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Effect of Double-application of Three Single-step Self-etch Adhesives on Dentin Bonding and Mechanical Properties of Resin-dentin Area

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Clinical Relevance

Double-application enhanced the hardness of bonding layer and resin-dentin interface for all the adhesives used; however, it only improved the bond strength to dentin for some of the single-step self-etch adhesives.

SUMMARY

Purpose: This study investigated whether double-application influences the bond strength of single-step self-etch adhesives and the mechanical properties of the resin-dentin area.

Materials and Methods: Three single-step self-etch adhesives (EXL-683, experimental, 3M ESPE; Clearfil Tri-S Bond, Kuraray Medical; G BOND, GC) were applied on dentin surfaces by using the single-application or double-application method. The manufacturers' instructions stated use of the single-application method. Resin composite (Clearfil AP-X, shade A3, Kuraray Medical) was then placed and light cured for 40 seconds. The microshear bond test was carried out and the bond strength data were analyzed using one-way ANOVA with post hoc. The hardness (H) of the bonding layer, resin-dentin interface and dentin beneath the interface at depths of 10 µm and 100 µm were measured with a nanoindentation device (ENT-1100, Elionix). The H data were analyzed using two-way ANOVA, one-way ANOVA and the independent *t*-test at a significance level of α=0.05.

Results: The mean bond strength with single-applications of EXL-683, Clearfil Tri-S Bond and G BOND were 39.1, 36.9 and 30.0 MPa, respectively, while with the double-application, they were 46.9, 40.2 and 32.2 MPa. Double-application for

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EXL-683 to dentin showed significantly higher bond strength than single-application α<0.05. However, there was no significant difference between the single- and double-application methods for the other two adhesives α>0.05. The H of the bonding layer was significantly influenced by both the adhesive material and application method α<0.001; the H of the bonding layer with a double-application was significantly improved compared to a single-application. The H of the resin-dentin interface was significantly affected by the application method α<0.001, but not by the adhesive material (α>0.05). Neither the H value of dentin at 10 µm nor a depth of 100 µm was affected by the adhesive material or application method (α>0.05).

Conclusions: The double application of EXL-683 did improve the bond strength to dentin. The hardness of the bonding layer and resin-dentin interface was significantly affected by the double application for each material used.

INTRODUCTION

Two-step self-etch adhesive technology was followed by the development of single-step self-etch adhesives. The advantage of the single-step self-etch adhesives is that a relatively simple procedure is involved, one that minimizes the steps of the bonding process. However, some previous studies have reported that the bond strength of single-step self-etch adhesives to dentin does not exceed that of two-step self-etch adhesives.[1-3] Controversies over the performance of these adhesives with regard to technique sensitivity also have been indicated.[4] Water, solvents and adhesive monomers mixed into a single-step self-etch adhesive resin may result in reduced mechanical properties and, accordingly, poor bonding performance.[5] It has been reported that the application of two coats (double-application) of a single-step self-etch adhesive increases the tensile bond strength to sound dentin,[6] and the multiple consecutive coating of another single-step self-etch adhesive can reduce nanoleakage.[7]

Hardness measurement by the nanoindentation method has been used to measure the hardness and elastic modulus of the dentin adhesives and resin-dentin area.[5,8-10] Van Meerbeek and others reported that the hardness of the resin-dentin inter-diffusion zone was significantly lower than that of unaltered dentin.[9] This zone is regarded as having a strain capacity capable of relieving stresses between the changes shrinking the composite restoration and the rigid dentin substrate and, thereby, improving conservation of the dentin bond.[9] Therefore, the effect of double-application of current single-step self-etch adhesives on the bond strength and mechanical property of the resin-dentin area is of crucial interest.

The current study investigated whether double application influenced the bond strength of single-step self-etch adhesives and the mechanical properties of the resin-dentin area. The null hypothesis is that the bond strengths of single-step self-etch adhesives and mechanical properties are not significantly different between the single-application and double-application method.

METHODS AND MATERIALS

The adhesives and their compositions used in the current study are listed in Table 1. Three single-step self-etch adhesives, Clearfil Tri-S Bond (Kuraray Medical, Tokyo, Japan), G BOND (GC, Tokyo, Japan) and EXL-683 (experimental, 3M ESPE, St Paul, MN, USA) were used.

Twenty-one extracted intact human molars stored for less than three months at 4°C in normal saline saturated with thymol were used in this *in vitro* study.

Sectioning of the Teeth

The roots of the teeth were cut out at the cemento-enamel junction using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water coolant. The samples were then cut parallel to the proximal surfaces through the central part of the teeth, and the resulting specimens were further sectioned by cutting in the same direction to provide two slices, each 2.0 mm thick. Accordingly, 42 dentin slices were totally obtained from 21 teeth. Thirty slices were used for the microshear bond test, six for the nanoindentation test and six for SEM observation (Figure 1).

Microshear Bond Strength Tests

Thirty sectioned slices were randomly assigned to six equal groups of five dentin slices each. The tooth slices were polished with #600 SiC paper to create a standard smear layer for the bonding procedures. Then, the adhesives were applied on the dentin surfaces by using one of the two methods: single-application (EXL-683/Single, Tri-S Bond/Single, G BOND/Single) or double application (EXL-683/Double, Tri-S Bond/Double, G BOND/Double). The single-application method followed the manufacturers' instructions. In the double application, the adhesive was applied two times; for each coating, the application time and air drying were followed according to the manufacturers' instructions. The second adhesive layer was applied without photo curing the first layer. Prior to light irradiation of the bonding resin on each specimen, two Tygon tubes (Norton Performance Plastics, Granville, NY, USA), 0.5 mm in height and 0.75 mm in diameter, were placed on the dentin surfaces. The bonding agents were then irradiated with a halogen light cure unit for 10 seconds and a hybrid restorative composite Clearfil AP-X, Shade A3 (Kuraray Medical), was inserted into the tubing lumens and irradiated for 40 seconds (Figure 1). The

Table 1: *List of Used Materials*

Materials (Manufacturers)	Composition	Lot #	Application Technique
Clearfil Tri-S Bond (Kuraray Medical, Tokyo, Japan)	MDP, Bis-GMA, HEMA, ethanol, photoinitiator, hydrophobic dimethacrylate, microfiller, water	00044B	Single: a (20 s) →b →c Double: a (20 s) →b →a (20 s) →b →c
G BOND (GC Company, Tokyo, Japan)	4-MET, methacrylic acid ester, acetone, water	0509201	Single: a (10 s) → b → c Double: a (10 s) →b →a (10 s) →b →c
EXL-683 (3M ESPE, St Paul, MN, USA)	phosphoric acid-methacryloxy-hexylesters, Bis-GMA, HEMA, ethanol, silane treated silica, 1,6-hexanediol dimethacrylate, copolymer of acrylic, water, itaconic acid, Phosphine oxide, camphorquinone	P-0282	Single: a (20 s) →b →c Double: a (20 s) →b →a (20 s) →b →c
Clearfil AP-X (Kuraray Medical, Tokyo, Japan)	Hybrid resin composite containing: Bis-GMA, TEGDMA, photoinitiator, microfiller	01112	c (40 s)

a: apply adhesive; b: air dry; c: light cure; s: second

Bis-GMA: bisphenol-A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA: triethyleneglycodimethacrylate; 4-MET: 4-methacryloyloxy ethyltrimellitic phosphate.

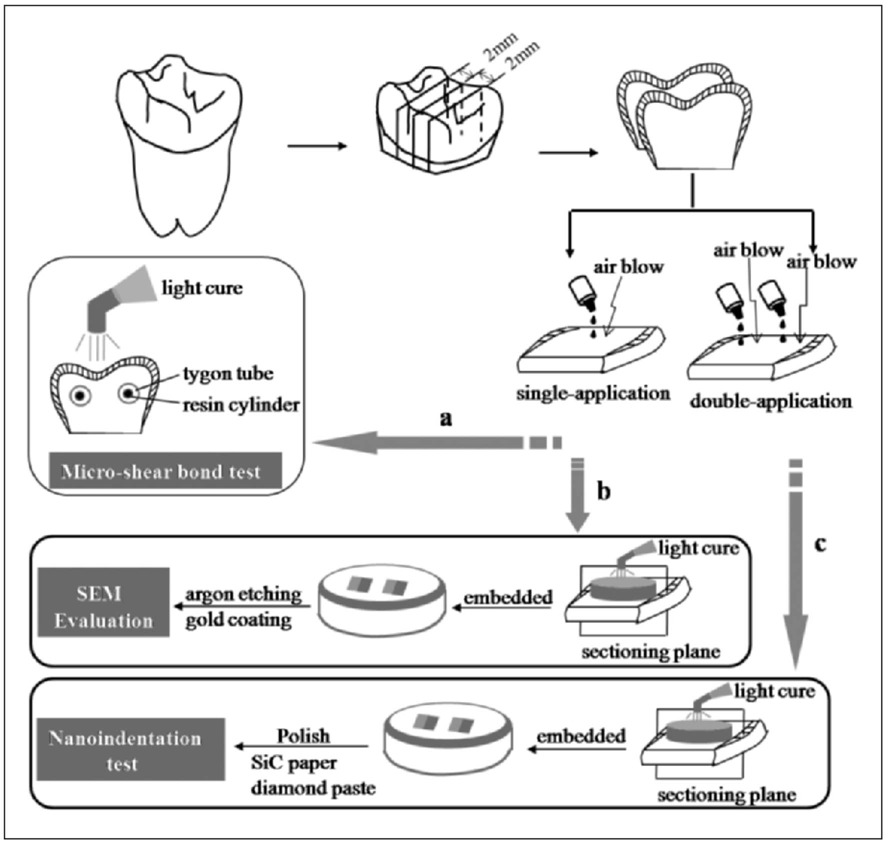


Figure 1. *Experimental procedure. The teeth were cut parallel to the proximal surfaces to provide two slices 2.0 mm thick. Each of the adhesives was applied to the dentin slice surfaces by single application or double application, then the specimens were further subjected to the micro-shear bond test (a) nano-indentation test (b) and SEM observation (c).*

tygon tubes around the composite cylinders were carefully removed after one hour using a steel blade, then the specimens were stored in distilled water at 37°C for 24 hours. In this manner, two resin cylinders were created on each tooth slice and 10 samples were created for each group. For the microshear bond test, a thin wire (0.2 mm in diameter) was looped flush between the load cell projection and the resin cylinder, touching the tooth surface. The force was applied at a crosshead speed of 1 mm/minute until failure (Figure 2). The shear force at failure was recorded and converted to shear bond strength in an MPa unit. Ten bond strength values were obtained for each group. The values of the microshear bond tests were then analyzed for normality and equal variances and were compared using one-way ANOVA with Dunnett's T3 at a 0.05 level of significance.

Failure Mode

After measuring the microshear bond strength, the specimens were gold sputter-coated and observed by scanning electron microscope (SEM) (JSM-5310LV, JEOL, Tokyo, Japan). The fractured surfaces were classified according to the following criteria: cohesive failure (CB): cohesive failure in bonding resin;

mixed failure (M_1): partly adhesive and partly cohesive in resin (either with bonding or composite); adhesive failure (A): failures between adhesive resin and dentin; mixed failure (M_2): partly adhesive and partly cohesive in dentin, either with partly cohesive in bonding resin. The results of the failure mode were statistically analyzed using the Pearson's Chi-square test at α=0.05 level.

Naonindentation Tests

Six dentin slices were used for the nanoindentation measurement. The tooth slice surfaces were polished using #600 SiC paper under running water and bonded with the corresponding adhesives used in the microshear bond test by the single-application or double-application method, followed by a composite buildup (Clearfil AP-X, shade A3). After storing in water at 37°C for 24 hours, the bonded specimens were sectioned into two halves perpendicular to the resin-dentin interface. The resulting samples were embedded in epoxy resin, with the resin-dentin interface facing out and polished using SiC papers under running water and diamond pastes with particle sizes down to 0.25 µm (Figure 1). After polishing, the nanoindentation test was performed using a nanoindentation device (ENT-1100, Elionix, Tokyo, Japan). The default temperature of the test chamber was set by the manufacturer at 27.5°C. Using the device software, hardness (H) was calculated following standard Elionix procedures. Indentations were made at the bonding layer (H1), the resin-dentin interface (H2), dentin at 10 µm (H3) and a 100 µm (H4) depth away from the interface (Figure 3). For the H measurement of the bonding layer, the positions of the indentation points were programmed at the approximate half-width of the bonding layer. The indentations were performed on points at a loading speed of 0.05 mgf/msec that reached a maximum loading of 500 mgf. The indents were then observed under the charge coupled device (CCD) microscope to confirm that they had appropriate shape. For each of the locations, the average of the H values of each 15 points was calculated. The hardness values were analyzed using two-way ANOVA (Univariate Analysis of Variance), one-way ANOVA with post-hoc and independent *t*-test at a 0.05 level of significance.

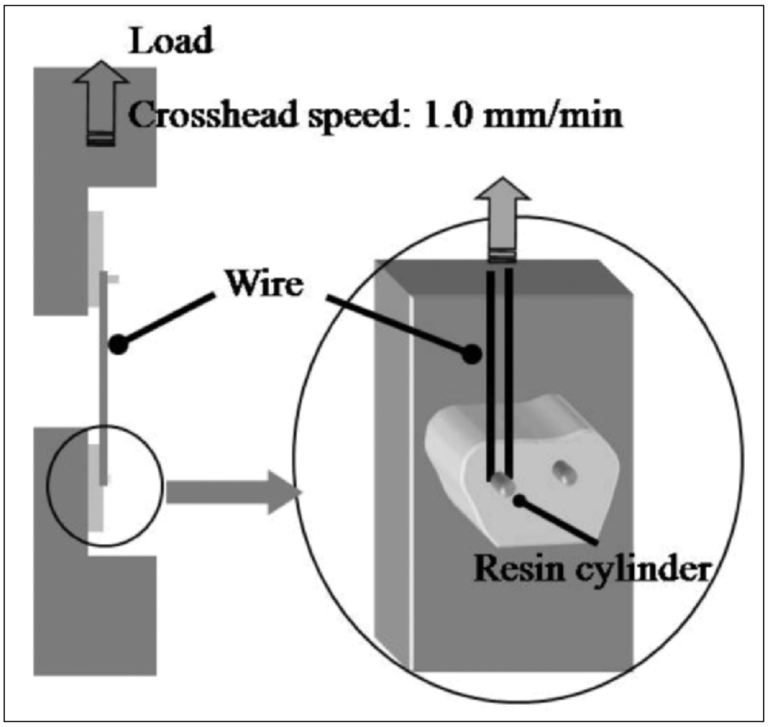


Figure 2. *A schematic of the microshear bond test apparatus.*

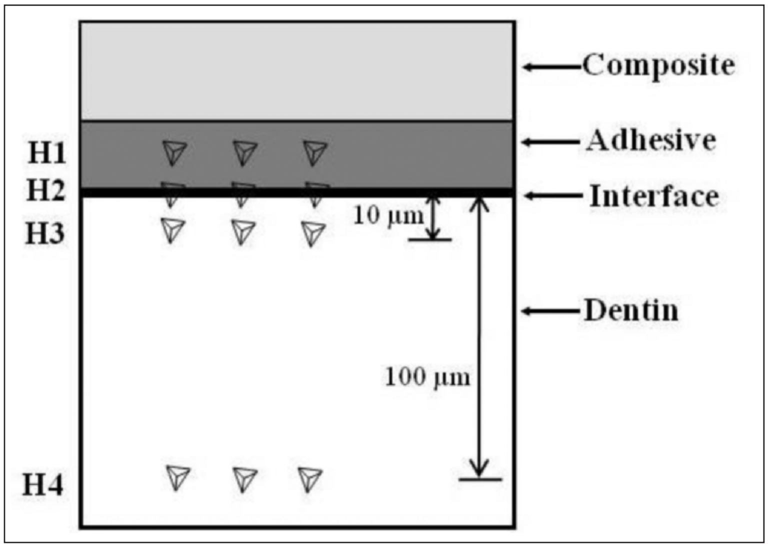


Figure 3. *A schematic of the positions of indentations in the nano-indentation test. H1: adhesive layer; H2: resin-dentin interface; H3: dentin at 10 µm depth; H4: dentin at 100 µm depth.*

SEM Observation of Resin-dentin Interface

The remaining six slices were morphologically used for SEM observation of the resin-dentin interfacial structures. The dentin surfaces were polished using #600 SiC paper under running water and bonded with three corresponding adhesive materials using either the single-application or double-application method followed by a composite buildup (Clearfil AP-X, shade A3). After storing in water at 37°C for 24 hours, the bonded specimens were sectioned perpendicular to the resin-dentin interface, and the resulting samples were embedded in epoxy resin with the resin-dentin interface facing out. The embedded specimens were polished using SiC papers and diamond pastes, with particle sizes down to 0.25 µm subjected to argon-ion beam etching, gold sputter-coated and observed under the SEM.

RESULTS

Microshear Bond Test

The results of the bond test are presented in Table 2. EXL-683/Double showed significant improvement of bond strength compared to EXL-683/Single (α=0.015), but there was no significant difference between the single- and double-application for the other two materials (α=0.918 and α=0.626, respectively). EXL-683 and Tri-S Bond had significantly higher bond strengths compared to G BOND, regardless of the application method.

Table 2: *Microshear Bond Strength Mean Values (SD) in MPa (n=10)*

	Adhesive		
	EXL-683	Tri-S Bond	G BOND
Single-application	39.1^{b} (3.27)	36.9^{b} (4.47)	30.0^{c} (3.20)
Double-application	46.9^{a} (5.26)	40.2^{b} (6.30)	32.2^{c} (2.78)

In each row or column, groups with different superscript letters are significantly different (p<0.05, one-way ANOVA with Dunnett T3).

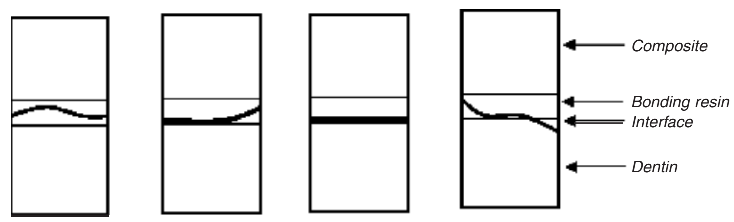
Failure Mode

Table 3 illustrates the results of the failure mode. The Pearson's Chi-square test indicated that statistically significant differences existed among adhesives (α<0.001). Although the failure pattern was different among the adhesives used, EXL-683 and Tri-S Bond showed a similar failure mode in single- and double-application methods. On the other hand, G BOND showed different failures between the single- and double-application.

Table 3: *Failure Mode Frequencies (n=10)*

	CB	MI	A	M2
EXL-683/Single	0	0	0	10
EXL-683/Double	0	1	0	9
Tri-S Double/Single	0	5	2	3
Tri-S Double/Double	0	3	4	3
G Bond/Single	1	4	3	2
G Bond/Double	3	0	0	7

CB: cohesive failure in bonding resin.
MI: partly adhesive and partly cohesive in resin (either with bonding or composite).
A: failure between adhesive resin and dentin.
M2: partly adhesive and partly cohesive in dentin, either with partly cohesive in bonding resin.



For EXL-683, mixed failure (type M2) was the dominant failure mode in single- and double-applications. For Tri-S Bond, adhesive failure (type A) and mixed failure (type M1 and type M2) were observed in most cases. In G BOND, mixed failures (type M1 and type M2) occurred most frequently for both application methods, with the remainder showing cohesive failure in bonding resin (type CB), which was not observed in either of the other two adhesives.

Table 4: *Mean Values (SD) of the Hardness of Resin-dentin Area for All Groups (mgf/μm²)*

Material	Location of Indents	Hardness: Single Application		Hardness: Double Application
EXL-683	H1	37.60 (1.8)^{b}	——	51.29 (3.8)^{A}
	H2	50.27 (2.0)^{c}	——	55.59 (3.7)^{C}
	H3	80.90 (5.3)^{d}		84.76 (2.7)^{D}
	H4	79.30 (3.6)^{e}		83.32 (8.3)^{E}
Tri-S Bond	H1	33.52 (2.0)^{a}	——	43.90 (2.5)B
	H2	50.18 (3.7)^{c}	——	59.13 (3.8)^{C}
	H3	85.98 (2.7)^{d}		85.73 (4.9)^{D}
	H4	82.47 (4.3)^{e}		83.51 (3.4)^{E}
G Bond	H1	36.84 (3.6)^{b}	——	42.56 (1.4)B
	H2	51.43 (4.7)^{c}	——	61.32 (7.3)^{C}
	H3	82.00 (5.6)^{d}		83.01 (6.3)^{D}
	H4	79.66 (5.9)^{e}		82.08 (8.1)^{E}

H1: adhesive layer *H2: resin-dentin interface*
H3: dentin at 10 μm depth *H4: dentin at 100 μm depth*

—— For each material-hardness data set (within materials), groups connected with line are significantly different. (n=15, p<0.05, independent t-test)

In each column, for similar nanoindnetation locations (between materials), groups with different letters are significantly different. (n=15, p<0.05, one-way ANOVA with Dunnett's T3 or Tukey HSD)

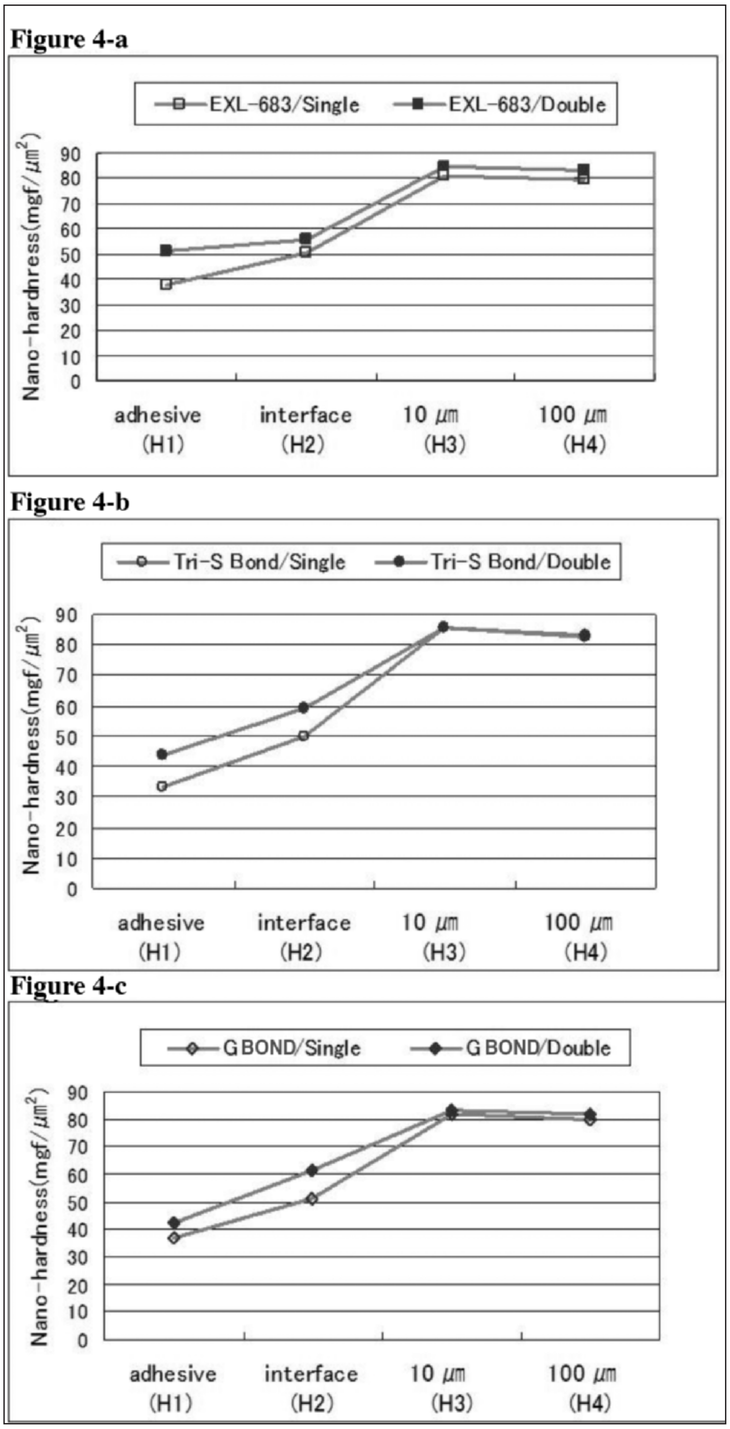


Figure 4a: *Nano-indentation hardness of the resin-dentin area of EXL-683.*
Figure 4b: *Nano-indentation hardness of the resin-dentin area of Tri-S Bond.*
Figure 4c*: Nano-indentation hardness of the resin-dentin area of G BOND.*

SEM Observation of the Resin-dentin Interface

Very thin hybrid layers (less than 0.5 µm thick) were observed for all three single-step self-etch adhesives in both the single- and double-application. The application method did not appear to affect the thickness of the hybrid layer; meanwhile, it was difficult to distinguish morphological differences at the resin-dentin interface between the tested materials on SEM micrographs (Figure 5).

Nanoindentation

The means and standard deviations of nano-indentation hardness results are included in Table 4. Two-way ANOVA (Univariate Analysis of Variance) showed that the H of the adhesive layer (H1) was significantly influenced by the adhesive material ($F=17.697$, $\alpha<0.001$) and the application method ($F=127.170$, $\alpha<0.001$). The interaction of these two factors was significant ($F=8.915$, $\alpha<0.001$). The H of interface (H2) was significantly affected by the application method ($F=38.123$, $\alpha<0.001$) but not the adhesive material ($F=2.296$, $\alpha=0.112$), and the interaction of the factors was not significant ($F=1.099$, $\alpha=0.342$). Both the H values of dentin 10 µm (H3) and 100 µm (H4) deep beneath the interface were neither affected by adhesive material nor the application method. One-way ANOVA with post-hoc tests showed that at the H1 location, the H of EXL-683 was significantly higher compared to the other two adhesives when applied in two coats ($\alpha=0.016$ and $\alpha=0.007$, respectively). When applied as a single coat, Tri-S Bond showed significantly lower hardness of H1 compared to the other two adhesives ($\alpha=0.001$ and $\alpha=0.029$, respectively). Independent *t*-tests within each adhesive showed that double application significantly improved the nanoindentation hardness of H1 and H2 over the single application in all adhesives ($\alpha<0.05$) but did not influence the hardness of H3 and H4 ($\alpha>0.05$).

DISCUSSION

In contemporary single-step self-etch adhesives, a significant amount of water and solvent, which is expected to be eliminated from the adhesive layer by sufficient evaporation prior to curing of the adhesive resin, is included in the adhesive container. Several lines of evidence showed the effect of water or the remaining solvents on the bonding performance of single-step self-etch adhesives.[8,11-12] One of the mechanisms by which those ingredients may adversely affect adhesive performance is decreasing polymerization efficacy and altering the mechanical properties.[5]

In the current study, the nanoindentation hardness of H1 and H2 significantly increased in the double application for all the three adhesives (Table 4), suggesting that the double application increased the mechanical properties of the adhesive layer and the resin-dentin interface, regardless of the acetone-based or ethanol-based nature of the adhesive materials.

Theoretically, it may be expected that the results of the bond strength test would reflect increased mechan-

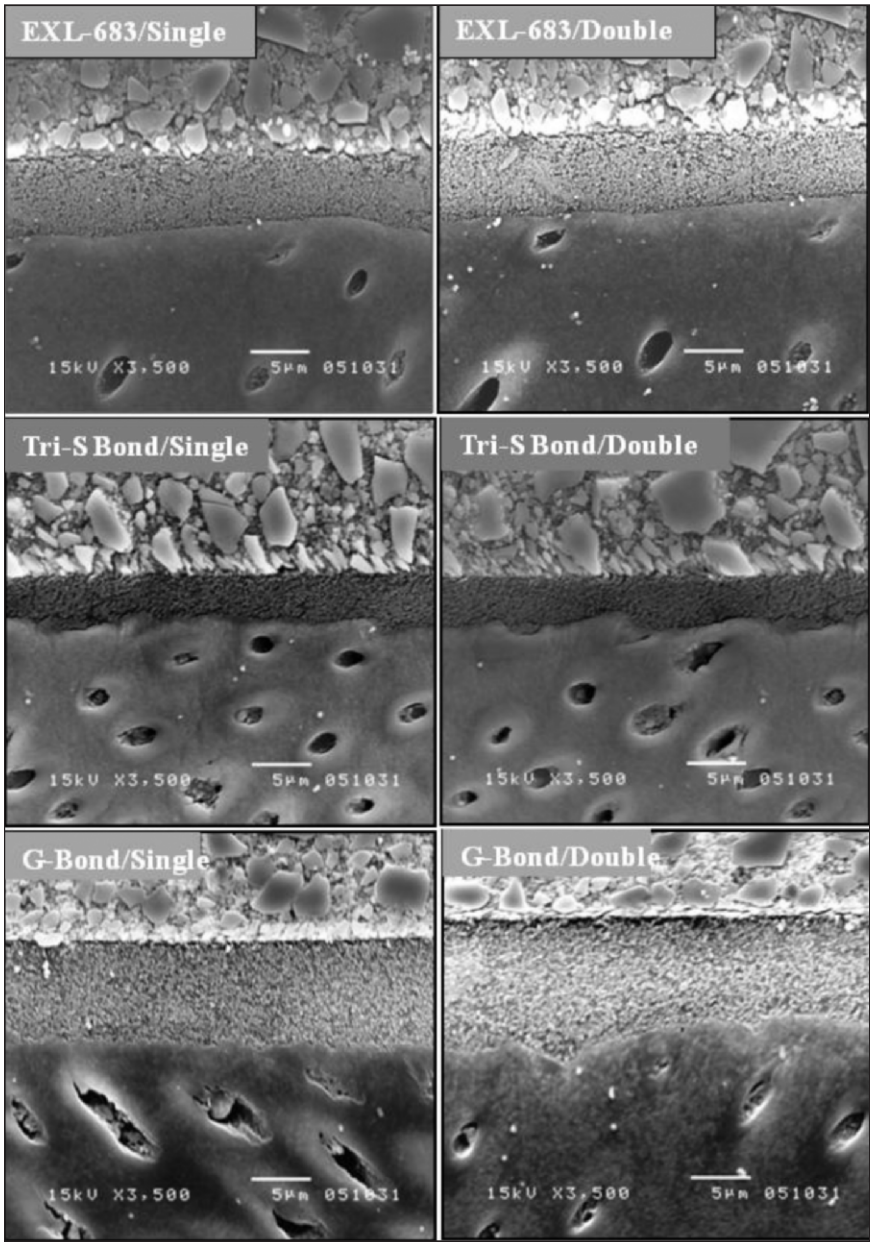


Figure 5. *The SEM micrographs of the resin-dentin interface.*

ical properties of the resin-dentin area after double application. However, only EXL-683/Double showed significant improvement of bond strength, while double application of the other adhesives showed little effect on the results (Table 2). Previous studies reported that tensile bond strengths to dentin significantly correlated with such mechanical properties of the resin as ultimate tensile strength or flexural strength but not with nanoindentation hardness and Young's modulus.[13-14] Thus, increased hardness of the resin-dentin interface may not be considered a determining factor for the increased bond strength of resin-dentin bonding.

EXL-683 showed a similar fracture mode (mostly type M2 failure) between the single- and double-application (Table 3). This may represent the stable mechanics of adhesive EXL-683 after light curing in both application methods. Additionally, the H1 value of EXL-683/double, over 50 mgf/μm^2, was significantly higher than that of the other adhesives (Table 4). EXL-683 also showed significantly higher bond strength in double application over the single-application. It was initially thought that the double application of EXL-683 might improve the priming effect on the tooth surface or it might effectively remove the water remaining on the surface. The ratio of cross-linked polymer within EXL-683 may be improved after the double application, which would enhance the mechanical properties of the adhesive, resulting in increased bond strength. It has been suggested that the filler contained in the adhesives would increase the mechanical strength of the adhesive layer.[15-18] Several researchers have discussed the optimum filler level for the maximum increase in bond strength.[15-17] In the current study, EXL-683 showed the greatest range of changes in hardness between single- and double-application among the three adhesives (Table 4); however, it is not known whether double-application of EXL-683 affected filler distribution in the current experiment.

According to the results, the double application of Tri-S Bond significantly improved H of the bonding layer and the underlying resin-dentin interface (Table 3), but it did not significantly affect the bond strength of this material (Table 2). However, when observing the fractured surfaces, the double application showed a tendency towards more frequent type A failures and fewer type M1 failures (Table 3). It might be due to the improved fracture toughness of the bonding resin by double application, resisting cohesive failure in the adhesive layer.

EXL-683 and Tri-S Bond both include HEMA, ethanol and water. Carvalho and others[20] reported that the solubility parameters of HEMA/ethanol mixtures can significantly improve bond strength to dentin by modifying the final degree of expansion of the dried matrix. This might explain the reason why, either in single application or in double application, the bond strength of EXL-683 and Tri-S Bond was significantly higher than that of G BOND.

The G BOND group presented no significant difference in bond strength between the single and double application (Table 2). G BOND is a HEMA-free, acetone/water based one-step self-etch adhesive. Acetone is more volatile than ethanol and does not form an azeotrope with water, so it may not promote water evaporation when compared to ethanol-based adhesives.[21] Lower polymerization efficiency due to incomplete removal of water and phase-inhomogenity from the adhesive layer (due to a lack of HEMA) may result in crack formation in the adhesive layer, leading to premature bond failure.[22-24] This can partly explain the lower bond strength and the observed mode of failure after the bond test. More type M2 and type CB failures were observed for double application compared to single application. Type CB was only found in G BOND specimens, but it could not be observed in the other two adhesives (Table 3).

It was difficult to detect variations in the characteristics of the hybrid layer among the adhesives on the SEM micrographs in the current study. The thickness of the hybrid layers observed was similar between application methods for each material and did not present any relation to the values of bond strength.

In this *in vitro* study, the effect of double application of single-step self-etch adhesive was detected and the null hypotheses were partially rejected. The effect of double application on bond strength was material-dependent; while improvement of the interfacial hardness by double application was adhesive material-independent.

CONCLUSIONS

The double application of EXL-683 improved bond strength on dentin; however, the double application did not improve the bond strength on dentin for the remaining two adhesives. The hardness of the bonding layer and that of the resin-dentin interface was significantly improved by the double application of each material used

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References

1. Ishikawa A, Shimada Y, Foxton RM & Tagami J (2007) Microtensile and micro-shear bond strengths of current self-etch adhesives to enamel and dentin *American Journal of Dentistry* **20(3)** 161-166.
2. Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H & Van Meerbeek B (2001) Microtensile bond strength of eleven contemporary adhesives to dentin *Journal of Adhesive Dentistry* **3(3)** 237-245.
3. De Munck J, Van Meerbeek B, Satoshi I, Vargas M, Yoshida Y, Armstrong S, Lambrechts P & Vanherle (2003) Microtensile bond strengths of one- and two-step self-etch adhesives to bur-cut enamel and dentin *American Journal of Dentistry* **16(6)** 414-420.
4. Chersoni S, Suppa P, Grandini S, Goracci C, Monticelli F, Yiu C, Huang C, Prati C, Breschi L, Ferrari M, Pashley DH & Tay FR (2004) *In vivo* and *in vitro* permeability of one-step self-etch adhesives *Journal of Dental Research* **83(6)** 459-464.
5. Van Meerbeek B, Willems G, Celis JP, Roos JR, Braem M, Lambrechts P & Vanherle G (1993) Assessment by nano-indentation of the hardness and elasticity of the resin-dentin bonding area *Journal of Dental Research* **72(10)** 1434-1442.
6. Nara Y, Nagakumi Y, Ito Y, Kisuki I & Kimishima (2003) Tensile bond strength of single-step self-etch adhesive system to cervical abrasion lesion dentin *Journal of Dental Research* **81(Special Issue B)** Abstract #55.
7. Hashimoto M, Sano H, Yoshida E, Hori M, Kaga M, Oguchi H & Pashley DH (2004) Effects of multiple adhesive coatings on dentin bonding *Operative Dentistry* **29(4)** 416-423.
8. Sadr A, Shimada Y & Tagami J (2007) Effects of solvent drying time on micro-shear bond strength and mechanical properties of two self-etching adhesive systems *Dental Materials* **23(9)** 1114-1119.
9. Sadr A, Shimada Y, Lu H & Tagami J (2009) The viscoelastic behavior of dental adhesives: A nanoindentation study *Dental Materials* **25(1)** 13-19.
10. Wei SH, Sadr A, Shimada Y & Tagami J (2008) Effect caries-affected dentin hardness on shear bond strength of current adhesives *Journal of Adhesive Dentistry* **10(6)** 431-440.
11. Ikeda T, De Munck J, Shirai K, Hikita K, Inoue S, Sano H, Lambrechts P & Van Meerbeek B (2005) Effect of evaporation of primer components on ultimate tensile strengths of primer-adhesive mixture *Dental Materials* **21(11)** 1051-1058.
12. Jacobsen T & Söderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.
13. Hasegawa T, Itoh K, Koike T, Yukitani W, Hisamitsu H, Wakumoto S & Fujishima A (1999) Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system *Operative Dentistry* **24(6)** 323-330.
14. Takahashi A, Sato Y, Uno S, Pereira PN & Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin *Dental Materials* **18(3)** 263-268.
15. Li Y, Swartz ML, Phillips RW, Moore BK & Roberts TA (1985) Effect of filler content and size on properties of composites *Journal of Dental Research* **64(12)** 1396-1401.
16. Miyazaki M, Ando S, Hinoura K, Onose H & Moore BK (1995) Influence of filler addition to bonding agents on shear bond strength to bovine dentin *Dental Materials* **11(4)** 234-238.
17. Nunes MF, Swift EJ & Perdigão J (2001) Effects of adhesive composition on microtensile bond strength to human dentin *American Journal of Dentistry* **14(6)** 340-343.
18. Montes MA, de Goes MF, da Cunha MR & Soares AB (2001) A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats *Journal of Dentistry* **29(6)** 435-441.

19. Van Meerbeek B, Vargas MA, Inoue S, Yoshida Y, Peumans M & Lambrechts P (2001) Adhesives and cements to promote prevention dentistry *Operative Dentistry* **Supplement 6** 119-144.

20. Carvalho RM, Mendonça JS, Santiago SL, Silveira RR, Garcia FC, Tay FR & Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin *Journal of Dental Research* **82(8)** 597-601.

21. Yiu CK, Pashley EL, Hiraishi N, King NM, Goracci C, Ferrari M, Carvalho RM, Pashley DH & Tay FR (2005) Solvent and water retention in dental adhesive blends after evaporation *Biomaterials* **26(34)** 6863-6872.

22. Dickens SH & Cho BH (2005) Interpretation of bond failure through conversion and residual solvent measurements and Weibull analyses of flexural and microtensile bond strengths of bonding agents *Dental Materials* **21(4)** 354-364.

23. Cho BH & Dickens SH (2004) Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength *Dental Materials* **20(2)** 107-115.

24. Zheng L, Pereira PN, Nakajima M, Sano H & Tagami J (2001) Relationship between adhesive thickness and microtensile bond strength *Operative Dentistry* **26(1)** 97-104.