

# Microshear bond strength of two types of universal adhesives to primary dentin using self-etch and total-etch techniques

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## Abstract


**Background and Aim:** This study aimed to compare the microshear bond strength ( $\mu$ SBS) of two types of universal adhesives to primary dentin following self-etch (SE) and total-etch (TE) techniques.

**Materials and Methods:** In this in vitro experimental study, 50 extracted sound primary first and second molars were randomly assigned into five groups ( $n=10$ ). The groups were treated as follows: All Bond Universal (ABU) using the self-etch (SE) technique, Scotchbond Universal (SBU) using the SE technique, ABU using the total-etch (TE) technique, SBU using the TE technique, and Adper Single Bond 2 (ASB2) as the control group, after exposing the occlusal dentin. Composite cylinders were bonded to dentin and underwent  $\mu$ SBS test. Data were analyzed by two-way ANOVA and Tukey's test ( $\alpha=0.05$ ).

**Results:** All experimental groups showed significantly lower  $\mu$ SBS than the control group ( $P<0.05$ ). However, the difference in  $\mu$ SBS was not significant among the experimental groups ( $P>0.05$ ).

**Conclusion:** The results showed higher  $\mu$ SBS of ASB2 (fifth-generation adhesive) than both universal adhesives (SBU and ABU) in SE and TE techniques. The  $\mu$ SBS to primary dentin depended on both adhesive type and technique.

**Key Words:** Shear Strength; Dental Cements; Dentin; Tooth, Deciduous

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Received: 7 Jan 2023

Accepted: 20 April 2023

➤ **Cite this article as:** Rezvani Y, Ghasemi A, Bagheri Hariri A, Eftekhari L. F. Microshear bond strength of two types of universal adhesives to primary dentin using self-etch and total-etch techniques. J Iran Dent Assoc. 2023; 35(1-2):1-6.

## Introduction

Preservation of primary teeth in the dental arch is critical for managing the eruption of subsequent permanent teeth. Selection of appropriate restorative materials for primary teeth is also important, and clinical success of composite restorations mainly depends on adhesive systems that provide a stable bond to

composite and enamel and dentin substrates [1,2].

Dental adhesives are primarily used to provide retention for composite restorations or composite cements. Universal adhesives are relatively novel materials that were introduced aiming to simplify the clinical procedures [1]. Several modifications have been made in their

chemical formulation compared with previous adhesive generations to improve their properties. Following the advent of dentin bonding agents, greater attention was directed to differences between primary and permanent dentin, and the effect of such differences on the bond strength to resin materials. The diameter of dentinal tubules and peri-tubular dentin thickness in primary teeth are double the corresponding values in permanent teeth [2]. Thus, the amount of solid dentin used for bonding in primary teeth is significantly lower than that in permanent teeth. Such structural differences can be responsible for tooth hypersensitivity, susceptibility to trauma, and caries progression [2]. The bonding mechanism of adhesive systems is mainly based on replacement of the lost minerals with resin monomers, such that the polymer is micromechanically retained within the dental substrate [3].

At present, the adhesive systems available in the market can be divided into two groups of self-etch (SE) and total-etch (TE) [2]. In application of TE (etch and rinse) systems, the first step is to apply 15%-17% phosphoric acid gel on the enamel and dentin to eliminate the smear layer and expose the collagen fibrils, which increases the surface area and surface energy of enamel. The second step is to apply adhesive primer, which may be supplied in a separate bottle, or mixed with adhesive [2,4]. The main problem of TE systems is the possibility of degradation of collagen fibers in the process of drying of demineralized dentin, which can decrease the bond strength [5]. To prevent this problem, dentin should remain moist, which is not clinically easy [2]. At present, SE adhesives have gained increasing popularity due to difficult and multiple application steps of TE adhesives. One reason for success of SE adhesives is the chemical bonding of functional monomers to hydroxyapatite [2]. On the other hand, micromechanical bonding has been suggested to enhance the bond strength of SE adhesives [6]. It has been reported that phosphoric acid etching creates a thicker hybrid layer [7] and longer resin tags [6]. Smear layer removal after

etching increases the penetration depth of adhesives [7]. However, a significant correlation between increased bond strength and increased interface area has not been clearly documented [8]. SE adhesives were introduced for elimination of acid etching step, which has high technical sensitivity. Their acidic monomers etch the dental substrate [7]. Among such adhesives, all-in-one adhesives have combined all the procedural steps in one single step. Although they have improved bonding ability to dentin, their adhesion to enamel is still not satisfactory [7]. Thus, separate etching of enamel prior to the application of SE adhesives especially adhesives with a mild pH is recommended [7]. Nonetheless, accidental pre-etching of dentin is a risk factor that can decrease the bond strength [9].

Recently, universal adhesives were introduced to the market to decrease the complexity and number of procedural steps required for bonding, which can be used in both SE and TE modes. Universal adhesives are also known as multi-purpose or multi-mode adhesives because of their application method. Considering the gap of information in this regard, this study aimed to assess the microshear bond strength ( $\mu$ SBS) of two types of universal adhesives to primary dentin by the SE and TE techniques. The null hypothesis was that the  $\mu$ SBS of the two types of universal adhesives to primary dentin would be the same in SE and TE techniques.

## Materials and Methods

This in vitro, experimental study was conducted on 50 extracted sound primary first (D) and second (E) molars. The study protocol was approved by the ethics committee of Shahid Beheshti University of Medical Sciences. (Ethical code: 13940205/0310/144)

### Sample size:

The minimum sample size was calculated to be 7 in each group according to a study by Muñoz et al, [10] assuming the mean standard deviation of  $\mu$ SBS to be 6 MPa, effect size of 0.7, study power of 80%, and type 1 error of 0.05 using PASS 11 software. To increase the accuracy, 10 samples were used in each group (a total of 50 Ds and Es).

**Eligibility criteria:**

Deciduous first and second molars, which had no caries or erosion and were extracted for reasons unrelated to this study, were collected. The teeth were standardized regarding the mean duration of storage until the experiment, mean age of children, and the residual root length after physiological resorption. The teeth belonged to children between 9 to 11 years. Each group included 3 teeth of 9-year-olds, 3 teeth of 10-year-olds, and 4 teeth of 11-year-olds.

**Specimen preparation:**

The teeth were cleaned from soft tissues and bone residues, and were immersed in 0.5% chloramine T solution for 24 hours for disinfection. They were then stored in saline at 24°C. Two sections were then made in each tooth. The first section was made in the occlusal surface such that dentin closest to the dentinoenamel junction was exposed. The second section was made parallel to the cementoenamel junction and perpendicular to the longitudinal axis of the tooth such that specimens with 2 mm thickness were obtained (Figure 1). The surface of specimens was polished with 600- and 800-grit silicon carbide abrasive papers under water coolant to obtain a smooth surface and expose dentin. The residues were rinsed with water, and the specimens were stored in distilled water at 24°C.



**Figure 1.** Prepared dental substrate

The teeth were then randomly assigned to 5 groups (n=10). Table 1 presents the bonding agents used in this study and their composition. Group 1. All Bond Universal (ABU) in SE mode: The specimens were rinsed and dried such that

they did not have additional moisture. Over-drying was also prevented. Next, two coats of ABU (Bisco) was applied at the center of prepared specimen by a microbrush as instructed by the manufacturer. Afterwards, it was air-sprayed for 10 seconds in order for the excess solvent to evaporate. Light curing was performed conventionally by a halogen light curing unit (Optilux 501, Kerr, USA) for 10 seconds. The tip of the curing unit had minimum distance from the specimen surface.

Group 2. Scotchbond Universal (SBU) in SE mode: The process was the same as that in group 1 except that SBU (3M ESPE) was used in SE mode.

Group 3. ABU in TE mode: After rinsing and drying of the surface, 37% phosphoric acid was used for 15 seconds as instructed by the manufacturer. The surface was rinsed and excess water was removed such that the dentin surface remained slightly moist. The rest of the procedure was similar to group 1.

Group 4: SBU in TE mode: The procedure was the same as that in group 1 except that SBU was used.

Group 5 (control): Adper Single Bond 2 (ASB2) in TE mode: The procedure was the same as that in group 1 except that ASB2 (3M, USA) which is a fifth-generation bonding agent was used in TE mode as instructed by the manufacturer.

After bonding in all groups, silicone Tygon tubes with 0.7 mm diameter and 1 cm height (Tygon Norton Performance Plastic Co., Cleveland, OH, USA) were placed on the prepared resin and filled with composite resin (Valux Plus; 3M ESPE, St. Paul, MN, USA) with 66v% filler content. Composite resin was packed in the tubes and cured conventionally with a halogen light curing unit (Optilux, Kerr Co., USA) with a light intensity of 450 mW/cm<sup>2</sup> for 40 seconds. The specimens were then incubated at 100% humidity and 37°C temperature for 24 hours. The Tygon tubes were removed after 24 hours.

**Measuring the  $\mu$ SBS:**

A microtensile testing machine (Tensile Tester Bisco Schaumburg, IL, USA) was used for this purpose. The specimens were glued to the jig by cyanoacrylate glue. A stainless-steel wire with

**Table 1.** Bonding agents used in this study and their composition

Bonding agent	Composition	Manufacturer
Scotchbond Universal	10-MDP, Dimethacrylate resins, silane, initiator, filler, polyacrylic acid, copolymer, HEMA, ethanol, water	3M ESPE, St. Paul, MN, USA
All Bond Universal	10-MDP, Dimethacrylate resins, HEMA, Ethanol, water, initiator	Bisco Inc., IL, Schaumburg, USA
Adper Single bond 2	Dimethacrylate resins, initiator, HEMA, copolymer, filler, ethanol, water	3M ESPE, St. Paul, MN, USA
Composite resin (Valux Plus)	BIS-GMA, TEGDMA filler: 66% silica or zirconia(volume)	3M ESPE, St. Paul, MN, USA
AcidoGel 37%	Phosphoric Acid 37%	Denta Flux Korea

0.2 mm diameter was ligated around the composite cylinders from one end and connected to the load cell jig from the other end. The wire was positioned parallel to the horizon such that it was in contact with the tooth-composite interface. Microshear load was applied at a crosshead speed of 0.5 mm/minute until debonding. The load at debonding was recorded in Newtons, and divided by the composite cylinder cross-sectional area to calculate the  $\mu$ SBS in megapascals (MPa).

Statistical analysis:

Data were analyzed by SPSS version 17.

Considering the normal distribution of data as confirmed by the Kolmogorov-Smirnov test ( $P>0.05$ ) and homogeneity of variances as shown by the Levene's test ( $P=0.023$ ), two-way ANOVA, one-way ANOVA, and Tukey's test were applied for the comparisons at 0.05 level of significance.

## Results

Table 2 presents the  $\mu$ SBS values of the groups. One-way ANOVA showed a significant difference in  $\mu$ SBS of the groups ( $P=0.023$ ). Pairwise comparisons were performed by the Tukey's test. The results showed a significant difference in the mean  $\mu$ SBS of all four experimental groups with the control group ( $P<0.0001$ ) such that all universal adhesives showed a significantly lower  $\mu$ SBS than the control group. However, the difference between the experimental groups was not significant ( $P>0.05$ ).

Comparison of the four universal adhesive groups by two-way ANOVA showed no significant effect of adhesive type ( $P>0.05$ ), etching ( $P>0.05$ ), and their interaction ( $P>0.05$ ) on  $\mu$ SBS values.

## Discussion

This study assessed the  $\mu$ SBS of two types of universal adhesives to primary dentin by the SE and TE techniques. The null hypothesis was that the  $\mu$ SBS of the two types of universal adhesives to primary dentin would be the same in SE and TE modes. The results showed that the  $\mu$ SBS of ASB2 was significantly higher than that of ABU and SBU to primary dentin. Also, the mode of adhesive application (SE/TE) had no significant effect on  $\mu$ SBS of universal adhesives, and the  $\mu$ SBS of universal adhesives to primary dentin was not significantly different. Thus, the null hypothesis of the study was accepted. Evaluation of ABU and SBU in the present study was due to their widespread use in the clinical setting. Moreover, ASB2 is commonly used as the control group in investigations, and is also extensively used in the clinical setting [7,11]. Furthermore, evaluation of  $\mu$ SBS in the present study was due to the fact that macro tests have higher error rate and higher risk of cohesive fracture of specimens. Thus, macro tests do not allow measurement of high values of bond strength because cohesive failure occurs sooner than adhesive failure [1]. Moreover, several specimens are obtained from one tooth in  $\mu$ SBS test, which is another advantage.

**Table 2.**  $\mu$ SBS values (MPs) in the groups (n=10)

	Mean	Std. deviation	Maximum	Minimum
All Bond Universal self-etch	16.34	6.46	24.63	9.11
Scotchbond Universal self-etch	18.81	7.45	34.14	7.43
All Bond Universal Total-etch	22.32	5.56	32.80	15.84
Scotch bond universal Total etch	20.87	6.37	32.85	8.75
Adper Single bond 2 Total etch	43.18	9.67	53.61	24.14

The current study showed no significant difference in  $\mu$ SBS of SBU and ABU in SE and TE modes to primary dentin, which was in line with the results of Gre et al, [12] Chen et al, [13] Wagner et al, [14] and Muñoz et al, [10] but different from the findings of Marchesi et al, [15] Muñoz et al, [16] Kumari et al, [17] and Tekçe et al [18]. The methodology of Munoz et al. [16] was different from that of the present study. For instance, they ground dentin with up to 2500-grit abrasive papers. Thus, difference in the results may be attributed to different types of smear created. In some studies [8,11,18], the specimens were built-up to undergo microtensile and tensile bond strength tests while in the present study, composite resin was bonded to the teeth in Tygon tubes. Two studies [7,13] showed that although the acid etch technique revealed a significant difference in the long-term, no significant difference existed in bond strength between the SE and TE modes in immediate testing, which was in line with the present findings.

In the present study, both universal adhesives showed a significantly lower  $\mu$ SBS to dentin than the fifth-generation ASB2 bonding agent. The results of some studies [11,17] were in agreement with the present findings while some others [7,8,17,19] reported contrary results. Difference in the results can be due to changed chemical formulation of universal adhesives such that universal adhesives have higher concentrations of 10-MDP and bis-GMA which cause their greater hydrophobicity and decrease their adequate penetration into hydrophilic dentin. Furthermore, poly alkenoic acid copolymer which is a constituent of 3M adhesives is capable of water storage for hydrolysis and greater penetration depth of

bonding agent; thus, adhesives containing poly alkenoic acid have considerably lower technical sensitivity [19].

The present results showed no significant difference in  $\mu$ SBS between SBU and ABU, indicating that type of universal adhesive had no significant effect on  $\mu$ SBS to primary dentin. This finding was in line with some [8,15,17] and in contrast to the findings of some other studies [7,11]. Such variations in the results can be due to evaluation of different tooth types (primary versus permanent teeth) since the differences in  $\mu$ SBS values to primary teeth are often smaller than those to permanent dentin [2].

In studies conducted on dentin, depth of preparation and site of bonding to tooth surface are among the factors that can affect the  $\mu$ SBS [17]. In the present study, occlusal dentin at the dentinoenamel junction (superficial dentin) was used. Muñoz et al, [10] and Wagner et al. [14] used occlusal superficial dentin, Marchesi et al, [15] and Gre et al. [12] used deep dentin, and Kumari et al. [17] evaluated both superficial and deep dentin. Differences between superficial and deep dentin can also explain the variations in the results reported in the literature.

The main limitations of this study were inability to standardize the teeth in terms of time passed since their extraction, and in vitro design of the study, which limits the generalization of results to the clinical setting. Future studies are required to assess the  $\mu$ SBS of specimens over longer periods of time. Also, thermocycling should be performed in future studies to better simulate the clinical setting.

## Conclusion

Within the limitations of the present study, the results showed that ASB2 yielded a significantly

higher  $\mu$ SBS to primary dentin than ABU and SBU adhesives in SE and TE modes. No significant difference was found in  $\mu$ SBS of the two universal adhesives.

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