



## Microtensile Bond Strength of Different Bonding Agents to Superficial and Deep Dentin in Etch-and-Rinse and Self-Etch Modes

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### ABSTRACT

**Objectives:** This study aimed to assess the microtensile bond strength ( $\mu$ TBS) of etch-and-rinse (E&R), self-etch (SE), and universal adhesives to superficial and deep dentin.

**Materials and Methods:** In this in vitro study, 40 sound third molars were randomly divided into two main groups of superficial and deep dentin. Based on our classification, superficial dentin was right beneath the deepest occlusal groove, and deep dentin was 2mm beneath the deepest occlusal groove. Each group was divided into 4 subgroups (n=20) for application of Adper Single Bond 2 (ASB), Clearfil SE Bond (CSE), and Scotchbond Universal (SBU) in E&R and SE modes along with Charisma Smart composite resin on dentin. The specimens were incubated in distilled water at 37°C for 24 hours and their  $\mu$ TBS was then measured. The mode of failure was determined under a stereomicroscope at  $\times 40$  magnification. Data were analyzed by one-way ANOVA ( $\alpha=0.05$ ).

**Results:** The highest  $\mu$ TBS belonged to the superficial dentin/SBU/E&R group. The  $\mu$ TBS was significantly higher in superficial dentin than deep dentin for all adhesives ( $P=0.005$ ). There was no significant difference in mode of failure among the groups.

**Conclusion:** Based on the results obtained in the present study, type of bonding agent and application mode affected  $\mu$ TBS. In use of universal adhesive, E&R mode can improve  $\mu$ TBS.

**Keywords:** Composite Resins; Dentin-Bonding Agents; Dentin

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### INTRODUCTION

Currently, composite resins are the most popular tooth-colored restorative materials that are bonded to tooth structure using adhesives such as dentin bonding agents. Achieving an efficient bond to dentin decreases the risk of restoration failure, caries recurrence, and pulpal stimulation due to minimal cavity preparation [1]. The main mechanism of action of dentin bonding agents is through formation of resin tags in tooth

structure that cause micromechanical interlocking [2]. However, the tubular and highly heterogeneous dentin structure challenge efficient bonding to dentin; whereas, bonding to enamel is reliable because of the morphological structure of enamel [3]. The structure and composition of dentin are directly correlated with its bond strength. In deep dentin, a larger area is occupied by dentinal tubules, and the dentin structure has greater moisture. Thus, the bond strength to

deep dentin is expected to be lower than that to superficial dentin [4].

Depending on the application steps, bonding agents are used in etch and rinse (E&R) or self-etch (SE) modes [5,6]. In 5th generation bonding agents, 35%-37% phosphoric acid is used for 15-20 seconds to etch the surface, and the primer and bonding agent which are both supplied in one bottle are then applied [7]. However, risk of collapse of collagen fibers in the process of dentin surface drying and insufficient penetration depth of bonding agent into dentin structure are the main drawbacks of 5th generation bonding agents that can lead to bacterial leakage [6].

The etching step was eliminated in 6th generation bonding agents. However, the main advantage of 6th generation bonding agents is that they depend less on dentin moisture compared with previous generations. Nonetheless, a preliminary study indicated that 6th generation bonding agents did not provide sufficient bond strength to enamel. Thus, separate enamel etching is suggested with phosphoric acid first; however, care must be taken to only etch the enamel because separate etching of dentin with phosphoric acid would result in its excessive etching [7]. Recently, universal adhesives were introduced to the market that can be used in three modes of E&R, SE, and selective etch. Clinical applications of universal adhesives have greatly increased in the recent years due to their easy use [8].

The microtensile bond strength ( $\mu$ TBS) test is among the most widely used tests to evaluate bond strength [9]. It has several advantages including, many small samples can be prepared from one tooth, it allows better control of differences (*e.g.*, peripheral dentin versus central dentin), and enables better distribution of stress on small bonding areas [10].

There are larger funnel-shaped dentinal tubules with much less intertubular dentin and greater moisture in deep dentin that make it difficult to achieve sufficient bond strength. Many studies have evaluated the efficacy of E&R and SE bonding agents to different depths of dentin, but their comparison with universal adhesives applied with different application

strategies needs further investigations [9,11]. Thus, the aim of this in vitro study was to compare the  $\mu$ TBS of E&R, SE, and universal adhesives to superficial and deep dentin.

## MATERIALS AND METHODS

This study was approved by the Ethics Committee of Babol University of Medical Sciences (IR.MUBABOL,HRL.REC.1398.100). This in vitro experimental study was conducted on 40 sound third molars with no caries or defects. The teeth were cleaned from plaque and debris and stored in saline which was refreshed weekly until the experiment. Prior to the onset of study, the teeth were immersed in 0.5% chloramine T solution for disinfection for 24 hours. The teeth were then randomly divided into two groups of deep dentin and superficial dentin (N=20). In the deep dentin group, tooth crowns were sectioned 2mm below the deepest groove with a diamond disc (D&Z, Germany) and the occlusal section was used as the deep dentin specimen. In the superficial dentin group, the tooth crowns were sectioned right beneath the deepest occlusal groove. In the next step, the exposed dentin surfaces in all teeth were wet-polished with 600-grit silicon carbide abrasive paper for 20 seconds to obtain a standard smear layer.

### **SUPERFICIAL DENTIN GROUP:**

The teeth in superficial dentin group (n=20) were divided into four subgroups (N=5) as follows:

#### **Subgroup 1: Adper single bond 2 (ASB; 3M, ESPE, USA)**

The polished specimen surfaces were etched with 37% phosphoric acid (Pulpdent, USA) for 15 seconds, rinsed with water for 10 seconds, and covered with a cotton pellet to prevent dehydration of dentin. ASB was applied on the etched surface in two layers with a microbrush for 15 seconds according to the manufacturer's instructions, air thinned for 5 seconds, and light-cured for 10 seconds (VALO Ultradent Products Inc., South Jordan, UT, USA). Then, Charisma Smart composite resin (Heraeus Kulzer, Hanau, Germany) was applied in two layers with 2mm thickness with a total thickness of 4mm on the surface of specimens. Each layer was light-cured separately for 20

seconds. The light intensity of the LED was checked after curing every 5 specimens by a radiometer (Demetron/Kerr Corp, USA) to ensure a light intensity of 800mW/cm<sup>2</sup>.

**Subgroup 2: Clearfil SE bond (CSE; Kuraray, Japan)**

CSE primer was applied on polished surfaces, dried for 20 seconds, and then the bonding agent was applied, dried, and light-cured for 10 seconds. Composite resin was then applied as explained for subgroup 1.

**Subgroup 3: Scotchbond universal (SE) (SBU; 3M, ESPE, USA)**

Bonding agent was applied on the polished surfaces as instructed by the manufacturer, gently dried for 5 seconds, and light-cured for 10 seconds. Composite resin was then applied as explained for subgroup 1.

**Subgroup 4: Scotchbond universal (E&R) (SBU; 3M, ESPE, USA)**

The polished surfaces were etched with 37% phosphoric acid for 15 seconds, rinsed with water for 10 seconds, and covered with a cotton pellet to prevent dentin dehydration. Bonding agent was then applied on the etched surface for 20 seconds with a microbrush according to the manufacturer's instructions, dried with air spray for 5 seconds, and light-cured for 10 seconds. Composite resin was then applied as explained for subgroup 1.

**DEEP DENTIN GROUP:**

**Subgroup 5: ASB**

ASB was applied on deep dentin specimens as explained for subgroup 1.

**Subgroup 6: CSE**

CSE was applied on deep dentin specimens as explained for subgroup 2.

**Subgroup 7: SBU (SE)**

SBU was applied on deep dentin specimens as explained for subgroup 3.

**Subgroup 8: SBU (E&R)**

SBU was applied on deep dentin specimens as explained for subgroup 4.

Finally, composite resin was applied as explained for superficial dentin specimens. The specimens were then incubated at 37°C (Scientific, LTD, UK) for 24 hours.

**Measurement of  $\mu$ TBS:**

The specimens were cut in buccolingual and mesiodistal dimensions perpendicular to the

adhesive interface at a speed of 300rpm with 1mm cutting intervals under water coolant. Accordingly, four perfect rods were obtained from each tooth. Each rod was then attached to the jig of microtensile tester (Koopaa, Mashhad, Iran). The specimens were subjected to a tensile force at a crosshead speed of 0.5 mm/minute until failure. The force at failure was recorded in Newtons (N) and the cross-sectional area of the failure zone was measured by a digital caliper (Shinwasokuti, China). The load causing failure was divided by the cross-sectional area of the failure zone to calculate the  $\mu$ TBS in megapascals (MPa) as follows:

Bond strength (MPa) = Tensile force (N)/Cross sectional area (mm<sup>2</sup>)

The cross-sectional area was inspected under a microscope (Dewinter, Italy) at  $\times 40$  magnification and the mode of failure was determined as follows:

- 1- Adhesive (at the composite-tooth interface)
- 2- Cohesive (within dentin or composite)
- 3- Mixed

**Statistical analysis:**

The mean and standard deviation of  $\mu$ TBS were calculated for all groups, followed by data analysis. The Kolmogorov-Smirnov test was used to assess the normality of data distribution, which showed normal distribution, prompting the use of one-way ANOVA for comparisons. Pairwise comparisons were performed by the Tukey's post-hoc test. Independent t-test was also used to compare superficial and deep dentin groups. P<0.05 was considered statistically significant.

## RESULTS

Table 1 shows the  $\mu$ TBS values in all groups.

**Superficial dentin group:**

The highest and lowest  $\mu$ TBS in the superficial dentin group belonged to the SBU (E&R) and CSE subgroups, respectively. The difference in the mean  $\mu$ TBS of superficial dentin subgroups was statistically significant (P<0.001).

As shown in Figure 1, pairwise comparisons of the subgroups in the superficial dentin group showed that the CSE subgroup had significantly lower  $\mu$ TBS than the other subgroups, and the SBU (E&R) subgroup had significantly higher  $\mu$ TBS than the other subgroups (P<0.05).

**Table 1.** Comparison of microtensile bond strength (MPa) in superficial and deep dentin subgroups

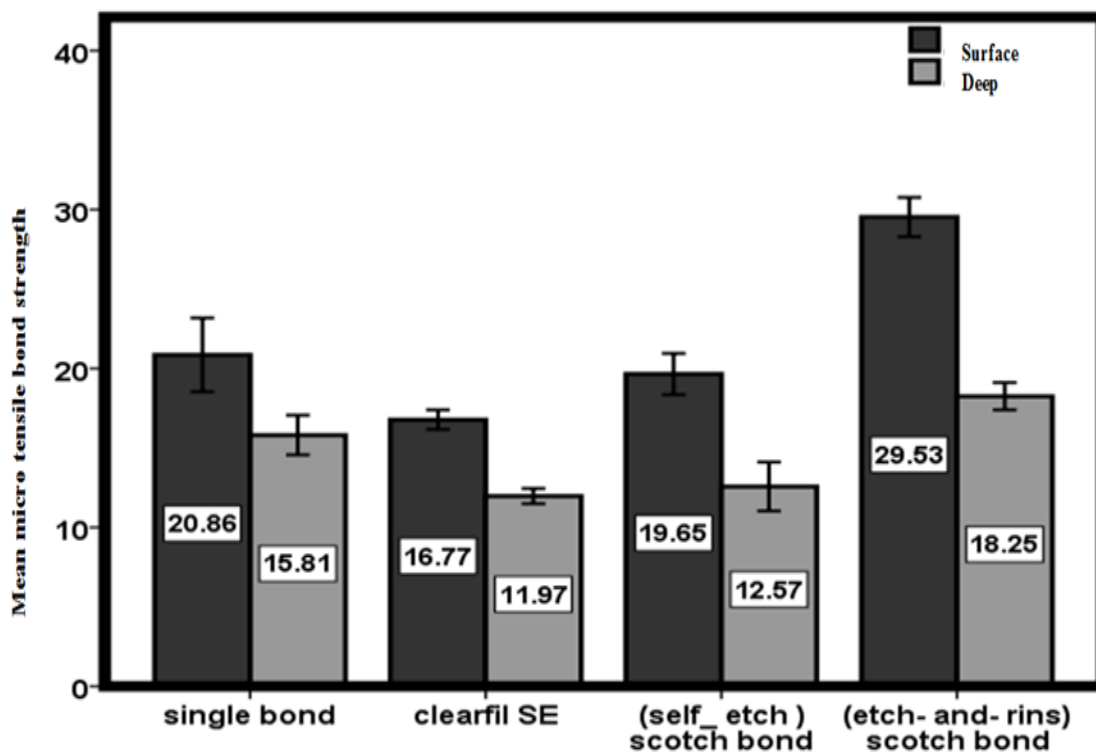
Tensile bond strength		Mean	SD	Min.	Max.	Sig.
Superficial	Single bond	20.85 <sup>a</sup>	4.96	13.08	29.75	<0.001
	Clearfil SE	16.77 <sup>b</sup>	1.31	14.02	18.93	
	Scotch bond (self-etch)	19.65 <sup>a</sup>	1.77	15.10	26.10	
	Scotch bond (etch and rinse)	29.52 <sup>c</sup>	2.62	25.50	34.61	
Deep	Single bond	15.80 <sup>a</sup>	2.67	10.80	19.47	<0.001
	Clearfil SE	11.96 <sup>b</sup>	1.03	10.15	13.64	
	Scotch bond (self-etch)	12.57 <sup>b</sup>	3.28	15.63	19.86	
	Scotch bond (etch and rinse)	18.24 <sup>c</sup>	1.83	15.11	22.25	

Similar letters indicate no significance between the means at the level of 0.05

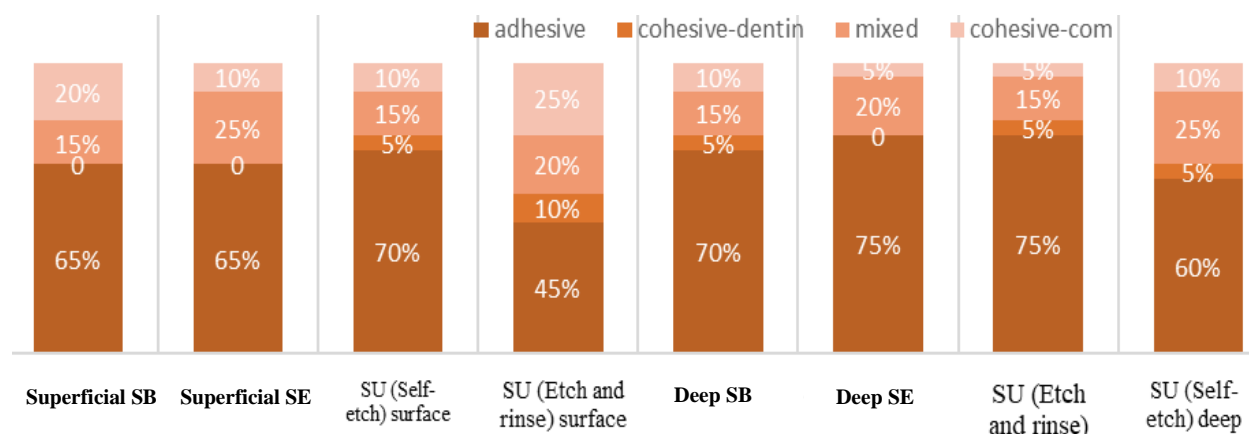
**Deep dentin group:**

In deep dentin group, the highest  $\mu$ TBS was noted in SBU (E&R) subgroup (18.24 MPa) and the lowest in CSE (11.96 MPa) subgroup. The difference in the mean  $\mu$ TBS of deep dentin subgroups was statistically significant ( $P<0.001$ ). Pairwise comparisons showed significant differences between all subgroups ( $P<0.05$ ) except between CSE and SBU (SE) subgroups ( $P>0.05$ ).

Independent t-test was also used to compare superficial and deep dentin groups which showed significantly higher  $\mu$ TBS in superficial dentin than deep dentin ( $P<0.001$ ). Figure 2 demonstrates the frequency distribution of different modes of failure. The groups and subgroups had no significant difference in mode of failure ( $P>0.05$ ). Cohesive failure within dentin was not seen in subgroups 1, 2 and 6.



**Fig 1.** Pairwise comparisons of microtensile bond strength of superficial and deep dentin subgroups



**Fig 2.** Frequency distribution of different modes of failure. SB: single bond; SE: Clearfil SE bond; SU: Scotch bond universal; Adhesive failure: failure of the tooth and composite interface; Cohesive failure: failure in composite or tooth; Mix failure: failure is a combination of the two

## DISCUSSION

This study assessed the  $\mu$ TBS of E&R, SE, and universal adhesives to superficial and deep dentin. The results showed significantly different  $\mu$ TBS of adhesives used in this study to superficial and deep dentin. Two-step E&R ASB, two-step SE CSE, and SBU with both E&R and SE modes were evaluated in this study. The results showed significantly different  $\mu$ TBS of these adhesives to superficial and deep dentin.

Tagami et al, [12] and Suzuki and Finger [13] reported that the bond strength to deep dentin was significantly lower than superficial dentin, which was similar to the results of the present study. The reason for the difference in bond strength to superficial and deep dentin can be due to different structure, chemical composition, and moisture level of superficial and deep dentin. Dentinal tubules are inverted cones that narrow from the area near the pulp towards the dentinoenamel junction. Each dentinal tubule is surrounded by highly mineralized peritubular dentin, the amount of which, increases towards dentinoenamel junction; thus, the moisture of superficial dentin is much lower than that of deep dentin [12,13]. Kumari et al. [9] used Single Bond Universal and Tetric N Bond to examine the effect of dentin depth on bond strength, and reported similar results; both bonding agents

showed higher bond strength to superficial dentin due to higher value of peritubular dentin in superficial dentin which plays an important role in hybrid layer formation. Pashley et al. [11] assessed the effect of dentin depth on bond strength in an animal study and reported results similar to the present findings.

In the present study, the two-step CSE had lower bond strength to superficial dentin than the two-step E&R SBU. In a study by Sofan et al, [7] and Navyasri et al, [14] the  $\mu$ TBS of a two-step self-etch bonding agent was lower than a two-step E&R and universal adhesive. They explained the reason to be the etching of dentin. Etching of dentin destroys minerals and creates a scaffold of collagen, almost empty of hydroxyapatite which is filled with resin after bonding; however, in two-step SE adhesives, the remaining smear layer blocks the entrance of the tubules due to low acidity of primer, which limits the hybridization of peritubular dentin and formation of resin tags [7,14].

According to the present study, the  $\mu$ TBS of SBU in SE mode was significantly higher than the two-step SE bond to superficial dentin. The reason for this difference is related to the composition of these two bonding agents and their pH values. According to Jacker-Guhr et al, [15] SBU contains 10-MDP functional

monomer and polyalkenoic acid copolymer, which can increase the bond strength by binding to calcium remaining in hydroxylapatite. Similar to SBU, the CSE bond contains 10-MDP monomer, but does not have a polyalkenoic acid copolymer in its composition; thus, the chemical bond of CSE is weaker than that of SBU.

SBU has a relatively low pH of 2.9 (ultra-mild); whereas, CSE bond has a pH of 1.9; thus, the calcium remaining in hydroxyapatite structure would be higher if SBU bonding agent is used. Thus, the bond strength also increases [6,16]. Yaseen et al. [17] showed that CSE bonding agent with a pH of 2.7 had higher bond strength than Contax bonding agent with a pH of 1.2, confirming the effect of pH on bond strength. Moreover, CSE has a water-based solvent; whereas, SBU has an acetone-water solvent, which can decrease the bond strength because acetone-based adhesives have higher vapor pressure that enables fast evaporation [3]. However, the bond strength of these two bonding agents to deep dentin was not significantly different; the reason for which can be the change in mineral composition of dentin, such that deep dentin has lower mineral content and resultantly less calcium [17]. Gre et al. [6] showed that the  $\mu$ TBS of SBU to deep dentin did not differ much in SE and E&R modes, which was opposite to the present findings. The reason is the difference in definition of deep dentin. In the study by Gre et al, [6] deep dentin was defined as 4mm lower than the deepest occlusal groove, while deep dentin specimens were 2mm deeper than the occlusal groove in the present study. The calcium remaining in hydroxyapatite increases the bond strength.

In this study, SBU was used in SE and E&R modes and the  $\mu$ TBS was significantly higher in E&R mode. According to some studies, the bond strength of universal adhesives was higher in E&R mode, because after etching of dentin, the movement of monomers on the etched surface is facilitated and the depth of the resultant hybrid layer increases. [6,16]. Overall, universal adhesives have shown reliable instant bond strength regardless of the mode of application [17].

According to Ramachandran et al, [18] the quality of the hybrid layer and the porosity of the bonded surface are effective in creating a suitable bond. This can justify lack of significant differences in bond strength of SBU in SE mode and ASB in E&R mode because universal adhesives have a functional MDP monomer that increases their bond strength.

HEMA hydrophilic monomer, present in the combination of all three bonding agents used in the present study, can increase the bond strength by creating favorable conditions for combining hydrophobic and hydrophilic bonding components [7]. However, it should be noted that this monomer is effective only if the bonding agent contains functional MDP monomers; otherwise as a hydrophilic monomer, it would result in reduction of bond strength in the long-term [18].

In the present study, all modes of failure were seen in all subgroups and the lowest frequency of adhesive failure was noted in SBU E&R bonding agent to superficial dentin, which may explain higher bond strength of this group than other groups [19]. Moreover, this bonding agent had the highest frequency of cohesive failures in dentin. In failures were generally deep dentin group, adhesive more common.

## CONCLUSION

According to our results, dentin depth affects  $\mu$ TBS and is significantly higher in superficial dentin compared to deep dentin. Moreover, we found that the type and mode of application of a bonding agent can affect bond strength. This was based on the observation that the universal adhesive showed higher bond strength than the two-step E&R and the two-step SE bonding agents. Additionally, in use of universal adhesive, the bond strength was significantly higher in E&R mode compared with SE mode.

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## CONFLICT OF INTEREST STATEMENT

None declared.

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