Research article
ISSN 2234-7658 (print) / ISSN 2234-7666 (online)
http://dx.doi.org/10.5395/rde.2012.37.3.155
RDE
Restorative Dentistry & Endodontics

Effect of moisture and drying time on the bond strength of the one-step self-etching adhesive system

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Objectives: To investigate the effect of dentin moisture degree and air-drying time on dentin-bond strength of two different one-step self-etching adhesive systems. ***Materials and Methods:*** Twenty-four human third molars were used for microtensile bond strength testing of G-Bond and Clearfil S^3 Bond. The dentin surface was either blot-dried or air-dried before applying these adhesive agents. After application of the adhesive agent, three different air drying times were evaluated: 1, 5, and 10 sec. Composite resin was build up to 4 mm thickness and light cured for 40 sec with 2 separate layers. Then the tooth was sectioned and trimmed to measure the microtensile bond strength using a universal testing machine. The measured bond strengths were analyzed with three-way ANOVA and regression analysis was done (p = 0.05). ***Results:*** All three factors, materials, dentin wetness and air drying time, showed significant effect on the microtensile bond strength. Clearfil S^3 Bond, dry dentin surface and 10 sec air drying time showed higher bond strength. ***Conclusions:*** Within the limitation of this experiment, air drying time after the application of the one-step self-etching adhesive agent was the most significant factor affecting the bond strength, followed by the material difference and dentin moisture before applying the adhesive agent. (*Restor Dent Endod* 2012;37(3):155-159)

Key words: Air-drying time; HEMA; Moisture control; One-step self-etching

Received April 24, 2012;
Last Revised July 5, 2012;
Accepted July 9, 2012.

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Introduction

The conventional adhesive systems have been multi-bottle types and they had a problem of technique sensitivity.[1] Recently, the development of dentin adhesive system is focused on the simplification of the procedure and this can reduce the operator-dependent variability on the bond strength.[2] The most simplified adhesive system is the all-in-one type and this includes all components in one bottle. The first one-step self-etching system was composed of two solutions: one part containing organic acid, resin, photoinitiators and stabilizer, and the other part containing water, 2-hydroxylethyl methacrylate (HEMA), and stabilizers.[3] Although the manufacturer advertised this product as an all-in-one system, this is a two bottle type one-step self-etching system since the two components should be mixed before use. Later, a true all-in-one system was introduced and every ingredient of the adhesive system was included in one-bottle. These one-step self-etching systems are becoming increasingly more popular and their market share is still growing.[4]

※This research was supported by the grant of 2007 Yonsei university school of dentistry research funds (7-2007-0070).

These early one-step self-etching products were easy to use, but they showed lower bond strength in *in vitro* experiments and it was reported that the adhesive layer acts as a semi-permeable membrane for water.[5] This property was affected by the hydrophilicity of the adhesive agent and HEMA, the main hydrophilic component in the adhesive system. Consequently, a HEMA-free one-step self-etching adhesive system was introduced (G-Bond, GC Corp., Tokyo, Japan). However the lack of HEMA in this product created a drawback known as phase separation. HEMA is the key component for crosslinking the hydrophilic and hydrophobic components of the adhesive system, and this is even more important in one-step self-etching adhesive system, because all components are in one bottle. The manufacturers of Clearfil S^3 Bond (Kuraray Medical Inc., Tokyo, Japan) insisted that their product did not show phase separation.

The main advantage of the one-step self-etching systems is that it is less technique sensitive than multi-step systems. However, according to recent studies, the bond strength of these products were affected by several factors: enamel surface treatment, smear layer thickness, grit size of the bur, moisture condition of the adhesive surface, drying time after application of the adhesives, and number of multiple coating.[6-9] The moisture control of the adhesive surface can be considered in two parts: the surface moisture before the application of the adhesive agent and the removal of the solvent after application. Acetone and ethanol are the most popular solvents used for the adhesive agents. Usually acetone is more volatile than ethanol and it is easier to remove after application, but there is less chance for ideal handling and this resulted in more technique sensitivity.[10] For the etch-and-rinse system, wet adhesive is essential for preventing the collapse of the exposed collagen fibers. However, the acid in the self-etching system is milder than phosphoric acid, and if the adhesive agent is diluted by the excess water on the adhesive surface, it may reduce the bond strength of the self-etching system.[9,10] On the contrary, Werner and Tani reported that the dentin wetness and relative humidity did not affect the bond strength of the one-step self-etching adhesive systems.[11]

The objectives of this experiment were to clarify the effect of the moisture control before and after adhesive agent application with two different one-step self-etching adhesive systems. The null hypothesis was 1) the bond strength of these two systems was not affected by either dentin moisture control or solvent drying time and 2) the bond strength of these two systems was not different.

Materials and Methods

Twenty-four extracted noncarious human third molars were used and 2 teeth were used for each group in this study. The roots were embedded in a mold with self-cured acrylic resin and they were sectioned and ground with #600 grit sand paper to expose a flat dentin surface perpendicular to the longitudinal axis of the tooth.

Experimental design

Two adhesive systems were used in this experiment (Table 1). Before the application of the adhesive agents, the tooth surface of one group was dried with a strong air blow at 2 cm away from the tooth surface and the other group was blot-dried with a moist cotton ball to provide a moist condition. Then the adhesive agent was applied following the instruction of the manufacturer. After the instructed waiting time, the teeth surfaces were dried for 1, 5, and 10 seconds to remove the water and solvent and light

Table 1. The adhesive systems used for this experiment and their instructions for use

Product	Composition	Manufacturer	Instruction for use
G-Bond	4-MET, UDMA, Phosphate monomer, DMA component, fumed silica filler, acetone, water, photo-initiator	GC Corp.	Before dispensing, shake the bottle thoroughly. Immediately apply to the prepared adhesive surfaces using the microbrush. Leave undisturbed for 10 sec. After application, dry thoroughly using oil free air under maximum air pressure . Light cure for 10 sec.
Clearfil S^3 Bond	10-MDP, HEMA, bis-GMA, water, ethanol, silinated colloidal silica, camphoroquinone	Kuraray Medial Inc.	Thoroughly wet brush tip with bond. Apply bond to the tooth surface and leave in place for 20 sec. Dry the entire surface sufficiently by blowing gentle air. Light cure for 10 sec.

4-MET, 4-methacryloyloxyethyl trimellitate; UDMA, urethane dimethacrylate; DMA, dimethacrylate; 10-MDP, 10-methacryloxydecyl di-hydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; bis-GMA, 2,2 bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl] propane.

http://dx.doi.org/10.5395/rde.2012.37.3.155

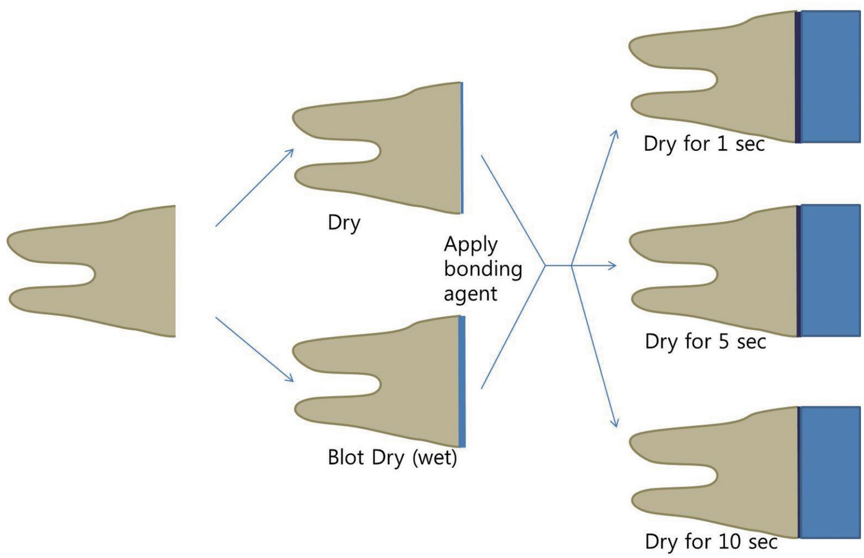

Figure 1. The schematic view of the experimental design. Wetness of the tooth surface was divided into dry and blot dry groups and drying times after adhesive application were into 1, 5 and 10 seconds groups for each adhesive agent.

cured for 10 seconds (Figure 1). Two layers of 2 mm-thick composites (Filtek Z350, 3M/ESPE, St. Paul, MN, USA) were built up and cured each layer for 40 seconds with LED light curing unit (Elipar S10, 3M/ESPE) with an intensity of 1,000 mW/cm^2.

After composite build-up, the samples were sectioned into 1 mm thick slices with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) and dumbbell shape specimens were prepared with high speed diamond bur. They were immersed in the 37¡É water for 24 hours and fixed on the testing jig with cyanoacrylate (Zapit, Dental Ventures of America Inc., Corona, CA, USA). Microtensile bond strengths were measured with an universal testing machine (EZ-test-500N, Shimadzu Co., Kyoto, Japan) at 1.0 mm/min crosshead speed (n = 10).

Statistical analysis

To evaluate the relation between bond strength and each factor (material, dentin wetness, air drying time) three-way ANOVA at a 0.05 significance level was used and regression analysis was done to compare the effectiveness of these factors with SPSS Ver. 19 (SPSS Inc., Chicago, IL, USA).

Results

Table 2 shows the microtensile bond strength results. Three-way ANOVA revealed significant differences in the effects of materials ($F = 39.7$, $p < 0.05$), dentin wetness before the adhesive agent application ($F = 8.8$, $p < 0.05$) and air drying time after the adhesive agent application ($F = 237.8$, $p < 0.05$). No interactions were found among these three factors.

According to the results of this experiment, Clearfil S^3 Bond showed higher bond strength than G-Bond and for the dentin wetness ($p < 0.05$), dry group showed higher bond strength than blot dry group ($p < 0.05$). For the air drying time, 10 seconds showed highest bond strength and followed by 5 and 1 seconds ($p < 0.05$). The regression analysis showed that the air drying time was the most critical factor on the bond strength, followed by the materials and dentin wetness (Table 3).

Discussion

For the lastest decades, one-step self-etching adhesive systems have been developed and the advantage of this system is the relatively simple procedure, which minimizes the steps of the adhesive and reduces the technique sensitivity.[12] In *in vitro* experiments, the moisture control on the tooth surface is relatively simple, but in clinical situations, it is not easy because cavity geometry is more complex and it is difficult to obtain water tight seal during the restorative procedure, especially when the cavity margin is located subgingivally. Thus revealing the effect of the moisture on the bond strength of the adhesive agent is important not only for the investigators but also for the clinicians. Following the result of this experiment, the adhesive agent, the moisture conditions before the application of the adhesive agents and the air drying time affected on the bond strength and the null hypothesis was rejected.

In this experiment, air drying time was the most im-

Table 2. The microtensile bond strength of this experiment

Materials	Dentin wetness	Drying time (seconds)	Mean ± SD (MPa)
G-Bond	Blot dry	1	10.4 ± 2.9
		5	23.7 ± 5.2
		10	31.1 ± 4.9
	Dry	1	12.1 ± 2.9
		5	26.8 ± 5.6
		10	37.0 ± 4.9
Clearfil S3 Bond	Blot dry	1	17.7 ± 3.5
		5	28.3 ± 4.7
		10	37.9 ± 4.4
	Dry	1	18.8 ± 2.9
		5	30.5 ± 4.7
		10	38.2 ± 5.0

Table 3. The regression analysis results of this experiment.

Model	Estimated coefficient	Standard error	Standardized Coefficient	*t* value	*p* value
(constant)	3.594	1.864		1.947	0.054
Materials	5.055	0.806	0.249	6.273	0.000
Dentin wetness	2.381	0.806	0.117	2.954	0.004
Drying time(1*5)	12.564	0.987	0.593	12.729	0.000
Drying time(1*10)	21.325	0.987	0.990	21.606	0.000

portant factor on the bond strength. Most self-etching system has water in the solvent to represent the acidity. Therefore if it is not removed completely, it is entrapped within the adhesive layer, and this results in catastrophic reduction of the bond strength, which is consistent with other experiments.[13,14] In G-Bond, one second air drying was not enough even though this product used acetone as a solvent. In previous studies, when this adhesive agent was not dried completely with strong air, water droplet remained in the adhesive layer, and this reduced the bond strength and increased the nanoleakage.[15,16] The bond strength increased when we dried the adhesive surface for 10 seconds, and this means that even five seconds was not enough time to remove the water and organic solvent.

Next significant factor affecting the bond strength was the material difference. In this experiment, Clearfil S3 Bond showed higher bond strength than G-Bond. Comparing the component of these two adhesive systems, Clearfil S3 Bond contains HEMA to prevent phase separation, 10-MDP as a functional monomer, and ethanol as the solvent. On the contrary, G-Bond is HEMA-free, and it contains 4-MET as a functional monomer and acetone as the solvent. All of these differences can influence the bond strength. Landuyt *et al*. reported that when 10% HEMA was added in adhesives, the bond strength of the one-step self-etching adhesive was improved.[17] Acetone, used in G-Bond, is known as a strong organic solvent for the functional monomer and hydrophobic resin monomer, and also as a good water chaser. However, this type of solvent is known to be more technique sensitive than ethanol.[5,18] In a view point of functional monomer, Tsuchimoto *et al*. reported that 10-MDP showed higher bond strength than 4-MET.[19] The combination of these factors probably resulted in the higher performance of Clearfil S3 Bond. The moisture condition of the tooth surface also affected the bond strength of one-step self-etching systems. This can be explained by the fact that the moisture on the dentin surface may dilute the adhesives, thus negating the etching effect of the adhesives. Such imperfect etching might decrease the potential for hybridization and finally lead to failure of the resin

http://dx.doi.org/10.5395/rde.2012.37.3.155

composite. Another explanation for the decrease in the dentin adhesive strength might be the imperfect polymerization of adhesives due to the excessive water present.[9]

When we compare the bond strength of the two adhesive systems, if the moisture control was ideal, that is, dry dentin surface before applying the adhesive agent and 10 seconds air dry performed, G-Bond and Clearfil S^3 Bond showed similar results, which were 37.0¡¾4.9 and 38.2¡¾5.0, respectively. However, if G-Bond was applied on a wet dentin surface, the decrease in the bond strength was greater than Clearfil S^3 Bond. This means that the G-Bond is more technique sensitive, and thus the operator should be more cautious when using this in complex clinical situations.

Conclusions

Within the limitation of this experiment, air drying time after the application of the one-step self-etching adhesive agent was the most significant factor affecting the bond strength, followed by the adhesive composition and dentin moisture before applying the bond.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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