

# The effect of storage and air-drying on solvent evaporation of three different one-bottle adhesive systems: a comparative in-vitro study

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## **Abstract:**

**Objectives:** The aim of this study is to clarify the relationship between the duration of storage and air-drying of one-bottle adhesive systems containing different solvents and the evaporation degree (ED) by gravimetric method.

**Materials and Methods:** The evaporation degree (ED) of XP Bond (Dentsply) which contains tertiary butanol and water, Prime and Bond NT (Dentsply) which contains acetone and water and Adper Single Bond (3M ESPE) which contains ethanol and water, was determined by storage respectively, 5, 10, 30, 60, 120 and 180 minutes and also without air-drying and after, respectively, 5- and 10-seconds air-drying using a gravimetric method.

**Results:** The evaporation degree (ED) increased with extension of air-drying time. Among the adhesives tested, Prime and Bond NT which contains acetone and water showed the largest ED, followed by Adper Single Bond which contains ethanol and water and XP Bond which contains tertiary butanol and water in this order.

**Conclusion:** Air-drying of one-bottle adhesive systems had a significant effect on the degree of solvent evaporation (ED). It is therefore beneficial to remove solvents of the one-bottle adhesive

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systems as much as possible by thorough, strong air-drying in order to achieve a strong adhesive layer at the interface.

**Keywords:** Dentin bonding agents, solvents, evaporation, storage, drying.

**Introduction:**

In an attempt to reduce clinical steps and save time, manufacturers simplified etch-and-rinse adhesives by mixing the components of the primer and the bonding resin into a single solution.<sup>[1]</sup> Such adhesives combine both hydrophilic and hydrophobic monomers which are blended, with a relatively high concentration of solvent, required to keep them in solution.<sup>[1,2]</sup>

It has been reported that one-bottle adhesive systems are more permeable to water from the oral environment and from the underlying bonded dentin, leading to faster degradation of resin–dentin bonds.<sup>[1,3]</sup>

The high quality of the hybridization process depends on optimal monomer infiltration between the collagen fibrils of the demineralized matrix and the removal of as much water and organic solvents as possible from the surface prior to curing.<sup>[4]</sup> Incomplete evaporation of solvents results in dilution, poor polymerization, phase separation of the resin components and after light-curing of the adhesives, residual water or solvents may become pathways for water movement within the hybrid or resin layers.<sup>[5]</sup>

Compressed air has been used to accelerate solvent and water evaporation, ensuring better conditions for resin polymerization, which relates to the durability of the resin-dentin bonds in the wet bonding technique.<sup>[4]</sup>

The solvent should indeed be removed to a sufficient degree during the short application time and subsequent air-drying, otherwise bond strength and stability decreases.<sup>[2]</sup> Thus, clinicians must be aware of the volatile characteristics and composition of adhesive systems to achieve effective bonding with one-bottle adhesive systems.<sup>[4]</sup>

Different solvents presented in the primer component or in simplified bonding agents are responsible for either carrying excess water out or infiltrating resin monomers into interfibrillar dentin.<sup>[6]</sup> As solvent is necessary to provide a proper infiltration of the resin monomer into demineralized collagen matrix, the bonding process still depends on its capacity.<sup>[7-9]</sup> Benefits offered by solvents rely on their properties of improving substrate wetting, aiding to impede the collagen fibrils collapse or to stiffen them.<sup>[8]</sup> However, solvents must be eliminated after having completed

their function because it has been demonstrated that residual solvent can lead to deterioration of the adhesive interface<sup>[7]</sup> by interfering with resin polymerization<sup>[6,8,10]</sup> and decreasing mechanical properties.<sup>[9-14]</sup>

Water, ethanol and acetone are basically the main solvents in commercial formulations.<sup>[8,11-13,15]</sup> A combination of some physical and chemical factors, namely vapour pressure, molar fractions, molar weight and solubility, is considered to determine the evaporation capacity of different solvents.<sup>[13,16-19]</sup> Although non-solvated resins may establish a more stable and less fragile adhesion to dentin compared to simplified solvated products, bonding durability to this substrate is still a clinical challenge.<sup>[18,20]</sup>

Previous studies have shown differences in the capacity of either experimental solvents or commercial formulations to evaporate<sup>[19]</sup>, but no comparative study is currently available. Such an investigation would be interesting because commercial formulations contain ingredients like initiators, co-monomers and/or nanoparticles which can affect solvent evaporation rate. Although previous reports have assessed experimental primers based mainly on 2-hydroxyethyl methacrylate (HEMA) and solvent, their performance might be different from commercial formulations since the latter present other ingredients that modify evaporation rate.

Gravimetric analysis, rely on the final determination of weight as a means of quantifying an analyte. Since weight can be measured with greater accuracy than almost any other fundamental property, gravimetric analysis is potentially one of the most accurate techniques of analytical methods available. These methods are among the oldest of analytical techniques, and they may be lengthy and tedious. Moreover there are many studies on evaporation degree of acetone and ethanol based adhesive systems but not enough literature on evaporation degree of tertiary butanol based adhesive system. The Null hypothesis of this study was that the time of storage and air-drying does not influence the Evaporation degree (ED) of one-bottle adhesive systems.

**Materials and methods:**

The following three one-bottle adhesive systems will be used: XP Bond (Dentsply) which contains tertiary butanol and water, Prime and Bond NT (Dentsply) which contains acetone and water and Adper Single Bond (3M ESPE) which contains ethanol and water.

Successively, the evaporation of adhesive components by storage in an oven at 37 °C and the

evaporation of adhesive components by air-drying will be determined using a gravimetric method.

#### Evaporation of adhesive components by storage in an oven at 37 °C

A piece of cover glass was weighed by a precision balance ( $X$ =weight of the cover glass), after which a drop of each one-bottle adhesive system was dispensed on the cover glass. The cover glass with the adhesive drop was weighed again ( $A+X$ =weight of adhesive drop plus the weight of the cover glass), after which it was placed in a dark oven at 37 °C to allow the solvent to evaporate. After a period of 5, 10, 30, 60, 120 and 180 minutes, the cover glass with the adhesive drop was weighed again in order to determine the weight loss due to evaporation of the volatile components of the one-bottle adhesive system with time ( $B_{\text{time}} + X$ =weight of one-bottle adhesive system drop at a specific time, plus the weight of the cover glass). Further weight measurements were carried out every 24 hours until the weight remained constant ( $C_{\text{storage}} + X$ =weight of one-bottle adhesive systems drop that remained constant after final storage, plus weight of cover glass).

The evaporation degree ( $ED_{\text{storage}}$  in wt%) of the one-bottle adhesive systems by storage in the oven was then calculated as follows: Takatsumi et al<sup>[2]</sup>

$$ED_{\text{storage}} (\text{wt}\%) = \frac{A - B_{\text{time}}}{A} \times 100$$

With  $A$  is the initial weight of the one-bottle adhesive drop and  $B_{\text{time}}$  is the weight of the one-bottle adhesive drop at each storage time. The respective weight of the adhesive drop at each time was calculated by subtracting the weight of the cover glass ( $X$ ) from the measured weight of the cover glass plus the adhesive drop at each time. As the weight  $C_{\text{storage}}$  actually represents the weight of the 'nonvolatile part' (NVP) of the one-bottle adhesive system, the percentage of NVP (in wt%) in each one-bottle adhesive system was calculated as follows:

$$NVP\% (\text{wt}\%) = \frac{C_{\text{storage}}}{A} \times 100$$

Correspondingly, the percentage of the 'volatile part' (VP) of the one-bottle adhesive system was obtained as follows:

$$VP\% (\text{wt}\%) = 100 - NVP\%$$

Each measurement was repeated five times for each one-bottle adhesive system.

#### Evaporation of adhesive components by air-drying

To evaluate the evaporation of adhesive components by air-drying, each adhesive resin applied on a cover glass was weighed after air-drying in simulation of the clinical application procedure. A

cover glass ( $X$ ) was weighed before the adhesive was applied. To standardize the application area as much as possible, the cover glass was placed on a acrylic block (25mm×25mm×15mm), precisely above a circle (radius of 16mm) drawn on the acrylic block. To keep the temperature of the cover glass constant, the glass block was stored in a 37 °C dry oven during the clinic-simulated application procedure. The adhesive was deposited on the glass within the circle using an applicator tip, and either not air-dried (0 second) or subsequently air-dried by a chip blower during 5 and 10 seconds, respectively. Immediately after air-drying, the weight of the cover glass plus the air-dried adhesive was measured ( $X + B_{\text{air-drying}}$ ). From this weight, the weight of solely the cover glass ( $X$ ) was subtracted to obtain the weight of the one-bottle adhesive system after air-drying ( $B_{\text{air-drying}}$ ). Finally, the cover glass with the air-dried adhesive was stored in the dark oven at 37 °C, with weight measurements every 24 hours, until the weight became constant ( $X + C_{\text{air-drying}}$ ). As mentioned above, the 'non-volatile part' (NVP) of the one-bottle adhesive system ( $C_{\text{air-drying}}$ ) was calculated by subtracting the weight of the cover glass ( $X$ ) from the constant ( $X + C_{\text{air-drying}}$ ). The percentage of NVP (wt%) in the one-bottle adhesive system could then be calculated as follows:

$$NVP\% (\text{wt}\%) = \frac{C_{\text{air-drying}}}{A_{\text{air-drying}}} \times 100$$

With  $A_{\text{air-drying}}$  the initial weight of one-bottle adhesive system glass and  $C_{\text{air-drying}}$  the final weight of the one-bottle adhesive system after storage.

As the NVP% was also calculated before, and is a constant, the initial weight of the one-bottle adhesive system applied on the cover glass ( $A_{\text{air-drying}}$ ) could be calculated as follows:

$$A_{\text{air-drying}} = \frac{C_{\text{air-drying}} \times 100}{NVP(\%)}$$

The evaporation degree by air-drying of the one-bottle adhesive system could then be obtained, as follows:

$$ED_{\text{air-drying}} (\text{wt}\%) = \frac{A_{\text{air-drying}} - B_{\text{air-drying}}}{A_{\text{air-drying}}} \times 100$$

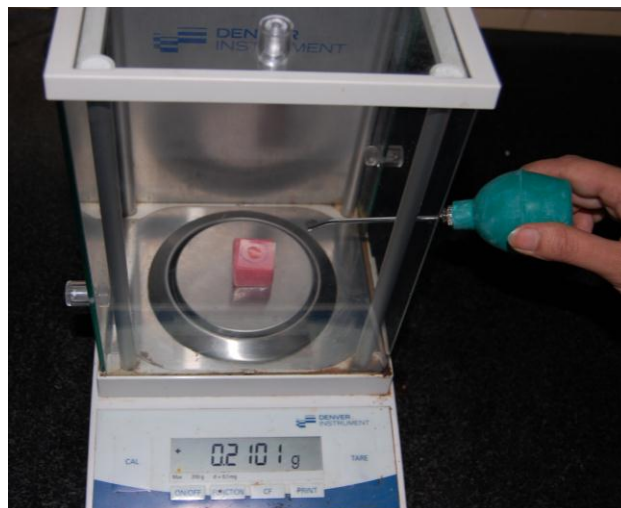
With  $A_{\text{air-drying}}$  the initial weight of the one-bottle adhesive system applied on the cover glass and  $B_{\text{air-drying}}$  the weight of the one-bottle adhesive system after air-drying.

This series of measurements was repeated 10 times for each one-bottle adhesive system.

**Fig. 1:** Acrylic Block for standardizing the application area



**Fig. 2:** Air-drying of the adhesive drop on the acrylic block



**Table 1: Composition and Manufacturer of the adhesive system used in this study**

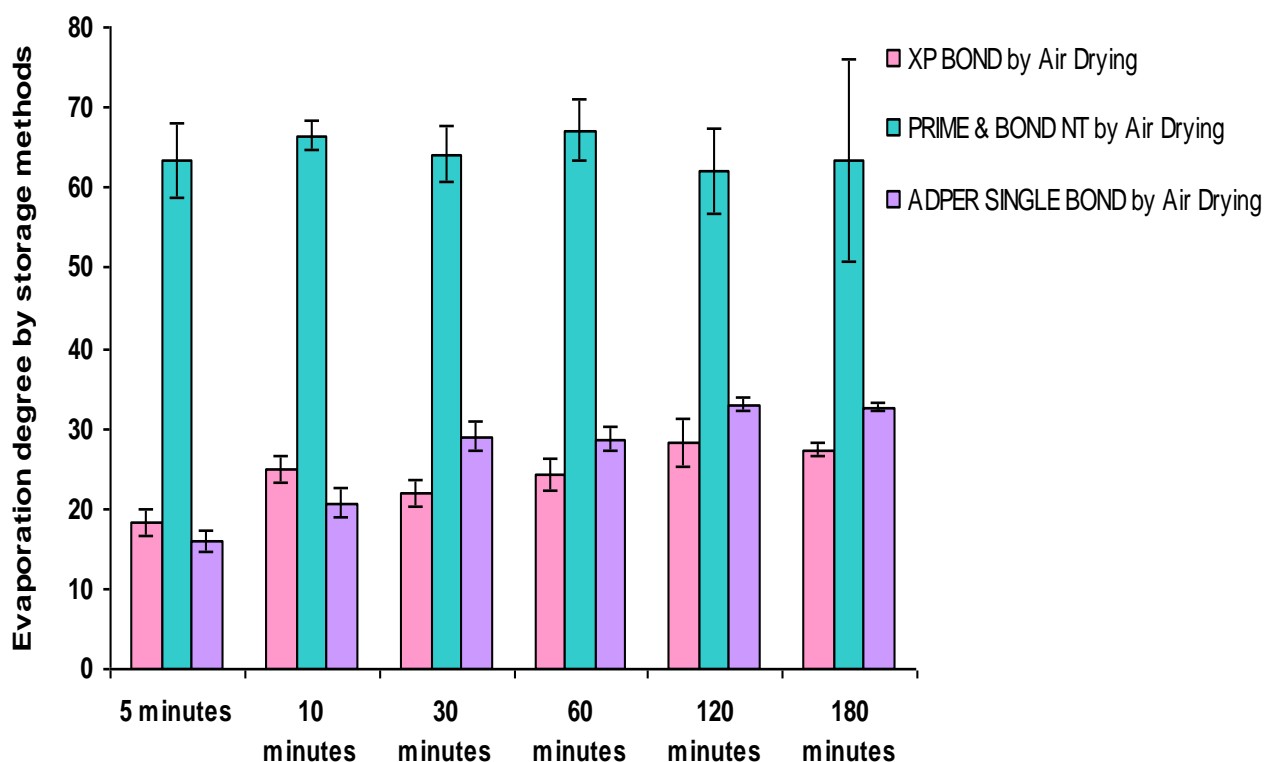
Adhesive system	Composition	Manufacturer
XP Bond	Phosphoric acid modified acrylate resin (PENTA), Carboxylic acid modified dimethacrylate (TCB resin), Urethane dimethacrylate (UDMA), Triethyleneglycol dimethacrylate (TEGDMA), 2-hydroxyethylmethacrylate (HEMA), Nanofiller, Camphorquinone, Ethyl-4-dimethylaminobenzoate (DMABE), Butylated Benzenediol, Tert-Butanol	Dentsply Detrey, Konstanz, Germany
Prime and Bond NT	Phosphoric acid modified acrylate resin (PENTA), di- and tri- methacrylate, cetylamine hydrofluoride, urethane di-methacrylate resin, silica colloidal and acetone	Dentsply Detrey, Konstanz, Germany
Adper Single Bond	2-hydroxyethylmethacrylate (HEMA), Bisphenol A glycidyl methacrylate (Bis-GMA), water, Polyacrylic acid, Coloidal Silica and ethanol	3M ESPE, St Paul, MN, USA

**Table 2: Effect of duration of storage and its effect on evaporation degree (ED) of one bottle adhesive systems containing different solvents by gravimetric method.**

Evaporation degree b storage methods	Methods			P value
	XP BOND by Air Drying	PRIME & BOND N' by Air Drying	ADPER SINGLE BOND by Air Drying	
5 minutes	18.32±1.68	63.27±4.66	16.01±1.33	<0.001**
10 minutes	24.95±1.70	66.43±1.80	20.72±1.82	<0.001**
30 minutes	21.96±1.71	64.21±3.42	28.92±1.82	<0.001**
60 minutes	24.21±2.02	67.21±3.87	28.68±1.37	<0.001**
120 minutes	28.21±2.84	61.98±5.30	33.00±0.76	<0.001**
180 minutes	27.29±0.77	63.37±12.68	32.61±0.51	<0.001**
Significance	<0.001**	0.770	<0.001**	-
R <sup>2</sup>	89.1%	30.8%	97.2%	

Significance is assessed at 5 % level of significance. Analysis of variance (ANOVA) has been used to find the significance of study parameters between three or more groups

**Fig. 3: Evaporation degree of different solvents by storage**

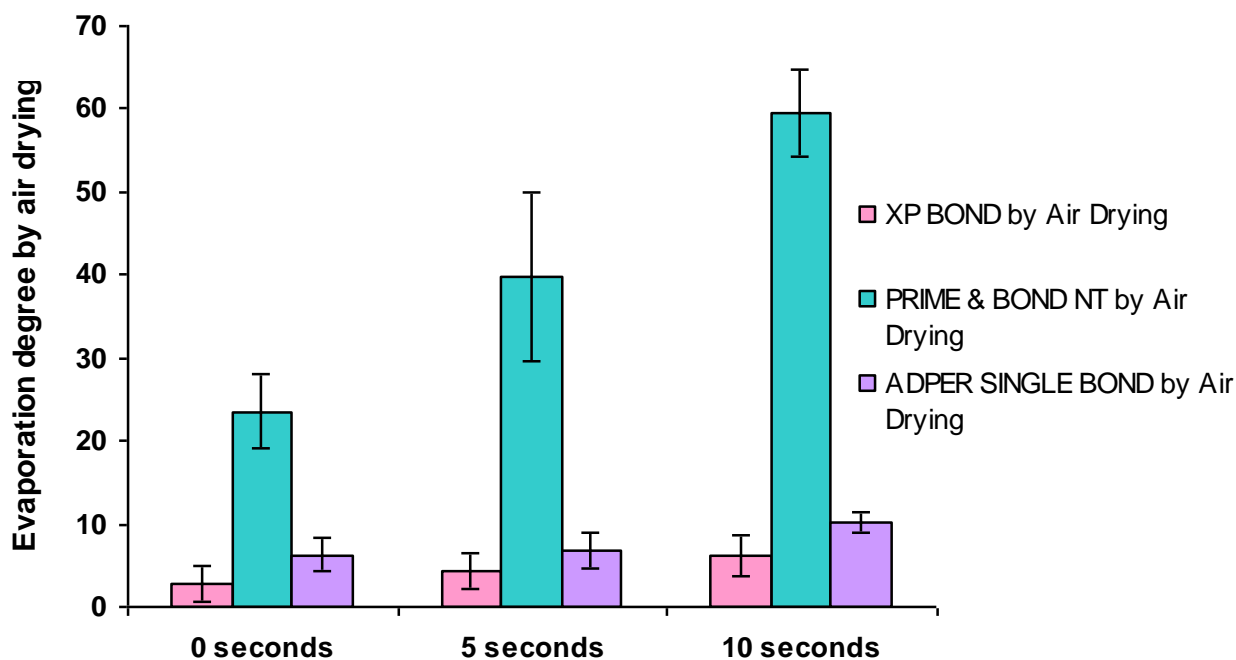


**Table 3: Effect of duration of air drying and its effect on evaporation degree (ED) of one bottle adhesive systems containing different solvents by gravimetric method.**

Evaporation degree by air drying	Methods			P value
	XP BOND by Air Drying	PRIME & BOND NT by Air Drying	ADPER SINGLE BOND by Air Drying	
0 seconds	2.74±2.07	23.54±4.38	6.29±1.98	<0.001**
5 seconds	4.29±2.21	39.86±10.16	6.80±2.16	<0.001**
10 seconds	6.13±2.53	59.51±5.35	10.23±1.14	<0.001**
Significance	0.009**	<0.001**	<0.001**	-
R <sup>2</sup>	54.1%	90.9%	99.4%	-

Significance is assessed at 5 % level of significance. Analysis of variance (ANOVA) has been used to find the significance of study parameters between three or more groups

**Fig. 4: Evaporation degree of different solvents by air-drying**



#### Method of Statistical Analysis

Descriptive statistical software namely SAS 9.2, SPSS 15.0, Stata 10.1, MedCalc 9.0.1 and Systat 12.0 were used for the analysis of the data in the present study. Results on continuous measurements are

presented on Mean SD (Min-Max) and results on categorical measurements are presented in Number (%). Significance is assessed at 5 % level of significance. Analysis of variance (ANOVA) has been used to find the significance of study parameters between three or

more groups of patients, Ridge Analysis has been performed for maximizing the Evaporation degree, Post-hoc Tukey test has been used to find the pairwise significance.

### Results:

#### Evaporation of adhesive components by storage in an oven at 37 °C

The ED<sub>storage</sub> of Prime and Bond NT abruptly increased in the first 5-minutes time interval, after which the increase in ED<sub>storage</sub> gradually decreased. Though the ED<sub>storage</sub> of Adper Single Bond and XP Bond also increased in the first 5 minutes, this increase was much more gradual than that of Prime and Bond NT

The weight of the all the adhesives became constant after 24 hours. Therefore, the weights at 24hours were recorded as the C<sub>storage</sub> of all the adhesive systems. The NVP% of XP Bond, Prime and Bond NT and Adper Single Bond were 56.71, 33.79 and 66.46 wt%. Hence, the VP% of XP Bond, Prime and Bond NT and Adper Single Bond were 43.28, 66.20 and 33.53 wt%, respectively.

Evaporation of adhesive components by air-drying. Longer air-drying significantly increased the evaporation of solvents. The largest ED<sub>air-drying</sub> was recorded for Prime and Bond NT, followed by Adper Single Bond and XP Bond in this order. Although a 10-seconds air-drying time seems sufficient for Prime and Bond NT, the 'volatile part' (VP) of the other two adhesives could not be completely eliminated with the help of this 10-seconds air-drying.

#### Discussion:

Solvents are substances capable of dissolving or dispersing one or more substances.<sup>[21]</sup> They are responsible for dilution of resin monomers, improving its diffusion throughout the demineralized matrix and represent an important role in removing moisture from the substrate during the evaporation.<sup>[22]</sup> In adhesive systems, water (boiling temperature 100° C), ethanol (78.3° C) and acetone (56-58° C) are solvents frequently used.<sup>[23]</sup> Recently a new solvent named tertiary butanol has also been used.

Due to the role attributed to solvents in the adhesive protocol, their evaporation capacity from different experimental products has been investigated.<sup>[24]</sup> There are also few studies with commercial adhesive systems, such as those of Abate et al.<sup>[11]</sup> and Lima et al.<sup>[13]</sup>, who have monitored this property directly from the product vials. In clinical practice, adhesive system vials are frequently left open

during the restorative procedure, which facilitates solvent volatilization. As the solvent evaporates from the open vial, the viscosity of the adhesive increases and the amount of residual vehicle to carry the resin monomer within the demineralized dentin matrix and involve collagen fibrils may not be sufficient, compromising the quality of the adhesive protocol.<sup>[13,25]</sup> On the other hand, solvent evaporation after application to dentin is extremely important because failure to remove excess solvent by gentle air drying may contribute to the degradation of the adhesive interface over time.<sup>[7-14]</sup>

In the present study, the tested products were grouped according to solvent type to allow better comparison. Based on the results of the proposed experimental design, it was observed that the acetone-based materials presented a greater evaporation capacity compared to all other products. These results are in accordance with those of Yiu et al.<sup>[20]</sup> after examining the solvent and water retention in dental adhesive blends after evaporation. Commercial formulations presented less solvent evaporation compared to the acetone-based products. In this study, solvent evaporation was mainly responsible for product mass loss.

Result in our study indicated that when adhesive is stored in an oven at 37° C, all the one-bottle adhesive systems lost weight most rapidly during the first 5 minutes of storage, after which the degree of weight loss gradually decreased. This decrease in weight loss due to evaporation of the solvent corresponded to a visual increase in viscosity of the one-bottle adhesive systems. This weight loss at the first 5 minutes time interval was clearly least pronounced for Adper Single Bond which contains ethanol followed by XP Bond which contains tertiary butanol and Prime and Bond NT which contains acetone, and must be explained by the difference in solvents between them. Similar results were reported by Takatsumi et al<sup>[2]</sup> and Fabio et al<sup>[22]</sup> in their studies.

The use of air-drying after the application of the adhesive improves the evaporation of solvent and water, reducing the thickness of adhesive layer and becoming more uniform.<sup>[26]</sup> The time of air-drying application should be observed according to the manufacturer's instructions and it can vary between 5 and 10 seconds. The solvent remainder in the adhesive can compromise its polymerization due to dilution of monomers and results in the permeability of the adhesive interface.<sup>[27]</sup> A similar result was seen with acetone based Prime and Bond NT adhesive system in this study.

The result observed in this study when different adhesives were evaluated, Prime and Bond NT revealed the largest ED<sub>air-drying</sub>, followed by Adper Single Bond and XP Bond, in this order. The largest ED of Prime and Bond NT must be ascribed to the longest application time (per manufacturer's instructions) and the largest percentage of volatile part (VP%) of the adhesive. On the other hand, the smallest ED<sub>air-drying</sub> in XP Bond must again be ascribed to the difference in volatile components between the one-bottle adhesive systems.

It is clear that other components of the adhesive system have the potential to limit spontaneous evaporation of the solvent. Pashley et al.<sup>[19]</sup> have claimed for the effect of monomer (HEMA) as a solute and as solvents evaporate over time, HEMA concentration increases, which lowered the pressure vapour of the experimental bonding mixture in the present study. Although the ED<sub>air-drying</sub> of XP Bond and Adper Single Bond after air-drying was the smallest among the three one-bottle adhesive systems tested in this study, especially after 10-seconds air-drying. This must probably be explained by the addition of HEMA to the adhesive formulation, which functions in part as a solvent and allows a much lower amount of actual solvents like water and ethanol (in case of Adper Single Bond) and water and tertiary butanol (in case of XP Bond) than the amount of solvents (water and acetone in case of Prime and Bond NT) needed in HEMA-free adhesives. The results of the present investigation indicate that HEMA is an adhesive system component that potentially interferes with solvent evaporation.<sup>[22]</sup>

Ideally, solvents and water (from the moist demineralized dentin) should be completely eliminated from the dentin surface before light-curing. On this basis, there is often an air-drying process recommended as part of the clinical regimen for dentin bonding while using adhesives that contain solvents. However, the removal of solvents with a simple air-drying stream is not an easy task to be accomplished under clinical situations. As water/solvent evaporates from the adhesive, the monomer density is found to increase sharply, creating a monomer concentration gradient which acts as a barrier for further solvent evaporation and thus, reduces the ability of water and solvents to evaporate from the adhesive.<sup>[19]</sup>

On the other hand, the presence of residual solvent might have an adverse effect on the performance of the resin–dentin bonds. It was already demonstrated that high solvent concentration within the adhesive polymer prior to light-curing prevents the

attainment of a high cross-linking polymer inside the hybrid layer<sup>[28,29]</sup> and leads to pores in the interfacial layers,<sup>[30]</sup> affecting the overall performance of resin–dentin bonds.<sup>[31]</sup>

There is a solvent concentration at which maximum conversion is reached; more or less solvent than this amount would decrease monomer conversion,<sup>[24]</sup> and this seems to be related to the viscosity of the adhesive film.<sup>[32]</sup> It is likely that non-solvated versions of adhesive systems might present a lower degree of conversion due to the increased viscosity of the solution. An increased viscosity restricts the mobility of reactive components during polymerization.<sup>[33]</sup> On the other extreme, excess of solvents would cause a dilution of the components preventing the collision of reactive components. In the present investigation no attempt was made to determine the amount of residual solvent in the adhesive films after using the two different modes of air-drying and this deserves further investigations.

Cardoso et al.<sup>[34]</sup>, used two adhesive systems with different solvent and examined the effect of the time of application increasing the microtensile bond strength and correlating with the rate of adhesive evaporation. It was verified that when increasing the application time, the bond strength presented better results and the time of application depends on the kind of solvent used in the adhesive system. This needs to be further investigated in the near future with the present study.

In addition to the properties of the main solvent and other components of each adhesive system, other factors such as cavity configuration and drying time can also influence solvent evaporation from adhesive systems. A recent study<sup>[21]</sup> has shown the effect of cavity configuration. Even when long air-drying was employed, there was evidence of ineffective drying and pooling of adhesive when applied in narrow Class I cavities, which resulted in solvent retention. This needs to be further investigated.

To show the importance of correct application of adhesive systems,<sup>[35]</sup> tested in vitro the effect of errors of primers application in dentin permeability. It was verified that in order to obtain an adequate sealing of dentinal tubules, it is necessary to follow the manufacturer's instructions, in accordance with Peutzfeldt & Asmussem<sup>[21]</sup> that demonstrated to reach a satisfactory result: the use of adhesive systems must be made as the manufacturer advice.

The kind of solvent present in adhesive system can be responsible for determining how humidity should be the substrate. It is important to verify what kind of solvent



is present in the adhesive system formulation, for surface wet pattern to be adjusted and thus provide a better performance of the adhesive system. However, besides the solvent used, monomer, initiator and other components also must be analysed.<sup>[27]</sup>

#### Conclusion:

Air-drying of one-bottle adhesive systems had a significant effect on the degree of solvent evaporation (ED). Solvent evaporation should be facilitated as spontaneous evaporation was not adequate to any tested material over storage time. Acetone and water based Prime and Bond NT (Dentsply) had better evaporation degree (ED) than that of tertiary butanol and water based XP Bond (Dentsply) or ethanol and water based Adper Single Bond (3M ESPE)<sup>®</sup> after 10 seconds of air-drying. It is therefore beneficial to remove solvents of the one-bottle adhesive systems as much as possible by thorough, strong air-drying in order to achieve a strong adhesive layer at the interface.

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