

Influence of a heating device and adhesive temperature on bond strength of a simplified ethanol-based adhesive system

Influência de um dispositivo de aquecimento e da temperatura do adesivo na resistência de união de um sistema adesivo à base de etanol

Marcos Paulo Marchiori CARVALHO^a, Rachel de Oliveira ROCHA^a, Ivo KREJCI^b,
Tissiana BORTOLOTTTO^b, Fábio Ecke BISOGNO^a, Alexandre Henrique SUSIN^{a*}

^aUFMS – Universidade Federal de Santa Maria, Santa Maria, RS, Brazil

^bDivision of Cariology and Endodontics, School of Dentistry, University of Geneva, Geneva, Switzerland

Resumo

Introdução: O aumento da temperatura do adesivo tem sido pesquisado como forma de melhorar a evaporação do solvente, reduzir a viscosidade e melhorar a permeação dos monômeros na dentina. **Objetivo:** Investigar a influência de diferentes métodos de aquecimento na resistência de união à dentina sob um ensaio de microtração de um adesivo de condicionamento ácido total. **Material e método:** Vinte e quatro terceiros molares hígidos foram seccionados transversalmente a fim de expor a superfície plana da dentina. As amostras foram condicionadas ácido fosfórico a 37% e divididas em três grupos (n=8). São eles: 1) Controle - onde o adesivo (Adper Single Bond 2, 3M ESPE) foi aplicado em temperatura normal (25°C); 2) Dispositivo de aquecimento - O adesivo foi aquecido em um dispositivo específico até alcançar a temperatura de 37°C e só então aplicado à dentina; 3) Ar quente - Usou-se jato de ar aquecido a 50°C por 10 segundos a uma distância de 10cm para facilitar a evaporação do solvente. Os espécimes foram restaurados com resina composta (Filtek Z250 A2, 3M ESPE) e preparados para o ensaio de microtração, após 24h de armazenamento em água destilada. Os dados obtidos foram submetidos para análise de variância (ANOVA) e teste de Tukey (p<0,05). **Resultado:** Não houve diferença significativa entre os grupos (p>0,05). As médias da resistência de união para o grupo controle, dispositivo de aquecimento e ar quente foram, respectivamente, 48,5 (± 5,2), 40,35 (± 4,9), e 47,2 (± 5,3). **Conclusão:** Os diferentes métodos de aquecimento não influenciaram significativamente na resistência de união imediata à dentina de um Sistema adesivo de condicionamento ácido total.

Descritores: Adesivos dentinários; dentina; temperatura; adesão.

Abstract

Introduction: Increased adhesive temperature has been reported to promote solvent evaporation, reduce viscosity, and improve monomeric permeation into dentin. **Objective:** The aim of this study was to determine the influence of different heating methods on the microtensile bond strength of an etch-and-rinse adhesive to dentin. **Material and method:** Twenty-four caries-free extracted human third molars were transversally sectioned to expose a flat dentinal surface. The samples were etched with 37% phosphoric acid gel and divided into three groups (n = 8): 1) Control - the adhesive system (Adper Single Bond 2; 3M ESPE) was applied at room temperature; 2) Warming device - the adhesive was warmed to 37°C in a custom device before application; and 3) Warm air - the adhesive was warmed to 50°C with an air jet after application on dentin. The specimens were restored with a composite resin (Filtek Z250 A2, 3M ESPE) and prepared for microtensile bond strength testing, after 24 h in water storage. The data were subjected to one-way ANOVA and Tukey's test (p < 0.05). **Result:** There was no significant difference among the groups (p > 0.05). The mean bond strength values in the control, the warming device, and the warm air groups were 48.5 (± 5.2), 40.35 (± 4.9), and 47.2 (± 5.3) MPa, respectively (p = 0.05). **Conclusion:** The different heating methods had no significant influence on the immediate microtensile bond strength of an etch-and-rinse ethanol-based adhesive to dentin.

Descriptors: Dentin-bonding agents; dentin; hot temperature; bond strength.

INTRODUCTION

The adhesive-dentin interface is the weakest link in the tooth-restoration complex¹. The interaction between dentin and resin monomers depends on surface conditioning², and optimal dentin bonding occurs when adhesive monomers infiltrate completely into the mineralized dentin fibril network after etching³. Procedure changes have been suggested to improve bonding performance^{4,5}. Bonding effectiveness of adhesive systems may be associated with their temperature of application. Increased adhesive temperature promotes superior solvent evaporation and reduces the adhesive viscosity, hypothetically ensuring a stable and lasting bond⁶⁻¹². Enhanced solvent evaporation limits the residual solvent¹³, enhances wettability^{11,14,15} — which influence the hybrid layer formation positively¹⁶ — and yields a highly reticulated polymer, with reduced water sorption and lower hybrid layer solubility¹⁷.

It is widely accepted that the polymerization rate of adhesive systems is also improved by a temperature rise up to 60°C, which promotes a more stable and less degraded resin-dentin interface over time, whereas low temperatures have a negative impact on both aspects^{7-9,11}.

Acetone, ethanol and water are commonly used as solvents to dissolve hydrophilic and hydrophobic monomers in the adhesive system. Use of warm air to raise adhesive temperature promotes solvent evaporation; consequently, the chemical content of the adhesive solution is altered⁹.

To preserve the integrity of the chemical composition of the adhesive system, a special device may be used to produce a controlled rise in adhesive temperature in a sealed chamber. Thus, resin-dentin interfaces can be formed with an optimal chemical balance of the adhesive system.

Given the effects of temperature on adhesives, the impact of adhesive heating in a sealed chamber on the bond strength of adhesives should be evaluated.

Therefore, the aim of this study was to evaluate the influence of different heating methods on the microtensile bond strength of an etch-and-rinse dentin adhesive. The null hypothesis tested was that the heating methods would not improve the bond strength of the adhesive to dentin.

MATERIAL AND METHOD

The study was approved by the local ethics committee (protocol n. 553.956). Twenty-four caries-free human third molars, extracted from young patients, were selected and stored in a 0.5% thymol aqueous solution at 4°C, until use in the study.

Preparation and Grouping

The teeth were sectioned transversally with a precision sectioning saw (Isomet 1000; Buehler, Lake Bluff, IL, USA), at 250 rpm, to remove the occlusal third of the crown and expose a flat dentinal surface. The dentin surface was polished with #600-grit silicon carbide paper in a circular polishing machine (Arotex S/A, Cotia, SP, Brazil) for 40 s to standardize the smear layer.

Random Allocation Software 2.0 (freeware) was used to randomly allocate the specimens into three groups according to the heating method (n = 8), as follows: 1) Control group - the adhesive was applied on dentin at controlled room temperature (25°C) according to the manufacturer's instructions; 2) Warming device group - the adhesive system was warmed to 37°C in a custom device before application to dentin; 3) Warm air group - the adhesive was warmed to 50°C with an air jet applied directly to the dentin, after adhesive application and before light-curing (Table 1).

Restorative Procedures

All the specimens were etched with a 37% phosphoric acid gel (3M ESPE, St. Paul, MN, USA), for 15 s, rinsed and dried, according to the manufacturer's instructions. Two layers of the etch-and-rinse adhesive system (Adper Single Bond 2; 3M ESPE) were applied according to the specifications of each group, dried with compressed air for 5 s, and light-cured with an LED unit (Emitter H; Schuster, Santa Maria, RS, Brazil) operating at 800 mW/cm² for 10 s. Restorations made in 1.0-mm increments totaling 5 mm in height were then fabricated on the specimens with a composite resin (Filtek Z250 shade A2, 3M ESPE, St. Paul, MN, USA), each increment being light-cured for 20 s.

Figure 1 outlines the sequence of microtensile stick preparation. The groups are illustrated in the gray box.

Bond Strength Test

After 24 h in distilled water at room temperature, the specimens were sectioned into stick-shaped beams with an approximate cross-sectional area of 1 mm², using a low speed diamond saw under continuous water cooling. This resulted in 15-20 beams per tooth, depending on coronal size and pulp chamber volume. The cross-sectional dimensions of the beams were measured using a digital caliper (Mitutoyo America Corporation, Aurora, IL, USA) to calculate surface areas prior to microtensile bond strength (μ TBS) testing. The specimens were attached to an apparatus using superglue gel (Cyanoacrylate Rite-Lok, 3M, Manchester, UK) and then subjected to tensile force at a crosshead speed of 0.5 mm/min until failure, using a universal testing machine (DL 1000; EMIC, São José dos Pinhais, PR, Brazil) equipped with a 50-kN load cell. Microtensile bond strengths (in MPa) were recorded, and the means and standard deviations of the groups were calculated. The bond strength (σ) was obtained using the formula $\sigma = F/A$, where **F** = load for specimen rupture (in N) and **A** = bonded area (in mm²).

The bonded interface of the fractured beams was observed under a stereomicroscope (Stereo CI 1500 ECO; Carl Zeiss, Jena, Germany) at 35 \times magnification to select beams exclusively with adhesive failure. Those beams that presented cohesive or mixed failures were excluded from the analysis.

Statistical Analysis

The microtensile bond strength values expressed in MPa were subjected to a Levene test to evaluate homogeneity of variances, and then analyzed using one-way ANOVA (factor: heating methods) and Tukey's test at a significance level of 5%. All the tests were conducted using a statistical software package (*Statistical Package for Social Sciences*, version 20, Chicago, IL, USA).

Table 1. Groups studied, materials and methodologic summary

Adhesive System / Composite	chemical composition	Methodology summary		
		G1 - Control group	*G2 - Warming device group - 37°C	**G3 - Warming air group - 50°C
Adper Single Bond 2	37% Phosphoric acid	Acid etching 15 s;	Acid etching 15 s;	Acid etching 15 s;
Batch number: N345318BRI		Rinse air-water spray 5s;	Rinse air-water spray 5s;	Rinse air-water spray 5s;
Exp Date: 2015/Jan	Silica, Silane, BisGMA	Water excess removal	Water excess removal	Water excess removal
	HEMA, Dymethacrilates	with absorbent paper;	with absorbent paper;	with absorbent paper;
	ethanol, water,	Adhesive applied (2 coats);	adhesive heat inside the chamber;	Adhesive applied (2 coats);
	camphoroquinone,	Photocure 10 s;	Adhesive applied (2 coats);	adhesive heat over dentin
	copolymers of polyal		Photocure 10 s;	Photocure 10 s;
	kenoic and polyacrilic			
	acids			
Filtek Z250 (A2)	BisGMA, BisEMA, UDMA	Composite resin restorations	Composite resin restorations	Composite resin restorations
Nt82566	TEGDMA, silica and	in 5 increments of 1 mm	in 5 increments of 1 mm	in 5 increments of 1 mm
Exp Date: 2015/Jan	Zirconia			

*Need time to heat 37° C-5 min (controlled by electronic display).

**Need time to heat 50°C-15 sec (controlled with digital thermometer).

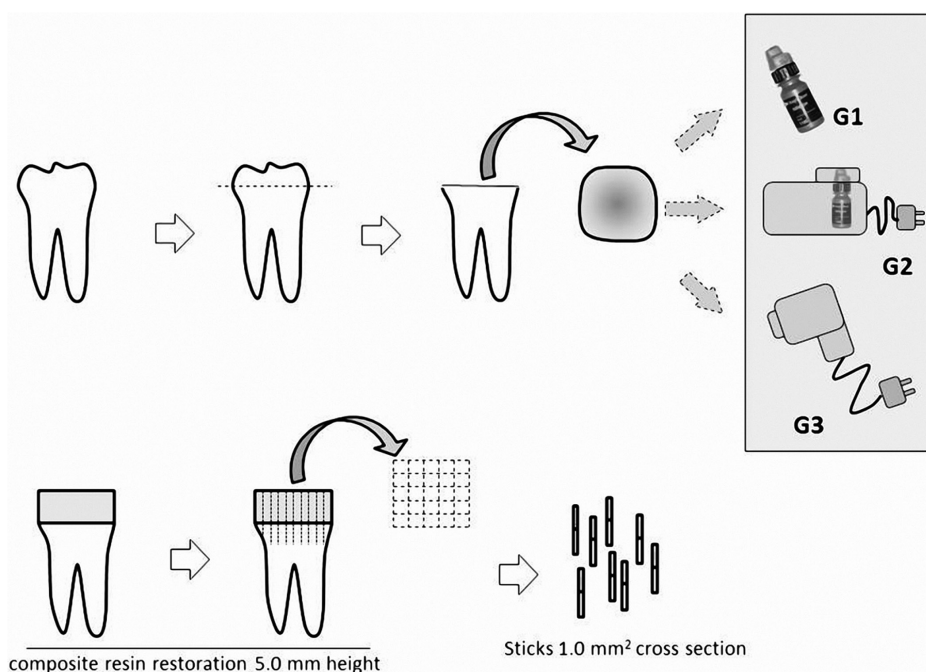


Figure 1. Schematic view of the methodology employed.

RESULT

ANOVA did not reveal significant differences among groups in regard to bond strength values ($p > 0.05$), indicating that the different heating methods were statistically similar. The mean bond strengths (and standard deviations) in the control, the warming device, and the warm air groups were 48.5 MPa (± 5.2), 40.35 MPa

(± 4.9), and 47.2 MPa (± 5.3), respectively (Table 2). The average number of viable sticks per tooth in each group is shown in Table 3.

In the groups where the heating methods were applied (warming device and warm air), the temperature was raised up to the limits set for the study. The temperature was raised to 37°C in five minutes in the specially designed warming device, and to 50°C in 15 s in the warm air group (Figures 2 and 3).

Table 2. Microtensile Bond Strength in Mpa, standard deviation (SD) - tested groups

Adper Single Bond 2	Control group (25°C)		Warming device group (37°C)		Warming air group (50°C)	
	MPa	SD	MPa	SD	MPa	SD
	48.52 a	5.2	40.35 a	4.9	47.21 a	5.3

Analyses of variance – Tukey’s test was applied at a level of 5% probability. Same letter indicate no statistically significant difference.

Table 3. Mean number of sticks obtained versus viable sticks per study group

Group / n	Obtained sticks per tooth	Viable sticks per tooth
Group 1 (n.8)	32	16
Group 2 (n.8)	34	18
Group 3 (n.8)	34	15

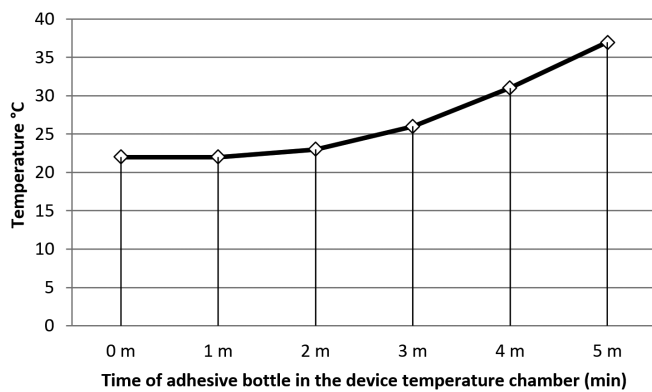


Figure 2. Warming device group. Temperature of the adhesive bottle in the device chamber according to time.

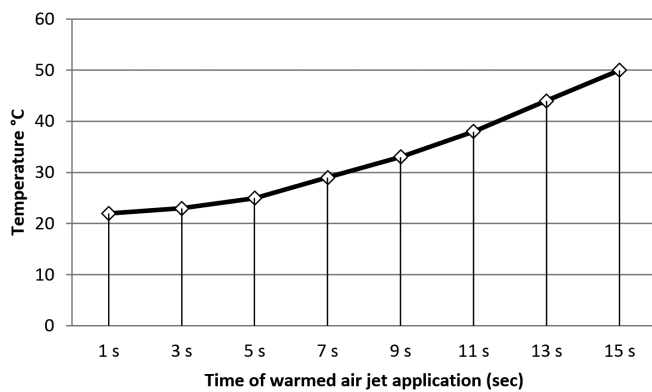


Figure 3. Warm air group. Temperature of the warm air jet according to time.

DISCUSSION

An increase in the evaporation rate and a decrease in the viscosity of the solvent are consequences of temperature elevation. During the bonding procedure, these effects immediately promote less residual solvent and improve the wettability of the tooth surface^{11,14,15}, positively affecting the hybrid layer formation. Therefore, at least theoretically, the enhanced bonding effectiveness of an adhesive system could be the result of a temperature increase altering the

physicochemical properties of the solutions involved¹⁶. Nonetheless, under the conditions of the present study, an increased temperature did not influence the bond strength values of the adhesive system tested. Thus, the null hypothesis was accepted, since none of the heating methods improved the microtensile bond strength of the dentin adhesive.

Changes in the temperature of solutions are usually achieved by applying warm air directly either on top of the adhesive or in a drying oven. Unlike other methods described in the literature, a specially designed device was used in this study to warm up the adhesive solution. An electronic display was used to maintain a controlled temperature, and a heating chamber housing the adhesive bottle allowed the temperature to rise to a controlled 37°C. Adhesives can be heated to appropriate levels by directing warm air from a special three-way syringe or hair dryer after application^{7,15,16}. Simple drying cabinets with temperature control displays can also be used. To our knowledge, this is the first study to test a custom-designed device with a sealed chamber to ensure a controlled increase of temperature, thus heating the adhesive solution precisely, up to the required temperature.

Microtensile bond strength testing is widely accepted as a method for assessing resin-dentin adhesion, since it allows the evaluation of small surface areas (~1.0 mm²) and multiple samples from a specimen¹⁷. No significant differences in microtensile bond strength were found among the groups tested in the present study. This finding contradicts some reports indicating that increased adhesive temperature immediately improves bond strength, regardless of the heating device or protocol used^{18,19}. This discrepancy may be explained by the possibility that the temperature increase promoted by the light-curing unit itself could have been enough to improve the physicochemical reactions and enhance monomeric permeation into the etched dentin^{16,20-22}. In addition, the solvent in Adper Single Bond 2 is based on ethanol, which reduces viscosity²³. Studies of acetone-based adhesives may present different results since the higher vapor pressure of acetone increases solvent evaporation^{22,24}. Furthermore, 37% phosphoric acid etching completely removes the smear layer and changes the energy surface of dentin². All of these factors could explain the lack of difference among the groups observed in the present study.

Both warming protocols could be relevant, considering that warmed air application to the adhesive could, in principle, be done clinically. A warming protocol presented in the literature as an option for *in vitro* tests has been found to increase bond strength values²⁴. Since heating promotes a forced evaporation of the solvent, it can alter the stoichiometric balance of the adhesive, thus affecting its permeation capability into the etched dentin^{10,19}. However, despite these advantages, warm air applied to heat up adhesives up to 20°,

30°C, and 50°C has failed to increase bond strength values in a previous study¹⁹. This latter finding corroborates that of the present study, mainly when considering some of the intrinsic characteristics of Adper Single Bond 2: (a) it is a one-bottle etch-and-rinse adhesive system wherein the solvent contains water and ethanol, rendering the adhesive less viscous, already favoring monomeric permeation, and (b) the 37% phosphoric acid etching prior to adhesive application completely removes the smear layer and promotes changes in the surface energy of the dentinal substrate. These factors could also explain the findings of the present study.

It is expected that studies assessing heated acetone-based adhesives could present different results from those found for ethanol-based ones. The explanation for this distinct behavior is probably linked to the vapor pressure of acetone, which is higher than that of ethanol, positively affecting solvent evaporation^{22,24}. The monomeric structure is not altered following a controlled temperature rise, and some interfacial degradation is expected to occur regardless of adhesive heating^{20,21,25}. Nevertheless, the long-term effects of heating on bond strength cannot be predicted based on the results of this study. To clarify the long-term influence of temperature on bonding, factors such as adhesive composition, solvent evaporation rates of acetone- and ethanol-based adhesives, and the clinical applicability of the proposed heating methods should also be considered.

CONCLUSION

In conclusion, the different heating methods used in this study had no influence on the microtensile bond strength of the etch-and-rinse ethanol-based adhesive system evaluated in this study. Therefore, adhesive heating seems pointless in the case of this adhesive system. Therefore, application of this adhesive according to its standard protocol and at room temperature would seem sufficient to achieve a satisfactory bond.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

*CORRESPONDING AUTHOR

Alexandre Henrique Susin, Departamento de Odontologia Restauradora, UFSM – Universidade Federal de Santa Maria, Rua Mal. Floriano, 1184, Centro, 97015-372 Santa Maria - RS, Brazil, e-mail: alexandre.susin@ufsm.br

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