Efeito da Temperatura de Evaporação do Solvente na Longevidade da Adesão de Adesivos Autocondicionantes à Dentina

Effect of Solvent Evaporation Temperature on Bonding Longevity of All-In-One Adhesives to Dentin

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Resumo

Avaliou-se a durabilidade da adesão de adesivos autocondicionantes à dentina após evaporação dos solventes com jato de ar frio e quente. Os sistemas adesivos *Clearfil 3S Bond* e *OptiBond All-In-One* foram aplicados em superfícies planas de dentina, a evaporação dos solventes feita com ar quente (60±2 ºC) ou ar frio (20±2 ºC), seguindo-se fotoativação (600mW/cm^2 por 10 s) e restauração com compósito (3 x 2 mm). Após armazenagem em água destilada (24 horas / 37 ºC), as amostras foram seccionadas para obtenção de paralelogramos de 0,8 mm^2 para testar em tração (0,5mm/min) em 24 horas e 6 meses. Dois paralelogramos de cada grupo experimental foram preparados para observar a nanoinfiltração na interface com a dentina. Os valores médios da resistência adesiva (em MPa) de cada grupo foram tratados por Análise de Variância de três fatores e teste de Tukey (5%), sendo o dente a unidade experimental. Foi observada maior adesão após evaporação dos solventes com ar quente (p=0.000) no tempo de 24 horas de armazenagem (0.003). Não houve diferenças para o tipo de adesivo (p=0.343) e nem para a interação adesivo X tempo X temperatura de evaporação do solvente (p>0,05). Concluiu-se que a durabilidade da adesão foi influenciada pela temperatura de evaporação do jato de ar.

Palavras-chave: Solventes. Dentina. Adesivos.

Abstract

Objective: To evaluate the bonding longevity of one-step self-etch adhesive systems to dentin, after solvent evaporation with warm or cold air-stream. Material and methods: Clearfil 3S Bond and OptiBond All-In-One adhesives were applied on flat dentin surfaces, solvent evaporation was performed with a warm (60±2 ºC) or cold air (20±2 ºC), the surfaces were light cured (600mW/cm^2 during 10 s), and blocks of composite resin were built (3 x 2mm). After storage in distilled water (24-hour at 37 ºC), the samples were sectioned into 0.8 mm^2 sticks and tested in tensile (0.5 mm/min) at 24-hour and 6-month periods. Two sticks from each experimental group were prepared for nanoleakage observation of the bond interface. The mean bond strength values of experimental groups (in MPa) were subjected to a three- way Analysis of Variance and post-hoc Tukey´s test (5%), using tooth as the experimental unit. Results: Higher bond strength was observed with warm air (p=0.000) for solvent evaporation, and 24 hour of water storage (0.003. No significant differences were observed for both the adhesive systems (p=0.343) and interactions. Conclusion: The bonding durability was influenced by the air temperature for solvent evaporation.

Keywords: *Solvents. Dentin. Adhesives.*

1 Introduction

Self-etch adhesives are less sensitive technique, as they do not require removal of the smear layer, despite a lower bond strength was reported when compared to the etch-and-rinse systems [1,2] due to the high concentration of solvents[3], which may impair polymerization[4].

Thus, some approaches have been tested to enhance the solvent removal from dental adhesives before light-curing, including prolonged air drying[5,6]. It was observed that prolonged air-drying times of 20-30s can increase the degree of solvent evaporation and bonding to dentin, depending on the adhesive system[7].

Also, higher immediate dentin bonding has been described to multi-mode adhesives when the solvent evaporation time was increased from 5s to 25s for the etch-and-rinse strategy, while lower nanoleakage to dentin was observed when Single Bond Universal was used[8]. However, it is worth emphasizing that the waiting time of 25s to evaporate solvents from dental adhesives may be clinically difficult to achieve.

Threfore, other alternatives have been studied, such as the use of a warm air stream that has shown to increase the immediate[5,9,10] and long-term dentin bonding[11]. These studies were performed predominantly on etch and rinse adhesives, thus further studies are required to evaluate whether or not this approach can improve the performance of all-in-one adhesives to dentin.

Therefore, the aim of this investigation was to compare the effects of the air stream temperature for solvent evaporation on the bonding durability and nanoleakage expression of all-in-one self-etch adhesives to dentin. The null hypothesis is that there may be no difference between the adhesive systems and air temperatures.

2 Material and Methods

2.1 Experimental design

An *in vitro study* with three factors: adhesive system, drying air temperature, and storage time, at two levels each was performed. Bond strength was the main response variable to compare the factors. Additionally, morphological analysis of the restorative interfaces to dentin was assessed. This research was approved by the Ethics Research Committee of the dental school (Protocol 0186/08).

2.2 Specimen preparation

Forty third molars (n=5) were disinfected in 0.5% chloramine, and a flat dentin surface was exposed after grinding the occlusal enamel on a # 180 grit SiC paper (Norton, Saint-Gobain, Curitiba, Brazil) and further polishment on wet # 600-grit silicon-carbide paper for 60 s to standardize the smear layer. Two one-step self-etch adhesive systems were tested (Table 1), as follows: Clearfil 3S Bond -S3 (Kuraray Medical Inc, Tokyo, Japan), and OptiBond All-In-One - OB (Kerr Co, Orange, USA).

Table 1: Adhesive systems, composition, and application modes

Material (Batch number)	Composition	Application Mode
Clearfil 3S Bond (00006)	10 MDP, Bis-GMA, 2-HEMA, di-camphoquinone, hydrophobic dimetacrilate, ethanol (<20%), water and silanated colloidal silica	**1**- Shake the bottle **2**-apply one coat of adhesive vigorously (20s); **3**- dry with warm/cold air stream (10s) **4**- 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, photoinitiators, accelerators and stabilizers	**1**- Shake the bottle; **2**- apply first coat of adhesive vigorously (20s); **3**- apply second coat of adhesive vigorously (20s); **4**- dry with warm/cold air stream (10s); **5**- light cure (10s).

Source: Research Data.

2.3 Restorative procedures and bond strength measurements

After the application of adhesives, the solvent evaporation was performed either with a warm (60±2 ^{o}C) or cold air (20±2 ^{o}C) for 10 s at a distance of 10 cm. In both cases, the air stream was generated by a commercially hair-dryer (SC831, Black & Decker, Uberaba, Brazil). The air speed was 5.50 m/s and the air flow rate 0.0138 m^3/s.[9] The adhesives were light-cured at 600 mW/cm^2 (UltraLux, Dabi Atlante, Rio de Janeiro, Brazil). Resin composite build-ups (Opallis, VL, FGM, Joinville, Brazil) were constructed on the bonded surfaces in 3 increments of 2 mm each that were individually light-cured for 30 s.

After storage in distilled water (37 ^{0}C/24 h), the teeth were sectioned perpendicularly to the bonded interfaces with a diamond saw in an Isomet 1000 machine (Buehler Ltd, Lake Bluff, USA) to obtain sticks of 0.8 mm^2. Each bonded stick was attached to a microtensile device (Odeme Biotechnology, Joaçaba, Brazil) with cyanoacrylate resin (Super Bonder Gel, Loctite, Itapevi, Brazil) and tested in tensile at 0.5mm/min (EMIC, São José dos Pinhais, Brazil) after 24 h and 6 months of water storage. The failure modes were evaluated at 40X (BEL Microimage analyser, Bel Photonics, Osasco, Brazil) and classified as cohesive (failure exclusive within dentin or resin composite), adhesive (failure at resin/dentin interface), or mixed (failure at resin/dentin interface that included cohesive failure of the neighboring substrates). Only the mixed type was considered for statistical purposes.

The mean bond strength of all sticks from the same tooth was averaged for statistical purposes, considering the tooth as the experimental unit. Data were subjected to a three way Analysis of variance (Adhesive System vs. air temperature vs. storage time) and the post-hoc Tukey´s test (alpha = 0.05).

2.4 Scanning electron microscopy for silver nitrate uptake evaluation

Two bonded sticks from each tooth 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 that was prepared according to the protocol previously described[9] and they kept stored in darkness for 24 h, rinsed thoroughly in distilled water, and immersed in photo developing solution for 8h (Kodak Dental Developer, Kodak, Curitiba, Brazil), 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 µm diamond paste (Buehler Ltd, Lake Bluff, 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.

3 Results and Discussion

3.1 Bond strength

No significant differences were observed for both the adhesive system (p=0.343) and the interactions. There were differences for the air temperature (p=0.003) and storage time (p=0.000). Table 2 shows the data according to the experimental conditions. Air-temperature and storage time data were grouped and analyzed together due to the lack of significance of the adhesive system.

Table 2: Mean (Standard deviation) for the experimental conditions (*).

Adhesive	Air Temperature	24 Hours	6 Months
Clearfil 3s Bond	Cold	31.64(6.44) Ab	19.79(2.80) C
	Warm	36.75(8.01) A	27.36(3.37) Abc
Optibond All In One	Cold	30.86(3.16) Abc	25.62(5.25) Bc
	Warm	36.27(7.42) Ab	29.21(4.05) Abc

(*) The same uppercase letters show similar means (p>0.05)

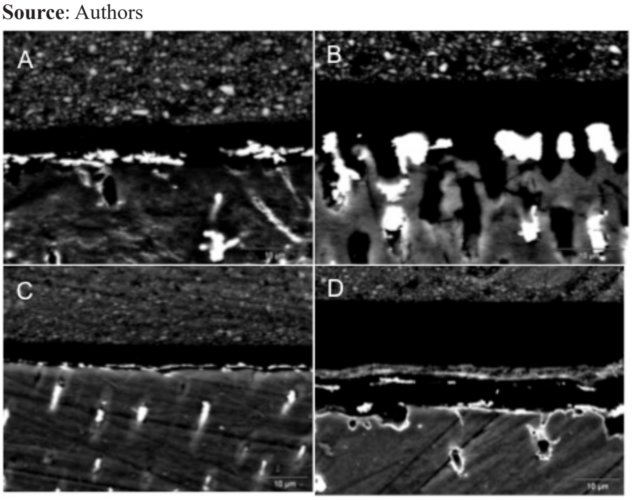
Source: Research Data.

According to the statistical analysis, higher bond strength was observed for warm-air for solvent evaporation when compared to cold-air, after 24 hours than 6 months. Table 3 shows the mean bond strength values of air-temperature and storage time. The failures after the bond strength test showed the prevalence of mixed fractures in all conditions, as can be seen in Table 4.

3.2 Scanning electron microscopy for silver nitrate uptake evaluation (SNU)

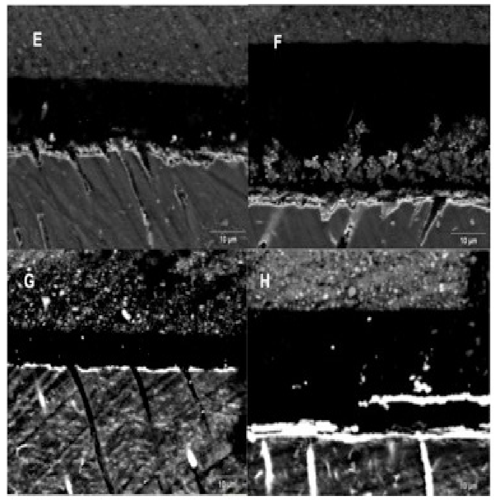
The Backscattered SEM images of the resin-dentin interface at 24 hour (Figure 1) and 6 months (Figure 2) are presented below. Silver nitrate update was observed in all experimental conditions.

Figure 1: Backscattered SEM images of the resin-dentin interface at 24 hours for Clearfil 3S Bond (A,B), and OptiBond All-In-One (C,D). Silver nitrate update (white hand) was observed in both the cold air dry stream (B,D) and warm-air dry stream groups (A,C). **Source**: Authors



Source: Research Data.

Figure 2: Backscattered SEM images of the resin-dentin interface at 6 months for Clearfil 3S Bond (E,F), and OptiBond All-In-One (G,H). Silver nitrate update (white hand) was observed in both the cold air dry stream F,H) and warm-air dry stream groups (E,G) **Source**: Authors



Source: Research Data.

A complex chemistry is needed to blend hydrophilic and hydrophobic monomers, solvents, water and additives to dental adhesives[3]. Water and solvents are indispensable because they provide the ionization medium for the self-etch activity[3]. Solvents act as a transport medium and lower resin viscosity. They allow greater penetration of resins into the micro porosities of the tooth surface, as well as enhance the mobility of radicals and growing polymer chains[13]. However, entrapped solvents within the polymerized adhesive and hybrid layer can compromise the formation of a strong polymer network[14].

Thus, attention should be given to the removal of as much solvent as possible from self-etch adhesives before light curing, and one approach was assessed in this study. Our results showed that apart from the adhesive system, higher bond strength was achieved when the solvent was evaporated with warm-air at 24h, when compared to 6-month period, and cold air-stream evaporation. Our 24h-results agree with previous studies[5,11] that used warm-air to evaporate solvents from dental adhesives, explained by differences in the kinetic of solvent evaporation as the warm air increases the energy of molecules that evaporate faster than the cold air.

It is known that as water/solvent can evaporate from the adhesive, the monomer density increases sharply and creates a monomer concentration gradient that acts as a barrier for further solvent evaporation, reducing the ability of water/solvents to evaporate from the adhesive layer[15]. In the case of simplified adhesives, it is even worse since the extent of solvent and water retention in polymer networks is correlated with the hydrophilicity of the resin blends[16]. Similar results on dentin bonding for all-in-one adhesives confirm this statement, which is highlighted by the scanning electron micrographs, where silver nitrate deposits kept entrapped within the resin-dentin bonds, predominantly in cold air groups of both adhesives.

Several attempts have been made to increase solvent evaporation from the adhesive layer, such as the use prolonged air-drying[5-7] Similar findings were observed in terms of the recommended clinical time for solvent evaporation (around 10 s) is rather short, and increasing the solvent evaporation time to more than 20s may have enhanced the bond strength of the adhesives, which is difficult to achieve clinically.

Thus, studies on increasing the air-drying temperature[5,1] for solvent evaporation of dental adhesives have been encouraged. Despite the good performance of the adhesives of the present study under warm air-drying at 24h, this fact was not observed after 6 months, probably because the solvents remained inside the polymer, as suggested by the photomicrographs. An earlier study has demonstrated that only periods longer than 24-96 h can ensure an almost complete solvent evaporation[17], which is clinically impossible.

The use of warm air to evaporate solvents from dental adhesives improved the resin-dentin bonds, as observed in previous studies[14,15], with better results when compared with other study[5]. However, the hydrophilicity of the adhesive layer cannot be changed by just using a warm air-dry stream or prolonged air-drying times[18].

These findings can lead to discussions about the real advantages of all-in-one adhesives [4]. Other strategies have been applied successfully to improve the bonding durability of dental adhesives to dentin, as the active application and the use of a hydrophibic layer[19]. Hydrophilic adhesives including the all-in-one system in the present study can attract water that plasticizes polymers, reducing their mechanical properties[20,21].

The nanoleakage was enhanced by the presence of solvent in *Clearfil SE Bond* and *Clearfil S³ Bond* dental adhesives[22]. The type of solvent (acetone, butanol, or ethanol/water) has shown to prioritarialy affect the evaporation rate of the adhesive, despite the time needed to do it[23]. All these strategies should be carefully considered to remove as much as solvent as possible before light curing, without compromising the dentin bonding durability.

4 Conclusion

The use os warm-air stream for solvent evaporation improved the bonding of all-in-one adhesive systems to dentin.

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