

Effect of Air-blowing Variables on Bond Strength of All-in-one Adhesives to Bovine Dentin

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This study evaluated the effect of air-blowing variables on the microtensile bond strength (μ TBS) of two all-in-one adhesives. A bonding agent was applied to the flat dentin surface of extracted bovine teeth, and the surface left undisturbed for 20 seconds. Gentle or intensive air-blowing was applied for five seconds, and the adhesive photopolymerized for 10 seconds. Resin composite paste was placed and cured after each bonding treatment. Specimens were subjected to μ TBS test with a crosshead speed of 1.0 mm/min. Data were statistically analyzed using ANOVA, followed by Bonferroni *post hoc* test. When Clearfil tri-S Bond was bonded to dentin, the μ TBS value of specimens applied with intensive air-blowing was significantly higher than that applied with gentle air-blowing ($p < 0.01$). On the other hand, with Fluoro Bond Shake One, the μ TBS value of specimens applied with intensive air-blowing was significantly lower than that applied with gentle air-blowing ($p < 0.01$).

Key words : Air-blowing, All-in-one adhesives, Microtensile bond strength

INTRODUCTION

For a span of more than 50 years, adhesive materials to tooth substrate have undergone rapid developments and advancements. Currently, dentin adhesion is routinely achieved by the use of dentin bonding systems. Most commercial adhesive systems adopt a self-etching primer, and they typically demonstrate high bond strengths to both enamel and dentin^{1–6}. However, these adhesive systems possess technique sensitivity due to their multiple application steps^{7–10}.

Recently, an all-in-one adhesive system has been developed to simplify the bonding procedure^{11–13}. This system is expected to minimize technique sensitivity as the bonding procedure comprises only one single step. However, while all-in-one adhesive systems simplify the bonding procedure, several studies have reported that the bond strength of all-in-one adhesive systems to tooth substrates was lower than that of two-step adhesive systems^{14–16}. The inadequate bond strength of all-in-one adhesive systems has been speculatively attributed to the multiple functions performed simultaneously by the bonding agent in the system: etching, priming, and bonding.

With a view to improving the bond strength of all-in-one adhesive systems, numerous *in vitro* studies have been carried out to investigate the effect of bonding procedure on bond strength to tooth substrates^{10,17–20}. According to Medina III *et al.*, the bond strength of an one-bottle adhesive to tooth substrates was affected by both moisture condition and

the number of coatings¹⁰. Pashley *et al.* also reported that bonding of an unfilled all-in-one adhesive might be improved by applying a second adhesive layer after light-curing the first layer¹⁷. Subsequently, Ito *et al.* reported that the bond strengths of two all-in-one adhesives increased with the number of coatings up to three layers, especially when each layer was light-cured¹⁸. On the other hand, Nakaoki *et al.* reported that the micro-shear bond strengths of four all-in-one adhesives showed no statistically significant differences between single-application and double-application methods¹⁹. Thus, the effect of the number of coatings on the bond strength of all-in-one adhesives is still under debate.

Among the factors that influence the bond strength of all-in-one adhesive systems, air-blowing variables are another important factor. This is because air-blowing pressure affects the thickness of the adhesive layer and the degree of polymerization. However, few studies have been carried out to investigate the relationship between air-blowing variables and bond strength of all-in-one adhesives^{7,10}. Therefore, this *in vitro* study sought to evaluate the effect of air-blowing variables on the microtensile bond strength (μ TBS) of two all-in-one adhesive systems to bovine dentin. The null hypothesis of this study was that the air-blowing variables would not have an effect on the bond strength of all-in-one adhesives to dentin.

MATERIALS AND METHODS

Materials used in this study are listed in Table 1. Sixteen extracted bovine teeth were used in this study. The teeth were cleaned and stored in refrigerator at -20°C until use. The labial surfaces of extracted bovine teeth were ground flat to dentin with wet 120-grit silicon carbide paper (Carbimet, Buehler Ltd., Lake Bluff, IL, USA) using a polishing machine (Lewel Specimen Polisher, Kasai Co. Ltd., Yokohama, Japan) under water irrigation. Ground surfaces were then finished with wet 600-grit silicon carbide paper. After which, specimens were randomly divided into four groups of four specimens each.

An adhesive tape (0.12 mm thickness) with a 6-mm-diameter opening was attached to the flat dentin surface to define the bonding area. Each adhesive system was applied to the dentin surface according to the following bonding procedure. A bonding agent was applied to the dentin surface, and the surface left undisturbed for 20 seconds. Then, either gentle or intensive air-blowing was applied for five seconds, and the adhesive layer was photopolymerized for 10 seconds.

A three-way syringe dental unit (J Morita Corp., Tokyo, Japan) was used for air-blowing. Air-blowing intensity was adjusted by using a stopper attached to the air syringe knob. The stopper was fabricated with a putty-type polyvinylsiloxane impression material (Exafine putty type, GC, Tokyo, Japan). Air-blowing intensity was measured on a scale with an electric balance when air stream was blown vertically over the electric balance at a distance 1 cm away. Gentle air-blowing was measured as 3 g (0.029 N), while intensive air-blowing was measured as 18 g (0.176 N), on the scale with the

electric balance.

After photopolymerizing the bonding resin, a laminated paper was peeled from the attached double-sided adhesive tape and a transparent acrylic tube (6 mm diameter \times 5 mm height) was placed onto the adhesive tape. Composite paste was placed into the acrylic tube (approximately 1 mm thickness) and then photopolymerized with a light-curing unit (Candilux, J Morita Corp., Tokyo, Japan) for 30 seconds. Composite paste (approximately 2 mm thickness) was further added to the existing composite surface and photopolymerized for 60 seconds, and then the translucent acrylic tube was removed.

After storing the bonded specimens in distilled water at 37°C for 24 hours, the roots were separated from their crowns using a diamond point (Bur No.105R, ISO size 22, Shofu Inc., Kyoto, Japan) and the pulps removed. Specimens were then serially sectioned — in a buccolingual manner — into 1-mm-thick slabs using a low-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. Two slabs were obtained from each specimen. Each slab was sectioned into beams of which the cross-sectional area was approximately 1 mm^2 using the low-speed diamond saw. Two beams were obtained from each slab. Accordingly then, 16 beam specimens were obtained for each experimental group.

Beam samples were attached to a testing device (Bencor Multi-T, Danville Engineering Co., San Ramon, CA, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA), and then mounted in a tabletop material tester (EZ Test, Shimadzu Corp., Kyoto, Japan) before being subjected to tensile testing at a crosshead speed of 1 mm/min. Data were subjected to two-way ANOVA with adhesive system and air-blowing vari-

Table 1 Materials used

Material	Manufacturer	Lot No.	Main components
Fluoro Bond Shake One	Shofu Inc. (Kyoto, Japan)		
Bond A		A-551F-3	Glass filler, acetone, water
Bond B		B-551F-3	HEMA, 4-AET, Bis-GMA, acetone, PI
Clearfil tri-S Bond	Kuraray Medical (Tokyo, Japan)	040219	MDP, HEMA, Bis-GMA, microfiller, ethanol, water, PI
Beautiful	Shofu Inc. (Kyoto, Japan)	010529	Bis-GMA, TEGDMA, S-PRG filler, MF glass filler, ultrafine filler, PI
Clearfil AP-X	Kuraray Medical (Tokyo, Japan)	00898A	Bis-GMA, TEGDMA, glass filler, microfiller, PI
S-PRG filler:	Surface reaction type Pre-reacted glass ionomer filler		
MF glass filler:	Multifunctional glass filler based on fluoroboric aluminosilicate glass		
Bis-GMA:	2, 2-Bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane		
TEGDMA:	Triethyleneglycol dimethacrylate		
HEMA:	2-hydroxyethyl methacrylate		
MDP:	10-methacryloxydecyl dihydrogen phosphate		
4-AET:	4-acryloxyethyl trimellitate		
PI:	Photoinitiator		

able as independent factors. Bonferroni *post hoc* test was used to compare the differences in μ TBS among the experimental groups ($p < 0.05$). Fractured surfaces of the specimens were also examined with an optical microscope at $\times 8$ magnification, and the failure modes were evaluated.

RESULTS

Table 2 shows the μ TBS test results. Two-way ANOVA revealed significant differences in the effects of adhesive system ($F=247.1$, $p < 0.0001$) and air-blowing ($F=298.3$, $p < 0.0001$), and significant differences were also found for the interaction between them ($F=685.4$, $p < 0.0001$).

Results of the *post hoc* test for the relationship between the air-blowing variables for each adhesive system were as follows. When Clearfil tri-S Bond was bonded to dentin, the μ TBS value of specimens applied with intensive air-blowing was significantly higher than that applied with gentle air-blowing ($p < 0.01$). On the other hand, with Shake One, the μ TBS value of specimens applied with intensive air-blowing was significantly lower than that applied with gentle air-blowing ($p < 0.01$).

Table 3 lists the failure mode results as observed using a microscope. All the specimens applied with Shake One and subjected to intensive air-blowing and those applied with Clearfil tri-S Bond and subjected to gentle air-blowing showed adhesive failure. On the other hand, specimens applied with Clearfil tri-S Bond and subjected to intensive air-blowing showed mixed failure, except for three specimens with cohesive failure. Specimens applied with Shake One and subjected to gentle air-blowing showed adhesive failure, except for two specimens with mixed failure.

DISCUSSION

Results of this study revealed that air-blowing variables indeed affected the bond strength of all-in-one

adhesives to bovine dentin. Gentle air-blowing decreased the bond strength of Clearfil tri-S Bond to dentin, while it increased that of Fluoro Bond Shake One. Hence, these results showed clearly that air-blowing variables – for the purpose of dispersing the bonding agent applied on dentin surface – produced contrary effects with each adhesive system. Based on the results obtained, the null hypothesis that air-blowing variables would not have an effect on the bond strength of all-in-one adhesives was rejected.

All-in-one adhesives contain hydrophilic and hydrophobic monomers, water, and volatile solvents. Resin monomers are dissolved in volatile solvents such as acetone, ethanol, or water. Water is included as an ionization medium to enable self-etching activity. Although solvent and water are important ingredients, their concentration levels matter too as they have an impact on adhesive performance. A high concentration of water may have an adverse effect on adhesive polymerization when water remains in the adhesive during polymerization. Likewise, a high concentration of solvent may cause inadequate resin polymerization when solvent evaporation is incomplete. Indeed, critical situations like these hinge on two factors: type of solvent contained in the adhesive and the air-blowing intensity.

In this study, ethanol contained in Clearfil tri-S Bond as a solvent is less volatile than the acetone contained in Fluoro Bond Shake One. Therefore, the solvent in Clearfil tri-S Bond might not be completely evaporated by gentle air-blowing and water might have remained in the adhesive due to incomplete evaporation of the solvent. It has been reported that water voids were formed along the bonding interface when the dentin surface became over-wet with water present in both the smear layer and resin of all-in-one adhesives^{21,22}. Consequently, intensive air-blowing might be necessary for adhesives that contain ethanol to eliminate both solvent and water. In the case of Fluoro Bond Shake One, the solvent could have been completely evaporated by gentle air-

Table 2 Results of μ TBS test

Air-blowing Intensity	All-in-one Adhesive System	
	Clearfil tri-S Bond	Fluoro Bond Shake One
Gentle	4.1 (2.4) ^a	13.1 (3.1)
Intensive	42.6 (3.8)	5.4 (2.9) ^a

Unit: MPa, (): SD, n=12

Values with the same superscript indicate no significant differences ($p > 0.05$)

Table 3 Failure modes of specimens after μ TBS test

Air-blowing Intensity	All-in-one Adhesive System	
	Clearfil tri-S Bond	Fluoro Bond Shake One
Gentle	0/0/0/12	0/0/2/10
Intensive	2/1/9/0	0/0/0/12

Cohesive failure in dentin/Cohesive failure in resin/Mixed failure/Adhesive failure

blowing. However, Cho and Dickens reported that an adhesive containing abundant acetone showed crack formation at the bonding interface²³. In other words, intensive air-blowing might over-dry and dehydrate the dentin surface when an acetone-containing adhesive is applied, because acetone is highly volatile. Fluoro Bond Shake One contained acetone as a solvent. Therefore, it was speculated that intensive air-blowing caused interfacial cracks to be formed and decreased the bond strength of Fluoro Bond Shake One.

Recently, self-etch adhesives are classified into three categories according to their acidity: mild ($\text{pH} \geq 2$), intermediate ($\text{pH} \approx 1.5$), and strong ($\text{pH} \leq 1$) self-etch adhesives^{22,24,25}. Both adhesives used in the present study belonged to the category of mild self-etch adhesives because their pH was 2.7. Mild self-etch adhesives dissolve the dentin surface only partially, such that a substantial number of hydroxyapatite crystals around exposed collagen fibrils may be available for additional chemical interaction with the functional monomers²². As a result, a very shallow hybrid layer of submicron dimensions is formed. However, current all-in-one self-etch adhesives have been proven to be acidic enough to penetrate the smear layer within the designated application duration without any intensive agitation. In an investigation on three adhesives with widely differing acidities, Tani and Finger reported that all the three tested adhesives were equally effective in bonding to dentin with smear layer thickness ranging from 0.9 μm to 2.6 μm ²⁶. In other words, despite the shallow hybrid layer, mild self-etch adhesives showed satisfactory bond strength to tooth substrate.

Several studies have reported that one-step self-etch adhesives consistently exhibited lower bond strength when compared to multi-step self-etch adhesives¹⁴⁻¹⁶. Two-step adhesive systems with self-etching primer show firm bond strength to dentin based on the following mechanism of action. Self-etching primer applied on prepared dentin surface dissolves the smear layer and demineralizes superficial dentin beneath the smear layer. Smear layer dissolved by the self-etching primer solution is then removed by air-blowing. Monomer in bonding agent sufficiently penetrates the dentin surface conditioned with self-etching primer, followed by photopolymerization. These independent bonding steps supposedly contribute to establishing a firm bond between two-step adhesives and dentin.

On the other hand, acidic monomers in all-in-one adhesive systems exhibit lower degree of dissociation in the bonding agent due to less water content compared to primers of two-step adhesive systems. As a result, application of one-step adhesives to dentin may show insufficient dissolution of the smear layer and superficial dentin, because the bonding agent of

one-step adhesives has lower etching ability than the self-etching primer of two-step adhesives. Thus, it is speculated that incomplete dissolution of the smear layer and insufficient penetration of resin monomers result in lower bond strength of one-step adhesives as compared to two-step adhesives. Furthermore, photopolymerized all-in-one adhesives have been demonstrated to act as permeable membranes, which permit water movement across the adhesive layer due to their high hydrophilicity²⁷. Their function as a water duct may contribute to gradual degradation of bond strength between dentin and all-in-one adhesives.

Film thickness of adhesive resins applied to dentin surface is affected by subsequent air-blowing. It is possible that gentle air-blowing produces thick adhesive resin layers including the dissolved smear layer. Due to presence of the smear layer, physical and mechanical properties of the photopolymerized adhesive layer may be adversely affected. Zheng *et al.* investigated the relationship between adhesive thickness and microtensile bond strength to dentin using commercially available two-step bonding systems²⁸. The results showed that the increase in bond strength of Clearfil Liner Bond 2V was directly proportional to the thickness of the adhesive layer; however, the bond strength of Single Bond decreased significantly with the increase of adhesive resin thickness. In light of this finding by Zheng *et al.*²⁸, it is therefore important to control the thickness of bonding agents — to which the bonding procedure has a contributory effect.

In this study, the transparent acrylic tube was placed in position after photopolymerizing the adhesive layer. This procedure prevented a pooling of bonding agent at the peripheral of dentin-tube interface. In clinical situations, bonding agents tend to pool at the cavity angle. If clinical conditions were to be simulated, it would be difficult to evaluate the effect of varied bonding thicknesses created by air-blowing. As such, in the present study, air-dispersed bonding agent was polymerized on a laminated paper, such that the original bonding area was clearly defined after removing the flash with the laminated paper. The results of our study showed that the bond strength of a thin adhesive layer with intensive air-blowing was significantly higher than that of thick adhesive layer for Clearfil tri-S Bond. However, the bond strength of a thick adhesive layer of Fluoro Bond Shake One with gentle air-blowing was significantly higher than that of thin adhesive layer with intensive air-blowing. These results indicated that the bond strength of all-in-one adhesive systems to dentin was influenced by adhesive thickness, which was consequently influenced by air-blowing intensity.

CONCLUSION

Based on the results obtained in the present study, it was concluded that intensive air-blowing after the application of Clearfil tri-S Bond produced significantly higher μ TBS to dentin than gentle air-blowing; however, use of intensive air-blowing showed significantly lower μ TBS with Fluoro Bond Shake One. It was thus suggested that for all-in-one adhesive systems, the type of solvent contained in the system would determine the air-blowing intensity suitability.

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