

Effect of alternative solvent evaporation techniques on mechanical properties of primer-adhesive mixtures

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ABSTRACT

This study evaluated the influence of the mode and time of solvent evaporation on the tensile strength (TS), flexural strength (FS) and elastic modulus (EM) of two adhesive systems: Scotchbond Multipurpose (SBMP) and Clearfil SE (CSEB). For this purpose, rectangular samples (2x1x7 mm) were prepared with 10 µL of primer and the solvents were evaporated with air spray at (23±1) °C, (40±1) °C and negative control (without spray). For each temperature, the times of 5, 20, 30, and 60 seconds were investigated. The statistical results showed that evaporation at 40±1 °C resulted in better EM for the

two adhesives tested and all the evaporation times evaluated. However, there were no significant differences between the times and modes of evaporation for TS. The results of this study indicate that evaporation at a temperature of (40±1) °C could improve the elastic modulus of both adhesives tested, regardless of the evaporating time.

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Keywords: dentin bonding agents- tensile strength- flexural strength.

Efeito de técnicas alternativas utilizando evaporação de solvente nas propriedades mecânicas da mistura primer-adesivo

RESUMO

Este estudo avaliou a influência do modo e do tempo de evaporação do solvente na resistência à tração (RT), resistência à flexão (RF) e módulo de elasticidade (MR) de dois sistemas adesivos: Scotchbond Multipurpose (SBMP) e Clearfil SE (CSEB). Para isso, amostras retangulares (2x1x7 mm) foram preparadas com 10 µL de primer e os solventes foram evaporados com aerossol a (23±1) °C, (40±1) °C e controle negativo (sem aerossol). Para cada temperatura, foram avaliados os tempos de 5, 20, 30 e 60 segundos. Os resultados estatísticos mostraram que a evaporação a (40±1) °C resultou

em melhor MR para os dois adesivos testados e todos os tempos de evaporação avaliados. No entanto, não houve diferenças significativas entre os tempos e modos de evaporação na RT. Os resultados deste estudo indicam que a evaporação a uma temperatura de (40±1) °C poderia melhorar o módulo de elasticidade de ambos os adesivos testados, independentemente do tempo de evaporação.

Palavras-chave: adesivos dentinários- temperatura- resistência à tração- propriedades de flexão.

INTRODUCTION

Dentin hybridization through monomeric interdiffusion is an essential mechanism to obtain an effective, intimate bond to the dentin tissue¹. However, the bond between the dental substrate and the restorative

material is affected by the presence of residual water or excess solvent. The residual solvent in these systems may act as a polymeric matrix plasticizer, interfering with the mechanical properties of these materials and consequently with the bonding interface^{2,3}.

The solvents in the composition of adhesive systems should be completely evaporated from the resin-infiltrated dentin matrix^{4,6}. Excess water or other solvents from inadequate evaporation may negatively affect the physical-mechanical strength of the adhesion layer due to the inhibition of the polymerization of these materials and the plasticization of the adhesive structure by the presence of solvent molecules⁵⁻⁷.

The influence of excess solvent on the bond strength and mechanical properties of adhesive systems has been tested in several *in vitro* studies, which reported quick, standardized results establishing the behavior of materials in conditions similar to those in clinical practice³⁻⁷. However, these studies evaluated only the pure adhesive solutions, without primer addition. There is therefore a need to evaluate the primer/adhesive mixture for better comparison with what actually happens in clinical practice.

It has been shown that mixing the adhesive with the primer solution decreases the mechanical resistance of adhesive systems because the polymerization process is inhibited by compounds in the primer^{8,9}. It is therefore important to determine the effect of solvent evaporation on the mechanical properties of primer/adhesive mixtures.

Considering the adverse effect of excess solvent on the polymerization of adhesive systems⁹, the creation of alternative techniques for better evaporating these solvents can provide greater adhesive longevity. Thus, the aim of this study was to test the hypothesis that the evaporation of the solvent at high temperatures may improve the mechanical properties of Scotchbond Multipurpose PlusTM and Clearfil SE BondTM.

MATERIALS AND METHODS

This study investigated two commercial adhesives: a conventional three-step adhesive system -

Scotchbond Multipurpose PlusTM (SBMP-3M ESPE, St. Paul, MN, USA) and a two-step self-etching adhesive - Clearfil SE BondTM (CSEB-Kuraray Medical Inc., Okayama, Japan).

These adhesive systems were selected because they are both the gold standard of their classes and have repeatedly shown excellent results in clinical and laboratory studies¹⁰⁻¹². Table 1 presents the composition of the adhesive systems and Fig. 1 shows the preparation of specimens for each adhesive.

2.1 Sample preparation

Bar specimens (1 mm × 2 mm × 7 mm) were prepared in a rectangular silicone mold from the mixture of primer and adhesive of the two systems used. A total 360 specimens were obtained (n = 5 for modulus of elasticity and flexural strength and n = 10 for maximum tensile strength).

The solvent was evaporated by air spray at room temperature (23±1) °C and by heated air spray (40±1) °C using a device with pressure, airflow, and controlled power to provide airflow at a constant temperature. The temperature of the hot air at the spray outlet was approximately 100 °C. A standard distance of 5 cm was set between the air spray outlet and the mold surface to obtain a temperature of (40±1) °C in the sample drying process.

Initially, 10 µL of primer of each adhesive system investigated was inserted in the silicone mold, followed by the primer evaporation process according to the mode and time proposed in the experimental design. After that, 20 µL of adhesive were added and carefully mixed with the primer for 60 s to prevent the incorporation of bubbles (as far as possible). The primer/adhesive ratio by weight was 1:3, in accordance with the protocol established by Burchardt and Merz¹³. The samples were dried

Table 1. Composition of the bonding agents

Adhesive system	Manufacturer	Composition*
Scotchbond Multipurpose Etch-and-rinse (SBMP)	3M ESPE, St Paul, MN	Primer: HEMA, polyalkenoic acid polymer, water Adhesive: Bis-GMA, HEMA, tertiary amines, photoinitiator (CQ)
Clearfil SE BOND Self-etch (CSEB)	Kuraray, Osaka, Japan	Primer: MDP, HEMA, photoinitiator, water hydrophilic dimethacrylate Adhesive: MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, photoinitiators, colloidal silica

Abbreviations - BisGMA: bisphenol-A glycidyl dimethacrylate; HEMA: 2-hydroxyethylmethacrylate; CQ: camphorquinone; MDP: 10-methacryloyloxydecyl dihydrogen phosphate *As informed by the manufacturers.

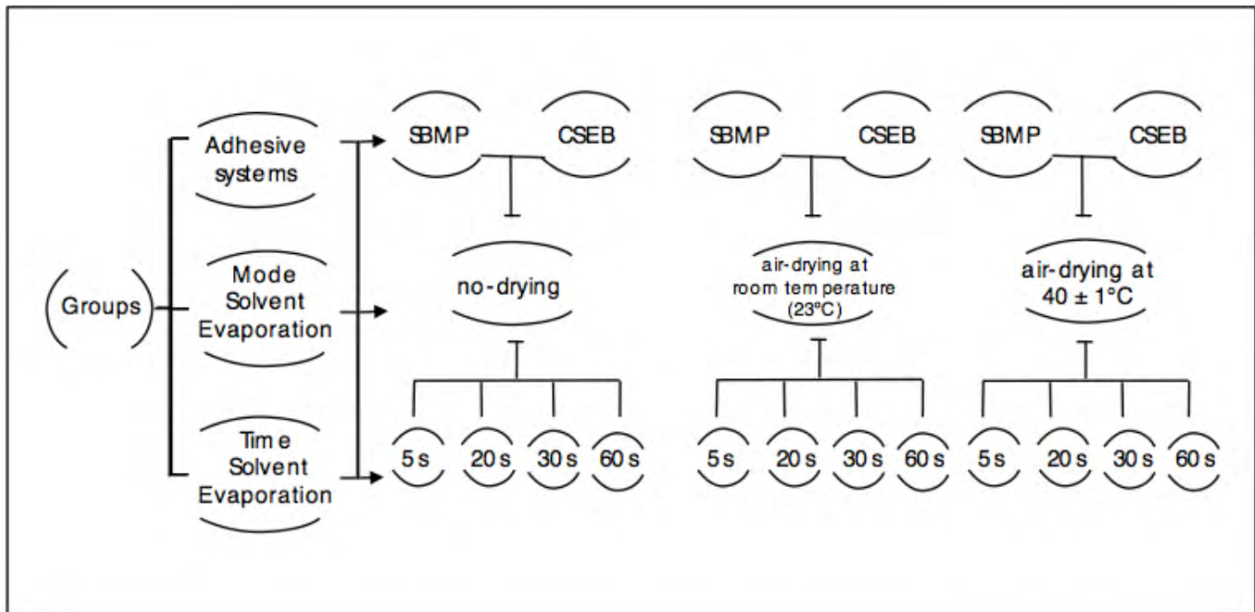


Fig. 1. Experimental design.

at different temperatures and photopolymerized using the Vip Bisco (Vip Curing Lights - BISCO Dental Products IL, USA) with 600 mw/cm² of light intensity.

The light was activated with a holder coupled to the light sources to standardize a 4 mm distance between the light guide tip and the material, which simulated a medium cavity depth. The polymerized samples were removed from the silicon mold and stored in distilled water for 24 h at 37 °C.

2.2 Measurement of tensile strength (TS)

The dimensions of the specimens were measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). Bar-shaped specimens were attached to a device with cyanoacrylate adhesive and tested under tension in a Universal Testing Machine (EMIC Ltda., São José dos Pinhais, SP, Brazil) at 0.5 mm/min speed until rupture. The TBS values were calculated in MPa by dividing the load at rupture by the cross-sectional area.

2.3 Measurement of flexural strength (FS) and elastic modulus (EM)

The specimens were subjected to a three-point bending flexural test to measure flexural strength (FS) and elastic modulus (EM). The three-point bending test was performed in a universal testing machine (Instron model 4411, Instron Corp., Canton, MA, USA) according to ISO 4049 specifications.

Before the test, the dimensions of each specimen were recorded with a caliper. The Bluehill 2 software (Instron Corp., Canton, MA, USA) was used to calculate EM (GPa) and FS (MPa) values according to the dimensions and tension in each case. The maximum loads were obtained and the flexural strength (σ) was calculated in megapascals (MPa) by using the formula:

$$\sigma = 3FL/(2BH^2)$$

where F is the maximum load (in newtons); L is the distance between the supports (in millimeters); B is the width of the specimen (in millimeters) and H is the height (also in millimeters).

The modulus of elasticity (GPa) was calculated as:

$$E = FL^3/4BH^3d$$

where F is the maximum load; L is the distance between the supports; B is the width of the specimen, H is the height of the specimen, and d is the deflection (in millimeters) corresponding to the load F.

2.4 Statistical analysis

The data were analyzed with one-way ANOVA and Tukey's post-hoc test at a 99.5% confidence level. In addition, they were subjected to multivariate analysis of variance (MANOVA) with repeated measures and a Lambda Wilks test ($p < 0.05$).

RESULTS

Based on the elastic modulus results (Table 2), the evaporation at $\pm 40^\circ\text{C}$ resulted in a higher elasticity modulus, regardless of the time of application of the air spray and the adhesive system used. The use of the heated air spray led to 1.59 GPa mean modulus value, which was statistically higher than the value found for the groups where spray at room temperature (1.40 GPa) or evaporation without application of air spray (1.37 GPa) were used.

As for the influence of time on the elastic modulus, the increase of the evaporation time did not affect the behavior of the adhesive systems investigated. There was an exception for the time of 5 s for the CSEB self-etching adhesive system, which showed a significant reduction in the elasticity modulus when compared to the time of 30 s for the same group (1.60 GPa) and 60 s for the conventional

adhesive system (1.56 GPa). The specimens from the other groups presented EM with statistically similar results (Table 3).

Flexural strength analysis showed that the change in the solvent evaporation regime did not lead to any significant results. However, comparing the systems evaluated, the CSEB self-etching adhesive system (84.04 MPa) was found to have better properties than the SBMP etch-and-rinse system (62.44 MPa) (Table 4).

The interaction between adhesive system, mode, and evaporation time did not present statistical difference concerning maximum tensile strength (Table 5).

DISCUSSION

The solvent present in adhesive systems acts as a transport vehicle by decreasing the viscosity

Table 2. Elastic modulus (EM) of the different solvent evaporation types

Air Spray	Evaporation times	Adhesive system	M/SD (GPa)	M/SD (GPa)*
No drying (ND)	5	SBMP	1.33 (0.09)	1.37 (1.02) B
		CSEB	1.10 (1.01)	
	20	SBMP	1.26 (1.00)	
		CSEB	1.30 (0.06)	
	30	SBMP	1.40 (0.08)	
		CSEB	1.60 (0.08)	
	60	SBMP	1.52 (1.02)	
		CSEB	1.41 (1.02)	
Air-drying at room temperature 23° C (AD23)	5	SBMP	1.35 (1.02)	1.40 (0.09) B
		CSEB	1.41 (0.09)	
	20	SBMP	1.36 (1.02)	
		CSEB	1.40 (1.01)	
	30	SBMP	1.36 (0.08)	
		CSEB	1.39 (1.00)	
	60	SBMP	1.56 (0.08)	
		CSEB	1.37 (0.06)	
Air-drying at 40°C \pm 1°C (AD40)	5	SBMP	1.52 (0.09)	1.59 (1.32) A
		CSEB	1.62 (1.31)	
	20	SBMP	1.61 (1.20)	
		CSEB	1.52 (0.16)	
	30	SBMP	1.60 (0.18)	
		CSEB	1.66 (0.68)	
	60	SBMP	1.60 (1.32)	
		CSEB	1.59 (1.32)	

*M: Mean; SD: Standard deviation; Means followed by different letters differ from each other ($p \leq 0.05$).

Table 3. Elastic modulus (EM) of the different solvent evaporation times

Evaporation times	Air Spray	Adhesive system	M/SD (GPa)	M/SD (GPa)*
5	No drying (ND)	SBMP	1.33 (0.09)	1.46 (0.09) A
		CSEB	1.10 (1.01)	
	Air-drying at room temperature 23° C (AD23)	SBMP	1.35 (1.02)	
		CSEB	1.41 (0.09)	
	Air-drying at 40°C ± 1°C (AD40)	SBMP	1.52 (0.09)	
		CSEB	1.62 (1.31)	
20	No drying (ND)	SBMP	1.26 (1.00)	1.41 (1.01) A
		CSEB	1.30 (0.06)	
	Air-drying at room temperature 23° C (AD23)	SBMP	1.36 (1.02)	
		CSEB	1.40 (1.01)	
	Air-drying at 40°C ± 1°C (AD40)	SBMP	1.61 (1.20)	
		CSEB	1.52 (0.16)	
30	No drying (ND)	SBMP	1.40 (0.08)	1.46 (1.00) A
		CSEB	1.60 (0.08)	
	Air-drying at room temperature 23° C (AD23)	SBMP	1.36 (0.08)	
		CSEB	1.39 (1.00)	
	Air-drying at 40°C ± 1°C (AD40)	SBMP	1.60 (0.18)	
		CSEB	1.66 (0.68)	
60	No drying (ND)	SBMP	1.52 (1.02)	1.48 (1.02) A
		CSEB	1.41 (1.02)	
	Air-drying at room temperature 23° C (AD23)	SBMP	1.56 (0.08)	
		CSEB	1.37 (0.06)	
	Air-drying at 40°C ± 1°C (AD40)	SBMP	1.60 (1.32)	
		CSEB	1.59 (1.32)	

*M: Mean; SD: Standard deviation; Means followed by different letters differ from each other (p<0.05).

of the adhesive, thereby enabling the resinous monomers to penetrate the micropores formed by acid etching^{13,14}. However, incomplete solvent evaporation significantly compromises adhesion efficacy^{3-7,14}, considering that excess solvents can lead to a dilution of the components that prevent the collision of reactive components. This hinders the attainment of high cross-linking polymers inside the hybrid layer, which may result in a porous hybrid layer structure with less mechanical properties¹⁵. The results showed that both drying time and temperature had a significant effect on the mechanical properties of the two adhesive systems tested. The application of hot air spray (40 ± 1°C) produced better elasticity modulus values, regardless of the adhesive system used. The elastic modulus represents the relative stiffness of the material as well as its resistance to plastic deformation, which is directly related to the formation of more crosslinks in

the polymer structure.² The increase in the modulus as a result of the heated air spray is probably due to the efficient removal of the solvent, leading to a more efficient polymerization process^{3,4,14}. This phenomenon may be also explained based on the formulation of the two adhesive systems investigated in this study, in which the primer formulation contains HEMA and water. The presence of HEMA is important because it can expand the dried dentin collagen and improve adhesion. However, the presence of HEMA in these systems improves the retention of solvent and water in polymer networks due to its influence on resin polarity. The presence of HEMA provides the high number of OH groups in the polymeric chains and improves the hydrogen bonding sites between polymer and solvent, hindering solvent evaporation¹⁶. In this case, the use of temperature for solvent elimination is important to better evaporate the water in the system.

Table 4. Flexural strength (FS) according to adhesive system interaction, time and type of solvent evaporation

Air Spray	Evaporation times	Adhesive system	M/SD (MPa)	M/SD (MPa)*
No drying (ND)	5	SBMP	60.55 (22.09)	72.52 (35,71) A
		CSEB	84.42 (18.09)	
	20	SBMP	60.11 (19.04)	
		CSEB	80.40 (19.99)	
	30	SBMP	64.57 (31.05)	
		CSEB	81.01 (22.09)	
60	SBMP	63.01 (22.09)		
	CSEB	86.04 (21.01)		
Air-drying at room temperature 23° C (AD23)	5	SBMP	62.09 (18.09)	73.77 (22.50) A
		CSEB	88.04 (21.01)	
	20	SBMP	64.21 (25.87)	
		CSEB	83.00 (21.01)	
	30	SBMP	65.31 (24.00)	
		CSEB	82.81 (21.01)	
60	SBMP	58.65 (28.01)		
	CSEB	86.04 (21.01)		
Air-drying at 40°C ± 1°C (AD40)	5	SBMP	59.88 (19.09)	73.44 (20.16) A
		CSEB	88.35 (21.07)	
	20	SBMP	65.77 (20.34)	
		CSEB	79.04 (18.00)	
	30	SBMP	64.63 (19.53)	
		CSEB	85.24 (20.01)	
60	SBMP	60.50 (22.23)		
	CSEB	84.09 (21.01)		

*M: Mean; SD: Standard deviation; Means followed by different letters differ from each other ($p \leq 0.05$).

In these adhesives with HEMA/water mixtures, the water evaporates more rapidly than the HEMA, which is a relatively non-volatile component. Thus, the concentration of HEMA increases as the water evaporates, reducing the vapor pressure of the water and making the adhesive more viscous, which makes it difficult to remove the last remaining water^{16,17}. The difference in the properties of the two adhesive systems compared in this study may be due to their composition. The primer in the self-etching adhesive system (CSEB) contains acid monomers, HEMA, and water, while the primer in the conventional three-step adhesive system (SBMP) contains only water and HEMA. The incorporation of acid monomers in the simplified adhesive systems is necessary to condition the dental structures. However, such monomers may increase the water absorption by these systems due to the interaction of monomers and water¹⁸.

The interaction between adhesive and time shows that the increase of water concentration results in an adhesive interface with loss of mechanical properties, higher water absorption, and a lower degree of conversion^{18,19}. The increase in water concentration results in increased ionization of acid monomers, which may reduce their reactivity and the polymerization of the systems^{18,20}. Thus, the lowest mechanical property exhibited at 5 s for all adhesives may be due to the high concentration of residual water that did not allow adequate polymerization of these monomers.

Tensile strength evaluates the cohesion in the polymer layer formed after polymerization. Thus, increasing cohesion indicates a formation of a hybrid layer with lower microporosity, which is consequently less susceptible to infiltration². In this study, there was no statistical difference in tensile strength, indicating that the evaporation obtained was not

Table 5. Maximum tensile strength (TS) according to adhesive system interaction, time and type of solvent evaporation

Air Spray	Evaporation times	Adhesive system	M/SD (MPa)	M/SD (MPa)*
No drying (ND)	5	SBMP	90.05 (5.81)	93.84 (8.05) A
		CSEB	95.34 (9.77)	
	20	SBMP	95.32 (9.76)	
		CSEB	91.11 (8.96)	
	30	SBMP	94.08 (7.87)	
		CSEB	95.14 (6.65)	
60	SBMP	94.45 (9.83)		
	CSEB	95.23 (5.75)		
Air-drying at room temperature 23° C (AD23)	5	SBMP	91.25 (6.72)	94.33 (6.21) A
		CSEB	96.44 (9.09)	
	20	SBMP	95.42 (4.67)	
		CSEB	92.23 (8.93)	
	30	SBMP	94.18 (5.45)	
		CSEB	95.24 (6.33)	
60	SBMP	94.55 (3.26)		
	CSEB	95.33 (5.23)		
Air-drying at 40°C ± 1°C (AD40)	5	SBMP	99.33 (9.01)	94.10 (8.93) A
		CSEB	94.06 (9.34)	
	20	SBMP	94.19 (9.20)	
		CSEB	90.32 (8.08)	
	30	SBMP	93.14 (9.06)	
		CSEB	94.08 (9.32)	
60	SBMP	93.23 (8.09)		
	CSEB	94.45 (9.34)		

*M: Mean; SD: Standard deviation; Means followed by different letters differ from each other ($p \leq 0.05$).

sufficient to improve material cohesion. The result does not corroborate previous studies that show the increase of cohesive resistance against the change in solvent evaporation^{3-7,14}. However, those studies did not use the primer/adhesive mixture, which may be responsible for the difference observed.

Similarly, the evaluation of flexural strength showed no statistical difference regarding the different times and modes of solvent evaporation. For flexural strength, it was observed that the CSEB adhesive system obtained better results than the SBMP. The evaluation of this property is important for clinical practice, considering that the adhesive layer must undergo the least flexing when submitted to masticatory loads and this mechanical property can evaluate the resistance of the material to deformity as well as the tensile strength².

As mentioned above, the adhesive systems used have

different primer compositions. The CBSE contains, along with HEMA and water, acidic monomers that are hydrophilic and favor higher water adsorption by the primer of the adhesive system, making it less viscous. The lower the viscosity of a fluid, the faster and more easily it will move and thus allow greater interaction among the monomers, resulting in a more complete polymerization reaction with a better quality of crosslinks and providing greater cohesion to the polymers¹⁴.

According to the results of this study, evaporation time did not provide a positive correlation between TBS and EM. The EM of the adhesives tested was increased with hot air evaporation, while flexural strength did not change. It is therefore believed that the evaporation of the solvent with heated air spray provides a greater number of crosslinks, but a better quality between these bonds could not be obtained.

CONCLUSIONS

The results of the present study show that:

- The different solvent evaporation modes and temperatures did not affect the flexural strength of the adhesives tested.
- The CSEB system obtained better results than

CBMP regarding flexural strength.

- Hot air spray evaporation (40°C) obtained better results of elasticity modulus (relative rigidity of the material), i.e., a greater number of crosslinks regardless of the adhesive system and time of evaporation.

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CORRESPONDENCE

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