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Comparative Evaluation of Application of Universal Bonding on the Microtensile Bond Strength of Light Cure and Dual Cure Composites Bonded to Dentin

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ABSTRACT

Objectives: There is limited knowledge on the efficacy of universal adhesives when used in different etching modes. The aim of this study was to evaluate the microtensile bond strength (µTBS) to dentin using universal adhesives with light-cure and dual-cure composites.

Materials and Methods: In this in vitro experimental study, the occlusal third of 60 caries-free human molars were removed and the exposed surfaces were treated and allocated to 10 groups (N=6). Two different bonding agents (Single Bond Universal, G-Premio) were used to bond 2 different composite resins (Z250 light-cure and CoreFlo DC dual-cure) to dentin, using etch-and-rinse (E&R) and self-etch (SE) modes. Single Bond 2 was applied in E&R mode on wet dentin to serve as control. The specimens were sectioned into 1×1 mm^2 sticks with a precision saw. A microtensile testing machine was used to measure the µTBS of the specimens with a crosshead speed of 0.5 mm/min. Data were analyzed using three-way ANOVA and Bonferroni post-hoc test (α=0.05).

Results: The Single Bond Universal group in SE mode with light-cure composite yielded the highest (39.24MPa), and the G-Premio SE group with dual-cure composite produced the lowest (13.65MPa) mean µTBS ($P<0.05$). The dominant failure mode was adhesive, in all groups.

Conclusion: The bonding agent had a significant effect on µTBS of composite to dentin, but different composites and different etching modes were not significantly different in this respect.

Keywords: Composite Resins; Dental Bonding; Tensile Strength

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INTRODUCTION

The use of direct composite resins has increased due to increased propensity of patients to tooth-colored and conservative restorations. However, success of composite restorations greatly depends on the type of adhesive and composite resin [1]. Dental adhesive systems are divided into two groups of etch-and-rinse (E&R) and self-etch (SE) systems [2,3]. Although E&R adhesives are considered as the gold standard, SE adhesives have become prevalent due to optimal efficacy and ease of use [4-6]. Self-etch bonding agents have advantages such as reduced post-operative sensitivity due to chemical bonding, reduced technical sensitivity, and enhanced application. Their ability to bond to dentin has been increasingly improved by chemical interactions, but the adequacy of enamel etching in SE mode is especially questionable in single-step adhesives [9-11]. For this reason, selective enamel etching has been suggested, especially in mild types. Enamel etching without affecting the exposed dentin is difficult; in addition, etching of dentin before bonding is risky. Although it facilitates adhesive penetration by removing the smear layer and smear plug, the adhesive cannot penetrate into the entire depth and may have a negative effect [12,13]. Universal adhesives were designed in accordance with the all-in-one concept, with the aim of alleviating problems and providing a product for all conditions. They are easier to apply and are available in single bottle form, allowing the dentist to choose an appropriate adhesive based on the conditions of the prepared cavity and their clinical judgment. Universal adhesives contain various monomers and primers, such as silane and methacryloy-loxydecyl dihydrogen phosphate (MDP) to improve the efficiency of their bonding to ceramic and tooth surfaces [14-18].

Use of dual-cure composite resins would save time when repairing a tooth crown as a single-bulk restoration. However, due to their more acidic nature, most SE systems are chemically incompatible with dual-cure composites. To eliminate this problem, some bonding systems come with activators in separate bottles; however, they are not often effective.

To date, few studies have examined the bond strength of universal adhesives with dual-cure composites. Some studies have shown that universal adhesives may be as compatible with dual-curing composites as light-cure composites; however, universal adhesives are not compatible with all types of dual-cure composites and some incompati-bilities have not been reported by the manufacturers [2, 5].

The purpose of this study was to compare dentin bond strength with two universal adhesives in two different etching modes. The null hypothesis was that etching modes would not affect the microtensile bond strength (μTBS) of dual-cure composites to dentin.

MATERIALS AND METHODS

In this in vitro, experimental study, 60 caries-free extracted third molar teeth were evaluated. The teeth were stored in thymol 0.01% and examined 3 months after extraction based on previous investigations [9,19]. For easy assessment, each tooth was mounted in an acrylic mold (Acroparse, Tehran, Iran). The occlusal surface enamel was removed utilizing a diamond bur (Tizkavan, Tehran, Iran) under water coolant perpendicular to the longitudinal axis of the tooth. This was done in order to obtain a smooth dentin surface, free of any residual enamel. To ensure a uniform dentin surface with standard roughness and smear layer, the specimens were polished with 180- and 600-grit silicon carbide sandpaper (Sof-Lex, Germany) for one minute, and were subsequently kept in distilled water at 37°C for 24 hours before being used [20]. Two universal adhesives, namely Single Bond Universal (3M ESPE, St. Paul, MN, USA) and G-Premio (GC, Tokyo, Japan), were used for bonding to nanohybrid composite (Z250XT, 3M, USA) and Core Flo DC composite (Bisco, Schaumburg, USA) in two etching modes. Single Bond 2 (3M, St Paul,USA) was used as the control group in E&R mode. The samples were cured by a LED curing unit (Kerr, Orange, CA, USA) with an intensity of 1200mW/cm^2 from 1mm distance for 10 seconds. The radiation intensity of the light curing unit was measured by a radiometer (LM_100, Monitex, Xianyang, China). The products were selected on the basis of the prevalence of use. In the next step, the 60 teeth were randomly divided into five equal groups as follows:

Group 1: G-Premio was used in the E&R mode as instructed in Table 1.

Group 2: Single Bond Universal was used in E&R mode as instructed in Table 1.

Group 3: G-Premio was used in SE mode as instructed in Table 1.

Group 4: Single Bond Universal was used in SE mode as instructed in Table 1.

Group 5: Single Bond 2 was used in E&R mode as instructed in Table 1.

Then, each group was equally divided into two subgroups.

Table 1: Materials used in this study and their application instructions

Material	Type	Main components	Application instruction
G-Premio Bond (GC Corporation, Tokyo, Japan)	Strong intermediate pH: 1.5	MDP Acetone Dimethacrylate Phosphoric acid ester monomer Photo-initiator BHT MDTP	Etching technique: Etch for 15s, then rinse and dry. Apply bond to the entire cavity wall with the applicator brush. Leave undisturbed for 10s after the end of application. Dry thoroughly for 5s with air under maximum air pressure. Light-cure bond with 1200mW/cm^2 LED for 10s SE mode: Apply adhesive to tooth surface by scrubbing action for 20s. Dry the adhesive for 5s. Light cure for 10s
Single Bond Universal (3M ESPE, St Paul, MN, USA)	Mild universal pH: 2.7	MDP phosphate monomer, dimethacrylate resins, HEMA Vitrebond copolymer, filler, ethanol, water, initiators and silane	1. E&R mode: Apply etchant for 15s. Rinse for 10s. Apply adhesive using a scrubbing action for 20 s. Dry the adhesive for 5s. Light cure for 10s. 2. SE mode: Apply adhesive to tooth surface by scrubbing action for 20 s. Dry the adhesive for 5s. Light cure for 10s
Adper Single Bond 2 (3M, St Paul,USA)	Etch and rinse resin	Adhesive: Bis-GMA, HEMA, dimethacry-lates, ethanol, water, photo-initiator, methacrylate functional copolymer of poly-acrylic and polyita-conic acids, 10% by weight of 5nm-diameter spherical silica particles	1. Apply etchant for 15s 2. Rinse for 10s 3. Blot excess water 4. Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air thin for 5s 6. Light-cure for 10 s at 1200mW/cm^2
Core Flo DC Bisco Inc (Bisco, Schaumburg, USA)	Dual cure	Glass filler, bisphenol A diglyciylmethacrylate, fused silica, amorphous silica triethylene glycol dimethacrylate	One 4-mm-thick layer is applied on the dentin surface and light-polymerized for 40s. The sample is then left to self-polymerize for 4 min before being stored in distilled water
Filtek Z250 (3M ESPE, St. Paul, MN, USA)	Light resin	Bis-GMA, UDMA, Bis-EMA Zirconia/silica Fillers (without silane treatment)	One 1.5mm-thick layer is applied on the dentin surface and light-polymerized for 40s

UDMA: urethane dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2hydroxyethyl methacrylate; BHT: butylated hydroxytoluene; MDTP: methacryloyloxydecyl dihydrogen thiophosphate; Bis-MEPP: bisphenol-A ethoxylate dimethacrylate; TEGDMA: triethylene glycol dimethacrylate

The first and second subgroups were restored with light-cure and dual-cure composites, respectively as follows: The matrix bond was mounted on the teeth to encircle the bonding area, and then Z250 composite or Core Flo dual-cure composite were applied in 2 layers of 2 mm and cured with a LED curing unit with an intensity of 1200mW/cm^2 for 40 seconds.
The specimens were kept in distilled water at room temperature for 24 hours. They were then cut with a diamond disc (Mashhadnomvo, Mashhad, Iran) at a speed of 300rpm under water coolant in both mesiodistal and bucco-lingual directions to obtain narrow beam-shaped samples from each dentin-composite group with an approximate cross-section of 1×1mm^2. Two completely intact composite-dentin specimens obtained from each tooth were selected. The prepared specimens were fixed in the jig of a universal testing machine (STM 50; Santam, Iran) with cyanoacrylate glue to assess microtensile strength. The bonding interface of the samples was positioned perpendicular to the direction of application of tensile force. The tensile force was applied to the resin-dentin bonding interface at a speed of 0.5mm/minute until fracture took place and the values displayed by the device were recorded. The µTBS was expressed in megapascals (MPa) [5].
In order to determine the failure mode, each specimen was evaluated under a stereo-microscope (SZ40; Olympus, Tokyo, Japan) at ×40 magnification and classified as follows: adhesive (failure at the composite bonding or dentin bonding interface), cohesive (failure in dentin or composite), or mixed (a combination of adhesive and cohesive).
From each group, one sample was used to assess the hybrid layer morphology by a scanning electron microscope (SEM). The samples were mounted on an aluminum stub by a conductive adhesive tape (double-sided carbon tapes) and then placed in a sputter-coater (JFC-1100E Ion Sputter, Jeol, Japan) and coated with gold palladium alloy for 10 minutes. The samples were analyzed under an SEM (JSM-840A, Jeol Ltd., Tokyo, Japan) at ×500 and ×3000 magnifications.
The data were collected taking into account the objectives of the study and analyzed in SPSS version 21 (IBM Corporation, Armonk, NY, USA) using descriptive statistics and statistical tests including three-way ANOVA, and Bonferroni post-hoc test. The significance level was set at 0.05 in all tests.

RESULTS

Descriptive results of the comparison of µTBS according to the composite type, and etching and bonding mode are presented in Table 2. The highest µTBS was related to Single Bond Universal in SE mode with light-cure composite (39.24±10.15 MPa) and the lowest µTBS was related to G-Premio in SE mode with dual-cure composite (13.56±11.9MPa).

Table 2. Mean microtensile bond strength (MPa) of the tested groups

Adhesive	Mode	Curing	Mean±SD
Single Bond Universal	E&R	Light cure	17.68±7.15
	SE		39.24±10.15
	E&R	Dual cure	19.45±9.27
	SE		21.31±18.96
G-Premio	E&R	Light cure	26.94±20.63
	SE		19.41±10.91
	E&R	Dual cure	14.52±6.11
	SE		13.65±11.95
Single Bond 2	E&R	Light cure	21.69±10.57
	E&R		32.81±16.41

E&R: Etch and rinse; SE: Self-etch; SD: Standard deviation

Three-way ANOVA showed that the etching mode had no significant effect on µTBS (P=0.159). Also, there was no significant difference in µTBS between different composites (P=0.098), but different bonding agents had a significant difference regarding µTBS (P=0.008, Table 3).
According to three-way ANOVA, the two-way and three-way interactions were significant; therefore, subgroup analysis was performed to compare the different bonding agents (Table 4).
The frequency of fracture modes in 120 samples was 46% adhesive, 28% cohesive in dentin, 15% mixed, and 9.1% cohesive in composite (Table 5). In the SEM view of the E&R samples, the hybrid layer was clearer, and resin tags were readily visible. However, in the SE samples, these characteristics were not as prominently evident.

Table 3. Comparison of microtensile bond strength by the type of adhesive, application mode, and composite type using 3 way analysis of variance

Source	Type III Sum of Squares	df	Mean Square	F	P
Adhesive	1725.48	2	862.74	5.05	0.008
Mode	342.72	1	342.72	2	0.159
Composite	475.21	1	475.21	2.78	0.098
Adhesive vs mode of application	1508.69	1	1508.69	8.84	0.004
Adhesive vs composite	1089.4	2	544.7	3.19	0.045
Mode of application vs composite	257.96	1	257.96	1.51	0.221
Adhesive vs mode of application vs composite	1036.16	1	1036.16	6.07	0.015

Mode: Etch and rinse or self-etch; Compsoite: Light cure and dual cure

Table 4. Mean difference and standard error (SE) of microtensile bond strength in subgroup analyses

Mode	Composite	Adhesive 1	Adhesive 2	Mean Difference (1-2)	SE	P
Etch and rinse	Light-cure	Single Bond Universal	G-Premio	-9.253	5.33	0.257
			Single Bond 2	-4.008	5.33	1
		G-Premio	Single Bond 2	5.245	5.33	0.983
	Dual-cure	Single Bond Universal	G-Premio	4.938	5.33	1
			Single Bond 2	-13.365*	5.33	0.041
		G-Premio	Single Bond 2	-18.303*	5.33	0.003
Self-etch	Light-cure	Single Bond Universal	G-Premio	19.746*	5.33	<0.001
			Single Bond 2	-	-	-
		G-Premio	Single Bond 2	-	-	-
	Dual-cure	Single Bond Universal	G-Premio	7.654	5.33	.154
			Single Bond 2	-	-	-
		G-Premio	Single Bond 2	-	-	-

Table 5. Comparison of the frequency of failure modes in the study groups based on the type of adhesive, etching mode, and composite type

Adhesive	Mode	Composite	Fracture			
			Cohesive composite N(%)	Mixed N(%)	Adhesive N(%)	Cohesive dentin N(%)
Single Bond Universal	E&R	L	1 (8.3)	0(0)	4(33.3)	7(58.3)
		D	0(0)	1(8.3)	5(41.7)	6(50)
	SE	L	3(25.0)	6(50.0)	0(0)	3(25)
		D	0(0)	1(8.3)	6(50.0)	5(41.7)
G-Premio	E&R	L	5(41.7)	3(25.0)	1(8.3)	3(25)
		D	0(0)	1(8.3)	7(58.3)	4(33.3)
	SE	L	0(0)	1(8.3)	10(83.3)	1(8.3)
		D	0(0)	2(16.7)	10(83.3)	0(0)
Single Bond 2	E&R	L	2(16.7)	2(16.7)	5(41.7)	3(25)
		L	0(0)	2(16.7)	8(66.7)	2(16.7)

L: Light cure; D: Dual cure; E&R: Etch and rinse; SE: Self-etch

DISCUSSION

Clinicians are looking for an appropriate and uncomplicated adhesive with low technical sensitivity. This demand has led the manufacturers to produce more popular adhesives (universal or multipurpose), introduced as the latest generation of adhesives, as a single bottle rather than a mixed adhesive [1, 7]. Universal adhesives can be used in E&R, SE, and elective etch modes for bonding of different direct and indirect restorative materials. While the composition of such adhesives is similar, there may be some differences in the amount of water and solvent and in the amount of MDP and di-methacrylate resins in different adhesives. It appears that these differences may affect the viscosity and wettability of each adhesive and, ultimately, the ability of the resin monomer to reach decalcified dentin [20]. Nonetheless, limited information on their various etching modes is also available, and the findings are controversial.

The manufacturers claim that universal adhesives are compatible with these composites. In the present study, a µTBS test was performed, which is a valid method to precisely measure the µTBS in areas smaller than 1 mm2 [5]. Our results showed that Single Bond Universal in SE mode had higher bond strength than E&R mode and, on the other hand, the µTBS with light-cure composite was higher than that with dual-cure composite. These findings in Single Bond Universal may be due to the presence of polyalkenoic copolymer (vitrebond). The primary chemical bond forms between the carboxyl groups in this copolymer and the calcium in the hydroxyapatite remaining around the exposed collagen fibers. On the other hand, Single Bond Universal includes MDP, which is capable of forming a chemical bond with hydroxyapatite and forms a stable nanolayer on the adhesive interface [21,3]. The total-etch systems expose the organic matrix by removing the mineral content; therefore, the formation of MDP-Ca salts for chemical bonding is difficult.

This chemical bond preserves the continuity of the collagen fibers between MDP-Ca and forms a stable layer. The lower solubility of calcium salt is highly desirable for bonding. Self-etch mild adhesives only interact with the enamel and dentin surface [22].

A previous study showed that etching prior to the application of Single Bond Universal significantly increased the penetration of resin into the dentin lattice, but did not increase the bond strength, consistent with the present study [23].

On SEM micrographs obtained from etched specimens, Single Bond Universal resin tags were different from the SE mode of this adhesive. In addition, the etching step in universal adhesives does not significantly increase the µTBS but can lead to creation of longer resin tags and a thicker hybrid layer.

In a study conducted by Peuman et al, [11] various factors such as type and age of tooth, degree of dentin demineralization, type of bond strength test, dentin moisture, and composition of the material in which the specimens, had an effect on bond strength [7]. Diniz et al. [22] found that the highest bond strength in universal adhesives was obtained in the E&R mode and the lowest bond strength was obtained in the self-etch mode in single bond universal bonding, which was different from the present findings, possibly due to the use of enamel as a substrate.

In the present study, we found that G-Premio in E&R mode was stronger than in SE mode, and the light-cure composite showed higher bond strength than the dual-cure composite.

This finding may be related to the acetone solvent in G-Premio bonding. Acetone has a higher evaporation rate than alcohol, resulting in faster evaporation. On the other hand, presence of butylated hydroxyl toluene in G-Premio may be a factor responsible for its higher bond strength as it serves as an inhibitor and improves resin durability. G-Premio contains 3 functional monomers of MDP, 4META, and MDPT, which make it suitable for bonding to all substrates [22]. Many universal adhesives are classified as ultra-mild (pH>2), which cannot etch well in SE mode, and the etched morphology is restricted to superficial layers, which eventually results in low monomer penetration. Nevertheless, most common adhesives have chemical bonding potentials. In addition, SE adhesives that are applied actively increase the external diffusion of solvents, which may be useful for adhesives with low evaporation of solvents (water and alcohol). Evaporation of the solvent creates a space that increase the cross-linking and degree of conversion, and enhance other mechanical properties [10].

In line with our findings, Munari et al [24] found that etching was not required to increase the bond strength of universal adhesives. The concentration of acetone in G-Premio is between 25% and 50%. As shown by previous studies, [10, 25] the type of solvent can affect the bond strength. As the acetone content increases, the bond strength decreases. Subsequently lower concentrations of alcohol solvent in Single Bond Universal could be the reason for better clinical outcomes. Acetone-containing systems are thinner after evaporation and, in addition to the thin adhesive layer, they are more vulnerable to degradation [25]. Systematic reviews have shown that mild universal adhesives (Single Bond Universal) do not have a significant difference in bond strength when used in different etching modes and tend to be more durable. The acidity of G-Premio is moderate to strong, which may affect the bond strength of dual-cure composites [2, 5, 26]. According to the present results, the μTBS did not depend on the type of composite used. Although the light-cure composite was stronger than the dual-cure composite, this difference was not statistically significant.

In a study conducted by Michaud P-L, it was found that the bond strength of light-cure composites was higher than dual-cure composites [5]. Previous studies [5, 27] have shown that all universal adhesives are not compatible with all dual-cure composites and, on the other hand, the use of activator in some universal adhesives has an adverse effect. On the other hand, the lower bond strength of dual-cure composites was not only due to bonding interactions, but also due to the chemical composition of composites. For example, some light-cure composites contained silica and zirconium, and some dual-cure composites had barium-glass and silica, which could affect the results [5, 27].

This study was performed on a light-cure and a dual-cure composite and two common bonding agents and thus, the results could not be generalized to all materials. In case of a dual-cure composite, the delay in light activation may affect the bond strength; thus, all specimens were exposed to light immediately before a self-adhesive reaction was initiated. The etching mode is determined by the size and depth of the cavity. If the entire substrate is dentin, SE or selective etch modes re recommended to minimize postoperative sensitivity and to inhibit the deterioration of resin and dentin interface over time. If the enamel is dominant or the exposed dentin is small, complete etching technique should be used to achieve effective bonding [1].

CONCLUSION

Type of adhesive may affect the bond strength of composite to dentin. The etching mode can also affect the bond strength of some universal adhesives.

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CONFLICT OF INTEREST STATEMENT

None declared.

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