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Effect of different air-drying time on the microleakage of single-step self-etch adhesives

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Objectives: This study evaluated the effect of three different air-drying times on microleakage of three self-etch adhesive systems. ***Materials and Methods:*** Class I cavities were prepared for 108 extracted sound human premolars. The teeth were divided into three main groups based on three different adhesives: Opti Bond All in One (OBAO), Clearfil S[3] Bond (CSB), Bond Force (BF). Each main group divided into three subgroups regarding the air-drying time: without application of air stream, following the manufacturer's instruction, for 10 sec more than manufacturer's instruction. After completion of restorations, specimens were thermocycled and then connected to a fluid filtration system to evaluate microleakage. The data were statistically analyzed using two-way ANOVA and Tukey-test ($\alpha = 0.05$). ***Results:*** The microleakage of all adhesives decreased when the air-drying time increased from 0 sec to manufacturer's instruction ($p < 0.001$). The microleakage of BF reached its lowest values after increasing the drying time to 10 sec more than the manufacturer's instruction ($p < 0.001$). Microleakage of OBAO and CSB was significantly lower compared to BF in all three drying time ($p < 0.001$). ***Conclusions:*** Increasing in air-drying time of adhesive layer in one-step self-etch adhesives caused reduction of microleakage, but the amount of this reduction may be dependent on the adhesive components of self-etch adhesives. (*Restor Dent Endod* 2013;38(2):73-78)

Key words: Air-drying; Microleakage; Self-etch adhesive

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Introduction

Recently introduced dental adhesives offer simplified clinical application and eliminate technique sensitivity.[1] The major benefit of the one-step self-etching systems is that they are less technique sensitive than multi-step systems. While the adhesive system becomes simpler, careful product management is still required in order to attain optimum bonding procedures. However, according to recent studies, the bond quality of these products were affected by several factors, such as 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.[2-5] The etching effect of the self-etch adhesives attributed to hydrogen ions derived from the acidic monomers.[6] Water is an essential component in the adhesives, which allows them to generate the hydrogen ions.[7] 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.[8] Since self-etching adhesives contain acidic functional monomers and water, which

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might interfere with subsequent resin polymerization, air-drying time might be an influential factor in determining dentin bond qualities.[7] The monomers are dissolved in organic solvents such as ethanol and acetone, which improve monomer penetration between the collagen fibrils of demineralized dentin and play an important role in the removal of water during solvent evaporation.[1,9] The high quality of the hybridization process depends on successful monomer infiltration into the dentinal matrix and the removal of water and solvents from the surface prior to polymerization.[10] 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.[11] Residual solvents may compromise the polymerization of adhesives and also directly affect the bond integrity, providing defects within the polymerized adhesive and pathways for nanoleakage.[12-14] Marginal leakage of resin composites has been a significant concern for clinical practitioners that can be resulted in pulp pathology, hypersensitivity, secondary decay, degradation of bond area, and marginal staining.[15] However, complete evaporation of solvents is difficult to achieve. A clinical approach to accelerate solvent evaporation recommended by the manufacturers is the use of an air stream. Although air drying of applied adhesive is recommended to evaporate solvents in the adhesives, variations in air-drying time probably occur in clinical application particularly in complex cavity with high C-factor.[7-9] This study evaluated the influence of the air-stream duration on the microleakage of different self-etch adhesive systems in Class I composite resin restorations with a fluid filtration method. The null hypothesis was that there was no difference in the leakage of self-etch adhesive systems with various times of solvent evaporation.

Materials and Methods

One hundred and eight noncariouse human premolars, extracted for orthodontic reasons were selected under a protocol approved by the Ethics Committee of Mashhad University of Medical Sciences (900221/2011). Teeth were disinfected in 0.5% chloramine T solution and stored in distilled water until starting the experiment. Class I cavities (2 × 2 × 4 mm) were made using straight fissure diamond burs (SS White Burs, Inc., Lakewood, NJ, USA) in a high-speed handpiece under copious water spray. The roots removed at 2 mm below the cement-enamel junction (CEJ) with an aluminum oxide disc (Sof-Lex, 3M ESPE, St. Paul, MN, USA) mounted on a low speed handpiece.

Prepared teeth were then divided randomly into three main groups (*n* = 36) based on three different self-etch adhesives which was used:

Group 1: Opti Bond All in One (OBAO, Kerr Corp, Orange, CA, USA)

Group 2: Clearfil S^3 Bond (CSB, Kuraray Medical Inc., Tokyo, Japan)

Group 3: Bond Force (BF, Tokuyama, Tokyo, Japan)

Adhesives were applied on the dentin surface according to the manufacturer's instruction (Table 1). Each of the three main groups was subdivided into three subgroups A, B and C (*n* = 12) for application of different duration of air stream for adhesive evaporation:

Subgroup A: without application of air stream

Subgroup B: according to the manufacturer's instruction

Subgroup C: 10 seconds more than manufacturer's instruction

Primed dentin surfaces were dried with oil-free compressed air at 25℃ (normal temperature) with 0.2 MPa air pressure for three different times from 5 cm above the dentin surface using a three-way syringe attached to a

Table 1. Chemical formulation and manufacturer's directions of the adhesives applied

Materials	Composition	Application procedures
Opti Bond All in One (OBAO)	GPDM, GDM, HEMA, Bis-GMA, water, ethanol, acetone, silica filler, CQ, sodium hexafluorosilicate	Apply two coats with agitation for 20 sec each Dry with air pressure for 5 sec Light cure for 10 sec
Clearfil S3 Bond (CSB)	10-MDP, HEMA, Bis-GMA, water, ethanol, silanated colloidal silica, CQ	Apply for 20 sec Dry with air pressure for 5 sec Light cure for 10 sec
Bond Force (BF)	Methacryloyloxyalkyl acid phosphate, HEMA, Bis-GMA, TEGDMA, water, isopropyl alcohol, Glass Filler, CQ	Apply for 20 sec Dry with moderate, strong air pressure for 5 sec after weak air pressure for 5 sec Light cure for 10 sec

HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol-A-diglycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; CQ, camphorquinone; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; GPDM, glycerol phosphate dimethacrylate; GDM, glycerol dimethacrylate.

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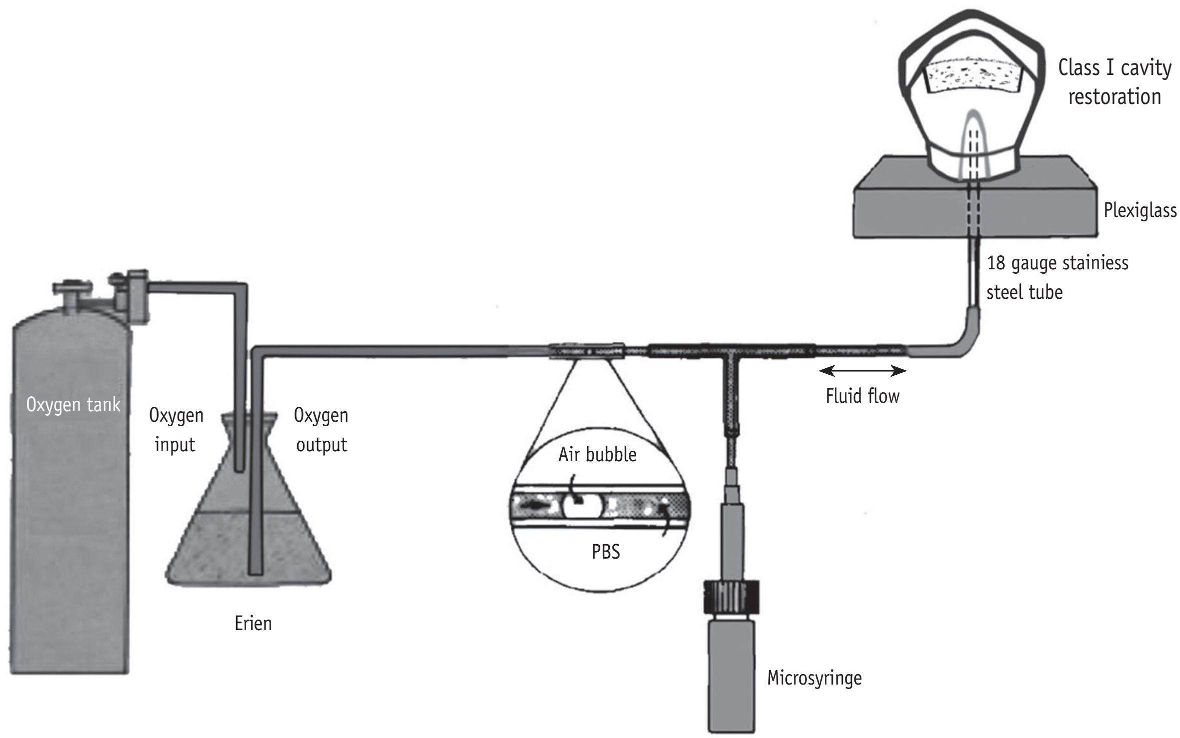

Figure 1. Schematic illustration of the fluid filtration apparatus used to measure the microleakage of the resin composite restorations bonded with the assigned adhesives in this study.

dental unit. After polymerization of the adhesives using a light curing unit (Optilux 500, Demerton-Kerr, Orange, CA, USA), cavities were restored 1 mm incrementally with the resin composite supplied by the same manufacturer with the assigned adhesive, group 1, Herculite XRV (Kerr); group 2, Clearfill APX (Kuraray); group 3, Estelite Sigma quick composite (Tokuyama). After storage in 37℃ distilled water for 24 hours, the restorations were finished and polished with aluminum oxide discs (Sof-Lex) and the specimens were thermocycled (Nemo Inc, Mashhad, Iran) at 5℃ and 55℃ ± 2℃ for 500 cycles. For leakage assessment, the specimens were assigned in a fluid filtration system. For connecting the specimens to a fluid filtration system, the canal orifices from apically position were widened with 2 and 3 Gates-Glidden drills and pulp tissue was deleted carefully. The pulp chambers were then rinsed with saline solution. Measurement of fluid movement was made through the 18-gauge needle in the Plexiglass connected to the cut surface of crown segments (Figure 1). Evaluation were performed by measurement of fluid flow through each specimen under 23/4 psi of water pressure every 2 minutes for four times (2, 4, 6, 8 minutes)after connecting the samples to fluid filtration system.

These four fluid flow values were averaged to a single mean value for raising the accuracy of extracted data (μL/min/cmH_2O). Data were analyzed statistically using two-way ANOVA and Tukey test ($\alpha = 0.05$).

Results

The results of the leakage measurements are presented in Tables 2 and 3. ANOVA test demonstrated a statistically significant interaction between the type of adhesive and air-drying time ($p < 0.001$). It showed that the leakage of the restoration restored with all the adhesives were significantly decreased when the air-drying time increased from no drying to the time recommended by the manufacturer's instruction ($p < 0.001$). Microleakage of BF reached its lowest values after increasing the drying time to 10 seconds more than the manufacturer's instruction ($p < 0.001$), but in OBAO and CSB groups increasing in drying time over the manufacturer's time suggestion had no significant effect on their microleakage (Table 3). Tukey test showed a significant difference in leakage of OBAO and BF, also between CSB and BF in all three drying time ($p < 0.001$). However, there were no significant differences in microleakage of OBAO and CSB in different drying time (Table 3).

Table 2. The ANOVA results of this experiment

Model	df	Mean square	F value	*p* value
Adhesive system	2	4.647×10^{-9}	75.180	0.001
Drying time	2	4.928×10^{-8}	797.443	0.001
Adhesive system X Drying time	4	4.187×10^{-10}	6.774	0.001

Table 3. Comparison between microleakage (Unit: µL/min/cmH$_2$O, mean ± SD, *n* = 12) of adhesive systems in each drying-time

Materials	Drying-time		
	No air-drying	Manufacturer's instruction	10 sec more than manufacturer's instruction
OBAO	$(7.01 \pm 1.02) \times 10^{-5\ a\ A}$	$(9.35 \pm 1.59) \times 10^{-6\ a\ B}$	$(5.48 \pm 1.44) \times 10^{-6\ a\ B}$
CSB	$(6.34 \pm 1.48) \times 10^{-5\ a\ A}$	$(8.96 \pm 3.80) \times 10^{-6\ a\ B}$	$(4.30 \pm 2.28) \times 10\text{-}^{6\ a\ B}$
BF	$(9.40 \pm 1.18) \times 10^{-5\ b\ A}$	$(3.19 \pm 0.72) \times 10^{-5\ b\ B}$	$(1.34 \pm 0.41) \times 10^{-5\ b\ C}$

Means within each group with the same superscript letter are not significantly by Tukey test (small letter, column; capital letter, row). OBAO, Opti Bond All in One; CSB, Clearfil S3 Bond; BF, Bond Force.

Discussion

The results of this study showed that microleakage of the single-step self-etch adhesives varied when the duration of air-drying was altered. Dye, bacteria, and radioisotope penetration methods, and light microscopic or scanning electron microscopic (SEM) methods have been used for measurement of leakage around the restorative materials that they provide qualitative information only.[15] Fluid filtration method, introduced and developed by Pashley's group, has been extensively used for 30 years for research purpose to understand the physiology of dentin, as well as the effects of various restorative treatments on dentin permeability. It permits quantitative, nondestructive measurement of microleakage in a longitudinal manner. The fluid filtration method permits quantitative measurement of leakage. Fluid filtration systems enhance reliability, reproducibility and comparability.[16] Inherent solvents of the adhesives behave like a transporter media and lower resin viscosity in adhesive solutions, allowing the resin monomers to penetrate into the demineralized porous dentin surfaces.[17] However, residual solvent can inhibit polymerization and weakens the mechanical properties of the adhesive resin, so they should be removed from the adhesive to a sufficient degree.[14,18] The tested adhesives contain different types of solvents, OBAO, acetone and ethanol; BF, isopropyl alcohol; CSB, ethanol. Water is a poor solvent for hydrophobic monomers, so alcohol or acetone is added to the solution.[19] Alcohol has higher vapor pressure compared to water and provides better evaporation by air-drying.[14] Water-alcohol mixtures form hydrogen bonds between their molecules that facilitate evaporation of the solvent compared to pure water.[20] Acetone has higher vapor pressure and lower boiling temperature when compared to alcohol, which translates into faster evaporation of it.[9] However, it was reported that even for an acetone-based adhesive, spontaneous evaporation exhibits only after 5 minutes of its application, which is clinically unacceptable.[19] Furthermore, the adhesives used in the present study contain HEMA, which is a co-monomer that acts as a wetting agent and diffusion promoter for resin.[21] One drawback of HEMA is that it strongly retains water in hydrogels which is difficult to evaporate. One of the methods used to improve the solvent evaporation rate is air-drying prior to light irradiation.[22,23] This study evaluated the effect of different air-drying time on microleakage of single-step self-etch adhesives. The data in the present study suggested that increasing in air-drying time from no drying to the time recommended by the manufacturers' instructions time led to significant decrease in microleakage for three adhesives. Whereas increasing air-drying time more than manufacturer's instruction resulted in significant decrease in microleakage only for BF adhesive. So these differences may be due to various compositions and the main application protocol of manufacturer. Air-drying provides better solvent evaporation and leaves only the priming resin at the dentin surface, resulting in higher degree of conversion.[24,25] Jacobsen *et al.* reported that the bond strength of all-in-one adhesives was affected by the air-drying time and reached its highest values after an air-drying time of 10 seconds or longer.[22] Argolo *et al.* evaluated the influence of the dwell time between the application of ethanol-based adhesive and light activation on the dentin bond strength and degree of conversion.[26]

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They found that longer dwell time (60 seconds) resulted in better bond strength and greater percentage of conversion and this may be due to better solvent volatilization. Furuse *et al*. evaluated the influence of the degree of solvent evaporation on the bonding capacity of one-step, self-etching adhesives, and also reported more adhesive failures were observed with shorter air-blowing durations than manufacturer's instructions.[27] For OBAO and CSB increasing in drying-time over the manufacturer's instruction did not decrease the microleakage. Ogura *et al*. evaluated the effect of warm air-drying on dentin bond strength, and found prolonged warm air-drying detrimental to some adhesives.[28] This finding indicated that some residual solvent is required for improving the degree of cure. However, the microleakage of BF reached its lowest values after increasing the drying time to 10 seconds more than the manufacturer's instruction. The vapor pressure (at 25℃) of isopropyl alcohol is 44 mm Hg, for ethanol is 54.1 mm Hg and for acetone is 200 mm Hg.[29] These indicate that evaporation of isopropyl alcohol by air-drying is more difficult than acetone and ethanol. In the present study CSB and OBAO exhibited better sealing ability compared to BF. Functional monomer of CSB is 10-MDP (10-methacryloxydecyl dyhidrogen phosphate). Yoshida *et al*. investigated interaction of 10-MDP with dentin and reported that this monomer interact chemically with hydroxyapatite and its bond appeared very stable, as confirmed by the low solubility of its calcium salt in water.[6] Higher bond strength of the 10-MDP to dentin may improve sealing properties of the CSB adhesive.[30] Good sealing ability of OBAO may be related to its application method, because the manufacturer recommends application of two coats of adhesive before air-drying. Previous studies demonstrated that by applying more coats of adhesives, the bond strength can be improved and microleakage was decreased.[31-33] It is purposed in future research to work about other properties of self-etch adhesives with regarding the role of different solvents and dentinal substrates. Also, doing a long term clinical trial about this subject will be necessary for supporting the *in vitro* studies.

Conclusions

Under the conditions of the current investigation, it can be concluded that the optimal drying time of some adhesives may deviate from the manufacturer's directions for use. This is especially important in clinical situations that cavity complexity might interfere with the access of air stream to each part of the bonded surface, so clinicians should be aware of the probably solvent present in adhesives and carefully evaporate it particularly in cavities with complex geometry to achieve best clinical results.

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

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