

Effect of prolonged air drying on the bond strength of adhesive systems to dentin

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This in vitro study evaluated the effect of air-drying time on degree of solvent evaporation (DE), dentin microtensile bond strength (μ TBS), and degree of conversion (DC) of 5 adhesive systems: Adper Single Bond 2, XP Bond, Prime & Bond 2.1, OptiBond Solo, and Adper Easy One. For DE testing, 20 μ L of each material was submitted to measurements in a digital balance after an air stream of 3, 5, 10, 20, 30, or 60 seconds; the weight loss was computed and converted to a percentage (DE). For μ TBS testing, 50 sound human molars were divided into groups ($n = 5$). The 5 adhesive systems were applied either in accordance with manufacturers' instructions for solvent drying time (control) or with a prolonged drying time (20-30 seconds). After composite resin was built up on the hybridized surfaces, the teeth were stored for 24 hours and then sectioned to obtain beams that were loaded until fracture. For DC testing, specimens of each adhesive and air-drying condition ($n = 3$) were evaluated

by means of attenuated total reflectance Fourier transform infrared spectroscopy. Data were submitted to 2-way analysis of variance, t test, and Spearman test for correlation analysis. Prolonged air drying resulted in significantly greater DE than did the time suggested by the manufacturers. The adhesives XP Bond and Adper Easy One showed significantly greater μ TBS with prolonged air drying. The DC was not affected by air-drying time. No statistically significant correlation was found between DC and μ TBS values. Depending on the material, bond strength can be improved by prolonged air-drying times.

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Hydrophilic volatile solvents are present in dental adhesive systems to dissolve, ionize, and carry the functional monomers in dentin to create a resin-impregnated collagen matrix known as the *hybrid layer*. Solvents also serve as a vehicle to remove water from substrate in wet dentin.¹

Air drying is considered the most simple and effective method to achieve solvent evaporation.²⁻⁶ However, this effect seems to be dependent on, among other factors, the type of solvent. Ethanol and acetone are common solvents added to adhesive systems. The evaporation process is modulated by solvent vapor pressure, and the combinations of solvents, substrate moisture, monomers, and inhibitors create distinct evaporation rates for different products; therefore, complete solvent elimination by air drying can be difficult to achieve.²⁻⁸ Consequently, some residual solvent remains trapped in the adhesive.^{4,8}

The presence of residual solvent prior to light curing can not only interfere with the polymerization of adhesive, reducing the degree of conversion, but also reduce the mechanical properties of adhesive layer.^{2,5,8-11} In addition, residual solvent produces an adhesive layer with a porous structure that enhances water sorption, thereby compromising bonding quality and longevity by hydrolytic degradation.^{12,13}

Furthermore, manufacturers' instructions concerning the time of air drying necessary to evaporate solvent seems to be insufficient to promote optimal mechanical properties.^{8,14} However, it is still unclear whether bond strength actually can be compromised, as some studies have shown that insufficient air-drying protocols lead to reduced bonding, but others observed that this effect is material dependent.¹⁵⁻¹⁷

Therefore, the aim of the present study was to evaluate the effect of air-drying time on solvent evaporation, dentin bond strength, and degree of conversion of different adhesive systems. The null hypotheses were that prolonged air drying will not influence degree of conversion or dentin bond strength of the tested adhesive systems.

Materials and methods

Five adhesive systems were evaluated in this study. Compositions, classifications, and manufacturers' instructions are shown in Table 1.¹⁸⁻²²

The influence of air drying on the degree of evaporation (DE) of solvents in the adhesive systems was evaluated indirectly by a gravimetric method measuring the weight loss.⁹ Dentin bond strength was measured by microtensile bond strength (μ TBS), and the degree of conversion (DC) was determined by means of Fourier transform infrared (FTIR) spectroscopy.

Degree of evaporation

Standard aliquots of each material (20 μ L) were dispensed on a previously weighed glass sheet (50 \times 90 mm) with a micropipette (Kacil Industria e Comercio, Ltda.) and immediately submitted to measurements in a digital balance (SAE 200, Robert Bosch, LLC) with accuracy of 0.0001 g. After initial mass was recorded, the glass sheet was removed from the balance, and air drying was performed with moisture- and oil-free air from an air syringe connected to a standard apparatus with controlled 80-lb air pressure. Air drying was performed at a 20-cm distance for 3, 5, 10, 20, 30, or 60 seconds.

After each drying time was completed, the glass sheet with adhesive was placed in the balance, and the final mass was recorded. All measurements ($n = 6$) were performed with the balance closed and protected from light at standardized conditions in an acclimatized room (25°C, 50% relative humidity). The DE (in percent) for each time was calculated by the following equation: $(\text{initial mass} - \text{final mass} / \text{initial mass}) \times 100$.

Microtensile bond strength

After the protocol was approved by the local research ethics committee (0036.0.243.000-11), 50 sound extracted human third molars were selected for the

Table 1. Composition of tested materials and manufacturers' instructions for use.¹⁸⁻²²

Material (manufacturer), approach, and batch No.	Composition	Instructions
Adper Single Bond 2 (3M ESPE), 2-step total etch, N169797BR	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, polyalkenoic acid copolymer, silica nanofiller	Acid etch for 15 s. Rinse for 10 s. Apply 2-3 consecutive coats with gentle agitation, using fully saturated applicator. Gently air thin for 5 s to evaporate solvents. Light cure for 10 s.
Prime & Bond 2.1 (DENTSPLY Industria e Comercio, Ltda.), 2-step total etch, L539734D	PENTA, UDMA, Bis-GMA, cetylaminefluoride, acetone cetylamine hydrofluoride, acetone, nanofiller (amorphous silicon dioxide, 8 nm), resin R5-62-1, T-resin, D-resin	Acid etch for 15 s. Rinse for 15 s. Apply with fully saturated applicator for 20 s. Apply brief air flow (5 s maximum). Light cure for 10 s.
OptiBond Solo (Kerr Corporation), 2-step total etch, 3648795	Bis-GMA, GDMA, GPDM, HEMA, silica, sodium hexafluorosilicate, ethanol (15%-25%)	Acid etch for 15 s. Rinse for 15 s. Apply for 15 s, using light brushing motion. Air thin for 3 s. Avoid pooling. Light cure for 20 s.
XP Bond (DENTSPLY International), 2-step total etch, 1011000650	TCB resin, PENTA, UDMA, TEGDMA, HEMA, stabilizer, ethyl-4-dimethylaminobenzoate, photoinitiator, silica, t-butanol	Acid etch for 15 s. Rinse for 15 s. Apply and wet all surfaces uniformly. Leave undisturbed for 20 s. Evaporate solvent for at least 5 s. Light cure for 10 s.
Adper Easy One (3M ESPE), 1-step self-etch, 344579	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, polyalkenoic acid copolymer, silica nanofiller, stabilizers, photoinitiator, ethanol, water	Apply for 20 s. Air thin for 5 s. Light cure for 10 s.

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; GDMA, glycerol dimethacrylate; GPDM, glycerol phosphate dimethacrylate; PENTA, dipentaerythritol penta-acrylate monophosphate; TCB, butan-1,2,3,4-tetracarboxylic acid di-2-hydroxyethylmethacrylate ester; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

study. Crown portions were sectioned with diamond disc under running water in a cutting machine rendering flat surfaces. Dentin surfaces were ground with 600-grit sandpaper for 60 seconds to obtain a standardized smear layer. Teeth were randomly shared among 10 groups (n = 5) encompassing 5 adhesive systems and 2 air-drying times. Bonding systems were also tested in a randomized manner (Random allocator, Randomness and Integrity Services, Ltd.)

The adhesive systems were applied either in accordance with the manufacturer's instructions (control) or with a lengthened solvent evaporation time: 30 seconds for Prime & Bond 2.1 and 20 seconds for the other adhesives (prolonged). The duration of the prolonged protocols was based on the number of seconds needed to achieve a significant difference during DE testing

(see Results section). Air drying was performed with a moisture- and oil-free air stream located 20 cm away from the surface and delivered under controlled air pressure. After light curing of the adhesive (LED light, Emmiter C, Schuster), composite resin blocks (Filtek Z100, 3M ESPE) were incrementally built up on the hybridized surfaces. Each increment was cured for 40 seconds (Emmitter C). A single operator performed all restorative procedures, using the adhesive systems in a randomized order.

After 24 hours' storage in distilled water 37°C, teeth were sectioned in 2 perpendicular axes (IsoMet 1000, Buehler), rendering specimens with a cross-sectional area of around 0.9 mm². Specimens were then fixed to metallic devices (ODMT03d, Odeme Dental Research) with cyanoacrylate adhesive (Pegamil, Anaerobicos

S.R.L.) and submitted to the test at a crosshead speed of 1 mm/min in a universal testing machine (EMIC 2000, EMIC Equipamentos e Sistemas de Ensaios, Ltda.) until fracture. Bond strength values were calculated in megapascals.

Fractured specimens were analyzed under 200× magnification (Discovery.V12 stereomicroscope, Carl Zeiss Microscopy) and classified as cohesive (dentin or resin), adhesive, or mixed failures. Specimens that fractured before being tested were computed as pretesting failures, to which a value of 4 MPa was assigned.²³

Degree of conversion

To allow analysis of the DC, standard aliquots of 20 µL of each adhesive system were dispensed on an acetate sheet and submitted to air drying with either the control or

Table 2. Mean (SD) degree of evaporation (%) by material and air-drying time.

Material	Air-drying time (s)					
	3	5	10	20	30	60
Adper Single Bond 2	5.9 (0.4) ^a	6.9 (1.0) ^a	7.4 (1.0) ^a	12.4 (1.4) ^{b,c}	11.6 (1.8) ^b	14.3 (1.6) ^c
XP Bond	12.5 (1.6) ^a	13.9 (0.8) ^a	17.7 (0.9) ^b	17.4 (1.3) ^{b,c}	21.3 (0.5) ^c	17.5 (2.5) ^c
Prime & Bond 2.1	64.8 (2.1) ^a	68.7 (2.9) ^{a,b}	68.2 (1.3) ^{a,b}	68.2 (1.5) ^{a,b}	71.4 (5.0) ^{b,c}	75.9 (0.6) ^c
OptiBond Solo	2.9 (1.2) ^a	4.0 (0.4) ^{a,b}	4.6 (0.4) ^{b,c}	5.9 (0.7) ^c	5.8 (0.8) ^c	5.8 (1.0) ^c
Adper Easy One	2.8 (0.4) ^a	2.9 (0.5) ^a	4.6 (0.6) ^{a,b}	5.6 (1.1) ^{b,c}	6.4 (1.4) ^{b,c}	7.2 (2.3) ^c

Means with different superscript letters within each row are significantly different ($P < 0.05$; 1-way analysis of variance).

prolonged protocol, as previously described. Immediately after it was apportioned, the material was placed over the zinc selenide crystal plate of an attenuated total reflection device (MIRacle, PIKE Technologies) and submitted to FTIR (Vertex 70, Bruker Corporation) to evaluate DC before and after light curing in the medium infrared working range. The spectral range was 600–4000 cm^{-1} with 32 scans at 4 cm^{-1} .

The percentage of nonreacted double bonds was determined by the ratio between aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) absorptions before and after light curing. The DC was determined by subtracting the residual percentage of aliphatic bonds from 100%. Three specimens were evaluated for each material and condition (control and prolonged air drying).

Statistical analysis

The DE data were submitted to 1-way analysis of variance and a Tukey test ($\alpha = 0.05$). As the goal of the study was to evaluate the effect of air-drying time on the performance of the adhesive systems, the different materials were not compared among each other and ranked. Instead, statistical analysis was performed for each material by air-drying protocol. Accordingly, DC and μTBS data were submitted to a t test ($\alpha = 0.05$) with 1 factor (air-drying time) for each adhesive system. The correlation between μTBS and DC was determined by a Spearman test.

Results

Degree of evaporation

Mean DE values are shown in Table 2. The air-drying time of 20 seconds produced

Table 3. Mean (SD) microtensile bond strength (μTBS ; in MPa) and No. of pretesting failures/test specimens (PF/TS) by adhesive system and air-drying protocol.

Material	Control protocol		Prolonged protocol		P value
	μTBS	PF/TS	μTBS	PF/TS	
Adper Single Bond 2	48.27 (10.67)	0/43	39.85 (4.47)	0/42	0.164
XP Bond	47.61 (3.67)	0/41	56.42 (5.72)	0/43	0.027 ^a
Prime & Bond 2.1	25.58 (8.61)	14/28	35.75 (11.64)	6/32	0.283
OptiBond	52.39 (8.10)	0/39	49.22 (5.04)	0/37	0.486
Adper Easy One	11.28 (5.16)	23/43	23.56 (7.74)	16/27	0.022 ^a

^aStatistically significant difference in bond strengths between protocols ($P < 0.05$).

significantly higher DE than did the time suggested by the manufacturers (control) for all adhesive systems except Prime & Bond 2.1, which first showed a significant rise in DE with 30 seconds of air drying. In addition, 60 seconds of air drying produced mean DE values similar to those at 20 seconds for all materials except Prime & Bond 2.1, for which the 60-second value was similar to the DE at 30 seconds.

Microtensile bond strength

Mean μTBS values for experimental groups are shown in Table 3. Despite the tendency toward higher bond strength values with prolonged air-drying conditions, statistically significant differences were only found for XP Bond and Adper Easy One ($P < 0.05$). Pretesting failures occurred only with Prime & Bond 2.1 and Adper Easy One and were more frequent in the control protocol than the prolonged air-drying protocol. A mixed failure mode was predominant in all experimental conditions.

Degree of conversion

Table 4 shows mean DC values for the experimental groups. DC was not affected by air-drying time. No statistically significant correlation was found between DC and μTBS values.

Discussion

The results of the present study clearly show that the solvent air-drying time suggested by the manufacturers for solvent evaporation produced less solvent evaporation than prolonged air-drying times (20 seconds for water/ethanol/*t*-butanol-based systems and 30 seconds for the acetone-based system). As expected, acetone produced significantly higher DE than other materials even at the first measurement, so it took slightly longer to achieve a significant difference with the prolonged drying times (30 seconds). The times needed to achieve significant change in DE were considered to be the prolonged protocols for μTBS and

DC evaluations. Longer times than those do not seem to be necessary with the methodology applied, as the evaporation degree obtained with 60 seconds was similar to that obtained with 20 and 30 seconds.

The prolonged air-drying time significantly increased the μ TBS values of Adper Easy One and XP Bond, but not those of the other materials, so the first null hypothesis was partially accepted. Thus, the effectiveness of increasing air-drying time to improve solvent evaporation is material dependent due to differences in the composition and application strategies of each system.^{2,6,8,24,25} One-step self-etching systems seem to be more sensitive to variations in the duration of airflow because of compositional aspects, such as type and amount of organic solvent, presence and amount of water, and type and hydrophobicity of monomers. Moreover other components, such as comonomers, initiators, and filler particles, can influence solvent evaporation.²⁶ The amount of water in those systems would demand a longer airflow to allow solvent evaporation.²⁷⁻²⁹ However, the optimal duration, pressure, distance, and temperature of airflow for various types of adhesive systems are not fully elucidated, and the manufacturer's instructions do not discuss these aspects.^{4,5,12,17,24,29-31}

When applied in accordance with the manufacturer's instructions, Adper Easy One produced extremely low μ TBS values compared to those commonly reported for total-etch and even self-etching systems.^{9,11,12,29,32} However, significantly higher values were found with the prolonged air-drying protocol, which is probably related to improved solvent evaporation and consequently a less defective adhesive interface.^{2,4,5,12,14,30,31} The improvement in μ TBS also could be simply due to time; that is, prolonged airflow could have provided more time for monomer diffusion, leading to better interaction between acidic monomers and the substrate.²⁷ The improvement could even be the result of a combination of both phenomena. The reduction in the number of pretesting failures in this group, compared to the control group, can be considered additional evidence that prolonged air drying produced interfaces that were more resistant.

Table 4. Mean (SD) degree of conversion (%) by air-drying protocol and adhesive system.

Material	Air-drying protocol		P value
	Control	Prolonged	
Adper Single Bond 2	59.59 (6.51)	60.48 (1.99)	0.842
XP Bond	43.38 (5.99)	39.80 (8.48)	0.593
Prime & Bond 2.1	68.06 (2.08)	54.72 (5.48)	0.059
OptiBond Solo	61.95 (4.15)	71.26 (3.16)	0.054
Adper Easy One	51.27 (4.24)	62.15 (5.60)	0.075

Prolonged air drying also improved μ TBS values for XP Bond, which has been observed in other studies.^{6,33,34} Several researchers have proposed modifications in total-etch adhesive systems to enhance solvent evaporation, thereby producing adhesive interfaces that are mechanically stronger and more resistant to degradation.^{6,20,35-39}

Despite the concern expressed by some researchers that prolonged or higher pressure airflow will produce extremely thin adhesive layers, that result does not seem to have occurred in the present study, since μ TBS values were not reduced significantly by prolonged air drying.^{12,17,29} Results from studies that evaluate the influence of air drying on adhesive systems must be compared cautiously, since several different parameters can be considered, including time, pressure, distance, and temperature of airflow. In addition, different materials may produce disparate results under the same protocol, as observed in the present study.^{2,6,11,12,17,29}

Despite existing evidence suggesting that greater solvent evaporation promotes a greater degree of conversion and greater bond strength, in the present study no such correlation (DC \times μ TBS) was observed.^{2,9,15,29,30,38,40} The DC of the evaluated adhesive systems was not influenced by the air-drying time; this result partly agrees with previous studies, in which no increase in the DC of a total-etch system was observed even after complete solvent removal.^{9,11,41} On the other hand, the air-drying time used in the present study also seemed short enough that it did not negatively influence DC by oxygen polymerization inhibition or viscosity reduction.^{13,42,43}

It is noteworthy that the time of 20 seconds, besides promoting adequate solvent evaporation, is also clinically acceptable.

Conclusion

The results of the present study indicated that the initial bond strength of adhesive systems can be improved by prolonging the air-drying time. However, this influence is material dependent, so manufacturers should revise their instructions for their own materials accordingly. Long-term evaluations are needed to assess the influence of prolonged air drying on the longevity of the bonds achieved.

Author information

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Manufacturers

- Anaerobicos S.R.L, Martinez, Argentina
54.11.4848.5555, www.anaerobicos.com
- Bruker Corporation, Billerica, MA
978.439.9989, www.bruker.com
- Buehler, Lake Bluff, IL
800.283.4537, www.buehler.com
- Carl Zeiss Microscopy, Dublin, CA
800.442.4020, www.zeiss.com
- DENTSPLY Industria e Comercio, Ltda., Petropolis, Brazil
55.0800.721.1200, www.dentsply.com.br
- DENTSPLY International, York, PA
800.877.0020, www.dentsply.com
- EMIC Equipamentos e Sistemas de Ensaio, Ltda., Sao Jose dos Pinhais, Brazil
55.42.3035.9400, www.emic.com.br
- Kacil Industria e Comercio, Ltda., Recife, Brazil
55.81.3268.5922, www.kacil.com.br
- Kerr Corporation, Orange, CA
800.537.7123, www.kerrdental.com
- Odeme Dental Research, Luzerna, Brazil
55.49.3521.4016, odeme.com.br
- PIKE Technologies, Madison, WI
608.274.2721, www.piketech.com
- Randomness and Integrity Services, Ltd., Dublin, Ireland
353.87.417.5108, www.random.org
- Robert Bosch, LLC, Farmington Hills, MI
917.421.7209, www.bosch.us
- Schuster, Santa Maria, Brazil
55.55.3222.2738, www.schuster.ind.br
- 3M ESPE, St. Paul, MN
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