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Effect of Air-drying Time of Single-application Self-etch Adhesives on Dentin Bond Strength

Y Chiba • K Yamaguchi • M Miyazaki
K Tsubota • T Takamizawa • BK Moore

Clinical Relevance

The air-drying time of single-step self-etch adhesives was a significant factor influencing dentin bond strengths.

SUMMARY

This study examined the effect of air-drying time of adhesives on the dentin bond strength of several single-application self-etch adhesive systems. The adhesive/resin composite combinations used were: Adper Prompt L-Pop/Filtek Z250 (AP), Clearfil Tri-S Bond/Clearfil AP-X (CT), Fluoro Bond Shake One/Beautifil (FB), G-Bond/Gradia Direct (GB) and One-Up Bond F Plus/Palfique Estelite (OF). Bovine mandibular incisors were mounted in self-curing resin and wet ground with #600 SiC to expose labial dentin. Adhesives were applied according to each manufacturer's instructions followed by air-drying time for 0 (without air-drying), 5 and 10 seconds. After light irradiation of the adhesives, the resin composites were condensed into a mold (ø4x2 mm) and polymerized. Ten samples per test group were stored in 37°C distilled water for 24 hours; they were then shear tested at a crosshead speed of 1.0 mm/minute. One-way ANOVA followed by Tukey's HSD tests (α=0.05) were done. FE-SEM observations of the resin/dentin interface were also conducted. Dentin bond strength varied with the different air drying times and ranged from 5.8 ± 2.4 to 13.9 ± 2.8 MPa for AP, 4.9 ± 1.5 to 17.1 ± 2.3 MPa for CT, 7.9 ± 2.8 to 13.8 ± 2.4 MPa for FB, 3.7 ± 1.4 to 13.4 ± 1.2 MPa for GB and 4.6 ± 2.1 to 13.7 ± 2.6 MPa for OF. With longer air drying of adhesives, no significant changes in bond strengths were found for the systems used except for OF. Significantly lower bond strengths were obtained for the 10-second air-drying group for OF. From FE-SEM observations, gaps between the cured adhesive and resin composites were observed for the specimens without the air drying of adhesives except for OF. The data suggests that, with four of the single-application self-etch

Yasushi Chiba, DDS, graduate student, Nihon University Graduate School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

Kanako Yamaguchi, DDS, graduate student, Nihon University Graduate School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

*Masashi Miyazaki, DDS, PhD, assistant professor, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

Keishi Tsubota, DDS, PhD, instructor, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

Toshiki Takamizawa, DDS, PhD, instructor, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

B Keith Moore, PhD, professor, director of Graduate Dental Materials, Indiana University School of Dentistry, Department of Restorative Dentistry, Division of Dental Materials, Indianapolis, IN, USA

*Reprint request: 1-8-13, Kanda Surugadai, Chiyoda-Ku, Tokyo 101-8310, Japan; e-mail: miyazaki-m@dent.nihon-u.ac.jp

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adhesive systems, air drying is essential to obtain adequate dentin bond strengths, but increased drying time does not significantly influence bond strength. For the other system studied, the bond strength of the non-air dried group was not significantly different from the five second drying time, but prolonged drying was very detrimental to bond strength. For all five of the systems studied, a five-second air-drying time appeared to be appropriate.

INTRODUCTION

Conventional dentin bonding systems require the sequential application of conditioner, dentin primer and bonding agent in several clinical steps. To reduce technique-sensitive and materials-related factors that affect bond strength, a self-etch approach involving either one- or two-step application has been developed (Kitasako & others, 2000; Van Meerbeek & others, 2003). Application methods of newly available adhesive systems have been simplified, and manufacturers' instructions have been clarified to help achieve optimum clinical results. Recently introduced single-application self-etch systems combine all three bonding steps into a single-application (Miyazaki & others, 2001; Miyazaki, Iwasaki & Onose, 2002a). Theoretically, the acidic adhesive dissolves the smear layer, incorporating it into the mixture, and the acidic adhesive demineralizes and penetrates into the superficial dentin, then hardens after light irradiation. In these adhesives, both the primer and bonding resin components are incorporated into the two-component assemblies that are mixed before application or in a single vial, along with water, ethanol or acetone (Ikemura & others, 2003a). In some products, functionalized microfillers are added to improve bonding properties and to form a thin film of adhesive (Ikemura & others, 2003b).

Self-etch adhesives are applied to the tooth surface prior to resin composite placement to ensure maximum adhesion by improving monomer penetration into the hydrophilic dentin substrate and to improve wettability of the tooth surface by the resin components (Shimada & others, 2003; Wang & Spencer, 2004). While the system becomes simpler, careful product management is still required in order to attain optimum bonding procedures (Miyazaki, Onose & Moore, 2000; Miyazaki & others, 2002). Although air drying of applied adhesive is recommended to evaporate solvents in the adhesives, variations in air-drying time probably occur in clinical application. Since self-etching adhesives contain acidic functional monomers and water, which might interfere with subsequent resin polymerization, air-drying time might be an influential factor in determining dentin bond strengths (Miyazaki & others, 1999).

The proper application of adhesives plays an important role in getting good bond strength, but the effect of the clinical application technique of a single-application self-etch adhesive has not been well explored. This study evaluated the influence of single-application, self-etch adhesive drying times on bond strength to bovine dentin by measuring shear bond strength. Field emission scanning electron microscopy (FE-SEM) observation on the resin/dentin interface was also conducted.

METHODS AND MATERIALS

Materials Tested

Single-application, self-etch adhesive systems with the combination of resin composites used were Adper Prompt L-Pop/Filtek Z250 (AP, 3M ESPE, St Paul, MN, USA), Clearfil Tri-S Bond/Clearfil AP-X (CT, Kuraray Medical, Tokyo, Japan), Fluoro Bond Shake One/Beautifil (FB, Shofu Inc, Kyoto, Japan), G-Bond/Gradia Direct (GB, GC Corp, Tokyo, Japan) and One-Up Bond F Plus/Palfique Estelite (OF, Tokuyama Dental, Tokyo, Japan) as listed in Table 1. The application protocols suggested by each manufacturer are listed in Table 2. All adhesive systems were used in combination with the manufacturers' restorative resins.

A visible-light activating unit Optilux 501 (Sybron Dental Specialties Kerr, Danbury, CT, USA) was used, and the power density (800 mW/cm^2) of the curing light was checked with a dental radiometer (Model 100, Demetron/Kerr) before curing the specimens.

Bond Strength Test

Mandibular incisors extracted from two-to-three year old cattle and stored frozen (-20°C) for up to two weeks were used as a substitute for human teeth (Nakamichi, Iwaku & Fusayama, 1983; Fowler & others, 1992; Schilke & others, 1999). After removing the roots with a low-speed saw (Isomet, Buheler Ltd, Lake Bluff, IL, USA), the pulps were removed and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of the bovine incisors were ground on wet 240-grit SiC paper to a flat dentin surface. Each tooth was then mounted in cold-curing acrylic resin (Resin Tray II, Shofu Inc) to expose the flattened area and was placed in tap water to reduce temperature rise from the exothermic polymerization reaction. Final finish was accomplished by grinding on wet 600-grit SiC paper. After ultrasonic cleaning with distilled water for one minute to remove the excess debris, these surfaces were washed and dried with oil-free compressed air.

A piece of double-sided adhesive tape with a 4-mm diameter hole was firmly attached to define the adhesive area of the dentin for bonding. The adhesive was applied on the dentin surface according to the manufacturers' instructions. Primed dentin surfaces were dried with oil-free compressed air with 0.2 MPa air pressure for 0, 5 and 10 seconds from 5 cm above the dentin surface using a three-way syringe; they were then irradiated with the curing unit. A Teflon (Sanplatec Corp, Osaka, Japan) mold, 2.0-mm high and 4.0-mm in diameter, was used to form and hold the restorative resin onto the dentin surface. The resin composite was condensed into the mold and cured for 40 seconds. The finished specimens were transferred to distilled water and stored at 37°C for 24 hours.

Ten specimens per group were tested in a shear mode using a shear knife-edge testing apparatus in an Instron testing machine (Type 4204, Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area. After testing, the specimens were examined in an optical microscope SZH-131 (Olympus Ltd, Tokyo, Japan) at 10x magnification to define the location of the bond failure (Fowler & others, 1992). The type of failure was determined based on the percentage of substrate-free material, adhesive failure, cohesive failure in adhesive and cohesive failure in dentin.

Statistical Analysis

The results were analyzed by calculating the mean shear bond strength (MPa) and standard deviation for each group. The statistical analysis was done to show how the bond strengths were influenced by air-drying times. The data for each group were tested for homogeneity of variance using Bartelett's test and subjected to ANOVA followed by the Tukey's HSD test at p=0.05 within each adhesive system. Statistical analysis was carried out with the Sigma Stat software system (Ver 2.01, SPSS Inc, Chicago, IL, USA).

Scanning Electron Microscopy

For ultrastructure observation of the resin/dentin interface by FE-SEM, bonded specimens stored in 37°C distilled water for 24 hours were embedded in self-curing

Table 1: *Materials Tested*

Code	Adhesive	Main Components (Manufacturer)	Lot #	Restorative	Lot #
AP	Adper Prompt L-Pop (3M ESPE)	Methacrylated phospholic eters, Bis-GMA,CQ, initiator, stabilizer, 2-HEMA, polyalkenoic acid, water	127613	Filtek Supreme (A2B)	3AU
CT	Clearfil Tri-S Bond (Kuraray Medical)	MDP, bis-GMA, HEMA, initiator ethanol, stabilizer, filler	040219	Clearfil AP-X (A2)	00987A
FB	Fluoro Bond Shake-One (Shofu Inc)	PRG, fluoroaluminosilicate glass, 4-AET, 4-AETA, bis-GMA initiator, water, solvent,	A: MS-13 B: MS-13	Beautifil (A2)	020135
GB	G-Bond (GC Corp)	4-MET, UDMA, acetone, water silanated colloidal silica, initiator	031015	Gradia Direct (A2)	0407051
OF	One-Up Bond F Plus (Tokuyama Dental)	MAC-10, HEMA, MMA, multifunction methacrylic monomer fliuoroaluminosilicate glass, water photo initiator (aryl borate catalyst)	A: 551F-2 B: 551F-2	Palfique Estelite (A2)	J279

Table 2: *Application Protocols of Single-application Adhesive Systems*

Adhesive System	Application Protocol
Adper Prompt L-Pop (Blister-Packed)	Activate blister pack by emptying the liquid out of the red blister into the yellow blister. The activated solution was applied to dentin for 15 seconds with moderate finger pressure. Use a gentle stream of air to dry and apply a second coat of adhesive. Gently air dry and light irradiation for 10 seconds.
Clearfil Tri-S Bond (Single Bottle)	Dispense one drop of liquid into well. Apply to dentin for 20 seconds. Relatively strong stream of air to dry and light irradiation for 10 seconds.
Fluoro Bond Shake One (Two Bottles)	Mix equal amounts of bond agent A and B. Apply to dentin for 20 seconds. Briefly air dry and light irradiation for 10 seconds.
G-Bond (Single Bottle)	Apply sufficient amount of adhesive for 10 seconds. Strong air dry and light irradiation for 10 seconds.
One-Up Bond F Plus (Two Bottles)	Mix equal amounts of the bond agents A and B until a pink homogenous liquid mixture is obtained. Apply to dentin for 10 seconds with agitation and light irradiation for 10 seconds.

Table 3: *Shear Bond Strength (Mean (SD) in MPa) to Bovine Dentin*

	Air-drying Time Time of Adhesive		
Code	0 seconds	5 seconds	10 seconds
AP	5.8 (2.4)[a] [10/0/0]	13.1 (2.7)[b] [10/0/0]	13.9 (2.8)[b] [9/1/0]
CT	4.9 (1.5)[c] [10/0/0]	15.2 (2.6)[d] [3/3/4]	7.1 (2.3)[d] [6/1/3]
FB	7.9 (2.8)[e] [10/0/0]	13.9 (2.4)[f] [8/2/0]	13.8 (2.4)[f] [9/1/0]
GB	3.7 (1.4)[g] [10/0/0]	10.4 (2.0)[h] [9/1/0]	13.4 (1.2)[h] [3/3/4]
OF	13.7 (2.6)[i] [5/4/1]	13.6 (3.6)[i] [7/2/1]	4.6 (2.1)[j] [7/3/0]

SD: standard deviation, n=10
Values with the same letter in each adhesive system are not significantly different at p>0.05.
Failure mode: [adhesive failure/cohesive failure in adhesive/cohesive failure in dentin]

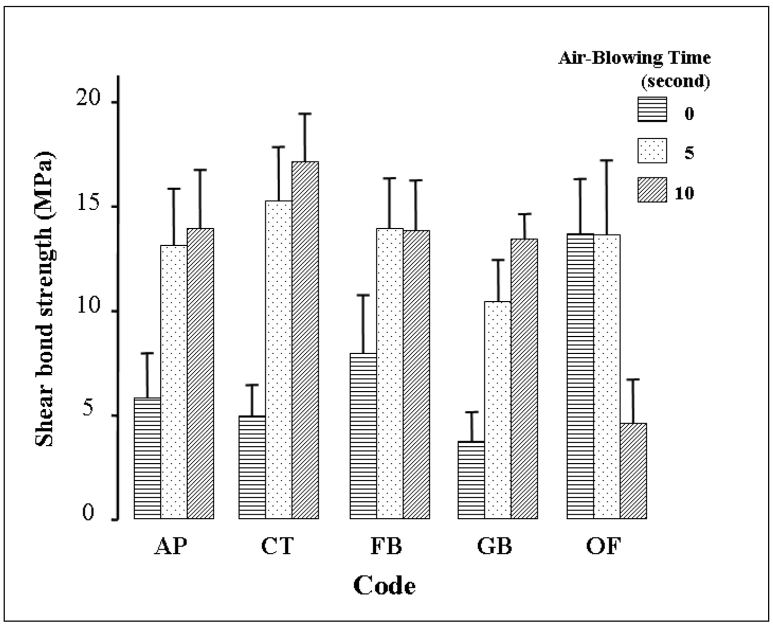

Figure 1. *Effect of air-drying time time of adhesives on shear bond strength to bovine dentin.*

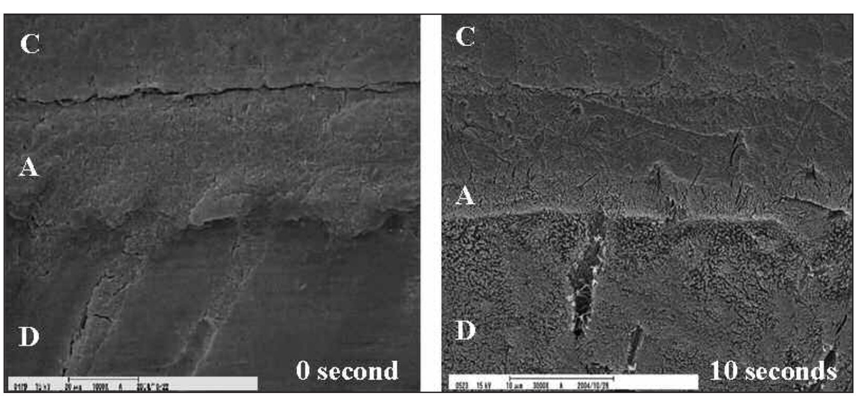

Figure 2a. *SEM photomicrographs of resin/dentin interface of a) AP, b) CT, c) FB, d) GB, and e) OF (left; 0 second air-drying time, right; 10 seconds air-drying time; original magnification, 3,000x).*

epoxy resin (Epon 812, Nisshin EM, Tokyo, Japan) and stored at 37°C for 12 hours. The embedded specimens were then sectioned to the diameter of the resin composite post, and the surfaces of the cut halves were polished with an Ecomet 4/Automet 2 (Buehler Ltd) using SiC papers of 600, 1200 and 4000-grit size, successively. The surface was finally polished on a special soft cloth using diamond paste (Buehler Ltd) with a grit size of 0.1 µm. All SEM specimens were dehydrated in ascending concentrations of *tert*-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes and 100% for 2 hours), then transferred to a critical-point dryer for 30 minutes. These surfaces were then subjected to Argon-ion beam etching (Type EIS-200ER, Elionix Ltd, Tokyo, Japan) for 30 seconds, with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2) directed perpendicular to the polished surface. The surfaces were coated in a vacuum evaporator, Quick Coater Type SC-701 (Sanyu Denshi Inc, Tokyo, Japan), with a thin film of Au. The specimens were observed in FE-SEM (ERA 8800, Elionix Ltd).

RESULTS

The results of the shear bond strength tests with different air-drying adhesive times applied onto the dentin surface are shown in Table 3 and Figure 1. Dentin bond strengths varied with the different air-drying times and ranged from 5.8 ± 2.4 to 13.9 ± 2.8 MPa for AP, 4.9 ± 1.5 to 17.1 ± 2.3 MPa for CT, 7.9 ± 2.8 to 13.8 ± 2.4 MPa for FB, 3.7 ± 1.4 to 13.4 ± 1.2 MPa for GB and 4.6 ± 2.1 to 13.7 ± 2.6 MPa for OF. The bond strengths of each bonding system, except for OF, were significantly lower ($p<0.05$) when the adhesive applied dentin surface was not air dried (0 second air-drying time). For OF, there appeared to be a maximum drying time of 5 seconds. All failures of groups with 0 seconds air-drying time for each adhesive system except for OF were adhesive at the dentin surface. There was no clear correlation between drying time and failure mode among the systems studied.

Figure 2 shows the results of FE-SEM observations of the resin/dentin interface with different air-drying times. These observations seemed to correspond with the results of the dentin bond strength tests. For the systems that obtained high bond strengths with 10-second air-drying time, good adaptation between adhesive and dentin were observed. On the other hand, gaps between adhesive and resin composite were observed for specimens made without air drying of the adhesives. Many cracks and pores were observed inside the adhesive of GB when the adhesive was not air dried.

DISCUSSION

The introduction of self-etching primer improves the efficacy of the dentin bonding system with the reduction of clinical steps (Kitasako & others, 2000; Van Meerbeek & others, 2003). Self-etching primers usually contain acidic functional monomers and water to enhance the wettability of hydrophobic resins. Primed tooth surfaces are usually dried on the tooth surface, because the remaining liquid primer on the adhesive surface may act as inhibitors of polymerization of the bonding agent, which is placed on the primed surface (Miyazaki & others, 1999). This study demonstrated that the air drying of single-step, self-etch adhesives applied on the dentin surface generally had a significant effect on bond strengths. With no drying, lower bond strengths were obtained for all the systems used except OF. An explanation of the reduction in bond strengths seen with no air drying was that solvents, such as water and ethanol, might act as inhibitors for the polymerization of resin components in adhesive (Cho & Dickens, 2004). Following the application of these adhesives, air drying is required in order to remove solvent from the dentin surface. FE-SEM observations on the resin/dentin interface corresponded to the results of bond strength tests. When the adhesives were not air dried, small pores and cracks, which could lead to catastrophic failure of the bonding, were observed inside the cured adhesives. The adequate bonding of adhesive materials to dentin depends not only on adequate penetration of the adhesive into dentin, but also on the mechanical properties of the cured adhesive.

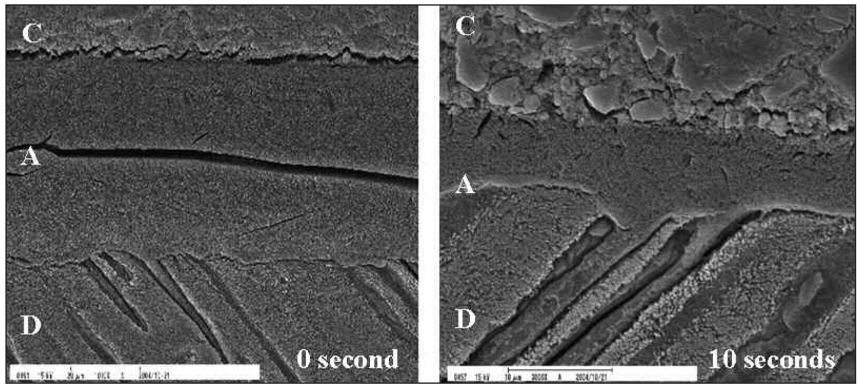

Figure 2b. *SEM photomicrographs of resin/dentin interface of CT (left; 0 second air-drying time, right; 10 seconds air-drying time; original magnification, 3,000x).*

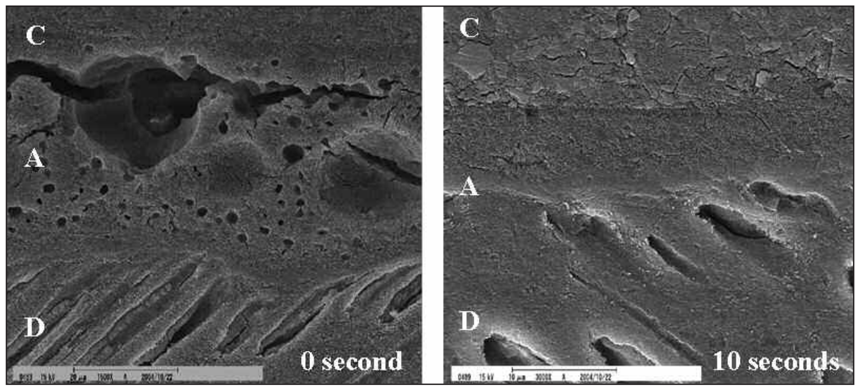

Figure 2c. *SEM photomicrographs of resin/dentin interface of FB (left; 0 second air-drying time, right; 10 seconds air-drying time; original magnification, 3,000x).*

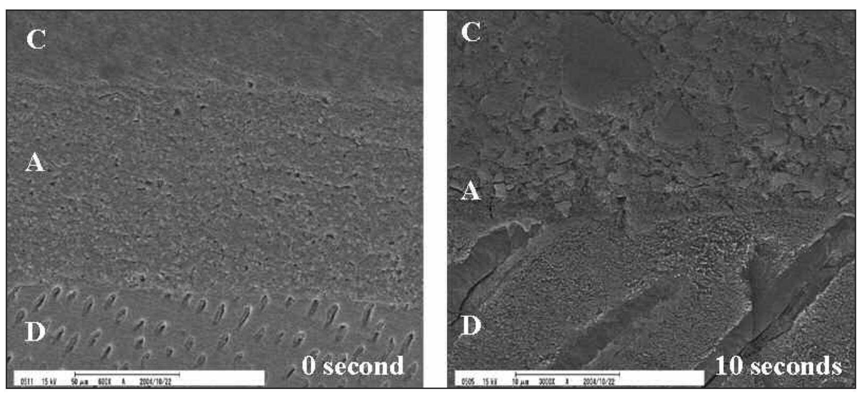

Figure 2d. *SEM photomicrographs of resin/dentin interface of GB (left; 0 second air-drying time, right; 10 seconds air-drying time; original magnification, 3,000x).*

Hypothetically stronger resins might lead to stronger bonding to dentin (Takahashi & others, 2002). The strength of cured adhesive resin is dependent on the composition, degree of conversion and length of the polymer chain. Unreacted resin monomer remaining in adhesive resins may alter their mechanical properties. Thus, the evaluation of mechanical properties of the adhesive resin is of importance to the durability of bonding to dentin (Yamada, Miyazaki & Moore, 2004). From the results of previous studies, increasing the initial acetone concentration of single-step adhesives resulted in lower bond strengths (Cho & Dickens, 2004). The higher content of acetone might lead to pores in the cured adhesive layer. The same phenomenon was observed for specimens made without air-drying time of the applied adhesive, especially for the GB adhesive system.

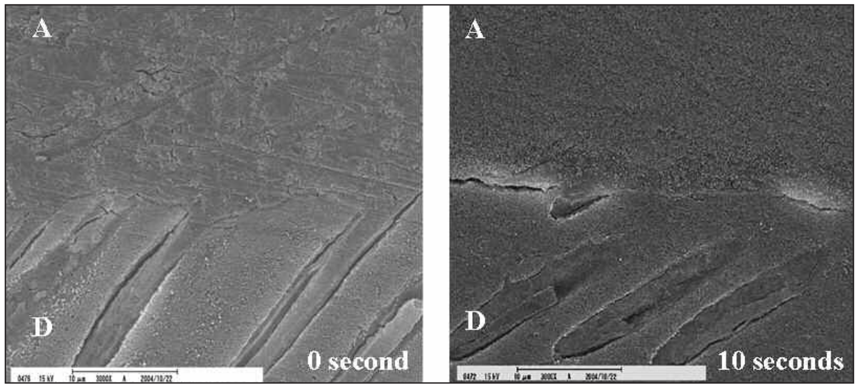

Figure 2e. *SEM photomicrographs of resin/dentin interface of OF (left; 0 second air-drying time, right; 10 seconds air-drying time; original magnification, 3,000x).*

The etching effect of self-etch adhesive is related to the acidic functional monomers that interact with the mineral component of tooth substrate and create a continuum between tooth surface and adhesive by simultaneous demineralization and resin penetration. The single-step system has to contain water and water-soluble hydrophilic monomers such as 2-hydroxyethyl methacrylate (HEMA), so that the acidic monomer can dissociate and penetrate into hydrophyllic dentin. Water is an essential component in adhesives, in order to generate the hydrogen ions required for effective dissolution and demineralization. Protons in solution derived from acidic monomer then interact with the mineral component of tooth substrate. However, improper polymerization of resin composites might occur when they were placed on single-step, self-etch adhesive. When resin composites are placed on cured adhesives without air drying, the remaining water and acid will act as an inhibitor of polymerization of the resin composites (Tay & others, 2001). An adverse interaction between acidic monomers and the light polymerized catalyst is thought to not normally occur, since the accelerators, such as aromatic sulfinic acid sodium salts, are incorporated together with initiator systems in the adhesives (Nyunt & Imai, 1996). Though the light polymerization reaction is much faster than chemical polymerization reaction, the remaining solvents inside the adhesive might behave as obstacles for the polymerization of resin composites.

It has been suggested that the etching effect of self-etch adhesives should be consumed and neutralized by reaction with hydroxyapatite in the dentin substrate (Camps & Pashley, 2000). When the composites were placed on cured adhesives without air drying, the remaining acid may have acted as an inhibitor of polymerization of the resin composites. This phenomenon was seen for most of the FE-SEM specimens for the no air-drying groups. Though gaps between adhesive and composite were observed, good adaptation between dentin and adhesives was observed.

Not all single-application, self-etch adhesives used in this study performed similarly with increasing air-drying times. For the adhesive system OF, no significant differences were found for dentin bond strengths between the 0- and 5-second air-drying time groups. Though the applied adhesive was not air dried, the remaining solvents, such as water, might not be an obstacle for polymerization of the adhesive system OF. This is presumably caused by the excellent polymerization ability of the dye-sensitized photopolymerization system employed in this adhesive. The initiator system OF contains dye-sensitizer, co-initiator and borate derivative (Table 1). The energy transfer reaction from dye-sensitizer to co-initiator occurs by light irradiation to form an excited state of co-initiator. The polymerizable radical species is then formed by reaction of the borate derivative with the activated co-initiator, with hydrogen ions derived from dye-sensitizer as well as acidic functional monomers (Kitasako & others, 2000). According to a study on the polymerization characteristics of tributylborane catalyst, the presence of a certain amount of water resulted in a higher level of polymerization activity (Okamoto, Takahata & Saeki, 1998). The unique features of the polymerization reaction of the borate derivative might relate to the results of the bond strength test. The significant reduction in bond strength observed for OF with extended drying time remains a subject for further study.

Considering the findings of this study, the benefits of using single-step, self-etch adhesives in terms of simplifying clinical procedures might be negated by the technique sensitive factors that can lead to reduced bond strengths. For the systems studied, in general, a 5-second air-drying time appears appropriate. The elimination of air drying or prolonged air drying are detrimental to some adhesives used in this study. Though the clinical performance of dentin bonding systems have been improved, the general practitioner needs to be cautioned about careful management of technique factors during clinical procedures, such as the air-drying time of applied adhesives.

CONCLUSIONS

The simultaneous etching of enamel and dentin simplifies and shortens clinical bonding procedures. However, the results of this study indicate that some self-etching primer bonding systems show dentin bond strengths that are less than adequate if the enamel surface was not air dried or if air drying was prolonged for more than 5 seconds after application of self-etching primer. Clinicians using these simplified systems must be aware of technique factors that can influence bond strengths.

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