Original Article

ANNALS OF DENTISTRY
UNIVERSITY OF MALAYA

www.adum.edu.my

Ann Dent UM 2019, 26:1-7
DoI: 10.22452/adum.vol26no1

Shear Bond Strength and Failure Mode of Different Dental Adhesive Systems

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KEYWORDS

Dental adhesive; Dental composite; Total-etch bonding system; Self-etch bonding system; Shear bond strength; Failure mode

ABSTRACT

The study aimed to evaluate the shear bond strength (SBS) and to analyse the failure mode at the resin-dentine interface of different dental adhesive systems. A total of 75 sound premolar teeth were selected and randomly assigned into five different adhesive groups (n=15): OptiBond Solo (OBS, total-etch), OptiBond Versa (OBV, two-bottles, self-etch), Adhe SE Ivoclar (ADHE, two-bottles, self-etch), G-Bond (GB, one-bottle, self-etch), and OptiBond All in One (OBO, one-bottle, self-etch). The occlusal surface of each tooth was flattened and composite resin cylinder (4x2 mm) was built up on the flat dentine surface using a custom made mould. The specimens were then subjected to 500 thermal cycles between 5 °C and 55 °C and dwell time of 20s. The SBS test was conducted using a universal testing machine at a crosshead speed of 0.5 mm/min. Data were analyzed using one-way ANOVA and Dunnett T3 Test. Failure mode was determined as adhesive, cohesive or mixed mode using a stereomicroscope and the data were analyzed using Fisher's exact test. The total-etch (OBS) had significantly higher value of SBS than the two self-etch (ADHE and GB) adhesive systems. Within the self-etch systems, OBV showed significantly higher SBS value compared to ADHE and GB. There were no statistically significant differences between types of failure mode ($p > 0.05$) and adhesive group. It can be concluded that the different compositions in the self-etch adhesive materials may contribute to the different SBS value. The failure modes detected within all tested groups did not show clinically important differences.

INTRODUCTION

Over the years, durability and reliability of adhesive restoration have been enhanced by the advancement of adhesive systems. Development started from the Buonocore era to the present. There were several factors that characterized the change of adhesive systems, namely enamel etching, dentine etching, modification of smear layer and handling properties. Adhesive systems are usually categorized by generation based on when they were introduced, with the newer generations performing better than earlier generations (e.g., self-etch and total etch) [1,2]. There is also another classification that uses the number of bottles or steps rather than generation.

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This type of classification is based on total-etch and self-etch mode application [3,4]. The recent emergence of different types of adhesive bonding agents in the market have resulted in dentists facing difficulty in choosing the most suitable material for their patients. There are many factors to analyze before a decision can be made. One of the factors to be considered is the clinical performance of an adhesive bonding agent. The clinical performance of current adhesives has significantly improved with resin restorations contributing to a highly predictable level of clinical success [5]. Other than that, the simplicity of application during clinical practice is another factor that may influence clinical judgment of the dentist as it may shorten clinical procedure time. Among contemporary adhesives, self-etch adhesives have been a popular choice, especially due to their convenience in application [6].

One of the ways to determine performance of dental adhesives is through bond strength tests. The rationale behind this testing method is that the stronger the adhesion between tooth and biomaterial, the better it will resist stress imposed by resin polymerization and oral function [7]. There are several types of bond strength that have been studied by researchers, for instance shear bond strength, tensile bond strength, microshear and microtensile bond strength.

There are many new adhesive systems available in the market with each product claimed by their manufacturers to perform better compared to others. Hence, as clinicians, we need to balance between simplification of clinical steps and clinical success together with material's durability in choosing the preferred material. However, there is lack of studies that evaluate the bonding efficiency for the latest adhesive systems. Therefore, the objective of the present study was to evaluate the shear bond strength (SBS) of different dental adhesive systems. In addition, the failure mode at the resin-dentine interface of these systems was analysed. The null hypothesis was that there is no difference in shear bond strength within all five different adhesives groups.

MATERIALS AND METHODS

In this study, five dental adhesive systems [OptiBond Solo (total-etch); OptiBond Versa (two-bottles, self-etch); Adhe SE Ivoclar (two-bottles, self-etch); G-Bond (one-bottle, self-etch); and OptiBond All in One (one-bottle, self-etch)] were used. The chemical formulations of the adhesive systems, etchant and dental composite are shown in Table 1.

Approximately ninety freshly extracted sound human premolars were collected from several clinics in Malaysia. However, only teeth which were free from decay, cracks or restorations were selected for this study. Soft and hard tissue deposits were cleaned with pumice and removed using ultrasonic scaler. The teeth were then disinfected using 0.5 % Chloramine T for a week, stored in distilled water at 4 °C until further use.

Seventy-five premolars were selected from the total number of teeth collected. The root surfaces were first marked at 2.0 mm below the cementoenamel junction. Each specimen was embedded vertically in a clear cold curing epoxy resin (Mirapox® 950-230 A, Balakong, Malaysia) at the level previously marked.

Each specimen was cut approximately 2.0 mm from the central fissure with a slow speed precision cutter (Metkon®, Bursa, Turkey) to expose the flat occlusal dentine surface. The dentinal surface of each tooth was further polished for 60 seconds with 400 grit and 600 grit silicon carbide papers which were placed on the polishing machine (Isomet, Buehler; Lake Bluff, IL, USA).

The adhesive system's testing area was confined using a laminated adhesive tape and a 5.0 mm puncher was used to form the central orifice. Adhesive agent was applied according to the manufacturer's instructions. The dentinal surface of each specimen received different preparative treatments prior to placement of the composite. In OBS group, the dentinal surfaces were etched with etchant for 15 seconds on the enamel and 10 seconds on the dentine. The dentinal surfaces were then rinsed thoroughly and gently air dried. This was followed by application of bonding agent for 15 seconds, air thinning for 3 seconds and light curing for 10 seconds. In OBV group, the dentinal surfaces were treated with a two-bottle system. Self-etching bonding agent primer was applied for 20 seconds using scrubbing motion and air thinned for 5 seconds. Next, adhesive was applied for 15 seconds, air thinned for 5 seconds and light cured for 10 seconds. In ADHE group, the dentinal surfaces were also treated with a two-bottle system; self-etching bonding agent primer was applied for 15 seconds. Following that, adhesive was applied, dispersed with air thinning and light cured for 10 seconds. In GB group, the dentinal surfaces were treated with a single bottle in self-etching mode. After G-Bond was applied, dentinal surfaces were left undisturbed for 5 seconds. The dentinal surfaces were dried thoroughly for 5 seconds and light cured for 10 seconds. In OBO group, the dentinal surfaces were also treated with single bottle self-etching mode. Two consecutive coats of OBO adhesive were applied and was scrubbed using a microbrush for 20 seconds, air dried for 5 seconds and then light cured for 10 seconds.

In ADHE group, the dentinal surfaces were also treated with a two-bottle system; self-etching bonding agent primer was applied for 15 seconds. Following that, adhesive was applied, dispersed with air thinning and light cured for 10 seconds. In GB group, the dentinal surfaces were treated with a single bottle in self-etching mode. After G-Bond was applied, dentinal surfaces were left undisturbed for 5 seconds. The dentinal surfaces were dried thoroughly for 5 seconds and light cured for 10 seconds. In OBO group, the dentinal surfaces were

also treated with single bottle self-etching mode. Two consecutive coats of OBO adhesive were applied and was scrubbed using a microbrush for 20 seconds, air dried for 5 seconds and then light cured for 10 seconds.

Light emitting diode (LED) light cure (Kerr, CA, USA) was calibrated with a radiometer (Caulk, Dentsply, USA) prior to usage in the restorative procedure. This was to ensure that energy emitted from the light cure was not less than 500 nm. A stainless steel split mould (2 mm in height, 4 mm diameter) was placed on the dentinal surface.

Table 1. Chemical formulations of the dental adhesive systems, etchant and composite used in is study.

Material (Manufacturer)	**Code**	**Type**	**Composition**
OptiBond S (Kerr, CA, USA) (CONTROL)	OBS -	Primer-adhesive (Total-etch)	Ethyl alcohol 20-25%, Alkyl dimethacrylate resins 55-60%, Barium aluminoborosilicate glass 5-10%, Fumed silica 5-10%, Sodium hexafluorosilicate 0.5-1%.
Gel Etchant (Kerr, CA, USA)		Etching agent	Phosphoric acid 37.5%, Water, Fumed silica, Dye colorant
Optibond Versa (Kerr, CA, USA)	OBV	Primer	Acetone 25-35%, Ethyl alcohol 4-15%, Hydroxyethylmethacrylate (HEMA) 30-50%
		Adhesive (Self-etch, two-bottle)	Ethyl alcohol 20-30%, Alkyl dimethacrylate resins 47-68%, Barium aluminoborosilicate glass 5-15%, Fumed silica 3-10%, Sodium hexafluorosilicate 0.5-3%
Adhe SE Ivoclar (Vivadent, Schaan, Liechtenstein)	ADHE	Primer	Phosphonic acid acrylate, Bis-acrylamide, Water, Initiators and stabilizers.
		Adhesive (Self-etch, two-bottle)	Dimethacrylates,Hydroxyethyl methacrylate, Highly dispersed silicon dioxide,Initiators and stabilizers, solvent, initiators.
G-Bond (GC Corporation, Tokyo, Japan)	GB	Self-etch (All in one)	Phoshoric acid ester monomer, 4-Methacryloxethyltrimllitic acid(4-MET), Dimethacrylate, acetone, distilled water, nano-silica fillers, photoinitiator
OptiBond All in One (Kerr, CA, USA)	OBO -	Self-etch (All in one)	Monomers glycerol phosphate dimethacrylate – self etching adhesive monomer Co-monomers including mono- and difunctional methacrylate monomers water, acetone and ethanol, camphorquinone based, three nano-sized fillers, sodium hexafluorosilicate and ylterbium fluoride
Herculite Ultra (Kerr, CA, USA)		Nano-hybrid composite	Uncured methacrylate esther monomers, titanium dioxide (TiO2) and pigments, 4-methoxyphenol (MEHQ), benzoyl peroxide (BPO), trimethylolpropane triacrylate and initiators

Modeling clay was used to stabilize the mould. The resin composite was incrementally applied inside the mould and each layer was light cured for 20 seconds to form a resin composite cylinder (Figure 1). The specimens were thermocycled with thermocycling machine (ATDM T6PD UM, Malaysia) following the ISO recommendation (ISO/IT 11405/2003). Samples were subjected to 500 thermocycles (5 °C and 55 °C, dwell time of 20 seconds) with 10 seconds transfer interval.

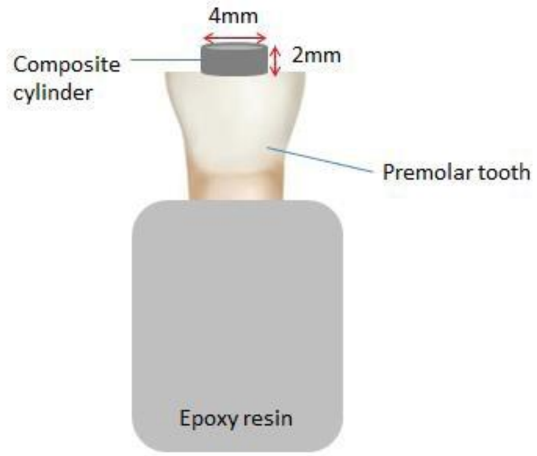

Figure 1. Schematic diagram of specimen with composite build-up occlusally

The specimen was position in the universal testing machine (UTM) (Shimadzu, Kyoto, Japan) with custom-made notched rod jig (Figure 2a and b). The long axis of the specimen was placed perpendicular to the direction of the applied force. Then shear loading test was performed at a crosshead speed of 0.5 mm per min. After testing, every debonded surface was examined under stereomicroscope using 25x magnification to determine the mode of failure. The mode of failure was classified as below:

a. Adhesive failure: failure at interface of adhesive and dentine or interface of composite and adhesive
b. Cohesive failure: Failure within the dentine or composite
c. Mixed failure which is combination of (a) and (b).

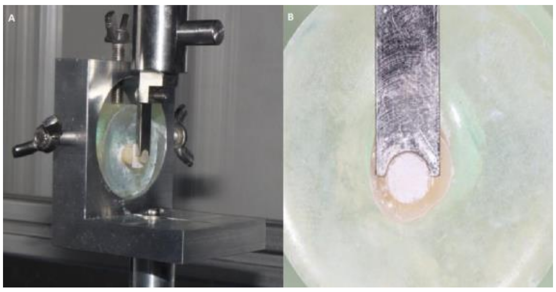

Figure 2. (A) Specimen alignment with the jig in UTM (B) Close up view of shear bond test jig.

Statistical Analysis

The data obtained were analyzed using Statistical Package for the Social Science (SPSS) software version 23 for Windows. Descriptive data of SBS were expressed as mean [± standard deviation (SD)] and subjected to analysis of variance (ANOVA) and post-hoc analysis (DunnettT3). The failure mode data were analyzed using Fisher's exact test.

RESULTS

(a) Shear bond strength of different adhesive groups

Descriptive statistics were performed and the shear bond strength (SBS) mean and standard deviation (SD) for all groups is presented in Figure 3. OBV group showed the highest mean SBS followed by OBS group with 17.45 MPa and 12.87 MPa respectively. The lowest mean SBS was observed in the ADHE group (8.09 MPa).

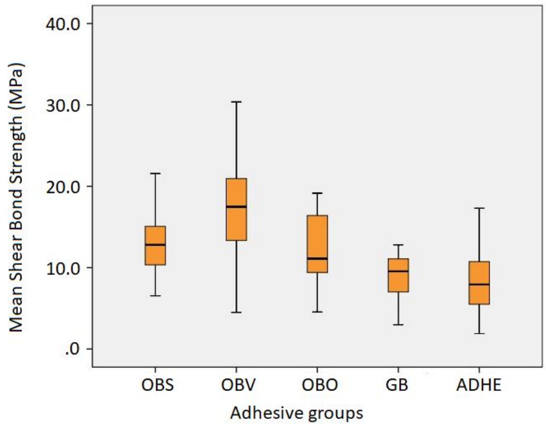

Figure 3. Mean shear bond strength of five different adhesive groups.

The p-value of the normality test (Kolmogorov-Smirnov) for SBS was more than 0.05 in all adhesive groups. Hence, the data can be assumed to be normally distributed. Statistical analysis was performed using One-way ANOVA. The p-value of Levene's test for equality of variances was 0.019, which was less than 0.05. Therefore, equality of variances was not assumed. Overall there was significant difference in SBS between the groups ($p < 0.001$). Thus post hoc test (Dunnett T3) was performed. The result of the post hoc test at 95% confidence level (Table 2) showed significant mean difference in SBS between OBS and GB group ($p = 0.04$) and ADHE group ($p = 0.03$) respectively. In addition, there was significant mean difference in SBS between OBV group with GB ($p = 0.01$) and ADHE ($p = 0.01$) groups.

(b) Mode of failure

The data of failure modes for all adhesive groups

Table 2. Post hoc test multiple comparisons (Dunnett T3) for mean shear bond strength.

(I) Group	(J) Group	Mean Difference (I-J)	p- value	95% Confidence Interval	
				Lower bound	Upper bound
OBS	GB	4.00*	0.04	0.08	7.93
	ADHE	4.78*	0.03	0.38	9.19
OBV	GB	8.59*	0.01	1.71	15.46
	ADHE	9.37*	0.01	2.28	16.45

* The mean difference is significant at the 0.05 level.

were analysed using Fisher's Exact Test. The test showed that there was no association between failure type and group of adhesive (p = 0.21). Therefore, in this section, only descriptive statistic was done. The percentage of failure can be observed in Figure 4. There was no cohesive failure in all adhesive groups. A higher percentage of adhesive failure was found in ADHE (46.67%) group followed by GB (33.33%) group. In comparison, OBO and OBS showed the lowest percentage of adhesive failure with 13.33% for both groups. However, most of the groups had more mixed failure as compared to adhesive failure.

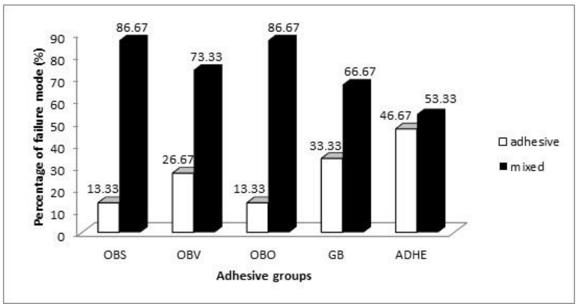

Figure 4. Percentage of failure mode in five different adhesive groups.

DISCUSSION

The null hypothesis was rejected because there was significant difference in SBS between different adhesive systems. Our results were in agreement with Knobloch et al. [8], who found that bond strength of total-etch to be higher than self-etch groups. Higher bond strength in total-etch (XP Bond system) group compared to self-etch group (Adper Single Bond 2 system) was also observed by Raposo and Santana [9]. Their findings can be explained by the solvent used in the XP Bond system (tert-butanol, or t-butanol) and by the hypothesis that this adhesive system is capable of forming not only a micromechanical bond, but also a chemical bond to the tooth structure [9].

In our study, different solvent was used for every adhesive group; OBS used ethanol (ethyl alcohol), GB group used acetone and water and ADHE group only used water as solvent. Cardoso et al. [10] found higher bond strength was achieved by the group with ethanol as a solvent in moist dentine. This observation is similar with our finding where OBS group had significantly higher mean SBS compared to GB and ADHE groups.

Villela-Rosa et al. evaluated the dentine SBS of four adhesive systems; Adper Single Bond 2 (etch and rinsed), Adper Prompt L-Pop (single step self-etch), Magic Bond DE (etch and rinse) and Self Etch Bond (single steps self-etch) [11]. Their study showed that Adper Single Bond 2 have the highest mean values of SBS in different dentine depth. On the other hand, Prompt L-Pop product, a self-etching adhesive, revealed higher mean values of SBS compared to Magic Bond DE and Self Etch Bond adhesives, a total and self-etching adhesive respectively. The explanation of high SBS in Adper Single Bond 2 and Adper Prompt L-Pop adhesives is the presence of polyacrylic acid, which promotes chelation with calcium and the formation of hydrogen bridges with dentine components. Whereas the low bond strength in Self-Etch Bond is related to the weak etching (pH = 5). They concluded that SBS of dentine is dependent on the material (adhesive system) and substrate depth.

The current study showed that OBV with pH ≈ 1.6, has a significantly higher bond strength compared to GB (pH = 2.0). Similar results are obtained by Sensi et al. [12] who studied six types of self-etch adhesive systems and one group of etch and rinse adhesive system. Higher bond strength was observed in self-etch with low pH (Optibond Solo plus with pH 1.2- 1.5). It was also considered as intermediated strength self-etch by Van Meerbeek et al. [13]. Cardoso et al. [10] suggest self-etch with low pH (1.2- 1.5), assure a better monomer penetration, which enhances the bond strength to dentine.

The lowest mean SBS was observed in the self-etching HEMA-free adhesive, GB group. In a recent study, phase separation among adhesive compositions was confirmed, as droplets entrapped during solvent evaporation from HEMA-free adhesives. This phenomenon could be explained by the evaporation of solvents such as ethanol and acetone, which affected the balance of solvents and resin monomer and caused water to separate from other compositions of the adhesive [14].

The present study observed that OBV had significantly higher SBS as compared to ADHE group. OBV had HEMA in its primer while ADHE is HEMA-free. This result is in agreement with Felizardo et al. [15] who found lower bond strength in HEMA-free self-etch as compared to the group with HEMA. In their discussion they also stated other factors that may influence bond strength such as solvent type. They found adhesive groups with only acetone as solvent had lower bond strength in comparison with group which has combination solvent (acetone and ethanol). This is because solvent evaporation from adhesives is influenced by the vapour pressure. As the vapour pressure of acetone is high, it volatilizes rapidly and may dehydrate the dentine. In our study, solvent in OBV were acetone and ethanol while for ADHE was water. The difference in the usage of solvent between these groups may contribute to a higher SBS in OBV group. This result can be further explained by Van Landuyt et al. [16] in a systematic review of chemical composition in adhesive agent, where the presence of water in self-etch adhesives is necessary to ensure the ionization of the acidic monomers. However, it is not as efficient as acetone or ethanol because of its lower vapour pressure. This condition may lead to lower SBS of ADHE group.

Koliniotou-Koumpia et al. evaluated a solvent free self-etch adhesive (Bond 1 SF), an ethanol self-etch adhesive (Futurabond M), and a water-acetone-ethanol self-etch adhesive (Optibond All-In-One) [17]. They found that solvent free self-etch (Bond 1 SF) had lower SBS as compared to the other two groups. In this study, both single step bonding agents (OBO and GB groups) are comparable in producing lower SBS. These results were in accordance with Yazici et al. who used five adhesive systems; Single Bond one-bottle total-etch, AQ Bond one-step self-etching, Clearfil SE Bond two-step self-etching, Tyrian SPE/One-step and Plus two-step self-etching in SBS of different dentine depth [18]. It was found that AQ Bond one-step self-etching adhesive produced significantly lower bond strengths to superficial dentine compared to the other groups. The reason for the low bond strength obtained by AQ Bond could be its relatively high pH (2.5), which might have been incapable of etching superficial dentine. Another reason could be related to its hydrophilic properties. It is known that one step self-etching adhesives are more hydrophilic than two-step self-etching adhesives and they attract more water. As it is difficult to evaporate water from these adhesives, water will rapidly diffuse back from the bonded dentine into the adhesive resin and subsequently, a lower mechanical strength.

It was revealed in our results that higher mixed failure was found in OBS and OBO groups and adhesive failure was highest in ADHE group. Our results support another study, which demonstrates high mixed failure in ethanol solvent group and ethanol-acetone-water solvent group, while higher adhesive failure was observed in free solvent group [17]. In our study, OBS is an ethanol based solvent while OBO has acetone-ethanol-water solvent and solely water in ADHE solvent. A study by Hara et al. found that only two types of failure at the interface of SBS which are adhesive and mixed failure [19]. These results were in disagreement with a review paper where pooled data revealed the incidence of cohesive failures in various tests including 45% in shear, 28% for tension, 13% for microshear and 12% for microtensile [20]. The mechanics of the test and the brittleness of the material involved explained cohesive failure rather than an indication of strong bonding.

CONCLUSION

Within the limitation of this study, OBS group had significantly higher SBS compared to GB and ADHE groups. OBV group had significantly higher SBS compared to GB and ADHE groups. No correlation between failure type and different adhesive system was observed.

ACKNOWLEDGEMENT

The authors would like to thank the University of Malaya for financing this project grant no: RG531-13HTM.

DECLARATION OF INTEREST

The authors report no conflicts of interest. The authors alone are responsible with the content of this article.

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