

The influence of air temperature for solvent evaporation on bonding of self-etch adhesives to dentin

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ABSTRACT

Objective: This study evaluated the effect of air temperature (warm or cold) for solvent evaporation on bonding and nanoleakage of self-etching adhesives to dentin. **Materials and Methods:** The adhesives Clearfil 3S Bond [S3], OptiBond All-In-One [OB], Adper SE Plus [AD], and Silorane adhesive [SI] were applied on dentin surfaces, and a warm ($60 \pm 2^\circ\text{C}$) or cold air ($20 \pm 1^\circ\text{C}$) was applied and light-cured. After water storage (24 h), the teeth were sectioned into sticks (0.8 mm^2) and tested in tensile. Then, they were immersed in a 50% solution of silver nitrate, photo-developed and the nanoleakage observed in a scanning electron microscope. The bond strength and nanoleakage pattern were analyzed by two-way analysis of variance and *post hoc* Tukey's test ($\alpha = 0.05$). **Results:** Higher bond strength and lower silver nitrate uptake were observed for the adhesives under warm condition ($P < 0.05$). AD and SI showed better adhesive results than S3 and OB ($P < 0.05$). **Conclusion:** The use of a warm air was useful to improve the bonding and diminish the nanoleakage of adhesive systems to dentin.

Key words: Bonding, dental adhesives, dentin

INTRODUCTION

Nowadays, the bonding of resin materials to tooth may be achieved by the etch-and-rinse or the self-etch strategies. They have been used directed to reduce the number of steps of the bonding protocol.^[1-3] Self-etch adhesives do not require the etching and rinsing steps, and their components may be presented into a single solution or in two bottles.^[2,3] They are less technique-sensitive because do not require removal of the smear layer and plugs.^[3]

Different researchers have observed that some self-etch adhesives presented lower bond strength to enamel and

dentin than the etch-and-rinse ones.^[1,3] They contain high concentration of solvents,^[4] which may interfere at the polymerization of the monomers.^[5] The higher the solvent content within the polymer, the lower the mechanical properties^[3,7] and the resin-dentin bond strength.^[7,8] The removal of solvents from self-etch adhesives before the light-curing is needed, and different strategies to achieve this purpose have been tested.^[9-13]

The use of a warm air around 60°C to evaporate solvents was reported to be able to increase the bond strength of two-step etch-and-rinse adhesives to dentin^[14] and reduced the degradation of resin-dentin

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interface over 6 months.^[15] However, this approach is controversial,^[9,11] and the use of this strategy to increase the bond strength and reduce the nanoleakage of self-etch systems to dentin is not known.

The aim of this study was to evaluate the influence of air temperature for solvent evaporation on bonding and nanoleakage of four self-etch adhesive systems to dentin. The null hypothesis is that there will be no difference between the adhesive systems and air temperatures on the studied parameters.

MATERIALS AND METHODS

Teeth and material selection

Forty human third molars were used after approval of the local Ethics Committee (Protocol 0186/08). The teeth were cleaned in 0.5% chloramine and their occlusal surface flattened using silicon carbide paper of #180 grit to expose dentin surfaces that were further polished on wet #600-grit silicon-carbide paper for 60 s to standardize the smear layer.

Four self-etch adhesive systems were tested: Two two-step self-etch (Adper SE Plus - AD and Silorane Adhesive - SI - 3M ESPE, St. Paul, MN, USA) and two one-step (Clearfil 3S Bond -S3 - Kuraray Medical Inc, Tokyo, Japan and OptiBond All-In-One - OB - Kerr Co, Orange, CA, USA). The composition, application mode, and batch number are described in Table 1.

Restorative procedures

After the application of each adhesive, the solvent evaporation was performed either with a warm ($60 \pm 2^\circ\text{C}$) or cold air ($20 \pm 1^\circ\text{C}$) for 10 s at 10 cm. The air stream was obtained by a hair-dryer (SC831, Black and Decker, Uberaba, MG, Brazil) at 5.50 m/s speed and $0.0138 \text{ m}^3/\text{s}$.^[8] air flow. The adhesives were light-cured using a quartz tungsten halogen light ($600 \text{ mW}/\text{cm}^2$; UltraLux, Dabi Atlante, Rio de Janeiro, Brazil). For S3, OB, and AD, resin composite build-ups (Opallis, VL, FGM, Joinville SC, Brazil) were built into 5 increments of 1 mm each. For SI, the restorations were build-up in the same way, but silorane-based composite was used (Filtek P90, 3M ESPE, St. Paul, MN, USA). The bonding procedures were carried out by the same operator at a room temperature of 20°C and constant relative humidity. Five teeth were used for each combination of adhesive system and air temperature.

Microtensile testing

The restored teeth were stored in distilled water ($37^\circ\text{C}/24 \text{ h}$), longitudinally sectioned in

mesio-to-distal and buccal-to-lingual directions across the bonded interface with a diamond saw in an Isomet 1000 machine (Buehler Ltd, Lake Bluff, IL, USA) to obtain sticks (cross-sectional area of approximately 0.8 mm^2). The number of premature debonded sticks per tooth during this step was recorded. The cross-sectional area of each stick was measured to calculate the bond strength in Mega Pascal (MPa).

The sticks were attached to a Geraldéi's device for microtensile testing (Odeme, Biotechnology, Joaçaba, SC, Brazil) using a cyanoacrylate resin (Super Bonder Gel, Loctite, Itapevi, São Paulo, SP) and tested in tensile (EMIC, São José dos Pinhais, PR, Brazil) at $0.5 \text{ mm}/\text{min}$. The failure modes were observed at 40X (BEL Microimage analyser, Photonics, Italy) and classified as cohesive (failure within dentin or resin composite, C), adhesive (failure at dentin/resin interface - A), or adhesive/mixed (at dentin/resin interface that included cohesive failure of the neighboring substrates, A/M).

Scanning electron microscopy for silver nitrate uptake evaluation (SNU)

Two bonded sticks from each tooth used were coated with two layers of nail varnish applied up to within 1 mm of the bonded interfaces. A total of 10 bonded sticks were analyzed for silver nitrate uptake as 5 teeth were used per experimental conditions. The specimens were re-hydrated in distilled water for 10 min prior to immersion in the tracer solution. Ammoniacal silver nitrate was prepared according to the protocol previously described by Tay *et al.*^[16] The sticks were placed in the ammoniacal silver nitrate in darkness for 24 h, rinsed thoroughly in distilled water, and immersed in photo developing solution for 8 h under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface.

All sticks were wet-polished with 600-grit SiC paper to remove the nail varnish. Specimens were polished with a 800-, 1000-, 1200-, 1500-, 2000-, 2500-grit SiC paper and 1 and $0.25 \mu\text{m}$ diamond paste (Buehler Ltd., Lake Bluff, IL, USA) using a polish cloth.

They were ultrasonically cleaned, air-dried, mounted on aluminum stubs, and sputter-coated with carbon only. Resin dentin interface were analyzed in a scanning electron microscope (Jeol 5800, Tokyo, Japan) operated in the backscattered electron model with an accelerating voltage of 12 KV.

Three pictures from each specimen were taken. The first picture taken was the one in the center of the stick.

Table 1: Adhesive systems, composition, and application modes

Material (batch number)	Composition	Application mode
Clearfil 3S bond (0006)	10 MDP, Bis-GMA, 2-HEMA, di-camphoquinone, hydrophobic dimetacrilate, ethanol (< 20%), water, and silanated colloidal silica	Shake the bottle Apply one coat of adhesive vigorously (20s); Dry with warm/cold air stream (10s) Light cure (10s)
OptiBond All In one (3075076)	Uncured methacrylate ester (33-43%), ethyl alcohol (4-9%), water, Acetone (35-45%), monomers, inert mineral fillers, ytterbium fluoride, photo initiators, accelerators, and stabilizers	Shake the bottle; Apply first coat of adhesive vigorously (20s); Apply second coat of adhesive vigorously (20s); Dry with warm/cold air stream (10s); Light cure (10s).
Adper SE plus (8BE and 8BB)	Primer (liquid A): Water (80%), HEMA, rose bengal, surfactant; Adhesive (liquid B): Methacrylate resin (UDMA, TEGDMA, TMPTMA, HEMA phosphate and MHP acidic monomers, zirconia, photo initiators	Apply liquid A; Vigorously apply liquid B (20 s); Dry with warm/cold air stream (10 s); Apply a second coat of adhesive; Dry with warm/cold air stream (20 s); Light cure (10 s)
Silorane system adhesive (8AP and 8AL)	Self-etch primer: Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers Bond: Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	Shake primer bottle briefly; Active rub during (15 s); Gentle air dispersion with warm/cold (10s); Light cure (10 s); Shake bond bottle briefly; Apply visibly thick layer; Gentle air dispersion with warm/cold until movement stops; Light cure (10 s)

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenolglycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, HEMA : 2-hydroxyethyl methacrylate, TMPTMA: Trimethylolpropane trimethacrylate, MHP: Methyl hydroxypropyl phosphates

Then, the other two pictures were taken 0.3 mm left and right from the first one by a blind technician. Only the most representative one, among the three, was chosen per stick. The relative percentage of silver nitrate uptake within adhesive and hybrid layer was measured using the UTHSCSA Image Tool 3.0 software (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, Texas).

Statistical analysis

The mean bond strength of all sticks from the same tooth was averaged for statistical purposes. The μ TBS mean for every testing group was expressed as the average of the five tooth used per group. The bond strength and silver nitrate uptake data were subjected to a two-way analysis of variance (air temperature vs. adhesive System) and the *post hoc* Tukey's test ($\alpha = 0.05$).

RESULTS

Microtensile testing

The mean cross-sectional area of the specimens

ranged from 0.87 to 1.02 mm², and no difference among groups was detected ($P > 0.05$). The premature debonded failures and the mode of fractures are shown in Table 2. An overall rate of premature failures was observed for the Optibond All-in-One adhesive.

Table 3 presents the overall means and the respective standard deviations of the resin-dentin bond strengths (MPa) for all experimental groups. The cross-product interaction adhesive vs. air temperature was not statistically significant ($P = 0.72$). The main factors adhesive and air temperature were statistically significant ($P = 0.03$ and $P = 0.02$, respectively). Higher bond strength values were observed for all adhesives when the solvent evaporation step was performed with a warm air [Table 3]. Two-step self-etch adhesives (AD and SI) showed higher bond strength to dentin regarding one-step bond strength (S3 and OB) [Table 3].

Silver nitrate uptake

The means and standard deviations of the silver nitrate uptake (%) for all experimental groups are shown in Table 4. The main factors adhesive and air temperature

Table 2: Number of specimens and their representative percentages (%) distributed according to the fracture pattern mode for each experimental condition

Adhesive	Air temperature	Fracture pattern		
		Adhesive/ mixed	Cohesive	Premature failures
Clearfil 3s bond	Cold	42 (79.2)	11 (20.8)	0 (zero)
	Warm	47 (88.7)	2 (3.8)	4 (7.5)
Optibond all in one	Cold	25 (46.3)	9 (16.7)	20 (37.0)
	Warm	44 (67.7)	11 (16.9)	10 (15.4)
Adper se plus	Cold	52 (86.7)	8 (13.3)	0 (zero)
	Warm	57 (93.4)	4 (6.6)	0 (zero)
Silorane adhesive system	Cold	54 (90.0)	6 (10.0)	0 (zero)
	Warm	66 (97.1)	2 (2.9)	0 (zero)

Table 3: Overall microtensile bond strength values and respective standard deviations (MPa) obtained in each experimental condition (*)

Adhesive	Air temperature	Mean (standard deviation)
Clearfil 3s bond	Cold	30.1 (6.9) c
	Warm	35.8 (4.4) b
Optibond all in one	Cold	29.4 (6.4) c
	Warm	35.2 (5.4) b
Adper se plus	Cold	34.2 (4.3) b
	Warm	39.4 (3.8) a
Silorane adhesive system	Cold	35.3 (4.6) b
	Warm	40.4 (5.1) a

(*) The same lowercase letters indicate means statistically similar ($P > 0.05$)

were statistically significant ($P < 0.02$), but not the cross-product interaction ($P = 0.63$). Statistically lower silver nitrate uptake was observed for all adhesives when the solvent evaporation step was performed with a warm air-stream [$P = 0.01$; Table 4]. One-step bond strength (S3 and OB) showed higher amount of silver nitrate than two-step self-etch adhesives (AD and SI) [$P = 0.02$; Table 4]. Representative SEM images of the resin-dentin interfaces from the experimental conditions are shown in Figure 1.

DISCUSSION

The self-etch adhesives are combination of hydrophilic and hydrophobic monomers, acetone, ethanol, and water or their combination.^[4] Although they offer a simplified technique, a complex chemistry exists in this combination.^[1,4] Water and solvents provide the ionization medium for the self-etch activity,^[4] and solvents reduce their viscosity to allow the penetration of resins into the porosities of the prepared tooth surface^[17] as well as enhance the mobility of polymer

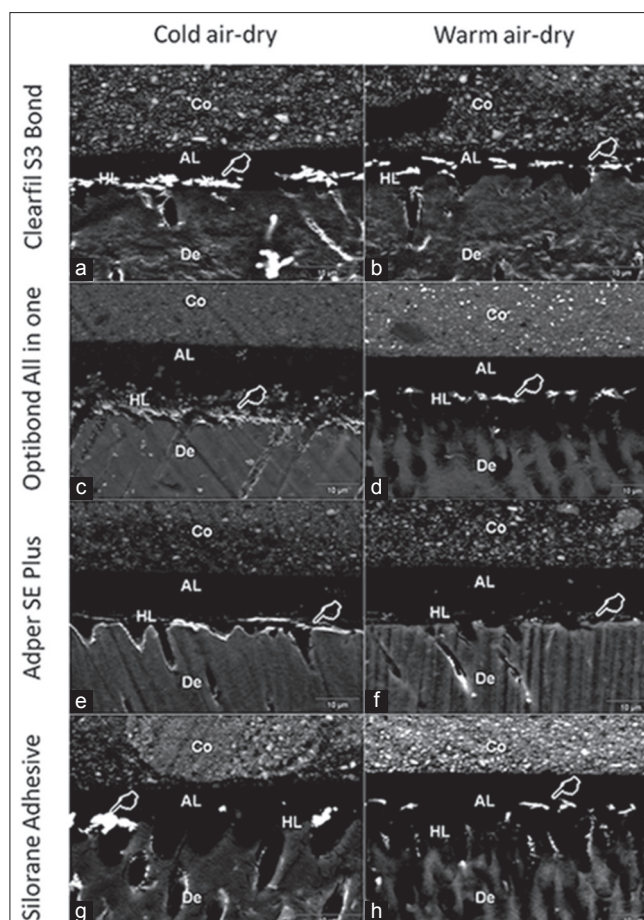


Figure 1: Representative backscattered SEM images of the resin-dentin interface bonded of all experimental conditions. The amount of silver penetration in the cold air dry stream groups [Figure a, c, e and g] was higher (see white hand) in comparison when warm air-dry stream was used [Figure b, d, f and h]. (Co = Composite, AL = Adhesive layer, HL = Hybrid layer, De = Dentin)

chains.^[18] But, residual solvents within the polymerized adhesive and hybrid layer may compromise the formation of the polymer network.^[11,19]

The removal of solvents should be done as much as possible from adhesive systems before the light-curing.^[20] This is a critical step because as the water/solvent evaporates from the adhesive, the monomer density increases and concentrate on the adhesive layer, which reduces further solvent evaporation.^[21]

This is critical for simplified adhesives since the extent of solvent and water retention in polymer networks seems to be directly correlated with the hydrophilicity of the resin blends.^[20] The recommended clinical time for solvent evaporation (around 10 s) is rather short as some studies have shown that longer times around 24-96 h were required to the highest solvent evaporation.^[10]

Table 4: Overall silver nitrate uptake values and respective standard deviations (%) obtained in each experimental condition (*)

Adhesive	Air temperature	Mean (standard deviation)
Clearfil 3s bond	Cold	18.1 (6.5) c
	Warm	12.4 (5.2) b
Optibond all in one	Cold	23.4 (5.2) c
	Warm	15.4 (4.3) b
Adper se plus	Cold	14.2 (3.4) b
	Warm	6.2 (3.2) a
Silorane adhesive system	Cold	13.5 (3.8) b
	Warm	8.3 (3.2) a

(*) The same lowercase letters indicate means statistically similar ($P>0.05$)

Different strategies were investigated to increase solvent evaporation such as the use of high-pressure or prolonged air-drying^[9-13] and increase in the air-drying temperature.^[9,11,14] The effect of the warm air-drying on solvent evaporation is controversial among the studies that did not observe advantages of using it^[9,11] and others that have noticed improvement on the resin-dentin bonds^[14] as well as observed in the present investigation. Different air temperatures were used for solvent evaporation, which explain their different results that employed half of the temperature^[9,11] than Klein-Júnior^[14] and in the present investigation. Usually, longer time to evaporation with warm-air around 38°C also helps to improve solvent evaporation and resin-dentin bond strength.^[9,11] However, an significantly increase of approximately 20% in the resin-dentin bonds could be detected when a warm air-stream at around 60°C was employed,^[14,15] and it is higher when compared with previous study.^[9]

Klein-Júnior *et al.*^[14] explained these results by the increasing of the kinetic energy of the molecules by the heat that changed their potential energy and state.^[22] When residual solvents remain within the resin-dentin interface, they act as a source of nanoleakage within adhesive interfaces.^[23] These are areas of incomplete polymerization and/or hydrogel formation^[16,23] and are prone to deposition of silver nitrate, as can be seen in the micrographs of the present investigation, mainly for the cold air dry groups.

However, the use of warm-air temperature for evaporating solvents did not change the hydrophilicity of the adhesive layer and so silver nitrate deposition was observed in these groups since the water flux from the underlying dentin still occurred.^[16,23]

Although in cold/ warm air-dry groups, the two-step self-etch showed lower silver nitrate uptake regarding

one-step self-etch, it was not a consensus in the literature^[24-28] but may be explained by the use of an hydrophobic coat as the second layer, which may have helped to prevent the water sorption.^[1,23] Simplified adhesives are highly hydrophilic and attract water that plasticizes the polymer and reduces the mechanical properties, including the bond strength to dentin.^[7,8]

This study showed that the use of a warm air-stream improved the bond strength and reduced the nanoleakage of the resin-dentin bonds, but it is also important to consider that other factors may influence the bonding performance of simplified adhesives to dentin, as the variability of the dentin substrate (superficial vs. deep dentin)^[29] and the use of nanofilled materials to enhance their performance to dentin.^[30] Also, further studies are needed to evaluate the effect of this strategy on the longevity of bonding to dentin by these adhesives.

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