Original Article
DOI: 10.22037/jds.v38i1.30796
ISSN 2645-4351

Long-term Comparison of Bond Strength Between One/Two Bottle and Self/ Total-Etch Universal Adhesive Systems

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(Submitted: 07 June 2021 – Revised version received: 25 August 2021 – Accepted: 13 September 2021– Published online: Winter 2021)

Objectives The aim of this study was to compare the shear bond strength and durability of one/two-bottle All-Bond Universal used in self-etch (SE) and total-etch (TE) modes on dentin discs.
Methods In this in vitro study, 144 human premolars were allocated to 12 groups for use of one-bottle or two-bottle adhesive in SE and TE modes and their assessment at three time points. Dentin discs with 2 mm thickness were prepared. They were polished with 600 and 800 grit silicon carbide abrasive papers. One/two-bottle All-Bond Universal bonding agent was used in SE and TE modes in the groups. Composite resin cylinders were made by the Tygon tubes on the bonding surface and then cured . Shear bond strength was measured by a universal testing machine at 24 h, and 6 and 12 months, and the mode of failure was determined under a stereomicroscope at x10 magnification. Data were analyzed by two-way ANOVA and Bonferroni test.
Results After 24 h and 6 and 12 months, the micro-shear bond strength was significantly lower in one-bottle SE compared with other groups. The two-bottle TE group showed the highest bond strength ($P<0.001$). In all groups, the bond strength significantly decreased at 12 months, compared with 24 h ($P<0.05$).
Conclusion Two-bottle TE system showed higher bonding durability and bond strength compared with other groups.
Keywords Shear Strength; Dental Bonding; Materials Testing

Introduction

In recent decades, patients demand more esthetic tooth-colored restorations. In this regard, composite resins can be used to restore the anterior and posterior teeth. These restorations are based on bonding to tooth structure. Therefore, bond strength to enamel or dentin is critical for restoration durability.[1, 2] Universal adhesives enable simple and practical bonding to tooth structure. They can be used with various clinical protocols such as self-etch (SE), total-etch (TE), and selective enamel etch, depending on the clinical condition.[3-5]. Universal adhesives can bond to direct and indirect restorations, like precious or non- precious alloys, zirconia, composite resins, silica or non-silica based ceramics, and stainless steel.[3, 4, 6-9] Also, they do not need separate primer application for better bonding.

The manufacturers of new dentin bonding agents have focused on reducing the steps of bonding procedure. Omitting the etching step not only shortens the working time but also prevents cavity contamination by blood and saliva during the washing of etchant and drying.[4] Therefore, the SE technique is less sensitive than the etch and rinse (ER) technique.[10] SE bonding agents have other advantages such as reducing the postoperative pain. This may be due to the residual smear plugs, which merely cover the dentinal tubules and cause less flow of dentinal fluid in comparison with ER adhesives. SE adhesives were introduced due to their compatibility with the hydrophilic nature of dentin, which can be an advantage. On the other hand, this may lead to more water sorption and their solubility in the long-term, which reduces the bonding characteristics, including mechanical properties[6], formulation stability[11, 12], bond strength[13], and structural stability.[14] Despite the manufacturers' claims on more hydrophobic features of universal adhesives compared with SE adhesives, there is insufficient information on hydrolytic stability of their polymer matrix in humid environments; it appears that water can soften the organic resin and increase the incidence of clinical failure.[15, 16]

Previous studies evaluated immediate bond strength of universal adhesives.[17, 18] But the durability and restoration stability in the oral environment are more important than immediate bond strength.[14] Few studies have been conducted comparing the bond strength of single-bottle and two-bottle adhesives.[19-21]

This study aimed to measure the shear bond strength of single-bottle and two-bottle All Bond Universal adhesive in SE and TE modes at 24 h, and 6 and 12 months.

Methods and Materials

This in vitro experimental study was conducted based on the methodology of Takamizawa et al.[22] The sample size was calculated to be 12 specimens in each group, considering power=0.8, $\alpha=0.05$ and at least 5 MPa difference in shear bond strength between the groups. Therefore, we considered the standard deviation for the microshear test to be 4.2 MPa. A total of 144 human premolars with no caries or abrasion were selected. All specimens were cleaned from the soft tissue remnants by a sickle scaler and were kept in saline at 25°C. Each tooth was cut into two slices with a 180-grit silicon carbide disc under water coolant. The first cut on each tooth was right below the enamel in the dentin, which was closest to the dentinoenamel junction. The second cut was parallel to the cementoenamel junction and was perpendicular to the longitudinal axis of the tooth. The thickness of specimens was 2 mm. They were kept in saline at room temperature for 1 week (25°C). The tooth surfaces were polished with 600 and 800-grit silicon carbide abrasive papers. The specimens were randomly allocated to 12 groups (12 samples per each). Figure 1 shows sample allocation to the groups. For the first group, a single-bottle bond and for the second group, a two-bottle bond was used in both SE and TE modes. Table 1 describes the manufacturer's instructions for each type of adhesive. Table 2 shows one and two-bottle All-Bond Universal ingredients.

Figure 1- Distribution of samples in the experimental groups

Table 1- Manufacturer's instructions for application of one- and two-bottle adhesive in self-etch and total-etch modes

Groups	Application steps according to the manufacturer's instructions (in single and two-bottle adhesives)
Single bottle self-etch	1- Pour one or two drops of the bonding in the pit and cover it. 2- Apply a layer of bonding on the wet surface of dentin with a microbrush. Using an air spray, remove the excess solvent for 10 s. 4. Use another layer of bonding and repeat steps 2 and 3 again. 5- Light cure for 20 s.
Single bottle total etch	1- Etch for 15 s and rinse thoroughly. 2. Remove excess water to keep a moist surface. 3- Apply one or two drops of the bonding in the pit and cover it. 4- Apply a layer of bonding on the wet surface of dentin with a microbrush. Remove the excess solvent for 10 seconds using an air spray. 6- Apply another layer of bonding and repeat steps 4 and 5 again. 7- Light cure for 20 seconds.
Two-bottle self-etch	1- Add equal amounts of Parts A and B of the primer in a bowl. Mix the two parts together, then cover the lid. 2- Apply a layer of bonding on the wet surface of dentin with a microbrush. Using an air spray, remove the excess solvent for 10 s. 4. Apply another layer of bonding and repeat steps 2 and 3 again. 5- Light cure for 20 s.
Two-bottle total etch	1- Etch for 15 s and rinse thoroughly. 2. Remove excess water to keep a moist surface. 3- Add equal amounts of Parts A and B of the primer in a bowl. Mix the two parts together, then cover the lid. 4- Apply a layer of bonding on the wet surface of dentin with a microbrush. Remove the excess solvent for 10 seconds using an air spray. 6- Apply another layer of bonding and repeat steps 2 and 3 again. 7- Light cure for 20 s.

Table-2- Ingredients of one-bottle and two-bottle All- Bond Universal

All- Bond Universal (one-bottle)	**All- Bond Universal (two-bottle)**	
Bisphenol A diglycidylmethacrylate (20- 50%)	Part A	Part B
Ethanol (30- 50%)	Magnesium NTG- GMA (5-10 %)	Bis-GMA (50- 75%)
MDP (5-25%)	Ethanol (>85%)	Ethanol (10- 30%)
2- Hydroxyethyl Methacrylate (5-25%)	Acetone	2- Hydroxyethyl Methacrylate (10- 30%)
		10- Methacryloyloxydecyl Dihydrogen Phosphate (10- 30%)
		Acetone

In the TE protocol, extra etching and rinsing were performed for the purpose of comparison with the SE protocol. At first, specimens were etched with 37% phosphoric acid gel (Maquira, Brazil) for 15 s. Then, they were rinsed and gently air dried such that some moisture remained on the dentin. After that, dentinal surfaces were conditioned with universal bonding by a micro-brush and air-dried for 10 s. Samples were cured for 20 s with a LED curing unit (Wood Packer Zone, Gulin Gunggxi, R. china) with 600 mW /cm2 light intensity.

In order to provide grip points for the shear test, we formed composite cylinders with 2 mm thickness using Valux composite (shade A3) on each specimen. Composite was applied in a transparent silicon tube (Tygon, Norton Performance) with 0.7 mm diameter as a mold. Each composite increment was perpendicularly cured for 40 s; then, they were exposed to extra light curing for 120 s at 45-degree angle from each side. The specimens were stored in distilled water at 37°C for 24 h in an incubator (Pars Azma Co., Iran). All specimens were attached to the universal testing machine jig (Tensile Tester; Bisco, Schaumburg, IL, USA) by cyanoacrylate glue, and the composite cylinders were tied by a 2.2 mm stainless steel wire which was connected to the other jig of the device. This wire was positioned parallel to the horizon, at the lowest point of the composite cylinder-tooth interface. Then, load was applied at a speed of 2.2 mm/min until the composite samples were debonded from the tooth surface (Fig. 2). After removing the Tygon tube, the samples were evaluated under a stereomicroscope (SZX 16; Olympus, Japan) at x10 magnification in order to determine the mode of failure.

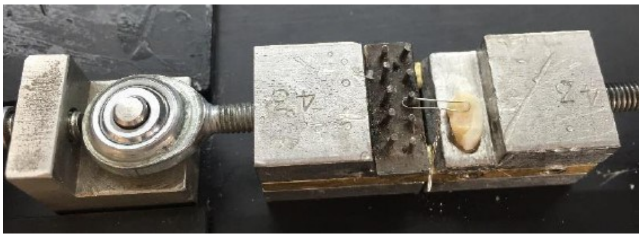

Fig. 2- Shear bond test by universal testing machine

The micro shear bond strength was evaluated at three time points of 24 h, 6 months, and 1 year. The Shapiro-Wilk test was used to evaluate normal distribution of data. Three-way ANOVA and Bonferroni test were used for statistical analysis.

Results

The Shapiro-Wilk test showed that the data had a normal distribution. Table 3 shows the mean shear bond strength of each group. According to the results, the highest bond strength was related to two-bottle TE at each time point, and the lowest bond strength was recorded for the single-bottle/SE.

Table 3- Descriptive data regarding the shear bond strength of the groups at different time points

Time	**All Bond Universal**	**Mean ± SD**	**Minimum (MPa)**	**Maximum (MPa)**
24 hour	1 bottle (self-etch)	16.72 ± 5.97	7.79	29.89
	1 bottle (total-etch)	31.91 ± 10.84	17.41	46.27
	2-bottle (self-etch)	33.94 ± 6.11	21.57	43.41
	2-bottle (total-etch)	39.47 ± 6.66	29.37	52.77
6 months	1 bottle (self-etch)	15.25 ± 4.85	8.05	21.83
	1 bottle (total-etch)	30.15 ± 3.78	22.37	42.37
	2-bottle (self-etch)	29.65 ± 5.59	22.35	38.73
	2-bottle (total-etch)	38.67 ± 4.33	29.37	43.67
12 months	1 bottle (self-etch)	8.34 ± 2.42	4.67	12.47
	1 bottle (total-etch)	23.81 ± 5.26	20.27	38.47
	2-bottle (self-etch)	20.99 ± 5.78	12.73	30.67
	2-bottle (total-etch)	32.34 ± 2.44	28.07	35.87

Two-way ANOVA was used to check the statistical difference between groups based on single/two-bottle, SE/TE, and time. There was a significant difference between all groups (P<0.001).

According to the pairwise comparisons by the Bonferroni test, the interaction effect of the number of bottles and etching mode on bond strength was significant at different time points (P=0.001). The bond strength change in TE groups was not similar to SE groups in use of single or two-bottle adhesive. The etching mode had a greater effect on single-bottle groups than the two-bottle groups (Fig. 3).

The interaction effect of time and bottle number on bond strength was not significant (P=0.636). The effect of time on decreasing the bond strength was similar between one and two-bottle groups (P=0.348).

In all samples, the micro-shear bond strength significantly decreased over time (Fig. 4). Table 4 shows the mode of failure in the groups based on the etching mode and number of bottles. Mixed failure was the most prevalent in all groups.

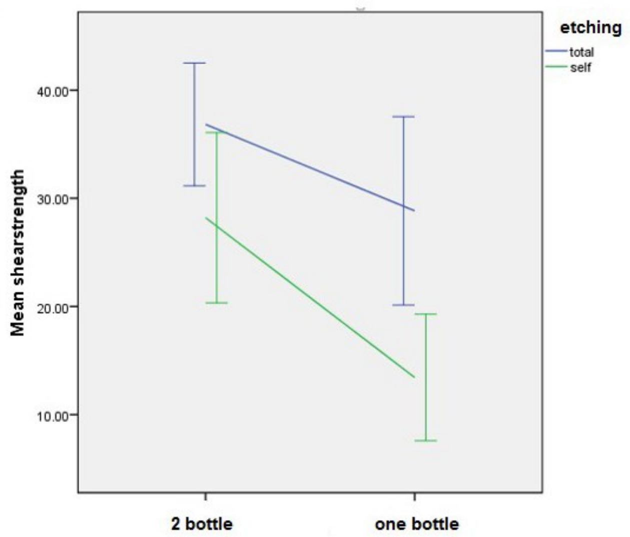

Figure 3- Comparison of shear bond strength in one/two-bottle systems with total-etch and self-etch modes

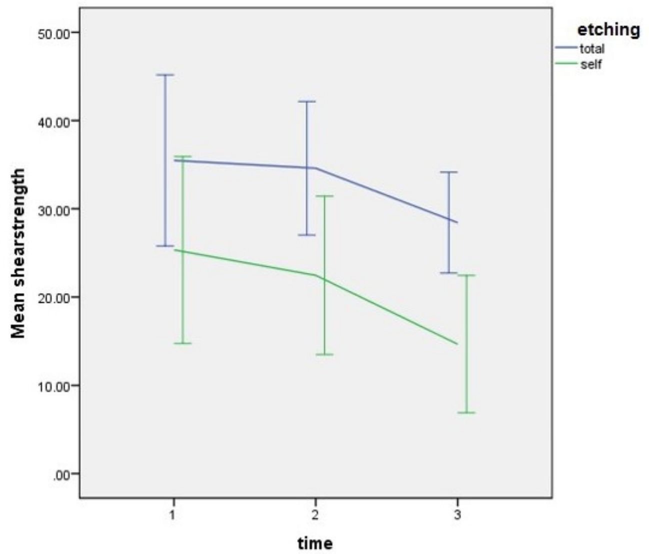

Figure 4- Interaction effect of time and etching on shear bond strength

Table 4- Distribution of modes of failure in the groups

Etching type/ Bottle	Cohesive failure	Adhesive failure	Mixed failure
Self-etch/one bottle	14.28	7.14	78.57
Total-etch/one bottle	7.14	14.28	78.57
Self-etch/two-bottle	9.5	21.75	68.75
Total-etch/two-bottle	9.5	21.75	68.75

Discussion

The Shapiro-Wilk test showed that the data had a normal distribution. Table 3 shows the mean shear bond strength of each group. According to the results, the highest bond strength was related to two-bottle TE at each time point, and the lowest bond strength was recorded for the single-bottle/SE.

Two-way ANOVA was used to check the statistical difference between groups based on single/two-bottle, SE/TE, and time. There was a significant difference between all groups (P<0.001).

According to the pairwise comparisons by the Bonferroni test, the interaction effect of the number of bottles and etching mode on bond strength was significant at different time points (P=0.001). The bond strength change in TE groups was not similar to SE groups in use of single or two-bottle adhesive. The etching mode had a greater effect on single-bottle groups than the two-bottle groups (Fig. 3).

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In all samples, the micro-shear bond strength significantly decreased over time (Fig. 4). Table 4 shows the mode of failure in the groups based on the etching mode and number of bottles. Mixed failure was the most prevalent in all groups.

Conclusion

At 1 year, the highest shear bond strength of All-Bond Universal was recorded in use of two-bottle TE while the lowest bond strength was recorded in one-bottle SE. In addition, the micro-shear bond strength of adhesives was lower at 1 year compared with 24 h.

Conflict of Interest

No Conflict of Interest Declared ■

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How to cite:
Azam Valian, Marzieh Nejatifard .Long-term Comparison of Bond Strength Between One/Two Bottle and Self/Total-Etch Universal Adhesive Systems. J Dent Sch 2020;38(1):14-19.