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Micro-shear bond strength of composite to deep dentin by using mild and ultra-mild universal adhesives

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ABSTRACT

Background: Considering the increasing demand for tooth-colored restorations, this study sought to assess the micro-shear bond strength (μSBS) of composite to deep dentin by using mild and ultra-mild universal adhesives.

Materials and Methods: This *in vitro*, experimental study evaluated 75 sound third molar teeth randomized into five groups (n = 15) of Clearfil self-etch (SE) Bond (control), Tokuyama Universal Bond (TUB), and All-Bond Universal (ABU) in etch and rinse (E and R) and SE modes. The occlusal surfaces of the teeth were trimmed to expose deep dentin at 0.5 mm distance from the pulp chamber. Adhesives were applied on the dentin surface according to the manufacturers' instructions, and Z350XT composite cylinders were bonded to dentin using Tygon tubes (0.9 mm internal diameter, 2 mm height). After incubation at 37°C and 100% humidity for 24 h, the teeth underwent μSBS test in a universal testing machine. The mode of failure was also determined under a stereomicroscope. Analysis of variance, Bonferroni test, and Fisher's exact test were applied for data analysis (alpha = 0.05).

Results: TUB in E and R mode (13.78 MPa) and ABU in SE mode (7.85 MPa) yielded the maximum and minimum μSBS values, respectively. No significant difference was noted in μSBS of tested universal adhesives in E and R and SE modes to deep dentin ($P > 0.05$). TUB yielded significantly higher μSBS than ABU ($P < 0.05$). No significant difference was noted in mode of failure of the groups ($P > 0.05$).

Conclusion: Type of adhesive affected the μSBS but etching mode of universal adhesives had no significant effect on their μSBS to deep dentin.

Key Words: Clearfil self-etch bond, Composite Resins, Dentin, Dentin Bonding Agents, Shear Strength

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INTRODUCTION

Due to the growing demand of patients for conservative, tooth-colored restorations, direct application of composite resins for tooth restoration has greatly increased in the past couple of decades; however, the success of composite restorations depends on the advances in adhesive systems.[1] Dental adhesive systems can be categorized into two groups of etch and rinse (E and R) and self-etch (SE) according to their bonding protocol. E and R adhesive

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systems are still the gold-standard dental bonding agents; however, attempts are ongoing by the manufacturers to simplify the application of adhesives by the development of efficient SE systems. The available SE bonding systems are categorized into three groups of strong (pH <1), moderate (pH between 1 and 2), mild (pH = 2), and ultra-mild (pH >2.5), depending on their depth of dentin demineralization.

Universal adhesives are the latest generation of dental adhesives available in the market.[2-4] They can be used for bonding to several types of substrates such as enamel, dentin, metal, and ceramic, and can be used in SE or E and R mode. Different manufacturers have produced different types of universal adhesives with different pH values.[1,2,5] The composition of universal adhesives is different from that of conventional SE adhesives, and most of them contain carboxylate monomers or phosphate compounds that can form ionic bonds with calcium present in the composition of hydroxyapatite.[6]

The presence of a high-quality hybrid layer is imperative for a strong bond between dentin substrate and resin restorative material. Dentin can be divided into superficial dentin at 0.5–1 mm distance from the dentinoenamel junction, deep dentin at 0.5 mm distance from the pulp chamber, and intermediate dentin located between the superficial and deep dentin at 0.5–1 mm distance from the superficial dentin.[7] Evidence shows that the density of dentinal tubules is different in superficial and deep dentin. Furthermore, the water content of superficial dentin is lower than that in deep dentin. The amount of collagen decreases from the outermost towards the innermost dentin. Thus, a reduction in bond strength is expected in deep dentin, compared with superficial dentin.[7,8]

Shear bond strength (SBS) and tensile bond strength tests are often used to measure the bond strength provided by different adhesives to dental substrate.[1] In these tests, increasing loads are applied to the bonding interface until debonding.[4,9]

Considering the variations in the pH value and acidity of different universal adhesives available in the market, controversy exists regarding the most efficient universal adhesive for the strongest bond to deep dentin. Concerning the gap of information regarding the bond strength of different universal adhesives in E and R and SE modes to deep dentin, this study aimed to compare the micro-SBS (μSBS) of two commonly used universal adhesives with different pH values in E and R and SE modes to deep dentin, in comparison with a conventional adhesive system. The first null hypothesis of the study was that there would be no significant difference in μSBS of universal adhesives to deep dentin in comparison with a conventional adhesive system. The second null hypothesis was that there would be no significant difference in μSBS of universal adhesives to deep dentin in SE and E and R modes.

MATERIALS AND METHODS

This *in vitro*, the experimental study evaluated 75 human third molars extracted for purposes not related to this study. The study protocol was approved by the ethics committee of Islamic Azad University, School of Dentistry, Khorasgan Branch (IR.IAU.KHUISF.REC.1397.107). Sample size was calculated to be 15 in each group assuming alpha = 0.05, beta = 0.10, and d = 5.8.

The inclusion criteria were extracted third molars with no caries, restorations, or cracks. After collection, the teeth were immersed in 0.1% thymol solution for disinfection. Soft tissue residues, debris, and calculus were removed by a scaler and a prophy brush, and the occlusal surface of the teeth was trimmed underwater coolant up to 1 mm distance from the pulp chamber. Next, a long fissure bur (Juya, Iran) was used to further remove dentin up to 0.5 mm distance from the pulp chamber to ensure accessing deep dentin. Deep dentin is located approximately 3 mm below the dentinoenamel junction, and the pulp chamber shadow can be seen through the dentin at 0.5 mm distance from the pulp chamber. The dentin surface was then polished with 600-grit waterproof silicon carbide abrasive paper (Starcke, Germany) for 60 s under copious water irrigation to create smear layer similar to clinical condition. At this point, the pulp chamber shadow was visible through the dentin. The teeth were then mounted in auto-polymerizing acrylic resin (Acropars, Malic Industries, Tehran, Iran) blocks with 2 mm diameter up to 2 mm below the trimmed area such that the buccal surface was perpendicular to the acrylic surface. The teeth were then randomly divided into five groups (n = 15) for the application of different adhesives in E and R and SE modes:

Group 1 (control): Clearfil SE Bond (Kuraray, Japan) with a pH of 2 was used in this group. Clearfil SE Bond primer was first applied, left for 20 s, and dried with mild airflow. Clearfil SE Bond bonding agent was then applied, followed by gentle airflow, and light-curing for 10 s.

Group 2: Preetching was performed with 35% phosphoric acid, which was applied on the surface for 10–15 s, and washed with air/water spray to eliminate the smear layer. (Tokuyama Universal Bond [TUB]; Tokuyama Dental, Japan) with a pH of 2.2 was then applied. For this purpose, the adhesive was dispensed from the bottles A and B in 1:1 ratio on a mixing pad and mixed. The adhesive was then applied on the surface and gently air-dried for 5 s.

Group 3: TUB in SE mode was applied in this group as explained for Group 2.

Group 4: Preetching with phosphoric acid was first performed as explained for Group 2. Next, (All-Bond Universal [ABU]; Bisco Inc., Schaumburg, IL, USA) with a pH of 3.3 was used. For this purpose, two separate coats of ABU were applied and scrubbed with a microbrush for 10–15 s per coat. Air-drying was then performed for 10 s followed by light-curing for 10 s.

Group 5: ABU in SE mode was applied in this group as explained for Group 4.

A plastic tube (Tygon tube; Norton Inc., USA) with an internal diameter of 0.9 mm and 2 mm height was then used to apply composite on the dentin surface. A2 shade of Z350XT Composite resin (3M ESPE, St. Paul, MN, USA) was condensed into the tube by a plugger. Care was taken to prevent the formation of voids or gaps at the composite-tooth interface. Next, the composite was light-cured for 20 s using a curing unit (Demi Plus; Kerr, USA) with 1100 mW/cm^2 light intensity. The teeth were then incubated at 37°C and 100% humidity for 24 h. To measure the μSBS, the teeth were placed in a universal testing machine (Instron, USA). The load was applied to the bonding interface at a crosshead speed of 0.5 mm/min until debonding. The maximum load causing debonding was recorded in Newtons and divided by the cross-sectional area of composite cylinders in square millimeters to calculate the μSBS in megapascals. The mode of failure was then determined under a stereomicroscope (SDF PLAPO 1XPF; Olympus; Japan) at x40 magnification. The debonded surface was also photographed. The mode of failure was categorized as adhesive (failure at the adhesive-substrate interface), cohesive (within the material mass), and mixed (a combination of adhesive and cohesive) [Figure 1].

The normal distribution of data was evaluated by the Kolmogorov–Smirnov test, which confirmed normal data distribution. Thus, a comparison of μSBS data was performed using one-way Analysis of variance (ANOVA), followed by Bonferroni test for pairwise comparisons. The mode of failure was compared among the groups by the Fisher's exact test. All statistical analyses were performed by SPSS version 24 at 0.05 level of significance.

RESULTS

Table 1 shows the mean μSBS of adhesives to deep dentin. TUB in E and R mode (13.78 MPa) and ABU in SE mode (7.85 MPa) yielded the maximum and minimum μSBS values, respectively. A significant difference was noted in the mean μSBS of the groups (one-way ANOVA; $P = 0.001$). Table 2 presents pairwise comparisons of the groups regarding μSBS. As shown, no significant difference was noted in μSBS of tested universal adhesives in E and R and SE modes to deep dentin ($P > 0.05$). TUB yielded significantly higher μSBS than ABU ($P < 0.05$).

Table 3 shows the mode of failure of the study groups. Mixed failure had the highest frequency. The Fisher's exact test revealed no significant difference in the mode of failure among the groups ($P = 0.949$).

DISCUSSION

The need for a reliable bonding agent is undeniable due to increased demand for tooth-colored durable restorations.[10] Dentin bonding has always been challenging due to the complex biological composition of dentin.[11] This study assessed the μSBS of composite to deep dentin by using mild and ultra-mild universal adhesives. The obtained μSBS values in all groups were lower than the corresponding values reported in some recent studies on bond strength to intermediate and superficial dentin.[1,10,12] This finding can be explained by the different organic and mineral composition, and water content of deep dentin (at 0.5 mm distance from the pulp chamber) compared with intermediate and superficial dentin, which affect the bond strength.[11,13] Superficial dentin has smaller number of dentinal tubules, and as we know, resin penetration into inter-tubular dentin increases the bond strength. Deep dentin, close to the pulp chamber, contains large funnel-shaped dentinal

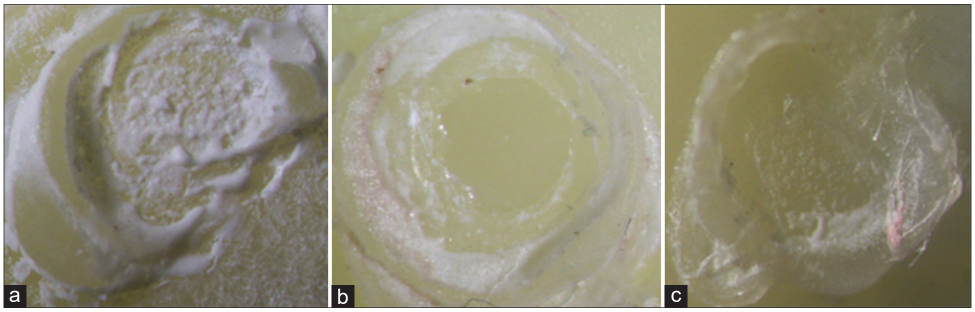

Figure 1: (a) Cohesive failure, (b) adhesive failure, (c) mixed failure

Table 1: Mean micro-shear bond strength (MPs) of adhesives to deep dentin

Variable	Mean±SD					*F*	*P*
	Control	Tokuyama Etch and Rinse	Tokuyama SE	All bond universal Etch and Rinse	All bond universal SE		
Bond strength	11.96±3.91	13.78±5.11	12.41±3.71	8.099±2.33	7.85±2.27	8.216	0.001

SE: Self-etch, SD: Standard deviation

Table 2: Pairwise comparisons of the groups regarding micro-shear bond strength (MPs)

Group (I)	Group (J)	Mean difference (I-J)	SD	*P*
Control	Tokuyama universal bond Etch and Rinse	−1.82	1.32	0.99
	Tokuyama universal bond SE	−0.44	1.32	0.99
	All bond universal Etch and Rinse	3.85	1.32	0.048*
	All bond universal SE	4.10	1.32	0.028**
Tokuyama universal bond Etch and Rinse	Tokuyama universal bond SE	1.37	1.32	0.99
	All bond universal Etch and Rinse	5.68	1.32	0.001**
	All bond universal SE	5.93	1.32	0.001**
Tokuyama universal bond SE	All bond universal Etch and Rinse	4.30	1.32	0.018**
	All bond universal SE	4.55	1.32	0.01*
All bond universal Etch and Rinse	All bond universal SE	0.24	1.32	0.99

*Significant difference at 0.05 level, **Significant difference at 0.01 level. SE: Self-etch, SD: Standard deviation

Table 3: Modes of failure of the study groups

Group	Mode of failure (%)			*P*
	Adhesive	Cohesive	Mixed	
Control	9 (60.0)	1 (6.7)	5 (33.3)	0.949
Tokuyama universal bond Etch and Rinse	9 (60.0)	1 (6.7)	5 (33.3)	
Tokuyama universal bond SE	8 (53.3)	2 (13.3)	5 (33.3)	
All bond universal Etch and Rinse	7 (46.7)	1 (6.7)	7 (46.7)	
All bond universal SE	8 (53.3)	0	7 (46.7)	

SE: Self-etch

tubules and resultantly lower amount of inter-tubular dentin. Considering the significant role of inter-tubular dentin in hybrid layer formation, and the fact that resin interlocking has a direct correlation with the available amount of inter-tubular dentin for bonding, lower bond strength to deep dentin is expected.[11] Kumari *et al*.[8] compared the mean bond strength of 7th generation bonding agents to superficial and deep dentin and reported a significantly lower bond strength to deep dentin. Singh *et al*.[7] measured the SBS of SE primers to superficial, intermediate, and deep dentin and reported a significant reduction in bond strength to intermediate and deep dentin, compared with superficial dentin.

The manufacturers of universal adhesives allow dental clinicians to opt for SE or E and R mode in use of these adhesives, depending on their clinical judgment.[2] However, the results of studies regarding the bond strength of universal adhesives in SE and E and R modes are controversial[1,7,14]. Kaczor *et al*.[15] evaluated the effect of different etching modes on nano-leakage of universal adhesives in a systematic review. They showed that the E and R mode significantly decreased the nanoleakage of Peak Universal and G-Bond Plus adhesives, among the 7 adhesives tested. However, the nanoleakage of ABU was lower in SE mode. In Prime and Bond Elect and Scotchbond Universal, no significant difference was noted in nano-leakage between the two etching modes. Atalay *et al*.[16] measured the bond strength of Scotchbond Universal adhesive in E and R and SE modes for noncarious cervical lesions. They concluded that this adhesive yielded acceptable clinical performance in all modes; however, its application in SE mode resulted in greater discoloration and marginal degradation than the E and R mode. On the other hand, De Paris Matos *et al*.[17] evaluated the 5-year application of Scotchbond Universal in noncarious cervical lesions, and concluded that its application in E and R was superior to its application in SE mode. Valizadeh *et al*.[18] measured the μSBS of Single Bond, Scotchbond Universal, and Clearfil SE Bond in E and R and SE modes, and reported that the μSBS values were maximum in SE mode; although the difference was not significant. The authors preferred the use of SE mode due to lower demineralization of dentin. The current results revealed a significant difference in μSBS of different adhesive systems to deep dentin. Thus, the first null hypothesis of the study was rejected. However, although the bond strength of tested universal adhesives to deep dentin was higher in E and R mode compared with SE mode, this difference was not statistically significant ($P > 0.05$). Thus, the second null hypothesis of the study was accepted. This result indicates that the application of TUB and ABU universal adhesives in E and R mode has no adverse effect on their μSBS to deep dentin. Takamizawa *et al*.[1] reported a reduction in bond strength of Clearfil SE Bond when applied in E and R mode. They also measured the bond strength of Prime and Bond Elect, Scotchbond Universal, and ABU to dentin and reported a higher SBS for Prime and Bond Elect when applied in E and R mode; however, the bond strength of the other two adhesives was not significantly different in E and R and SE modes. Their result regarding ABU was in agreement with our finding. On the other hand, Wagner *et al*.[3] found that preetching improved the penetration depth of universal adhesives into dentin; however, it did not affect the bond strength after 24 h of storage or 5000 thermal cycles. Chen *et al*.[5] evaluated the short-term performance of five universal adhesives namely Prime and Bond Elect, Scotchbond Universal, ABU, Clearfil Universal Bond, and Futurabond U to coronal dentin, and concluded that their application mode (E and R or SE) had no significant effect on their microtensile bond strength to dentin. Ahmed *et al*.[19] reported that among different universal adhesives, Futurabond and Tetric N-Bond Universal yielded higher bond strength in E and R mode compared with SE mode; however, Single Bond Universal showed no significant change in μSBS in SE and E and R modes. In general, the results of previous studies support an increase in bond strength in E and R application mode of adhesives for the more mineralized structure of enamel and simplified SE mode for dentin bonding.[1,13,20]

In the present study, TUB in E and R mode yielded the maximum μSBS (13.78 MPa) while ABU in SE mode yielded the minimum μSBS (7.85 MPa); the bond strength of Clearfil SE Bond (CSB) was 11.96 MPa.

High bond strength of universal adhesives is probably attributed to the presence of methacryloyloxydecyl dihydrogen phosphate (MDP) monomer in their structure. The chemical reaction of MDP with hydroxyapatite leads to the formation of a stable nano-layer, forming a strong phase on the adhesive surface and increasing the mechanical strength of bonding. Moreover, the stable structure of MDP-Ca along with the nano-layer explains the high bond strength of these adhesives. CSB was the first adhesive containing MDP. Evidence shows that MDP is effective in formation of a durable chemical bond to dentin.[19] The composition of CSB is similar to that of ABU, containing MDP functional monomer. Thus, the difference in their bond strength may be related to the difference in the amount of water, solvent, MDP, and dimethacrylate resins in their composition. Such differences can affect the viscosity and wettability of these adhesives, and change the penetration depth of resin monomer into decalcified dentin.[1] Also, evidence shows that increase in bond strength after removal of collagen from demineralized dentin surface depends on the type of bonding system and particularly the type of solvent in its composition.[21] TUB showed higher μSBS than other groups in our study. This adhesive was recently introduced to the market, and studies regarding its efficacy are limited. It is supplied in two bottles, and the manufacturer claims that it forms a strong bond to different substrates due to its unique composition.[12] It has phosphoric acid monomer (new 3D-SR monomer) instead of MDP in its composition. Furthermore, this monomer has several functional groups, which can bond to calcium and polymerized groups in each molecule, increasing the durability of the bond to calcium.[12] It appears that this monomer provides higher bond strength than MDP. Based on the current results and some previous investigations, the bond strength to deep dentin depends on the type of bonding agent,[1,10,19] which is *per* SE attributed to the composition of the bonding agent and ratio of its constituents.

In the E and R technique with phosphoric acid, the smear layer that is formed on dentin surface during cavity preparation is lost. At the same time, 3–5 μm of the dentin surface is demineralized. The collagen fibers covered with hydroxyapatite are exposed in this process, and a small network is formed, which enables micromechanical interlocking of monomers.[14]

In the SE mode, demineralization and monomer penetration into dentin occur at the same time, which decreases technical sensitivity and shortens the treatment time. The available SE bonding systems are categorized into three groups of strong (pH <1), moderate (pH between 1 and 2), mild (pH = 2), and ultra-mild (pH >2.5), depending on their depth of dentin demineralization.[18] The strong SE systems have a highly acidic pH and cause deep dentin demineralization. They create an etched pattern similar to that caused by phosphoric acid. Mild SE adhesives, however, partially and superficially demineralize the dentin surface, and thus, part of the smear layer and smear plug remain intact, and high amounts of hydroxyapatite crystals around the collagen fibers are preserved. The pH of SE adhesives is an important factor affecting the solubility of the smear layer and dentin demineralization. In total, mild (pH >2) and ultra-mild (pH >2.5) SE adhesives are highly effective for bonding to dentin.[19,20]

This study had some limitations. Due to its *in vitro* design, intra-tubular pressure of dentin, and humidity of the oral environment could not be simulated, which can affect the results and decrease their generalizability to the clinical setting. Also, not having a cutting machine to create samples with a size smaller than 1 mm, and using an alternative method for specimen preparation might have affected the results. Last but not least, thermocycling or long-term water storage were not performed to better simulate the oral environment. Future studies are required to address the above mentioned limitations to increase the reliability of the results.

CONCLUSION

The type of universal adhesive (mild/ultra-mild) affects the μSBS to deep dentin. TUB yielded maximum μSBS. Mode of application of universal adhesives (E and R or SE) had an insignificant effect on μSBS to deep dentin.

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Conflicts of interest

There are no conflicts of interest.

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