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Evaluation of the micro-shear bond strength of four adhesive systems to dentin with and without adhesive area limitation

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Abstract. The purpose of this study was to evaluate the bonding ability of four representative dentin-adhesive systems by applying the micro-shear bond strength (μ-SBS) test method and to evaluate the influence of adhesive area limitation on the bond strength. Two different adhesive application methods were used in the μ-SBS test (with and without adhesives area limitation), and four representative adhesive systems were used in this study. Each dentin surface was treated with one of the four representative adhesive systems, and with twenty samples per group (n=20), each of the four groups underwent a μ-SBS test. The results showed that the bond strength was significantly influenced by the adhesive application method (p<0.05), the adhesive type (p<0.05) and the interaction between the two factors (p<0.05). With regard to the four representative dentin-adhesive systems, 3-E&R has a much better bond quality compared to the other adhesive systems. Furthermore, the micro-shear bond strength test method of restricting the area of both the adhesive and the resin is more reliable for evaluating the bonding property of adhesives to dentin, and it is also adequate for comparing the different adhesives systems.

Keywords: Adhesive area limitation, adhesive system, dentin, adhesive, micro-shear bond strength

1. Introduction

Adhesive dentistry has generally revolutionized modern dentistry over the past decades. With continuously improving bonding technology, the adhesive-resin restoration strategy is being used more and more often in clinical practice due to the focus of less invasiveness and esthetic property [1]. Moreover, the long-term clinical success of adhesive restoration is primarily dependent on the bonding quality of adhesive systems to dentin, and the key parameter for evaluating the bond quality of different dentin-adhesives systems is bond strength [2]. However, it is difficult to obtain precise and consistent bond strength data due to the use of numerous testing techniques and parameters [3, 4]. For example, some factors affect testing results, such as different adhesive areas, the crossing speed, the

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selection and preparation of substrate, the direction of shear force, and so on [5, 6]. Therefore, an accurate and unified laboratory evaluation method is crucial for the effective clinical application of different adhesives, and just as important, differences in the testing procedures should be identified and standardized for consistency.

Recently, the micro-shear bond strength (μ-SBS) test has been advocated as a modified method for evaluating the bonding ability of dentin-adhesive systems [7-9]. Compared to the macro-shear bond strength test, the μ-SBS test is more advantageous; it has fewer internal defects as well as more homogeneous stress distributions at the interface due to the use of smaller specimen [10]. In addition, the μ-SBS test does not require an additional specimen trimming process after the bonding procedure, which conserves the integrity of the specimens and avoids pre-testing failures [11]. However, due to the unrestricted adhesive area, the traditional μ-SBS test technique still does not produce precise data. Traditionally, the adhesive is applied and cured on the entire substrate prior to the construction of the composite cylinder, instead of constraining the adhesive area to the substrate [12, 13]. More recently, Shimaoka, et al. [14] proposed that the adhesive area should be delimitated and constrained to the dentin substrate so as to equate the area between the adhesive and the resin and to eliminate differences in test results caused by traditional adhesive application technology. Furthermore, constraining the adhesive area has also been proposed to improve the accuracy of test data concerning the tensile bond strength, and it has been concluded that applying the adhesive on the whole dentin and without adhesive area limitation potentially modifies the local stress distribution at the adhesive/dentin interface [15]. However, no study to date has effectively tested the different categories of dentin-adhesives systems by applying adhesive area restriction.

In a previous study, an accepted classification of dentin adhesives divided adhesives into two systems: etch-and-rinse and self-etching systems. The classification was based upon whether or not a separate etching agent was used [16, 17]. An etch-and-rinse system is characterized by removing the smear layer by using an etching agent [18], and a self-etching system is characterized by eliminating the technique-sensitive rinsing step and reducing the operation time [19]. ART Bond (Coltene) and Adper Single Bond 2 (3M ESPE) are products used with etch-and-rinse systems, and they are commonly used in clinics. In contrast, Clearfil SE Bond (Kuraray) and Adper Easy One (3M ESPE) are both employed in self-etching systems and are the dentin-adhesive materials that are most commonly used in daily clinical practice. Furthermore, Clearfil SE Bond (Kuraray) is considered as the golden standard for self-etch adhesive systems, and Adper Easy One (3M ESPE) is a representative material of one-component self-etching adhesives.

Based on the above considerations, the purpose of this study is and to evaluate the bonding ability of the four above classes of dentin adhesives with and without adhesive area limitation and to evaluate the feasibility of adhesive area delimitation as a standardized laboratory examination method.

2. Experimental method

2.1. Tooth preparation

One hundred and sixty bovine teeth were used as substrates within three months of extraction. The selected bovine excluded the tooth that had caries, tooth discolored and other disordered and had been stored in a 1% aqueous solution of chloramine-T. The labial side (bonding side) of each embedded tooth was grinded with 120-grit silicon paper to expose flat approximately 4 mm diameter of dentin surface, and subsequently, polished the exposed dentin with 600-grit silicon paper for 20s to obtain the

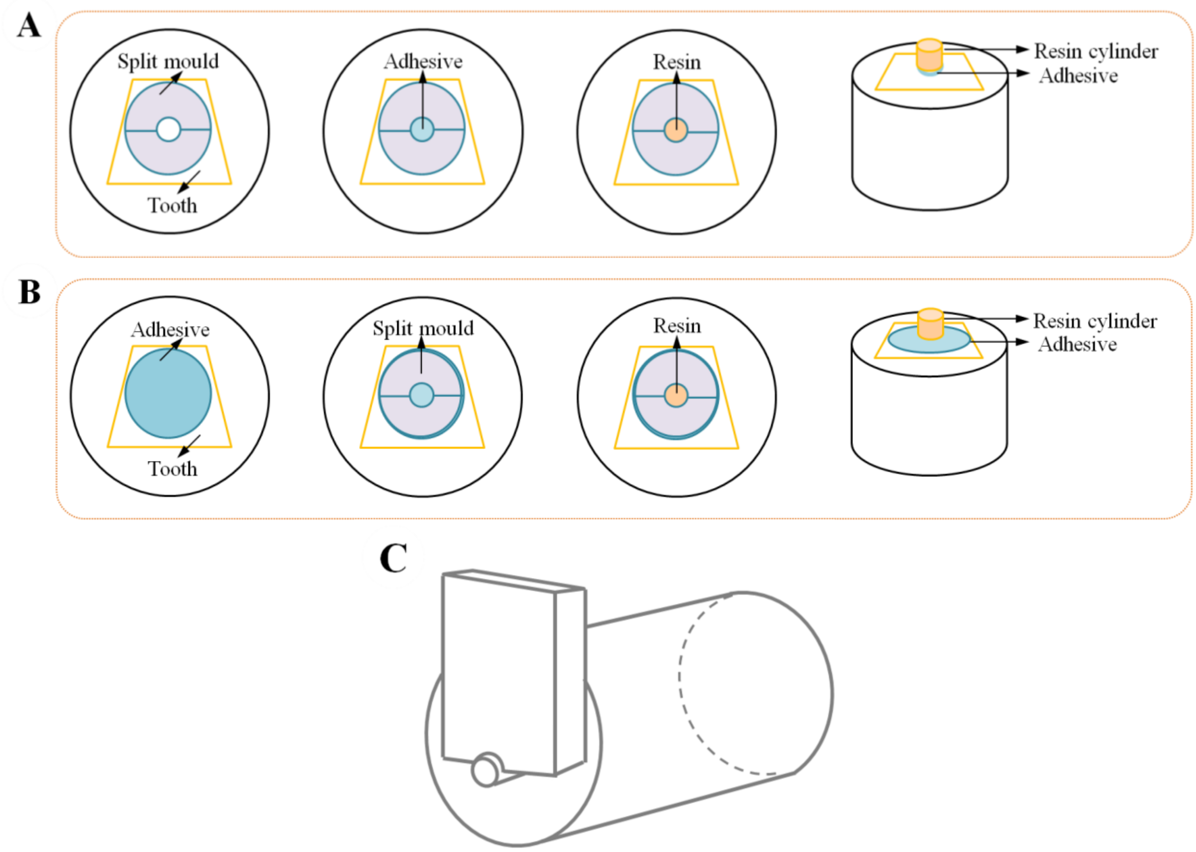


Fig. 1. The two adhesive application methods and the bond strength test apparatus. (A) The method of restricting both the adhesive area and the resin cylinder area. (B) The method of only restricting the resin cylinder area. (C) Diagram of the micro-shear test apparatus.

uniform smear layer.

2.2. Bonding procedures

160 prepared bovine teeth were divided into two groups for applying two different adhesive application methods. In Group I, the adhesive area was constrained to the substrate, which first restricted the bonding area by using a 1mm diameter PTEE split mold on the dentin, and then, the adhesive and composite were filled and cured, respectively, in this mold (Method I) (Figure 1A). In Group II, the adhesives were applied and cured on the entire dentin instead of the limited adhesive area, and then, the 1mm diameter PTEE split mold was used, followed by the construction of the composite cylinder (Method II) (Figure 1B). Each group was then divided into four sub-groups, and each group was treated with one of the four tested adhesives and the corresponding composite resins according to the manufacturers' instructions (Table 1).

2.3. Micro-shear bond strength test

Before testing, the diameter of each bonded resin cylinder was measured with an electronic digital

Table 1

Application of the adhesive materials and composites used in the study

Adhesives	Resins	Application
ARTBond (Coltene) Lot: E91488 (3-E&R)	Synergy D6 (Coltene) Lot: C11664	Apply etchant for 15s→rinse and dry→ apply primer for 30s→ gently blow with air → apply bond for 20s→light cure for 20s →place composite→ light cure for 40s
Adper Single Bond 2 (3M ESPE) Lot:M454303 (2-E&R)	Filtek Z250 (3M ESPE) Lot:N377177	Apply etchant for 15s→rinse and dry→ apply bond→ gently blow with air → light cure for 10s→place composite→ light cure for 20s
Clearfil SE Bond (Kuraray) Lot:071203 (2-SE)	AP-X (Kuraray) Lot:01171A	Apply primer for 20s→dry→Apply bond→ gently blow with air →light cure for 10s→place composite→ light cure for 40s
Adper Easy One (3M ESPE) Lot:493837 (1-SE)	Filtek Z250 (3M ESPE) Lot:N377177	Apply adhesive for 20s→ gently blow with air →light cure for 10s→place composite→ light cure for 20s

caliper (Guanglu, 1-29, China) to confirm the bonding area. The bonded assemblies were immersed in water at 37°C for 24 h, and afterwards, they were used for the μ-SBS tests at a crosshead speed of (1.0 ± 0.1) mm/min until failure. The direction of the applied force is from the cervical to the incisal site of the tooth (Figure 1C).

2.4. Mode of failure after micro-shear bond strength tests

After the μ-SBS tests, all of the de-bonded specimens were observed under a stereo microscope at 40× (Olympics SZX, Japan) and a scanning electron microscope (SEM ZEISS, EV018, Germany) to determine the failure modes. The failure modes were divided into adhesive failure, cohesive failure and mixed failure. The adhesive failure was defined that failure occurred at the dentin/adhesive interface or the adhesive/resin interface. The cohesive failure was defined that the failure occurred at the inside of the resin or dentin. And mixed failure included both adhesive failure and cohesive failure.

2.5. Statistical analysis

A two-way ANOVA was conducted to determine the effect of the adhesive application method, adhesive type, and the interaction of the two factors on the bond strength. For each adhesive application method (with and without adhesive area delimitation), one-way ANOVA tests were used to compare the bond strength of the different adhesive types. For all analyses, Tukey's post-hoc test was used for pairwise comparisons. Moreover, the frequency of the failure modes was analyzed by a Chi-square test. Calculations were handled by the software PASW Statistics 17 (SPSS Inc., Chicago, IL, USA), and all of the tests' accuracy was set at a significance level of 0.05.

3. Results

3.1. Micro-shear bond strength

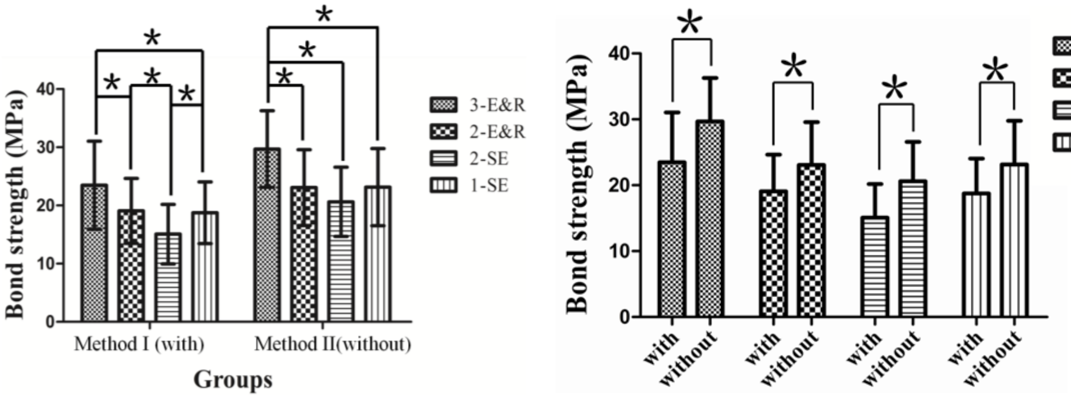
Descriptive statistics of the μ-SBS results are shown in Table 2 and Figure 2. The two-way ANOVA

Table 2

Micro-shear bond strength (Mean±SD, MPa)

Adhesives	Micro-shear bond strength	
	Method I(+)	Method II(-)
3-E&R	$23.48{\pm}7.57^{Cc}$	$29.69{\pm}6.59^{Dd}$
2- E&R	$19.07{\pm}5.59^{Aa}$	$23.08{\pm}6.51^{Ee}$
2-SE	$15.07{\pm}5.13^{Bb}$	$20.63{\pm}5.96^{Ee}$
1-SE	$18.74{\pm}5.30^{Aa}$	$23.15{\pm}6.63^{Ee}$

Note: Means followed by the same upper-case letters within any row are not statistically different (p>0.05). Means followed by the same lower-case letters within any column are not statistically different (p>0.05).



A B

Fig. 2. (A). The micro-shear bond strength of four dentin-adhesive systems with two Methods (with and without adhesive area limitation). Groups with ⋆ represent the significant difference of different adhesives. The bond strength can be distinguished with Method I. Only 3-E&R was observed the significant difference compared to the other adhesives with Method II. (B). The μ-SBS results comparison of a same adhesive with two different adhesive application methods (with and without adhesive area limitation). Groups with ⋆ represent the significant difference of μ-SBS test results of an adhesive with two different methods(Tukey's test, p<0.05). The dentin bond strength of each adhesive without adhesive area limitation distinctly exceeded the corresponding data that was obtained with adhesive area limitation.

indicated that the μ-SBS bond strength was significantly influenced by the adhesive application method, adhesive system type, and the interaction between the two factors (p<0.05). The one-way ANOVA revealed that the μ-SBS test result that was obtained without the adhesive area limitation was significantly higher than the corresponding group with adhesive area limitation, irrespective of the adhesive system types (p<0.05). Significant differences in bond strength were observed when different adhesive systems were applied, and 3-E&R exhibited the highest bond strength as compared to the other adhesives.

3.2. Mode of failure observation

The failure modes of the μ-SBS are shown in Figure 3. The chi-square test of the failure mode frequency of the μ-SBS test specimens indicated that the failure mode frequency was significantly influenced by the adhesive application method for 3-E&R and 1-SE (p<0.05), whereas there was not a significant difference between the two adhesive application methods for either 2- SE or 2- E&R. No cohesive failure was observed for Method I (restricting the area of both the adhesive and the resin).

The SEM observation showed that with Method I, the failure geometry was within the area of the

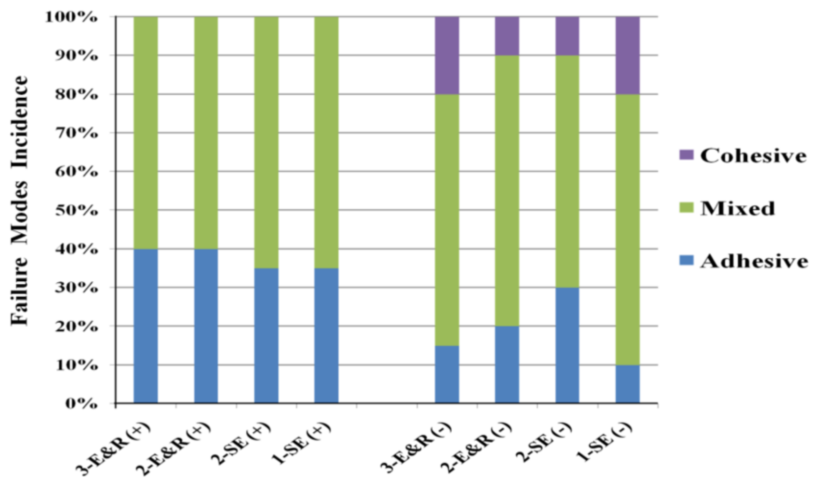


Fig. 3. The failure mode frequency of the μ-SBS test. The Groups with (+) represent the results obtained by the Method I (with adhesive area limitation) and groups with (-) represent the results obtained by Method II (without adhesive area limitation). The groups with Method II express higher cohesive failure percentages.

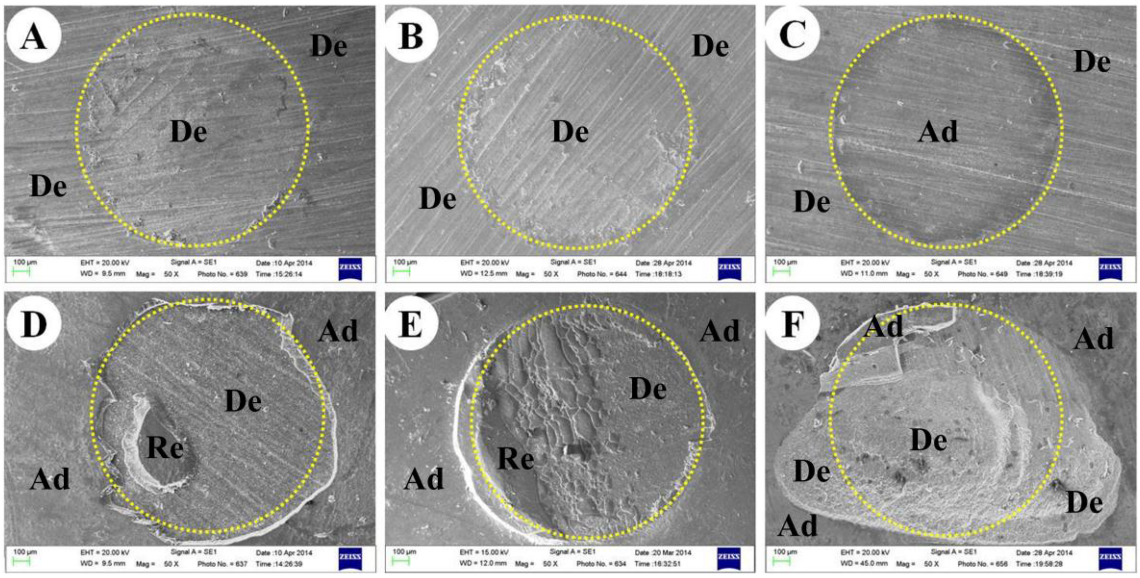


Fig. 4. SEM photomicrographs of the failure modes of the micro-shear test (50x). De: dentin, Ad:adhesive, Re: resin. (A), (B) and (C) are the failure modes with adhesive area limitaiton(restricting both the adhesive and resin cylinder area); (D), (E) and (F) are the failure modes without adhesive area limitaiton(restricting only the resin cylinder area).The dotted loops represent 1 mm of the resin cylinder area. (A) Adhesive failure occurred at the interface between the dentin and the adhesive. (B) Mixed failure. (C) Adhesive failure occurred at the interface between the adhesive and the resin cylinder. (D) Mixed failure; includes most part of the dentin-adhesive interface failure as well as a small part of cohesive failure in resin. (E) Mixed failure; includes the dentin-adhesive interface failure and the cohesive failure in resin. (F) Mixed failure; includes motly cohesive failure in dentin and only a little adhesive area between the adhesive and the dentin.

adhesive and the resin (Figures 4A-4C). Conversely, with Method II, the failure geometry extended beyond the resin area that was used to calculate the bond strength (Figures 4D-4F). The failure detail of each adhesive system is shown in Figure 5.

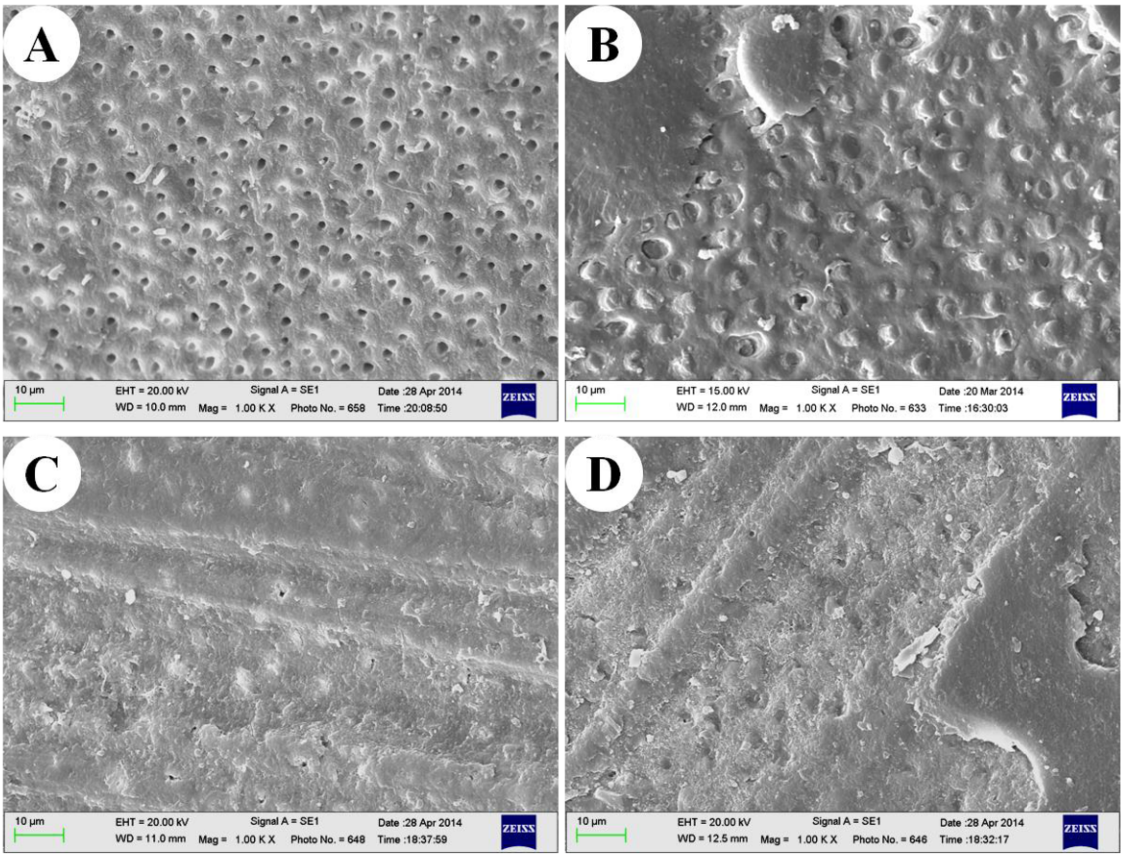


Fig. 5. SEM photomicrographs of the failure modes of the four adhesives (1000x). (A) Cohesive failure in the dentin can be observed in 3-E&R. The dentinal tubule can be observed clearly, and few resin-tags were observed. (B) The 2-E&R failure generally occurred at the adhesive/resin interface. The etched dentin was covered with the adhesive and the dentinal tubules were filled with resin tag. (C) The 2-SE failure tended to occur at the dentin/adhesive interface. It is hard to observe the open dentinal tubules. (D) The failure mode of 1-SE tended to occur at the adhesive/resin interface. Mixed failure was observed in 1-SE.

4. Discussion

The rapid development of adhesive restorative dentistry allows for more conservative and less invasive treatment. In addition, it overcomes the esthetical problems of placing restorations as well as lengthens the lifespan of the teeth. With the development of adhesive restorative dentistry, many new dental adhesive materials were produced. The etch-and-rinse adhesive system and the self-adhesive system are the most frequently employed in clinical works [20]. Before new adhesive materials can be put into clinical use, it must be evaluated through various laboratory methodologies; one such methodology is the μ-SBS test, which is one of the most essential tests for predicting the efficiency of new adhesives [20-22]. However, the reproducibility of testing results is affected by various factors regarding the inherent weakness of test institute [3]. Although ISO 11405 [23] strictly stresses that “a limitation of the bonding area is important,” in most μ-SBS tests, the step of restricting the area of both the adhesive and the resin was often ignored [22, 24-27], and only the area of the resin cylinder was taken as the bonding area to calculate the bond strength of tested materials. Therefore, the objective of this study was to compare the bond quality of different adhesive systems by employing the μ-SBS test with and without adhesive area restriction. From the results, it was determined that 3E&R has the highest bond strength. In addition, adhesive area limitation is the paramount step for controlling variability in μ-SBS tests.

Restricting the adhesive area in μ-SBS tests is crucial for evaluating and comparing the bonding quality of different dentin-adhesive systems. In this study, restricting the area of both the adhesive and the resin (Method I) and restricting the resin area only (Method II) were respectively applied, and the dentin bond strength of each adhesive without adhesive area limitation distinctly exceeded the corresponding data that was obtained with adhesive area limitation. This is probably due to the difference between the bonding area and the actual tested bonding area with Method II. In the process of the bond strength test, the actual tested bonding area includes not only the area of the resin cylinder but also includes the area of the cured adhesive around the resin cylinder, and the latter was not counted as the bonding test area to calculate bond strength, resulting in the higher nominal bond strength. This outcome was in accordance with R. Van Noort [15] and N. Pecora [28], and the researchers concluded that applying the adhesive on the whole dentin and without adhesive area limitation potentially affects the measurements of dentin tensile or shear bond strength. Moreover, the adhesive property of the different adhesives can be distinguished with Method I (Figure 2), but this is not the case for Method II. In most traditional μ-SBS tests [12, 13, 22, 24-27], the area of the resin cylinder was taken as the bonding area to calculate the bond strength of the tested materials. Therefore, the higher nominal bond strength was obtained and only if massive improvement in bond quality can be distinguished with method II.

The failure mode analysis is considered to be an important parameter for interpreting tests results. In fact, it has been reported that there is a direct positive correlation between the bond strength and cohesive failure [29]. In this study, Method II groups express a higher cohesive failure percentage (Figure 3). From the theoretical aspect, if the typical adhesive failure occurred between the adhesive and the resin cylinder, the bond strength obtained from the two different bonding area restriction methods should always be consistent; however, it is apparent that this testing bond strength cannot represent the bond strength of the adhesive to dentin. When the adhesive failure occurred between the dentin substrate and the adhesive, the bond strength obtained with Method II should obviously surpass the respective data obtained with Method I, and the resultant data obtained with Method II also cannot be identified as the bond strength of this adhesive to dentin. This is primarily because the actual testing area exceeds the bonding area that is calculated with Method II (Figures 4D-4F). In previous studies, the validity of nominal bond strength in μ-SBS tests has been questioned due to a high cohesive failure incidence rate [30]. However the reason for the common occurrence of premature cohesive failure prior to the interface failure is the improved bond strength of modern adhesives as well as the imprecision of bonding area restriction and calculation, which is in line with previous research [14, 15, 28]. Moreover, R. Van Noort [15] concluded that the stress concentration is situated around the circumference of the resin composite cylinder at the adhesive/resin composite interface when the method mode of adhesive flash is applied by finite element analysis. In this study, the analysis of the failure mode indicates that the cohesive failure rate of the method without adhesive area limitation is higher than the cohesive failure rate of the method with adhesive area limitation.

In addition, two methods (with and without adhesive area limitation) were used in μ-SBS test in this study in order to accurately evaluate and compare the bonding quality of different dentin-adhesive systems. In this study with Method I, the bond strengths of 3-E&R and 2-E&R were superior to that of 2-SE. Furthermore, the SEM image at 1000× can clearly distinguish the resin-tag that formed following the primer/adhesive application on the smear layer-free and demineralized dentine (Figures 5A and 5B), and this is perhaps ideal bonding to dentin, which is in agreement with other researchers [16, 20]. Although the bond strength of 2-SE is lower (possibly because its primmer with high viscosity has an adverse effect on the penetration of resinous monomers into the collagen network leading to the non-ideal hybrid layer formation), there is less adhesive infiltration (Figure 5C) and compromising the bond quality, which is in accordance with some studies [31]. Moreover, wet bonding is very technique-sensitive, and proper water content plays a crucial role in the bonding process. The all-in-one self-etch adhesive system incorporates acid monomers and hydrophilic and hydrophobic components in one bottle. In order to accomplish hard tissue demineralization and resin infiltration simultaneously, more acidic and more hydrophilic counterparts were incorporated, thereby decreasing the technique-sensitivity of wetting bonding.

In this and previous studies, the all-in-one self-etch system achieves acceptable dentin bond strength [32, 33], but the durability of the all-in-one self-etch system remains a matter of concern.

5. Conclusion

Based on the results of this study, it can be concluded that the adhesive limitation is a vital step for controlling the variability in micro-shear bond strength tests. Furthermore, from the aforementioned results, it was demonstrated that restricting the area of both the adhesive and the resin is a more reliable method for evaluating the bonding property of adhesives to dentin. With regard to the four representative dentin-adhesive systems, 3-E&R has the superior bond quality compared to the other adhesive systems.

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