



## Microshear Bond Strength of Scotchbond Universal Adhesive to Primary and Permanent Dentin: A Six-Month in Vitro Study

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### ABSTRACT

**Objectives:** This study aimed to assess the microshear bond strength (MSBS) of Scotchbond Universal adhesive, used in self-etch and etch-and-rinse modes, to primary and permanent dentin at 24 hours and six months.

**Materials and Methods:** A total of 88 composite micro-cylinders were divided into eight groups (n=11) as follows: (A) Etch-and-rinse, 24 hours, primary dentin; (B) Self-etch, 24 hours, primary dentin; (C) Etch-and-rinse, six months, primary dentin; (D) Self-etch, six months, primary dentin; (E) Etch-and-rinse, 24 hours, permanent dentin; (F) Self-etch, 24 hours, permanent dentin; (G) Etch-and-rinse, six months, permanent dentin; (H) Self-etch, six months, permanent dentin. The MSBS was measured by a testing machine at a crosshead speed of 0.5 mm/minute. Data were analyzed using three-way analysis of variance (ANOVA).

**Results:** The mean MSBS was 12.3±2.3 MPa in A, 18.8±4.1 MPa in B, 11.9±3.7 MPa in C, 16±2.9 MPa in D, 19.1±2.7 MPa in E, 22.8±4.1 MPa in F, 16.2±2.6 MPa in G, and 17.2±4.4 MPa in H. In the self-etch mode, the MSBS was significantly higher than that in the etch-and-rinse mode (P<0.001). The MSBS in permanent teeth was significantly higher than primary teeth (P<0.001). At six months, the MSBS significantly decreased in all groups (P<0.001).

**Conclusion:** The micro-shear bond strength of Scotchbond Universal adhesive decreases over time and depends on the type of tooth and the mode of application of the adhesive.

**Keywords:** Shear Strength; Scotchbond; Dentin Bonding Agents

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## INTRODUCTION

The increasing use of tooth-colored restorative materials highlights the need for a strong and durable bond to tooth structure [1]. New non-rinsing adhesives, commonly known as self-etch or etch-and-dry systems, have simplified the concept of dentin bonding. Self-etch systems, compared to etch-and-rinse systems, have advantages such as easy and fast application. Another clinical advantage of these adhesives is the absence of postoperative tooth hypersensitivity. On the other hand, self-etch adhesives decrease clinical iatrogenic procedural errors during etching, rinsing, and drying [2,3]. Self-etch adhesives partially dissolve the smear layer and cause less dentin demineralization compared to etch-and-rinse systems. The combination of the smear layer, resin, collagen, and minerals in the hybrid layer and superficial parts of resin tags prevents postoperative tooth hypersensitivity that is present in etch-and-rinse systems due to the lack of complete penetration of resin monomers into the collagen network [4]. A new group of adhesive systems was recently introduced to the market and named as universal or multi-mode adhesives. In these adhesives, all bonding components are supplied in one bottle. These adhesives can be used in self-etch and etch-and-rinse modes. The chemical composition of these adhesives contains silane monomer and phosphoric monomer, which enable the bond to mineralized tooth structures, metal, and porcelain [5]. The majority of universal adhesives contain acidic functional monomers such as 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP), which contains a polymerizable group and a phosphate group with the ability to form stable salt with calcium in the structure of hydroxyapatite. The stability of this calcium salt is related to the high bond strength of 10-MDP to enamel and dentin. Moreover, 10-MDP is a hydrophobic molecule, causing hydrophobicity of adhesive and subsequent reduction of water sorption. Addition of 10-MDP to adhesives enhances the chemical bond and decreases the hydrolytic destruction of bond compared to adhesives

without 10-MDP [6]. In-vitro studies have assessed the bond strength of different universal adhesives in self-etch and etch-and-rinse modes and have reported that the bond strength to enamel increases with the use of adhesives in the etch-and-rinse mode. However, some other studies have reported no significant difference in immediate bond strength of different universal adhesives to dentin in self-etch and etch-and-rinse modes [7].

The durability of the bond is a challenge in adhesive systems because the stability of the bond of restorative materials to tooth structure is related to clinical service and longevity of the restoration [7]. Evidence shows that although the primary resin-dentin bond strength is high, it decreases by 50% to 60% after one to two years in vitro and in vivo due to structural differences of dentin and enamel, microleakage, and the presence of Matrix Metalloproteinases (MMPs) in dentin [8-10]. Considering the relatively recent introduction of universal adhesives, studies on their mechanical properties, durability in wet environments, and aging by water storage are limited, and the available ones have been mainly conducted on permanent teeth. Therefore, this study aimed to assess the microshear bond strength of Scotchbond Universal adhesive in self-etch and etch-and-rinse modes to coronal dentin in primary and permanent teeth at 24 hours and six months.

## MATERIALS AND METHODS

This in-vitro experimental study has been approved by the Ethics Committee of Shahid Beheshti University of Medical Sciences (IR.SBMU.RIDS.1394.1). A total of 20 primary molars and 20 permanent premolars, extracted for orthodontic purposes within the past three months, were used in this study after obtaining consent from the patients. The teeth were sound and caries-free and were immersed in 0.5% chloramine-T solution for one week. The teeth were then immersed in distilled water at 4°C. The occlusal enamel was removed by a thin sectioning device with water coolant. Another section was made 2 mm beneath the superficial layer to prepare

**Table 1:** Composition and modes of application of the universal adhesive used in the present study according to the manufacturer's instructions

Material	Composition	Application mode
<b>Scotchbond Universal (3M ESPE, Seefeld, Germany; D-82229)</b>	MDP phosphate monomer, dimethacrylate resins	Self-etch: Scrub adhesive for 20 seconds; air-thin for 5 seconds; light-cure for 10 seconds
	HEMA, methacrylate functionalized polyalkenoic acid, filler, ethanol (pH=2.6)	Etch-and-rinse: Etch for 15 seconds; rinse for 10 seconds; air-dry for 5 seconds; scrub the adhesive for 20 seconds; air-thin for 5 seconds; light-cure for 10 seconds

HEMA :Hydroxyethyl methacrylate, MDP: Methacryloyloxydecyl dihydrogen phosphate

dentin discs with 2-mm thickness. All dentin discs were ground using 220-, 400-, 600-, and 800-grit abrasive papers, each used for 30 seconds with water coolant to obtain a smooth layer and uniform smear layer in all samples. The 20 primary dentin and 20 permanent dentin sections were randomly divided into eight groups (n=5) based on the mode of application of the bonding agent and duration of storage of samples in distilled water:

A: Etch-and-rinse, 24 hours, primary dentin; B: Self-etch, 24 hours, primary dentin; C: Etch-and-rinse, six months, primary dentin, D: Self-etch, six months, primary teeth.

E: Etch-and-rinse, 24 hours, permanent dentin, F: Self-etch, 24 hours, permanent teeth, G: Etch-and-rinse, six months, permanent dentin, H: Self-etch, six months, permanent dentin. According to the manufacturer's instructions, in each group, Scotchbond Universal adhesive (3M ESPE, Seefeld, Germany) was applied on the surface of the discs and light cured for 10 seconds using a light-curing unit (Optilux 50, Kerr, Danbury, CT, USA) with an intensity of 650 mW/cm<sup>2</sup> (Table 1). Tygon tubes with an internal diameter of 0.79 mm and height of 1 mm were placed on dentin surfaces, were filled with A2 shade of Filtek Z250 composite resin (3M ESPE, St. Paul, MN, USA), and were cured for 40 seconds. Two composite cylinders were placed on each dentinal section, while three composite cylinders were placed on one dentinal section in each group. Finally, 88 composite micro-cylinders were prepared. All discs were immersed in distilled water for 24 hours and were incubated at 37°C and in 100% humidity. After 24 hours, plastic Tygon tubes were cut using a scalpel. The 24-hour samples were subjected to

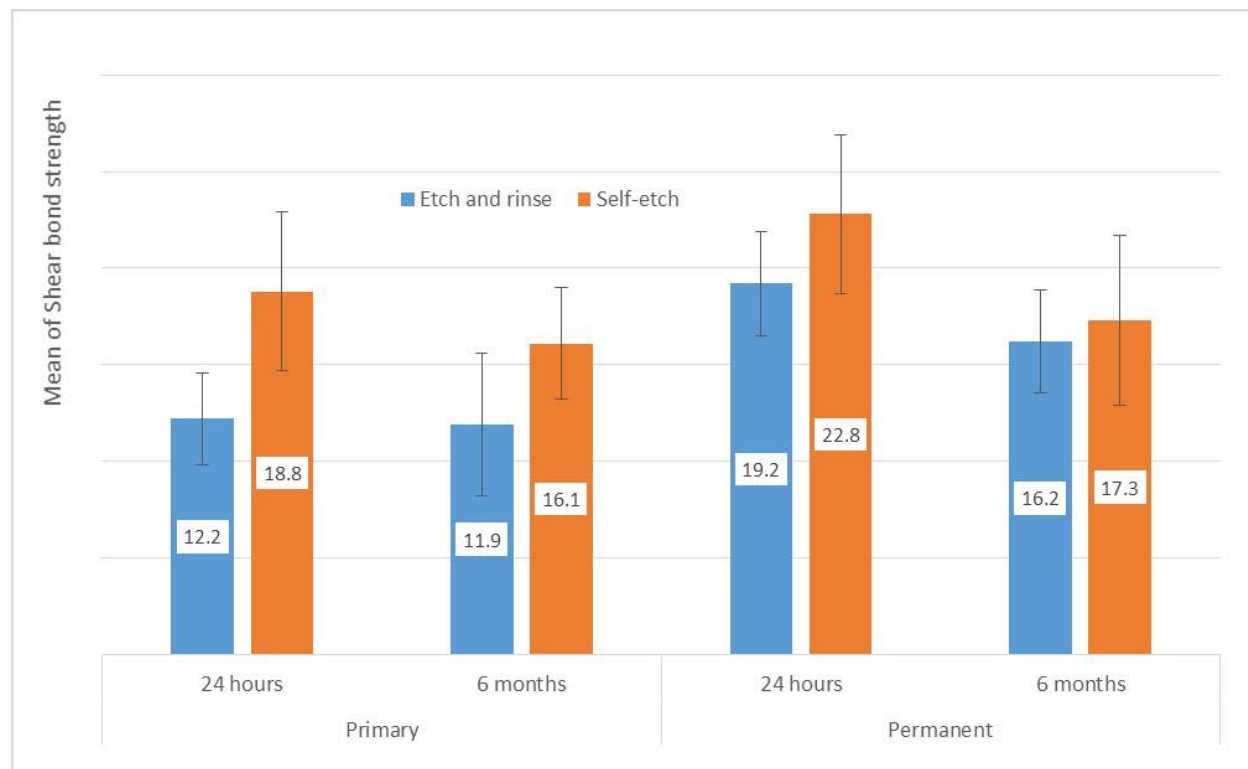
microshear bond strength testing in a universal testing machine (Bisco, Inc., Schaumburg, USA) at a crosshead speed of 0.5 mm/minute. The remaining 44 dentinal sections were immersed in distilled water and were incubated at 37°C and in 100% humidity. Distilled water was refreshed weekly. After six months, these samples were also subjected to microshear bond strength testing. Considering the presence of three influential factors, including type of tooth (primary or permanent), mode of application of adhesive (self-etch or etch-and-rinse), and duration of storage in distilled water (24 hours and six months), as well as the quantitative dependent variable of microshear bond strength, three-way analysis of variance (ANOVA) was used for data analysis. Type one error was considered as 0.05, and P<0.05 was considered statistically significant.

## RESULTS

Table 2 shows the mean and standard deviation (SD) of microshear bond strength in primary and permanent dentin with self-etch and etch-and-rinse modes of application of adhesive at six months and 24 hours.

**Table 2:** Mean and standard deviation (SD) of shear bond strength (MPa) in the studied groups (n=11)

Tooth	Time	Type	Mean±SD
Primary	24 hours	Etch-and-rinse	12.84±2.39
		Self-etch	18.84±4.1
	6 months	Etch-and-rinse	11.95±3.7
		Self-etch	16.09±2.91
Permanent	24 hours	Etch-and-rinse	19.19±2.7
		Self-etch	22.81±4.1
	6 months	Etch-and-rinse	16.20±2.68
		Self-etch	17.26±4.43



**Fig. 1.** Mean shear bond strength (MPa) in primary and permanent teeth with self-etch and etch-and-rinse modes of application at 24 hours and six months. Columns represent mean values and bars indicate standard deviation.

The results showed that the effect of adhesive on bond strength was significant, and the application of universal adhesive alone yielded a higher bond strength than the application of the adhesive with acid (Fig. 1;  $P < 0.001$ ). The effect of type of tooth on bond strength was also significant ( $P < 0.001$ ), and permanent teeth showed higher bond strength values than primary teeth (Fig. 1). Moreover, the duration of storage had a significant effect on microshear bond strength ( $P < 0.001$ ), and microshear bond strength at 24 hours was higher than that at six months (Fig. 1). None of the second-level and third-level interaction effects were significant ( $P > 0.05$ ).

## DISCUSSION

Considering the relatively recent introduction of universal adhesives, information on their properties and long-term performance are limited. On the other hand, demand for tooth-colored restorations has increased in pediatric dentistry, and since universal

adhesives can be used in one-step mode, their application can expedite restorative treatment of teeth, which is an advantage in pediatric dentistry. Scotchbond Universal is among the most commonly used universal adhesives available in the market. Thus, this adhesive was used in our study.

The conventional or macro-bond strength test with a bonding surface area larger than 3 mm<sup>2</sup> causes non-uniform stress distribution related to internal defects due to a larger area; this increases the amount of stress [11,12]. On the other hand, the microshear bond strength test assesses small bonding surface areas, and a higher number of samples can be obtained from one tooth. The samples do not need trimming or sectioning, and this method has lower technical sensitivity than the microtensile bond strength test [11,12]. Some previous studies have also discussed no difference or even superiority of this test over the microtensile test [11-14]. Thus, the microshear bond strength test was carried out in our study.

The results showed that microshear bond strength in all primary and permanent teeth at both 24 hours and six months was significantly higher in the self-etch adhesive group compared to the etch-and-rinse group. Self-etch one-step Scotchbond Universal is a mild adhesive. The 10-MDP molecules in the composition of this adhesive have a linear long alkyl chain and phosphoric acid ester group with the ability to chemically bond to hydroxyapatite in tooth structure [4,6]. Dentin contains less than 50% minerals, which decrease after acid-etching; this may be a possible reason for the compromised chemical bond of 10-MDP monomers. Moreover, the application of phosphoric acid as a separate step before the application of adhesive can result in deeper demineralization of dentin (3-6  $\mu\text{m}$ ) compared to the expected penetration depth of self-etch adhesive resins. However, the ability of the components of this bonding agent is limited for deep penetration between collagen fibers and into exposed dentinal tubules, and thus, collagen fibers may remain exposed [15]. The collagen network obtained following etching has low surface energy, which can also explain the reduction in shear bond strength [16]. Sabatini [17], van Landuyt et al [18], and Isolan et al [15] reported results similar to ours. Hanabusa et al [19], in their study on the bond of G-Bond Plus universal adhesive to dentin and enamel, stated that acid-etching definitely improves the bond to enamel, while no significant difference was noted in microtensile bond to dentin with the two modes of application of the adhesive [19]. Difference between their results and ours may be due to the use of different types of adhesives since they used G-Bond Plus, which is among the adhesives with lower etching power compared to Scotchbond Universal used in our study. Therefore, application of this adhesive on dentin surface in the etch-and-rinse mode may enhance its penetration. Moreover, this adhesive contains MET-4 instead of 10-MDP monomer. Nonetheless, Hanabusa et al [19] discussed that despite no difference in microtensile bond strength to dentin, infrastructure assessments by transmission

electron microscopy (TEM) revealed that the adhesive resin interface was porous, and the collagen network was not completely covered with resin. Thus, care must be taken in the use of universal adhesives in the etch-and-rinse mode because some concerns exist regarding the durability of the bond over time [19]. Wagner et al [2] reported increased penetration of three types of universal adhesives to dentin in self-etch and etch-and-rinse modes, despite no difference in microtensile bond strength. The controversy in the results of the two aforementioned studies and our study may be attributed to the different substrates used and the number of samples in each group.

In our study, the overall microshear bond strength of permanent teeth in both modes of application and at both time points was significantly higher than that of primary teeth. The bond strength of composite to primary dentin is not reliable due to structural, chemical, and morphological differences of primary and permanent teeth. Adhesives have a greater effect on primary dentin, causing more severe and deeper demineralization of intertubular dentin. Due to this effect as well as the lower mineral content of primary dentin compared to permanent dentin, a hybrid layer thicker by 25%-30% is formed, which results in subsequent incomplete penetration of adhesive resin and consequent formation of shorter resin tags in primary teeth. As a result, the bond strength values are lower in primary teeth compared to permanent teeth [12,20]. The results of this study are in line with those of previous studies reporting a higher bond strength in permanent teeth [21,22].

In our study, the microshear bond strength significantly decreased over time (from 24 hours to six months) in both primary and permanent teeth with both modes of application of adhesive. These findings may be attributed to the negative effect of water sorption on bond strength over time. Water molecules bond to polar areas of the polymer by hydrogen bonds and cause plasticization, swelling, and subsequent reduction of mechanical properties of the polymer [23,24].

Moreover, water storage and subsequent water sorption over time result in the formation of nanocavities in the polymer matrix and subsequent degradation of the adhesive matrix following the loss of accessory attachments of monomers [25-27]. This also causes a reduction in bond strength of adhesives. Reduction in bond strength over time can also be due to the failure of the bond between fillers and matrix [28]. According to the brochure provided by the manufacturer, Scotchbond Universal adhesive has superior hydrophobic properties. Nonetheless, this adhesive contains water, ethanol, hydroxyethyl methacrylate (HEMA), 10-MDP functional monomer, and polyalkenoic acid copolymer, which can possibly play a role in water sorption and subsequent reduction of bond strength over time [10,11,29,30]. Marchesi et al [3] reported a significant reduction in microtensile bond strength of a universal adhesive in self-etch and etch-and-rinse modes at six months compared to 24 hours. Evidence shows that universal adhesives devoid of polyalkenoic acid copolymer have higher durability, which further confirms our findings regarding the reduction of bond strength of Scotchbond Universal over time since it contains polyalkenoic acid copolymer. Mechanical properties of adhesive itself also play a significant role in bond strength. According to Takahashi et al [31], who support the use of adhesives without HEMA, reduction in bond strength over time in Scotchbond Universal, which contains HEMA, may be due to the decline of physical and mechanical properties of the adhesive itself.

On the other hand, scanning electron microscopic (SEM) studies of the interface of self-etch adhesives indicated the presence of an acid-base resistant zone against acid-base interactions, which can play an important role in secondary caries prevention [32]. In other words, self-etch adhesives have been designed to prevent secondary caries when applied in self-etch mode. Thus, logically, the application of etchant on dentin surface should be prevented prior to the application of self-etch adhesives.

## CONCLUSION

Within the limitations of this study, the results showed that the strength and durability of Scotchbond Universal adhesive were greater with its application in dentin in the self-etch mode compared to the etch-and-rinse mode. Bond strength in primary teeth is less than that in permanent teeth. The durability of the bond decreases over time in both primary and permanent teeth.

## CONFLICT OF INTEREST STATEMENT

None declared.

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