (Kuraray Noritake Dental, Okayama, Japan) were tested as control groups. The null hypotheses were as follows: (1) universal adhesives applied to primary enamel in ER mode show the same shear bond strength when compared to their respective control groups; (2) universal adhesives applied to primary dentin in SE mode show the same shear bond strength when compared to their respective control groups; and (3) there are no differences in the shear bond strength for primary enamel and dentin between different universal adhesives.

MATERIALS AND METHOD

The four universal adhesives examined were Scotch Bond Universal (SBU; 3M ESPE, St. Paul, MN, USA), Clearfil S³ Bond Universal (SEU; Kuraray Noritake Dental, Okayama, Japan), Tetric N-Bond Universal (TEN; Ivoclar Vivadent, Schaan, Liechtenstein), and All Bond Universal (BIS; Bisco, Schaumburg, IL, USA). Adper Single Bond 2 (SB_T; 3M ESPE, St. Paul, MN, USA) and Clearfil SE Bond (SE_S; Kuraray Noritake Dental, Okayama, Japan) were selected as ER and SE control groups, respectively. The compositions of these adhesives shown in Table 1 demonstrate that all of the study group adhesives contained Bis-GMA, HEMA, MDP, ethanol, and water, and that only the additional methacrylate monomers, the Vitrebond copolymer in Scotch Bond Universal, and possibly the photoinitiator systems were different.

Selection of tooth materials

Seventy-two noncarious human primary molars were stored in 0.1% thymol solution at 4℃ and were used within 6 months following extraction. The institutional review board of Seoul National University School of Dentistry reviewed and approved the protocol used in this study including informed consents (IRB No. S-D20170044).

Preparation of tooth specimens

The teeth were carefully cleaned with pumice and rinsed with water spray. Each root was sectioned with a low speed diamond saw (Isomet; Bueher-Met Lake Bluff, IL, USA) under running water 2 mm below the cementoenamel junction when present. The teeth were then cut in a perpendicular direction to the occlusal surface, providing two halves of the crown of the tooth, one of the lingual and one of the buccal surface (n=144). The lingual or buccal surface of each crown was positioned upwards and embedded in self-curing epoxy resin in the center of a pre-fabricated acrylic resin ring (2.5 cm in diameter, 1.0 cm in height, Taejin Acryl, Seoul, Korea). The buccal or lingual surface on the specimen was cut using Isomet under running water to flatten the overlying the hard tissue and to expose the flat dentin or enamel surface enough to bond according to the protocol. The surfaces for bonding on the buccal or lingual surfaces of the teeth were ground with water-cooled 600-grit silicon carbide paper on a polishing machine to produce a standardized smear layer. The specimens were examined using a stereomicroscope (Olympus SZX7, Tokyo, Japan) before and after cutting to ensure that the enamel or dentin surface was well exposed and they were free of any defects.

Allocation of the specimens

The teeth were randomly divided into two control groups and four study groups to measure SBS to enamel and dentin, providing 12 groups in total (n=12 in each group).

Adhesive and composite applications

The adhesive systems were applied using sterile microbrushes (Dentsply Caulk, Milford, DE, USA) on enamel and dentin as shown in Table 2. All adhesives were light polymerized for 20s using a LED curing unit (Valo; Ultradent, South Jordan, USA) emitting light at 1000 mW/cm^2 within 1 mm at the tip.

The specimens were then placed on a box-form base (3.0 cm in width, 3.0 cm in length, and 1.0 cm in height), which was prepared with polyvinyl siloxane impression material (Exafine; GC, Tokyo, Japan, Lot No. 1904241 and 1904252), in the Ultradent bonding jig (Ultradent, South Jordan, UT, USA). A polyethylene mold (Ultradent Bonding Assembly; Ultradent, South Jordan, UT, USA) was placed on the surface of the specimen which was indented in the shape of a circular hole (2.38 mm in internal diameter, 3 mm in height), and they were clenched in the jig. A blue colored resin composite (Light-Core; Bisco, Schaumburg, IL, USA, Lot No. 1800004627), containing Bis-GMA, Bis-EMA, and glass particles (>60%), was applied in a single increment into the mold with a Teflon coated instrument, producing cylinders of composites standing perpendicular the bonding surface and measuring 4.45 mm^2. The composite was polymerized for 20s using a Valo LED curing unit.

The specimens were aged for 24 hrs in distilled water at 37℃, and then thermocycled with a thermocycling machine (Taewon Tech, Bucheon, Korea) for 5000 cycles between 5℃ and 55℃ with a dwell time of 30s.

Shear bond strength test

SBS was measured using a shear bond tester (Bisco, Schaumburg, IL, USA), which is adopting the Ultradent notched rod, set at a crosshead speed of 0.5 mm/min. The force was applied to the adhesion surface of specimen, and the maximum fracture strength was measured in N, which were converted into MPa by dividing by the bonding area (4.45 mm^2). There were no pre-test failures in this study.

The fractured specimen interface was evaluated by a single examiner using an Olympus SZX7 stereomicroscope at x40 magnification to assess the failure mode, which was classified as cohesive (in resin, or in tooth material), adhesive (at the interface between resin and tooth material), or mixed (combination of cohesive and adhesive failure).

Statistical analysis

Statistical comparisons of SBS to enamel and dentin were made among each group for all six materials. The normality of the studied parameters was verified using the Kolmogorov-Smirnov test, and statistical analyses were performed based on parametric tests because all were normally distributed. The values of the studied parameters in each material group for either enamel or dentin were compared by ANOVA, and the homogeneity of variance was tested with Levene's test. According to the results of Levene's test, post hoc comparisons were performed using either Tukey's HSD or Dunnett's T3. Also, the independent t-test was performed to compare the shear bond strengths for each material to enamel and dentin. All statistical analyses were performed with an alpha significance level of 0.05 using SPSS 23 (IBM SPSS Statistics, New York, USA).

RESULTS

Adper Single Bond 2 showed statistically higher SBS than other adhesive group using the etch-and-rinse mode on enamel (Figure 1). Clearfil SE Bond and All Bond Universal had statistically higher SBS than the other adhesive groups using self-etch mode on dentin. There were significant differences in SBS of Adper Single Bond 2, Clearfil SE Bond, and All Bond Universal between bonding to enamel and dentin.

The analysis of the fracture modes revealed a predominance of the mixed failure mode (87.9%) as shown in Figure 2. The adhesive and cohesive failures were observed in 9.8% and 2.3% of the specimens, respectively.

Figure 1. Mean shear bond strength to enamel and dentin of human primary teeth. SB_T: Adper Single Bond 2 Etch-and-rinse; SE_S: Clearfil SE Bond Self-etch; SBU: Scotch Bond Universal Adhesive Etch-and-rinse on enamel and Self-etch on dentin; SEU: Clearfil S³ Bond Universal Adhesive Etch-and-rinse on enamel and Self-etch on dentin; TEN: Tetric N-Bond Universal Adhesive Etch-and-rinse on enamel and Self-etch on dentin; BIS: All Bond Universal Etch-and-rinse on enamel and Self-etch on dentin. Bars with the same letter did not differ significantly. *: Statistical differences in shear bond strength between enamel and dentin ($P < 0.05$).

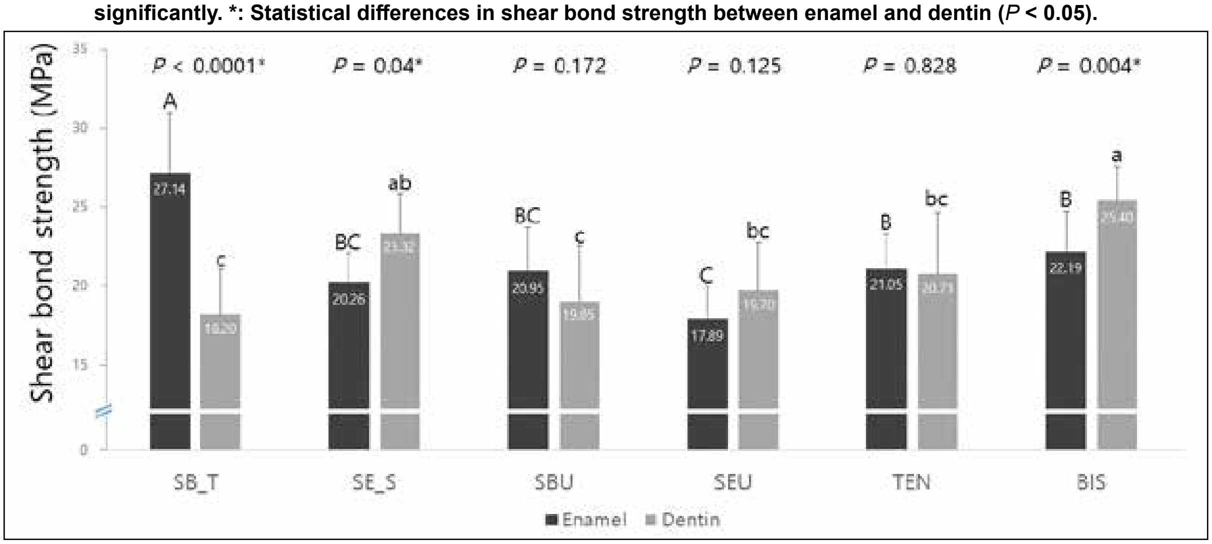

Figure 2. Failure mode distribution (%). SB_T: Adper Single Bond 2 Etch-and-rinse; SE_S: Clearfil SE Bond Self-etch; SBU_T: Scotch Bond Universal Adhesive Etch-and-rinse; SBU_S: Scotch Bond Universal Adhesive Self-etch; SEU_T: Clearfil S³ Bond Universal Adhesive Etch-and-rinse; SEU_S: Clearfil S³ Bond Universal Adhesive Self-etch; TEN_T: Tetric N-Bond Universal Adhesive Etch-and-rinse; TEN_S: Tetric N-Bond Universal Adhesive Self-etch; BIS_T: All Bond Universal Etch-and-rinse; BIS_S: All Bond Universal Self-etch. E: Bonding on enamel; D: Bonding on dentin.

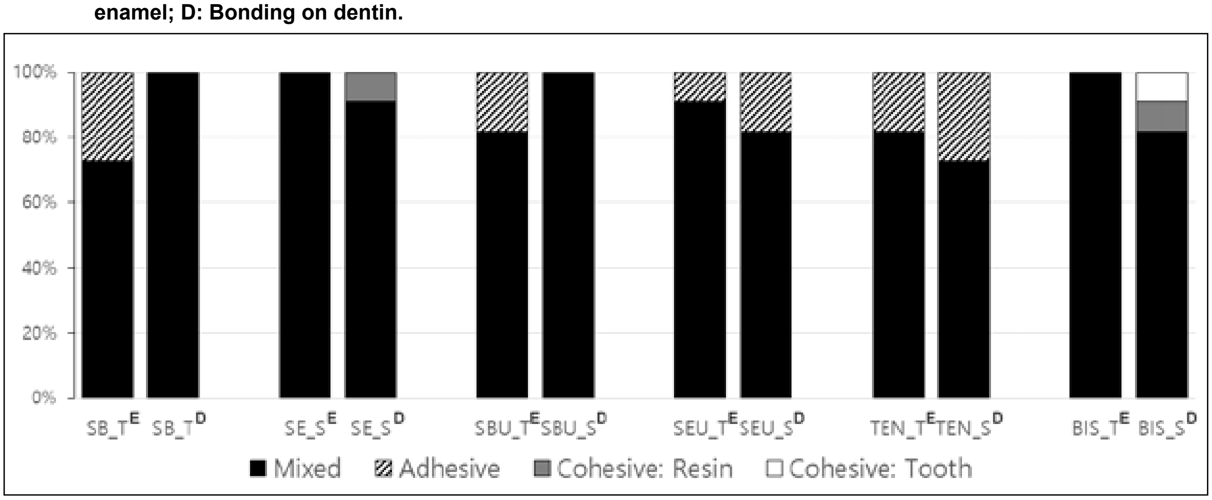

Table 1: Adhesive systems in this study. Abbreviations: Bis-GMA: bisphenol-glycidyl mehacrylate; HEMA: 2-hydroxylethyl methacrylate; MDP: 10-methacryloyloxy methacrylate

Group	Materials (Batch number)	Main components	Manufacturer
Control Groups	**Group SB_T:** Adper Single Bond 2 (N955153) [pH 3.6]	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer (Vitrebond™ copolymer), water, ethanol	3M ESPE, St. Paul, MN, USA
Study Groups	**Group SE_S:** Clearfil SE Bond (Primer: B90273 [pH 2.0], Bond: B70437)	Primer: MDP, HEMA, hydrophilic methacrylate, tertiary amine, water Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, tertiary amine, silanated colloidal silica, initiators	Kuraray Noritake Dental, Okayama, Japan
	Group SBU: Scotch Bond Universal (80410C) [pH 2.7]	MDP, dimethacrylate resins, HEMA, Bis-GMA, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane	3M ESPE, Neuss, Germany
	Group SEU: Clearfil S^3 Bond Universal (5X0037) [pH 2.3]	MDP, dimethacrylate resins, HEMA, Bis-GMA, ethanol, water, initiators, fillers, silane	Kuraray Noritake Dental, Okayama, Japan
	Group TEN: Tetric N-Bond Universal (X33272) [pH 2.5]	MDP, decandioldimethacrylate, HEMA, Bis-GMA ethanol, water, silicon dioxide, initiators, stabilizers	Ivoclar Vivadent, Schaan, Liechtenstein
	Group BIS: All Bond Universal (1800005212) [pH 3.2]	MDP, HEMA, Bis-GMA, ethanol, water, initiators	Bisco, Schaumburg, IL, USA

Table 2: Procedures applied in this study. Abbreviations: SB_T: Adper Single Bond 2; SE_S: Clearfil SE Bond; SBU: Scotch Bond Universal; SEU: Clearfil S^3 Bond Universal; TEN: Tetric N-Bond Universal; BIS: All Bond Universal.

Group	Materials	Enamel	Dentin
Control Groups	SB_T	**Etch + Rinse** Acid-etch with 35% phosphoric acid (15s) and rinse (10s); Gently air-dry (2s); Apply adhesive; Gently air-dry (5s); Light-cure (20s); Place restoration	**Etch + Rinse**
	SE_S	**Self-etch (2 steps)** Apply self-etching primer with agitation (20s); Gently air-dry (3s); Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration	**Self-etch (2 steps)**
Study Groups	SBU	**Etch + Rinse** Acid-etch with 35% phosphoric acid (15s) and rinse (10s); Gently air-dry (2s); Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration	**Self-etch (1 step)** Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration
	SEU	**Etch + Rinse** Acid-etch with 35% phosphoric acid (15s) and rinse (10s); Gently air-dry (2s); Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration	**Self-etch (1 step)** Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration

Group	Materials	Enamel	Dentin
Study Groups	TEN	**Etch + Rinse** Acid-etch with 35% phosphoric acid (15s) and rinse (10s); Gently air-dry (2s); Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration	**Self-etch (1 step)** Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration
	BIS	**Etch + Rinse** Acid-etch with 35% phosphoric acid (15s) and rinse (10s); Gently air-dry (2s); Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration	**Self-etch (1 step)** Apply self-etching adhesive with agitation (20s); Gently air-dry (5s); Light-cure (20s); Place restoration

DISCUSSION

In enamel bonding, Adper Single Bond 2 in ER mode showed the highest SBS, so the first null hypothesis was rejected. It was previously reported that Single Bond in ER mode showed higher microtensile bond strength than Clearfil SE Bond in SE mode, both on unprepared and prepared enamel.[19] The authors explained that phosphoric acid conditioning of enamel created microporosities and irregularity on enamel surface, providing reliable enamel bond strengths. However, another study reported that there was no difference in micro SBS of Single Bond in ER mode and Clearfil SE Bond in SE mode both on permanent and primary enamel,[6] which was not consistent with our results. A recent study using universal adhesives reported that Adper Single Bond 2 and Scotch Bond Universal in ER mode had higher microshear bond strength to enamel than Clearfil SE Bond both in SE and ER modes, and there was no significant difference between Adper Single Bond 2 and Scotch Bond Universal.[20] In our study, Scotch Bond Universal showed lower SBS than Adper Single Bond 2 on enamel with ER technique. Another study revealed that Clearfil SE Bond in SE mode showed higher SBS to enamel than Scotch Bond Universal and All Bond Universal in ER mode at both 24 hrs and 6 months.[21] In the present study, there were no significant differences between those adhesives in enamel bonding. Our study revealed that one-step self-etching primer Clearfil S^3 Bond Universal showed the lowest SBS on enamel, even with ER mode. Suda et al. compared SBS to enamel bonding for universal adhesives including Clearfil S^3 Bond Universal and two-step self-etch adhesives including Clearfil SE Bond.[22] In their study, Clearfil S^3 Bond Universal in ER mode showed lower SBS than Clearfil SE Bond in SE mode, which is consistent with our study. Universal adhesives include a hydrophobic and hydrophilic monomer mixture, and the residual water in the mixture may degrade the mechanical properties of the adhesive layer.[23] The authors explained that the weaker mechanical properties of universal adhesives may lead to lower bond fatigue durability compared to two-step self-etching adhesive Clearfil SE Bond, which has a relatively thicker adhesive layer.[22]

The second null hypothesis was also rejected because All Bond Universal and Clearfil SE Bond in SE mode had the highest SBS to dentin, while Adper Single Bond 2 in ER mode and Scotch Bond Universal, Clearfil S^3 Bond Universal, and Tetric N-Bond Universal in SE mode showed relatively lower SBS to dentin. A previous study reported that Scotch Bond Universal and All Bond Universal in SE mode showed higher microtensile bond strength on dentin than Clearfil S^3 Bond Universal in SE mode after 10000 thermocycles.[23] Another study of dentin bonding revealed that Clearfil SE Bond and Scotch Bond Universal in SE mode had higher microtensile bond strength than All Bond Universal in SE mode after 6 months of water storage.[24] Jang et al. studied the microtensile bond strength of universal adhesives to dentin, and Clearfil SE Bond and All Bond Universal in SE mode showed higher shear bond strength than Scotch Bond Universal in SE mode, which is consistent with our study.[25] Scotch Bond Universal contains another functional monomer, polyalkenoic acid copolymer (Vitrebond™ copolymer) and monofunctional resin monomer co-solvent, hydroxyethyl methacrylate (HEMA).[23] It was reported that both the polyalkenoic acid copolymer and HEMA repeatedly compete with 10-MDP for calcium coordination sites on the surface of apatite crystallites, possibly resulting in reduced nano-layering of 10-MDP-calcium salts within the resin-dentin interface.[26] All Bond Universal includes minimum amount of ethanol and water as its solvent, and 10-MDP as its functional monomer, resulting in very hydrophobic bond.[25] It may enhance its bonding performance by hindering water-permeable adhesive layer of compromising bond performance.[25]

As a result of our study, the third null hypothesis was rejected. Four different universal adhesives showed different SBS to enamel and dentin, possibly due to differences in their composition (Table 1). Scotch Bond Universal and Clearfil S^3 Bond Universal incorporated a silane in the formula, expecting to function as porcelain primer as well. However, it was reported that the silane in the presence of MDP (acidic monomer) and Bis-GMA was not stable for long term and not effective as porcelain primer.[27] In our study, All Bond Universal showed a difference between dentin bonding and enamel bonding. Interestingly, All Bond Universal in SE mode on dentin showed higher SBS than in ER mode on enamel, contrary to other universal adhesives.

The predominant failure modes for enamel and dentin were mixed type fractures, followed by adhesive fractures, for most all of the adhesives studied (Figure 2). Several studies using Adper Single Bond 2 or Clearfil SE Bond or universal adhesives showed similar fracture modes,[21, 23, 28, 29] while others reported that adhesive fractures were predominant.[4, 6, 24, 30] A study on the effects of thermocycling on the microtensile bond strength of Clearfil SE bond to dentin reported that more adhesive failures were observed and related to bond strength reductions occurring 5000 and 10000 thermocycles.[31]

Nevertheless, it has been reported that there is no correlation between the tooth-resin fracture mode and the bond strength in any of adhesive systems.[32, 33] Also, it was reported that there were no differences found in failure modes between the adhesive systems or the primary/permanent dentition.[6]

The shear bond strength test is the most commonly used bond strength test, mainly because of its easy of specimen preparation and simple test protocol.[34] However, both shear and tensile forces are induced during this test and the stresses are mostly concentrated at the point of loading, thus causing a premature failure in the dentinal substrate at loads far less than the tensile strength of dentin.[34, 35] Therefore, cohesive failures in the substrate were frequently observed with new adhesives showing improved bond strengths.[35] The microtensile bond strength test was introduced, and it was reported that the test would be a possible solution to evaluate the adhesion under clinically relevant conditions because of its ability to analyze bond strength values of up to 70 MPa with small percentage of cohesive failures.[36] However, the microtensile test has the difficulty in fabricating specimens with consistent geometry, and has the possibilities of easily damaged specimens and loss or fracture of post-fracture specimens.[36] In our study, the cohesive failures were observed in 2.3% of the specimens, and most of the failure modes were mixed or adhesive irrespective of the experimental groups, although the shear bond strength test we performed has limitations in terms of clinical accuracy. A further study using the microtensile bond strength test might be of value to assess the reliability of our results.

Thermocycling is thought to be useful for inducing artificial ageing processes by accelerated chemical degradation,[37] and expansion/contraction stresses caused by the coefficient of thermal expansion mismatch between the restorative materials and the tooth substrates.[38] A study reported that SBS of two step ER and SE adhesives to enamel and dentin was not influenced by 500 thermocycles.[39] We performed 5000 thermocycles, which is considered to correspond to 6 months of in vivo functioning.[37] However, it may not be enough for simulating long-term bonding efficacy, and some have suggested that the current thermocycling method might not be an accurate predictor of in vivo performance.[40]

The limitations of this study include that we performed the failure mode evaluation using a stereomicroscope, but the evaluation under SEM with high magnification may provide the better decision.[41] Although there was a difference in the application technique suggested by universal adhesive manufacturers, we tried to standardize the application methods to compare objectively, which may affect the results of this study. Also, comparison of SBS before and after thermocycling may be effective in explaining the influence of the thermal cycling on shear bond strength, which is one of the limitations of our study. The other limitations of this study are that we did not perform the shear bond strength test for the self-etch mode of the universal adhesives to enamel, and the number of sample for each group was small. It would be recommended if a combination of various bond strength tests could be investigated to assess the bonding effectiveness of new adhesive materials in the further studies.

CONCLUSION

Within the limitations of the present study, it was concluded that universal adhesives revealed lower SBS on primary enamel when using selective etch technique compared with Adper Single Bond 2 in ER mode. However, universal adhesives showed similar or higher SBS on primary dentin using selective etch technique compared to Adper Single Bond 2 in ER mode. Also, only one of the universal adhesives (All Bond Universal) showed a difference in SBS between enamel and dentin, with dentin being higher.

CONFLICT OF INTEREST

The authors do not have any financial interests in the companies whose materials are included in this article.

ACKNOWLEDGMENT

This work was supported by Research Resettlement Fund for the new faculty of Seoul National University.

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