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Microshear Bond Strength of Composite to Superficial Dentin by Use of Universal Adhesives with Different pH Values in Self-Etch and Etch & Rinse Modes

Mahtab Hosseini[1], Zahra Raji[2], Mehrdad Kazemian[2]

[1]Department of Operative Dentistry, Isfahan (Khorasgan) Branch, Islamic Azad University, Isfahan, Iran, [2]Department of Operative Dentistry, Borujerd Branch, Islamic Azad University, Borujerd, Iran

ABSTRACT

Background: This study aimed to assess the microshear bond strength (micro-SBS) of two universal adhesives with different pH values in etch-and-rinse (E and R) and self-etch (SE) modes to superficial dentin.

Materials and Methods: This *in vitro* experimental study evaluated 75 extracted sound human third molars. Superficial dentin was exposed by trimming the enamel and removing 0.5–1 mm of tooth structure beyond the dentinoenamel junction. A 600-grit abrasive paper was used to create smear layer. The teeth were randomly divided into five groups (n = 15). All-Bond Universal and G-Premio Bond were applied in E and R and SE modes in four groups. Clearfil SE Bond was used in the control group. Z350XT composite cylinders (0.9 mm diameter and 1.5 mm height) were then bonded to the prepared surface. The teeth were incubated at 37°C and 100% humidity for 24 h. Micro-SBS was measured by a universal testing machine, and the mode of failure was determined under a stereomicroscope. Data were analyzed using ANOVA, Bonferroni test, and Fisher's exact test (alpha = 0.05).

Results: The micro-SBS of Clearfil SE Bond was significantly lower than all other groups ($P < 0.05$). All-Bond Universal yielded the maximum micro-SBS in SE and minimum micro-SBS in E and R mode. All-Bond Universal showed significantly lower micro-SBS in E and R mode than SE mode ($P < 0.05$). No other significant differences were noted. The mode of failure was also significantly different among the groups ($P < 0.05$). Mixed failure had the highest frequency in G-Premio in E and R mode.

Conclusion: Type of adhesive and application mode affected the micro-shear bond strength to superficial dentin.

Key Words: Clearfil self-etch bond, dentin, shear strength

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Address for correspondence:
Dr. Mehrdad Kazemian, Department of Operative Dentistry, Isfahan (Khorasgan) Branch, Islamic Azad University, Isfahan, Iran.
E-mail: mehrkaz@gmail.com

INTRODUCTION

In the past decades, application of direct composite restorations has greatly increased due to higher demand of patients for tooth-colored, conservative restorations. At present, adhesive restorations with direct bonding to tooth structure are extensively used for the anterior and posterior teeth.[1,2] Success of bonded restorations largely depends on optimal bond strength between the tooth structure and restorative

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material. A high-quality restoration should provide optimal marginal seal, reinforce the tooth structure, and have long-term durability.[2]

The bond strength between the restorative material and tooth structure depends on a number of factors such as the amount of available enamel for bonding, smear layer, organic content of the tooth structure, and intratubular fluid.[3] A durable bond to enamel can be achieved; however, optimal bonding to dentin is more challenging due to its heterogenic nature.[3]

Despite the extensive literature available regarding the efficacy of universal adhesives and their bond strength to enamel and dentin, information regarding the quality of adhesive bonding provided by universal adhesives applied in self-etch (SE) and etch-and-rinse (E and R) modes by use of fatigue test is limited.[1]

Evidence shows a reduction in bond strength to dentin from the outermost to the innermost layer of dentin, and the shear bond strength (SBS) to superficial dentin is reportedly higher than that to deep dentin.[1,2] This finding is due to the difference in density of dentinal tubules in superficial and deep areas. Furthermore, the water content of the superficial dentin is much lower than that of deep dentin (due to increased mineral content of peritubular dentin). Moreover, the collagen content is minimal in deep dentin due to increased diameter of dentinal tubules.[1-3]

Universal or multimode adhesives are a relatively new type of one-step adhesives with two main features: first, they can be used for bonding to a wide range of substrates, and particularly for composite restorations when bonding to different substrates is required; second, they can be used in SE or E and R mode, depending on the clinicians' judgment.[1] Universal adhesives contain multipotential monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which are responsible for their durability and creation of an interface (hybrid layer) resistant to hydrolysis over time.[4] However, considering their relatively recent introduction to the market, no consensus has been reached regarding the superiority of one particular etching mode (SE or E and R) over the other in use of universal adhesives.[1] It has been reported that application of SE universal adhesives in E and R mode has no adverse effect on primary bond strength or fatigue resistance.[1] On the other hand, Clearfil SE Bond, which is a SE adhesive, has shown significantly lower SBS and fatigue resistance when applied in E and R mode.[1]

Although E and R adhesives are the oldest adhesives available in the market and are still the gold standard for dental adhesion, dental clinicians increasingly opt for simplified SE dental adhesives.[5] SE adhesives are categorized into four groups based on their acidity and the ability to demineralize dentin: weak adhesives (pH >2.5) capable of dentin demineralization by a few hundred nanometers, mild adhesives (pH of almost 2) with around 1 μm penetration depth, moderate adhesives (pH of 1–2) by 1–2 μm penetration depth, and strong adhesives (pH <1) with several micrometers penetration depth. The resin tags formed in dentinal tubules are only reliable when strong SE adhesives are used. Weak and mild adhesives cannot form reliable resin tags. They only demineralize the smear plugs to some extent and allow limited penetration of resin.[6] Several manufacturers produce universal adhesives with different pH values,[5] which calls for further investigation regarding their efficacy and clinical service.

Micro-SBS test is commonly used to assess the bond strength of different dentin bonding agents. This test has greater advantages than the SBS test.[7] Considering the different pH values of different universal adhesives available in the market, this study sought to assess the micro-SBS of different universal adhesives with different pH values in E and R and SE modes to superficial dentin, in comparison with a conventional adhesive system. The null hypothesis was that no significant difference would be found in micro-SBS of different adhesives to superficial dentin.

MATERIALS AND METHODS

This *in vitro* experimental study evaluated 75 extracted human third molars. The study was approved by the ethics committee of Islamic Azad University, School of Dentistry, Khorasgan Branch (IR.IAU.KHUISF.REC.1397.096). The sample size was calculated to be 75 (n = 15 in each of the five groups) assuming alpha = 0.05, beta = 0.1, and d = 5.8.

The inclusion criterion was sound extracted human third molars stored in distilled water since their extraction. The teeth were cleaned from tissue residues and debris by a prophy brush and mounted in auto-polymerizing acrylic resin (Acropars, Tehran, Iran) blocks measuring 2 cm × 2 cm. The

enamel surface of the teeth was trimmed by a trimmer (Dandiran, Tehran, Iran) underwater coolant to the dentinoenamel junction and was then reduced by 0.5–1 mm beyond the dentinoenamel junction by a long diamond fissure bur (Tees Kavan, Tehran, Iran) to obtain a homogenously smooth surface of superficial dentin. Dentin surface was then ground with 600-grit silicon carbide waterproof abrasive paper (Starcke, Germany) for 1 min underwater coolant to create smear layer, simulating the clinical setting. The teeth were then randomly assigned to five groups (n = 15) as follows:

Group 1 (control group): 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: Etching with 35% phosphoric acid (Ultra-Etch; Ultradent, USA) was first performed. The etchant was applied on the surface for 10–15 s and was then washed with air/water spray for 15 s to completely remove the smear layer. Next, G-Premio (GC, USA) universal adhesive with a pH of 1.5 was used. For this purpose, G-Premio Bond was applied and after waiting for 10 s, air-drying was performed for 5 s with maximum air pressure, followed by 10 s of light curing.

Group 3: SE application of G-Premio Bond with a pH of 1.5 as explained for Group 2.

Group 4: Etching with 35% phosphoric acid was first performed as explained for Group 2 and was followed by the application of All-Bond Universal (Bisco, USA) adhesive with a pH of 3.3. For this purpose, 2 separate coats of All-Bond Universal were applied. The preparation was scrubbed with a microbrush for 10–15 s/coat. Air drying was then performed for at least 10 s followed by 10 s of light curing.

Group 5: SE application of All-Bond Universal adhesive with a pH of 3.3 as explained for Group 4.

Table 1 shows the composition of adhesives used in this study. All adhesives were applied on the dentin surface according to the manufacturers' instructions. Next, a Tygon tube measuring 1.5 mm in height and 0.9 mm in diameter was used as a mold for the application of composite resin. The mold was placed on the prepared dentin surface and composite resin (Z350XT; Filtek, 3M ESPE, St. Paul, MN, USA) was applied into it by a plugger (Juya, Tehran, Iran). Before curing of composite, the mold was removed from the surface. To prevent the separation of composite cylinder from the surface when removing the mold, the composite cylinder was held in place by a plugger. It was then cured for 20 s by a LED curing unit (Demi Plus; Kerr Dental, Orange, CA, USA) with a light intensity of 1100 mW/cm^2.

Table 1: Composition of adhesives used in this study

Adhesive type	Composition and pH
Clearfil SE bond (Kuraray)	Primer: Water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate Bonding: MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N, N-diethanol p-toluidine bond, colloidal silica(pH=2)
GC G-Premio bond (GC)	MDP, 4-MET, MEPS, methacrylate monomer Acetone, water, initiator, silica. (pH=1.5)
All bond universal (Bisco)	Bis-GMA, 10-MDP, HEMA, ethanol, initiators, water (pH=3.3)

SE: Self-etch, MDP: Methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-Hydroxyethylmethacrylate, GMA: Bisphenol A-glycidyl methacrylate, MET: 4-Methacryloyloxyethyl trimellitic acid, MEPS: Methacryloyloxyalkyl thiophosphate methylmethacrylate.

The teeth were then incubated at 37°C and 100% humidity for 24 h and then underwent micro-SBS test in a universal testing machine (STM-20; Santam, Iran). Load was applied to the composite-tooth interface at a crosshead speed of 0.5 mm/min until fracture. Maximum load displayed on the monitor at the time of fracture (in Newtons) was recorded and divided by the cross-sectional area of the composite cylinder (in square-millimeters) to determine the micro-SBS value in megapascals (MPs). The mode of failure was determined under a stereomicroscope (SZX10; Olympus, Japan) at ×40 magnification and categorized as adhesive (debonding at the interface), cohesive (debonding within the tooth structure or composite mass), and mixed (a combination of adhesive and cohesive failures).

Data were analyzed by SPSS version 24 (SPSS Inc., IL, USA). Normal distribution of data was evaluated by the Kolmogorov–Smirnov test. Since data were normally distributed, the groups were compared regarding the micro-SBS using ANOVA. Pairwise comparisons were performed by the Bonferroni test. The mode of failure was compared among the groups by the Fisher's exact test. Level of significance was set at 0.05.

RESULTS

Table 2 shows the mean micro-SBS of different adhesive groups. As shown, the maximum micro-SBS value was noted in All-Bond Universal in SE mode, and the minimum micro-SBS value was recorded in All-Bond Universal in E and R mode. Considering the normal distribution of micro-SBS data ($P > 0.05$), ANOVA was applied to compare the micro-SBS of the groups, which revealed a significant difference ($P = 0.001$). Thus, pairwise comparisons were performed by the Bonferroni test [Table 3]. The micro-SBS of Clearfil SE Bond was significantly lower than all other groups ($P < 0.05$). All-Bond Universal showed significantly lower micro-SBS in E and R mode than SE mode ($P < 0.05$).

Table 4 shows the mode of failure in the study groups. The Fisher's exact test showed a significant difference in mode of failure of the study groups ($P < 0.05$). As shown, mixed failure had the highest frequency in G-Premio in E and R mode.

DISCUSSION

This study assessed the micro-SBS of two universal adhesives with different pH values to superficial dentin, in comparison with a conventional adhesive system. The results showed a significant difference in micro-SBS of the groups. Thus, the null hypothesis of the study was rejected. The micro-SBS of Clearfil SE Bond was significantly lower than all other groups ($P < 0.05$). All-Bond Universal yielded the maximum SBS in SE and minimum SBS in E and R mode ($P < 0.05$). No other significant differences were noted.

The current results showed optimally high micro-SBS to superficial dentin in all groups, which can be due to smaller number and narrower dentinal tubules as well as the lower water content of superficial dentin, which yields a high bond strength.[1-3] Moreover, due to higher amount of inter-tubular dentin in superficial dentin surface, a more appropriate hybrid layer is often formed, which brings about higher bond strength.[8]

The amount of sclerotic dentin is another parameter affecting resin penetration and bond strength. The amount of sclerotic dentin is higher in superficial dentin than deep dentin, which also explains higher bond strength.[9]

In this study, we confined the adhesive application area to the site of bonding of composite cylinder. Thus, the obtained micro-SBS values may be lower than those reported in studies that did not limit adhesive application to a specific area and applied adhesive over the entire dentin surface.[7] A previous study compared the micro-SBS of four adhesive systems to dentin, with and without limiting the adhesive application area. They reported that the micro-SBS was lower when the adhesive application area was limited to the composite-tooth structure interface, compared with the application of adhesive over the entire dentin surface.[7]

The three bonding agents evaluated in the present study, namely Clearfil SE Bond, All-Bond Universal, and G-Premio Bond are almost similar in terms of composition; but they are different in terms of pH since All-Bond Universal with a pH of 3.3 is categorized as a weak adhesive, G-Premio Bond with a pH of 1.5 is categorized as a moderate adhesive, and Clearfil SE Bond with a pH of 2 is categorized as a mild adhesive.[10-12] The present results showed that pH of different adhesive systems affected their micro-SBS to superficial dentin. The micro-SBS of Clearfil SE Bond (control group) with a pH of 2 was lower than that of G-Premio in SE and E and R modes, and also All-Bond Universal in SE mode. In G-Premio system with a pH of 1.5, no significant difference was noted in micro-SBS between its application in SE and E and R modes. The reason may be high acidity of this adhesive, and the resultantly strong demineralization, which minimizes the difference in the efficacy of SE and E and R modes of application of this adhesive. In All-Bond Universal with a pH of 3.3, the difference between SE and E and R modes was significant due to the low acidity of this adhesive. As the acidity of the adhesive system increases (pH decreases), the depth of demineralization and subsequently the resin

Table 2: Mean micro-shear bond strength (MPs) of different adhesive groups

Variable	Mean±SD					*F*	*P*
	Clearfil SE bond	GC G-Premio (E & R)	GC G-Premio (SE)	All bond universal (E & R)	All bond universal (SE)		
Micro-SBS	1.40±10.85	1.02±15.26	1.04±15.14	0.99±8.78	1.35±16.70	5.09	0.001

SBS: Shear bond strength, E & R: Etch and rinse, SE: Self-etch, SD: Standard deviation, GC: Corporation

Table 3: Pairwise comparisons of the groups regarding micro-shear bond strength (MPs) by the Bonferroni test

Group (I)	Group (J)	Mean difference (I-j)	SD	*P*
Clearfil SE bond	All bond universal (SE)	2.32	0.42	0.001*
	All bond universal (E & R)	−4.60	0.44	0.001*
	GC G-Premio (E & R)	−3.47	0.43	0.001*
	GC G-Premio (SE)	−3.38	0.43	0.001*
GC G-Premio (E & R)	All bond universal (SE)	5.79	0.44	0.001*
	All bond universal (E & R)	−1.13	0.43	0.119
	GC G-Premio (SE)	0.09	0.43	0.999
GC G-Premio (SE)	All bond universal (SE)	5.70	0.43	0.001*
	All bond universal (E & R)	−1.21	0.44	0.070
All bond universal (SE)	All bond universal (E & R)	−6.91	0.44	0.001*

*Statistically significant. E & R: Etch and rinse, SE: Self-etch, SD: Standard deviation, GC: Corporation

Table 4: Mode of failure in the study groups

Group	Mode of failure (%)			Total (%)	*P*
	Adhesive	Cohesive	Mixed		
Clearfil SE bond	6 (40)	2 (13.33)	7 (46.66)	15 (100.0)	0.040
GC G-Premio bond (E & R)	1 (6.66)	5 (33.33)	9 (60)	15 (100.0)	
GC G-Premio bond (SE)	2 (13.33)	5 (33.33)	8 (53.33)	15 (100.0)	
All bond universal (E & R)	8 (53.33)	3 (20)	4 (26.66)	15 (100.0)	
All bond universal (SE)	1 (6.66)	6 (40)	8 (53.33)	15 (100.0)	

E & R: Etch and rinse, SE: Self-etch, GC: Corporation

penetration increase. Therefore, when preetching is performed before the use of All Bond Universal, the demineralization depth increases but resin penetration remains the same, and it cannot penetrate into the entire created depth. Thus, the bond strength decreases.

All three adhesive systems evaluated in the present study contain 10-MDP, which is a multifunctional monomer with one hydrophobic and one hydrophilic end. It is the most hydrophobic functional monomer used in dental adhesives, which guarantees the bonding durability and prevents the hydrolysis of the adhesive interface over time.[4] Furthermore, hydrophobicity of 10-MDP improves its stability in the solution and increases its effective half-life. It is among the few monomers that form ionic bonds with calcium in the structure of hydroxyapatite in the tooth structure. This property helps in forming a resistant hybrid layer and is probably the reason for high clinical success rate of Clearfil SE Bond for over 8 years. Thus, universal adhesives containing MDP are also expected to bring about clinically successful results.[4,13]

In the present study, G-Premio with a mean micro-SBS of 15.26 MPa in E and R and 15.14 MPa in SE mode showed maximum micro-SBS after All-Bond Universal in SE mode. The presence of 4-methacryloyloxyethyl trimellitic acid in the composition of this adhesive is probably responsible for its high success rate. Four-methacryloyloxyethyl trimellitic acid is incorporated into the composition of bonding agents to serve as an adhesive agent as well as a demineralizing monomer. It enhances the wetting of metals such as amalgam and gold. Two carboxylic groups connected to an aromatic ring increase the acidity of this monomer, which explains its demineralizing and wetting effects. Nonetheless, the aromatic group is hydrophobic and balances the acidity and hydrophilicity of carboxyl groups. High acidity enhances the penetration depth of adhesive.[14]

In the present study, All-Bond Universal in SE mode yielded a mean micro-SBS of 16.70 MPa, which was the highest among all. It contains ethanol, which is not present in the composition of the other two adhesives evaluated in this study. A previous study evaluated the efficacy of ethanol for dentin wetting and its effect on adhesive bond strength before the application of universal adhesives. Application of ethanol resulted in higher micro-tensile bond strength even in long term. The mode of application of adhesive (SE or E and R) had no significant effect on the results. Thus, the presence of ethanol in the composition of All-Bond Universal may explain its optimal success rate.[15]

Evidence shows that mode of application of adhesives (SE or E and R) can also affect the resultant bond strength. Sabatini[16] evaluated the effect of phosphoric acid etching on SBS of FL-Bond II and Beauti Bond SE adhesives to enamel and dentin. They reported that preetching before the application of FL-Bond II SE adhesive on the enamel surface increased the bond strength; however, it had no significant effect on the bond strength of Beauti Bond to enamel. Preetching before the use of both adhesives on dentin decreased the bond strength. This can explain the lower micro-SBS of All-Bond Universal in

E and R mode in our study. Yousry *et al*.[17] evaluated the interface morphology and micro-SBS of SE and E and R adhesives to superficial and deep dentin. They reported that E and R adhesive systems yielded higher micro-SBS to deep dentin, compared with superficial dentin; however, the bond strength of SE systems was the same to superficial and deep dentin. The etching process eliminates high amounts of mineral content and decreases the amount of available calcium for the function of MDP monomer.

Another factor affecting the micro-SBS is resin penetration into dental substrate. The efficacy of preetching of dentin with phosphoric acid before the application of SE adhesives is a matter of debate since it may prevent adequately deep penetration of resin into etched dentin.[18] In this study, preetching before the application of All-Bond Universal decreased the micro-SBS; however, preetching had no significant effect on the bond strength of G-Premio.

Another factor with possible effect on micro-SBS to dentin is the cross-section of superficial dentin after trimming. Considering the mode of distribution of different types of dentin in a horizontal cross-section, we could not ensure homogenous presence of superficial dentin in our tested specimens. To date, no precise index has been suggested for this purpose to ensure homogenous presence of superficial or deep dentin. In previous studies, the presence of superficial dentin was confirmed by observation of dentinoenamel junction and exceeding it by 0.5–1 mm.

The results of assessment of modes of failure in the present study indicated the presence of an association between the micro-SBS values and the mode of failure. Higher frequency of adhesive failure in groups with lower micro-SBS confirmed weaker bond strength of adhesive to dentin in these groups. Future studies are required to trim the teeth longitudinally to ensure homogenous presence of superficial dentin. Furthermore, the adhesive surface should be photographed under an electron microscope to determine the depth of demineralization as well as the penetration depth of adhesive to cast a more reliable judgment in this respect.

CONCLUSION

Type of adhesive, and its application mode (SE versus E&R) both affected the micro-SBS to superficial dentin in All Bond Universal group but had no significant effect on micro-SBS in G-Premio Bond group.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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