



Microshear Bond Strength of Different Adhesive Systems to Dentin

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ABSTRACT

Objectives: The aim of this study was to compare the microshear bond strength (μ SBS) of various adhesive systems to dentin.

Materials and Methods: In this in vitro experimental study, 60 sound human third molars were divided into four groups. Dentin discs were prepared of middle-third dentin measuring 4 mm in diameter and 2 mm in thickness. Dentin surfaces were bonded with one of the four types of adhesives: (A) Single Bond (3M ESPE), Scotchbond Universal (3M ESPE) in etch and rinse (B) and self-etch (C) modes and (D) Clearfil SE Bond (Kuraray Noritake Dental). After the application of adhesive systems according to the manufacturers' instructions, composite cylinders (Vit-l-essence) were bonded to dentin surfaces. The μ SBS test was performed using a universal testing machine at a crosshead speed of 1 mm/min. Data were analyzed with one-way ANOVA and Tukey's test ($\alpha=0.05$).

Results: The μ SBS was the highest in self-etch Scotchbond Universal (15.8 \pm 6.08 MPa) followed by Clearfil SE Bond (15.24 \pm 4.6 MPa), etch and rinse Scotchbond Universal (11.68 \pm 4.07MPa) and Single Bond (11.24 \pm 3.74 MPa). A significant difference was only found between Single Bond and etch and rinse Scotchbond Universal groups ($P=0.04$).

Conclusion: Based on the results of this study, application of Scotchbond Universal in self-etch mode provides a reliable bond to dentin.

Keywords: Shear Strength; Dentin-Bonding Agents; Composite Resins

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INTRODUCTION

Use of composite resin restorations has significantly increased due to their optimal esthetic properties and non-invasive tooth preparation design. Long-term success of composite resin restorations depends on the

durability and strength of the bond between the tooth structure and composite resin [1]. Bonding durability is critical for the longevity of restorations. Degradation of the bonding interface can lead to gap formation at the interface. The success of adhesion in dentistry

depends on several factors, such as the type of substrate, type of adhesive, moisture, and operator's experience and skills [2]. Effective bonding to substrates with different properties is an important aspect in dentin adhesion [3].

Resin-dentin bonds are less durable than resin-enamel bonds, because dentin is a more complex substrate composed of both mineral and organic phases. Moreover, dentin moisture should be preserved to avoid collapse of the collagen matrix; thus, it is essential for successful bonding, but it also adversely affects the long-term bonding results [3,4].

According to the bonding substrate (enamel or dentin), dental adhesive systems are used in three clinical steps of etching, priming, and bonding [5]. Dental adhesives are classified into three major categories based on their clinical application mode. The first system is referred to as the etch-and-rinse system and can be employed in three-step and two-step approaches [6]. The second system is the self-etching primer system. The third system is known as all-in-one or one-step self-etching system [7].

The ultimate goal of adhesive dentistry is to enable simple and fast adhesive application with durable bonding to enamel and dentin. The manufacturers are constantly introducing new adhesive systems with claims of simple use, improved composition and the ability to bond to tooth structure [8]. One of the most recent novelties in adhesive dentistry is the introduction of 'universal' or 'multi-mode' adhesives. These are simplified adhesives, usually containing all bonding components in one single bottle. Universal adhesives may be applied either in etch-and-rinse or self-etching bonding modes, according to the manufacturers' claims. Besides, universal adhesives can be used with different restorative materials [9]. The manufacturers claim that there is no compromise on bonding effectiveness when either bonding strategy is used. Nevertheless, it is known that simplified adhesives are often associated with lower in vitro bond strength results and poorer in vivo longevity of restorations. These findings are

probably due to the complex formulation of simplified adhesives and their high content of solvents, which may impair complete solvent volatilization and consequently lead to inadequate adhesive polymerization [10].

Based on all the above, the aim of the current study was to evaluate the microshear bond strength (μ SBS) of different adhesive systems to dentin. The null hypothesis was that no significant difference in bond strength exists between the universal adhesives and other adhesive systems.

MATERIALS AND METHODS

After obtaining approval from the Ethics Committee in Research of the Health Sciences of Tehran University of Medical Sciences (6087), 60 sound extracted human third molars without any cracks or defects were collected, cleaned and disinfected in 0.5% chloramine solution for 1 week. Then, they were embedded in acrylic resin cylinders. The root of each tooth was cut and the occlusal enamel was removed by means of a diamond disc (Extec; Enfield, CT, USA). Dentin discs with 2 mm height were obtained from the middle part of the tooth crowns. The specimens were ground wet using 100, 400 and 1000-grit abrasive papers (Carborundum Abrasives; Recife, PE, Brazil) and incubated at 37°C for 48 hours.

They were then randomly divided into four groups (n=15) according to the adhesive system and the application protocol of adhesives on dentin surfaces:

Group A: Scotchbond Universal (3M ESPE, St. Paul, MN, USA) in self-etch mode: No acid-etching gel was used. The adhesive was rubbed on dentin surface with a microbrush for 20 s, followed by gentle air drying for 5 s and light curing for 10 s (woodpecker D; Guilin Woodpecker Medical Instrument Co., Guilin, Guangxi, China).

Group B: Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA): 37% phosphoric acid gel (Etch Royale; Pulpdent, Watertown, USA) was used to etch dentin surfaces for 15 s. The etched dentin surfaces were then rinsed for 10 s to completely remove the etching gel. Then, the adhesive was applied on the wet dentin with a

microbrush and rubbed for 20 s followed by gentle air drying for 5 s, and the second layer was applied and gently air-dried and light cured for 10 s.

Group C: Clearfil SE Bond (Kuraray Co. Ltd., Osaka, Japan): The self-etching primer was applied on dentin using a microbrush and was left in place for 30 s. Air drying for 5 s was done to remove excess solvent. Then, bonding was used with a microbrush and after that gentle air drying and light curing were performed for 5 and 20 s, respectively.

Group D: Scotchbond Universal (3M ESPE, St. Paul, MN, USA) in etch and rinse mode: First, 37% phosphoric acid gel (Etch Royale, Pulpdent) was used to etch the dentin surface for 15 s. Next, the etched dentin surfaces were rinsed for 10 s to completely remove the etching gel and then the adhesive was agitated on dentin with a microbrush for 20 s followed by gentle air drying for 5 s and light curing for 10 s. Detailed information on chemical composition of the adhesive systems is presented in Table 1. Following adhesive applications, Tygon tubes 1 mm in height and 1.2 mm in diameter were fixed on the surface. Composite resin (Vit-l-escence, Ultradent,

USA) was incrementally applied into the tubes. Each increment was polymerized for 20 s using a LED curing unit (woodpecker D; Guilin Woodpecker Medical Instrument Co., Guilin, Guangxi, China) with an intensity of 1000 mW/cm². The bonded specimens underwent thermocycling and were subjected to 1,000 thermal cycles between 5°C and 55°C with a dwell time of 20 s and a transfer time of 10 s. Specimens were loaded with shear force until fracture in a universal testing machine (Instron 3220; Instron Corporation, Canton, Massachusetts, USA) at a crosshead speed of 1.0 mm/min using a knife-edged chisel. The SBS in megapascals (MPa) was calculated by dividing the maximum load in Newtons by the cross-sectional area of the bonded surface in square millimeters.

Statistical analysis

Data were statistically analyzed using one-way ANOVA with bond strength data as dependent variable and adhesive type and application mode as factors. One-way ANOVA and the Tukey's post-hoc test were used to find groups with significant differences. The level of significance was set at 0.05 for all tests. SPSS version 21.0 (SPSS Inc., IL, USA) was used for statistical analyses in this study.

Table 1: Chemical composition of adhesives

Dentin Bonding Agent	Composition	Manufacturer	Batch No.
Adper Single Bond 2	Ethyl alcohol (25-30), silane treated silica (nanofiller) (10-20), bis-GMA (10-20), HEMA (5-10), glycerol 1,3-dimethacrylate (5-10), copolymer of acrylic and itaconic acids (5-10), water (5), diurethane dimethacrylate (1-5)	3M ESPE, St Paul, MN, USA	N300780BR
Scotchbond Universal	MDP, bis-GMA HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St Paul, MN, USA	502226
Clearfil SE Bond Primer	2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophilic aliphatic, dimethacrylate dl-camphorquinone, water, accelerators, dyes and others	Kuraray, Osaka, Japan	00147A
Clearfil SE Bond adhesive	2-hydroxyethyl methacrylate (25-35), 10-methacryloyloxydecyl dihydrogen phosphate (MDP) Expose to a gentle air stream Bisphenol A diglycidyl methacrylate Cure 10 s 2-Hydroxyethyl methacrylate Hydrophobic dimethacrylate dl-Camphorquinone N,N-diethanol-p-toluidine Silanated colloidal silica	Kuraray, Osaka, Japan	00114A

Table 2. Mean and standard deviation of μ SBS in different groups

Adhesive	Mean	Std. deviation	Maximum μ SBS	Minimum μ SBS
Scotchbond Universal in self-etch mode	15.80 ^a	6.08	26.37	8.05
Adper Single Bond 2	11.24 ^b	3.75	18.93	6.63
Clearfil SE Bond	15.24 ^{ab}	4.06	22.38	6.28
Scotchbond Universal in etch and rinse mode	11.68 ^{ab}	4.07	21.59	6.99

Similar superscripted letters indicate no significant difference in μ SBS values.

RESULTS

The mean and standard deviation of μ SBS values are shown in Table 2. As shown, Scotchbond Universal in self-etch mode showed the highest mean μ SBS value followed by the Clearfil SE Bond and Scotchbond Universal in etch and rinse mode; Adper Single Bond demonstrated the lowest mean μ SBS value. One-way ANOVA showed significant differences in bond strength values among the groups ($P=0.015$). Thus, the Tukey's test was applied for pairwise comparisons.

One-way ANOVA showed significant differences ($P=0.015$) in bond strength values between groups. Thus, Tukey's test was performed for pairwise comparisons. The results showed that there was a significant difference in μ SBS between Scotchbond Universal in self-etch mode and Adper Single Bond 2 ($P=0.04$); but there were no statistically significant differences between Scotchbond Universal in self-etch mode and Clearfil SE Bond and Scotchbond Universal in etch and rinse mode, and also between Adper Single Bond 2 with Clearfil SE Bond and Scotchbond Universal in etch and rinse mode ($P>0.05$, Table 2).

DISCUSSION

Dentin adhesion is a difficult challenge, while good adhesion to enamel is easy and predictable [11,12]. Therefore, a new type of adhesive known as "universal" or "multi-mode" adhesive was recently introduced to enhance dentin adhesion. Universal adhesives are recommended by dental material manufacturers for use with/without acid

pretreatment of enamel surfaces [13,14]. There is limited information as to whether the different etching modes provide equal bond strength to dentin [15]. Bond strength is one of the most important factors that affects the bonding durability [16].

Our study investigated the μ SBS by use of a universal adhesive in etch and rinse and self-etch modes compared with other adhesive types with the same application modes. Thus, the aim of this study was to evaluate the μ SBS of different adhesive systems to dentin. We found that Scotchbond Universal in self-etch mode resulted in higher μ SBS to dentin compared with other adhesives. Thus, the null hypothesis was rejected.

In this study, Scotchbond Universal in self-etch mode showed the highest mean μ SBS value (15.80 ± 6.08 MPa) followed by the Clearfil SE Bond (15.24 ± 4.60 MPa) and Scotchbond Universal in etch and rinse mode (11.68 ± 4.07 MPa), and Adper Single Bond 2 demonstrated the lowest mean μ SBS value (11.24 ± 3.75 MPa). The μ SBS in self-etch mode was higher than that in etch and rinse mode in use of universal adhesive, but there was no significant difference between them. Studies have shown that there is no significant difference in dentin bond strength of universal adhesives in etch and rinse or self-etch modes of application. However, there is presently a preference for self-etch adhesive systems for application on dentin due to shallower demineralization compared with 35% phosphoric acid, and elimination of the rinsing step after etching with phosphoric acid; this is one of the most critical steps during etch-and-

rinse adhesive system application [17]. In addition, 35% phosphoric acid removes calcium from the dentin surface, leaving a network of collagen fibers surrounded by water [18]. The removal of calcium from the dentin surface might avoid any potential ionic bonding between the calcium and phosphate and/or carboxylate groups present in the adhesive, decreasing the bonding ability to dentin, especially after aging [19].

Okada et al. [20] compared the bonding efficacy of two self-etch one-step adhesives, one self-etch two-step adhesive and one etch and rinse two-step adhesive. According to their findings, the μ SBS of self-etch adhesive was higher to dentin compared with others. But in contrast to our findings, Yousry et al. [21] concluded that etch and rinse adhesive compared with self-etch adhesive, had better results in μ SBS to dentin.

Since the application protocols of adhesive systems on dentin substrate and dentin moisture play a significant role in mechanical and biological behavior of the adhesive interface, in the present study, we investigated the μ SBS of Scotchbond Universal adhesive system applied on wet and dry dentin following etch and rinse and self-etch bonding modes. According to our results, the application of Scotchbond Universal on dry dentin by the self-etch technique resulted in the highest μ SBS value, with no significant difference with Clearfil SE bond. Our study showed that self-etch universal adhesive showed almost similar results to self-etch adhesive in μ SBS [22]. In our study, self-etch mode yielded superior bond strength results to dentin in comparison with etch and rinse mode irrespective of the type of adhesive (universal or conventional).

The chemical bonding potential in self-etch adhesives has a positive effect on dentin bonding because of the adhesion of functional monomers to hydroxyapatite [23]. Among the currently used functional monomers, 10-methacryloyloxydecyl dihydrogen phosphate (MDP) has demonstrated a very effective and durable bond to dentin, due to the low solubility of the calcium salt that forms on the hydroxyapatite surface. On the other hand,

micromechanical interlocking by means of good dentin hybridization (i.e. resin tags and hybrid layer) has been proposed to improve the bond strength of adhesives in self-etch mode [24].

The composition of Clearfil SE Bond is similar to that of Scotchbond Universal containing MDP as a functional monomer. Although the components of both materials are the same, there may be differences in the quantities and proportions of water, solvent, MDP, and dimethacrylate resins in the adhesives. There is a possibility that such differences affect the viscosity and wettability of each bonding agent, affecting the ability of resin monomers to penetrate into decalcified dentin [24].

Low-quality hybridization in dentin occurs following the application of total-etch approach, which is characterized by formation of a porous and poorly resin-infiltrated collagen network. Thus, in use of total-etch adhesives, dental clinicians should be careful about additional phosphoric acid etching of dentin because it can make the adhesive interface highly susceptible to biodegradation. [25].

The universal adhesive showed lower μ SBS to dentin in etch and rinse mode in our study. Generally, when the dentin surface is pre-etched with phosphoric acid, the resin components of the self-etch adhesive are prevented from penetrating into the exposed collagen network, leading to a reduction in bond strength [26,27]. Adper Single Bond had the lowest μ SBS among the adhesive groups in our study. One reason for significantly lower dentin bond strength of etch and rinse systems is the suboptimal infiltration of resin into the demineralized collagen network and subsequently poor adaptation of the bonding resin to the collagen fibrils. The lower bond strength of Adper Single Bond could be explained by the absence of MDP functional monomer in its composition [28].

CONCLUSION

Within the limitations of this in vitro study, we may conclude that universal adhesive in self-etch mode may yield a μ SBS superior or almost equal to that of universal adhesive in etch and

rinse mode and two-step self-etching adhesive when using dentin as substrate. Also, an etching step prior to universal adhesive application insignificantly decreases the SBS to dentin.

CONFLICT OF INTEREST STATEMENT

None declared.

REFERENCES

1. Lynch CD, Frazier KB, McConnell RJ, Blum IR, Wilson NH. Minimally invasive management of dental caries: contemporary teaching of posterior resin-based composite placement in U.S. and Canadian dental schools. *J Am Dent Assoc.* 2011 Jun;142(6):612-20.
2. Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, et al. State of the art etch-and-rinse adhesives. *Dent Mater.* 2011 Jan;27(1):1-16.
3. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. *Dent Mater.* 2011 Jan;27(1):17-28.
4. Münchow EA, de Barros GD, da Silva Alves L, Valente LL, da Silva Cava S, Piva E, et al. Effect of elastomeric monomers as polymeric matrix of experimental adhesive systems: degree of conversion and bond strength characterization. *Appl Adhesion Sci.* 2014 Dec;2(1):3.
5. Alex G. Is total-etch dead? Evidence suggests otherwise. *Compend Contin Educ Dent.* 2012 Jan;33(1):12-4.
6. Loguercio AD, Barroso LP, Grande RH, Reis A. Comparison of intra- and intertooth resin-dentin bond strength variability. *J Adhes Dent.* 2005 Summer;7(2):151-8.
7. Pashley DH, Tay FR, Carvalho RM, Rueggeberg FA, Agee KA, Carrilho M, et al. From dry bonding to water-wet bonding to ethanol-wet bonding. A review of the interactions between dentin matrix and solvated resins using a macromodel of the hybrid layer. *Am J Dent.* 2007 Feb;20(1):7-20.
8. de Souza Costa CA, Hebling J, Scheffel DL, Soares DG, Basso FG, Ribeiro AP. Methods to evaluate and strategies to improve the biocompatibility of dental materials and operative techniques. *Dent Mater.* 2014 Jul;30(7):769-84.
9. Tuncer D, Yazici AR, Özgünaltay G, Dayangac B. Clinical evaluation of different adhesives used in the restoration of non-carious cervical lesions: 24-month results. *Aust Dent J.* 2013 Mar;58(1):94-100.
10. Chen C, Niu LN, Xie H, Zhang ZY, Zhou LQ, Jiao K, et al. Bonding of universal adhesives to dentine--Old wine in new bottles? *J Dent.* 2015 May;43(5):525-36.
11. Eliguzeloglu E, Omurlu H, Eskitascioglu G, Belli S. Effect of surface treatments and different adhesives on the hybrid layer thickness of non-carious cervical lesions. *Oper Dent.* 2008 May-Jun;33(3):338-45.
12. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent.* 2003 May-Jun;28(3):215-35.
13. Sensi LG, Lopes GC, Monteiro S Jr, Baratieri LN, Vieira LC. Dentin bond strength of self-etching primers/adhesives. *Oper Dent.* 2005 Jan-Feb;30(1):63-8.
14. Nunes MF, Swift EJ, Perdigão J. Effects of adhesive composition on microtensile bond strength to human dentin. *Am J Dent.* 2001 Dec;14(6):340-3.
15. Wagner A, Wendler M, Petschelt A, Belli R, Lohbauer U. Bonding performance of universal adhesives in different etching modes. *J Dent.* 2014 Jul;42(7):800-7.
16. Oilo G. Bond strength testing--what does it mean?. *Int Dent J.* 1993 Oct;43(5):492-8.
17. Nicoloso GF, Antoniazzi BF, Lenzi TL, Soares FZ, Rocha RO. Is There a Best Protocol to Optimize Bond Strength of a Universal Adhesive to Artificially Induced Caries-affected Primary or Permanent Dentin? *J Adhes Dent.* 2016;18(5):441-446.
18. Marchesi G1, Frassetto A, Mazzoni A, Apolonio F, Diolosà M, Cadenaro M, Di Lenarda R, et al. Adhesive performance of a multi-mode adhesive system: 1-year in vitro study. *J Dent.* 2014 May;42(5):603-12.
19. Manfroi FB, Marcondes ML, Somacal

- DC, Borges GA, Júnior LH, Spohr AM. Bond Strength of a Novel One Bottle Multi-mode Adhesive to Human Dentin After Six Months of Storage. *Open Dent J.* 2016 Jun 6;10:268-77.
20. Okada H, Sadr A, Shimada Y, Tagami J. Micro-shear bond strength of current one-step adhesives to cementum and dentin. *Am J Dent.* 2009 Oct;22(5):259-63.
21. Yousry MM, ElNaga AA, Hafez RM, El-Badrawy W. Microshear bond strength and interfacial morphology of etch-and-rinse and self-etch adhesive systems to superficial and deep dentin. *Quintessence Int.* 2011 Oct;42(9):e96-e106.
22. Ikeda M, Kurokawa H, Sunada N, Tamura Y, Takimoto M, Murayama R, et al. Influence of previous acid etching on dentin bond strength of self-etch adhesives. *J Oral Sci.* 2009 Dec;51(4):527-34.
23. do Amaral RC, Stanislawczuk R, Zander-Grande C, Gagler D, Reis A, Loguercio AD. Bond strength and quality of the hybrid layer of one-step self-etch adhesives applied with agitation on dentin. *Oper Dent.* 2010 Mar-Apr;35(2):211-9.
24. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res.* 2004 Jun;83(6):454-8.
25. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, et al. Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine. *J Dent.* 2012 Jun;40(6):475-84.
26. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. *J Dent.* 2006 Jan;34(1):77-85.
27. Van Landuyt KL, Peumans M, De Munck J, Lambrechts P, Van Meerbeek B. Extension of a one-step self-etch adhesive into a multi-step adhesive. *Dent Mater.* 2006 Jun;22(6):533-44.
28. Takamizawa T, Barkmeier WW, Tsujimoto A, Berry TP, Watanabe H, Erickson RL, et al. Influence of different etching modes on bond strength and fatigue strength to dentin using universal adhesive systems. *Dent Mater.* 2016 Feb;32(2):e9-21.