Influence of Temperature on the Spreading Velocity of Simplified-Step Adhesive Systems

FLÁVIA BITTENCOURT PAZINATTO, DDS, MS* Luiz Marquezini Jr, DDS, MS* Maria Teresa Atta, DDS, MS, PhD[†]

ABSTRACT

Background: Flowability and viscosity vary for different adhesive systems owing to differences in their composition. These characteristics can be modified by environmental temperature.

Purpose: The purpose of this study was to determine the influence of temperature on the spreading (flow capacity) of simplified-step adhesive systems.

Materials and Methods: Spreading velocities of adhesive systems (Adper Single Bond and Single Bond Plus [3M ESPE, St. Paul, MN, USA]; Prime & Bond 2.1 and Prime & Bond NT [Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil]; Adper Prompt [3M ESPE]; and One Up Bond F [Tokuyama Corp., Tokyo, Japan]) were analyzed at intervals of 10, 15, 20, and 30 seconds at both 25°C and 37°C by placing 10 µL drops on a glass slide surface with an inclination of 45°. The spreading of each adhesive system was measured in millimeters per second.

Results: Data were analyzed by two-way analysis of variance and Student-Newman-Keuls tests. Regression analysis was used to determine a correlation between spreading velocity and time. Statistical significance was considered at a confidence level of 95%. Temperature influenced the spreading velocity, increasing it for Single Bond and Prime & Bond 2.1 and decreasing it for Adper Prompt (p < .05). No differences on spreading were observed for the other adhesives studied (p > .05). Regression analysis of each adhesive system demonstrated an inverse correlation between mean spreading velocity and time ($R^2 = .999$) on both temperatures.

Conclusions: Temperature increases yielded an increase of spreading for Single Bond and Prime & Bond 2.1. The influence of temperature on the spreading velocity was material dependent.

CLINICAL SIGNIFICANCE

Environmental temperature can influence the rate of spreading of the adhesive system in clinically relevant times and may influence adhesive thickness on cavity walls.

(J Esthet Restor Dent 18:38-46, 2006)

A dhesion requires an intimate contact between a liquid adhesive and a solid adherent.¹ A relevant criterion for resin restorations is good wetting of tooth sur-

faces by the adhesive systems.² According to Ruyter,³ adequate adhesion requires a good wetting capacity of the adhesive system, that is, a low contact angle between the adhesive and adherent. However, the wetting of a solid by a liquid is determined in part by the surface free energy of the solid and the liquid, the surface

*PhD student, Bauru Dental School, University of São Paulo, Bauru, São Paulo, Brazil *Assistant professor, Department of Operative Dentistry, Endodontics and Dental Materials of Bauru Dental School, University of São Paulo, Bauru, São Paulo, Brazil topography of the adherent, and the viscosity of the liquid.¹

Acid-etching of enamel and dentin causes different changes in the surface free energy of these substrates. The surface free energy of enamel is increased, facilitating wettability.³ However, the opposite occurs in dentin after mineral removal and exposure of organic components (collagen fibrils), which is responsible for reducing surface free energy.⁴ Hence, dentin requires an appropriate adhesive agent to increase the surface free energy of dentin, allowing good wettability and adhesion; this is usually performed by a primer agent or the first application of a single-bottle system.⁵ Additionally, the primer usually contains organic solvents that remove a fraction of the water present around the collagen fibrils after demineralization in the wet technique, allowing resin hydrophilic monomers to infiltrate the collagen network.

New dental adhesive systems (simplified-step systems) have been developed to be less time consuming as they combine the primer and adhesive in the same bottle (twostep systems). This is accomplished by creating a mixture of hydrophilic and hydrophobic monomers, with organic solvents. However, some simplified-step adhesive systems (single step, selfetching) present these hydrophilic and hydrophobic monomers in two bottles that need to be mixed before they are applied to the tooth substrate. The solvents in most adhesive systems are basically acetone, ethanol, or water (or a combination of the three), whereas the amount and type of resin monomers vary according to the manufacturer.

The heterogeneity of the composition of adhesive systems influences their surface free energy,^{1,6} and the concentration of each component contributes to the final viscosity of the material.⁷⁻⁹ The viscosity of each adhesive system developed is different and plays an important role in the wettability and spreading velocity on a solid surface.⁹ Wettability and the spreading velocity are also though to be dependent on the chemical composition of adhesive systems.¹⁰ The higher the viscosity of an adhesive, the more difficult it is to wet a substrate.¹¹

As the solvent evaporates from an adhesive system, the concentration of monomers increases.¹² The vapor pressures of solvents are temperature dependent, with acetone having the lowest vapor¹²; therefore, an increase of temperature may influence the pressure of solvents commonly used in dental adhesives.¹³ Thus, if more solvent evaporates from an adhesive system during its clinical application time, the remaining comonomers should be more viscous, which might inhibit wettability and adhesion.13 Increases in temperature decrease the viscosity of monomers.⁹ The influence of temperature on solvent

evaporation and monomer viscosities can be functionally evaluated by observing the spreading capacities of adhesive systems at several temperatures.¹³

Few have studied the adhesion mechanism between solids and liquids using an adhesive system as a wetting agent.¹⁴ Most studies have used water, which provides a low contact angle compared with that of adhesive agents.^{14,15} Water spreads and wets the dental substrates more effectively than most adhesives and tends to overestimate the wettability of substrates compared with solvated comonomers.

It is important to measure the spreading (flow capacity) of adhesive systems to a solid surface at different conditions of temperature to predict their behavior on cavity walls. The aim of this study was to test the null hypothesis that there is no difference in the spreading of adhesive systems even when environmental temperature changes.

MATERIALS AND METHODS

The spreading of six adhesive systems (see Table 1 for system and manufacturer information) on glass slides surfaces ($25 \times 75 \times 1$ mm) (Corning, Monterrey, Mexico) was evaluated. The spreading of each adhesive system was measured at a surface inclination of 45° to a horizontal surface to simulate an inclined surface for adhesion, such as that found in clinical restorative procedures after the removal of

| Adhesive System | Composition* | Batch No. |
|---|---|------------------|
| Adper Single Bond (3M ESPE Dental Products, St. Paul, MN, USA) | BIS-GMA, HEMA, polyalkenoic acid copolymer, dimethacrylates, camphorquinone, ethanol, water | 1FH |
| Adper Single Bond Plus (3M ESPE Dental Products) | Similar to classic Single Bond except for the addition of nanofillers | 005AA |
| Prime & Bond 2.1 (Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil) | Elastomeric BIS-GMA-diisocyanate adduct, UEDMA, BIS-DMA, PENTA, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone | 9875 |
| Prime & Bond NT (Dentsply Indústria e Comércio Ltda) | Di- and trimethacrylate resins, UDMA, PENTA, nanofillers (amorphous silicon dioxide), photoinitiators, stabilizers, cetylamine hydrofluoride, acetone | 0306000197 |
| One Up Bond F (Tokuyama Corp., Tokyo, Japan) | MMA, HEMA, coumarin dye, methacryloyloxyalkyl acid phosphate, methacryloxyundecane dicarboxylic acid (MAC-10), multifunctional methacrylic monomer, fluoramine silicate glass, photoinitiator (acrylborate catalyst), water | 455661C |
| Adper Prompt (3M ESPE) | Methacrylated phosphoric esters, BIS-GMA, initiators, stabilizers, HEMA, polyalkenoic acid, stabilizers, water | 133511 |
| BIS-DMA = bisphenol A dimethacrylate; BIS-GN | 1A = bisphenol A glycidyl methacrylate; HEMA = 2-hydroxyethyl methacryla | te; MMA = methyl |

BIS-DMA = bisphenol A dimethacrylate; BIS-GMA = bisphenol A glycidyl methacrylate; HEMA = 2-hydroxyethyl methacrylate; MMA = methyl methacrylate; PENTA = dipentaerythrytol pentacrylate phosphoric acid ester; UDMA = 1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylexane; UEDMA = urethane dimethacrylate . *As per manufacturers' information.

caries. This was performed using a protractor and a base to support the glass slides. A metallic millimeter ruler was placed on the back of the glass slides to measure the spreading of the adhesive systems.

Ten-microliter drops were placed onto the upper half of the glass slide with a micropipette (Pipetman, Gilson Medical Electronics S.A., Villiers Le Bel, France), and the spread of each drop was observed at 10, 15, 20, and 30 seconds and recorded in millimeters per second. For each adhesive system, the spreading was measured five times on the same glass slide. The glass slide was changed prior to testing another adhesive system. The measurements were made after the glass slide was cleaned with household detergent to remove any greasy contaminants, after which the slide was rubbed with acetone-soaked gauze and air blow dried. Groups and experimental conditions are summarized in Figure 1. None of the adhesives were light cured.

The experiment was carried out at controlled relative humidity at $50 \pm 5\%$ and controlled room temperatures of 25°C and 37°C. The temperatures were chosen to simulate the temperature of dentin in prepared teeth isolated by rubber dam, which is close to room temperature (25°C), and that when no rubber dam is used (37°C).

Statistical Analysis

Data were analyzed by a two-way analysis of variance (ANOVA; material and temperature). Multiple post hoc comparisons between pairs of means were performed using the Student-Newman-Keuls test. Regression analysis was used to determine a correlation between spreading velocity and time. Statistical significance was considered at a confidence level of 95%.

RESULTS

The results are shown in Table 2. Two-way ANOVA indicated a



Figure 1. Scheme of study groups and experimental conditions. ADP = Adper Prompt; OUB = One Up Bond F; PB2.1 = Prime & Bond 2.1; PBNT = Prime & Bond NT; SB = Adper Single Bond; SBP = Adper Single Bond Plus.

statistically significant difference for materials ($p \le .001$) but not for temperatures (p = .134). There was a significant interaction between material and temperature (p = .002). The Student-Newman-Keuls post hoc test showed significant differences between Adper Single Bond (SB), Prime & Bond 2.1 (PB2.1), and Adper Prompt (ADP) at both 25°C and 37°C (*p* < .001). When the temperature increased, SB and PB2.1 showed an increase in spreading velocity, whereas ADP showed a decrease. There were no statistically differences in the spreading velocity of the other materials with the different temperatures (p > .05).

Regression analysis of each adhesive system demonstrated a strong and inverse significant correlation between mean spreading velocity and time ($R^2 = .999$) at both 25°C and 37°C (Figures 2 and 3). The correlation between spreading velocity and time for the adhesive systems PB2.1 and PBNT could not be performed because the flow of these materials stopped before the shortest 10-second measurement time. Nevertheless, their mean spreading velocities for the temperatures studied were calculated as spreading distance per 30 seconds, as was done for all materials.

DISCUSSION

To minimize the high viscosity of monomers such as bisphenol A glycidyl methacrylate (BIS-GMA) or urethane dimethacrylate (UDMA), diluent monomers (triethylene glycol dimethacrylate [TEGDMA], 2-hydroxyethyl methacrylate [HEMA]) are included into adhesive systems formulations. The lower the viscosity of a solution, the more it facilitates wetting of substrates and promotes adhesion.¹¹ Additionally, hydrophilic monomers such as HEMA or 4-methacryloyloxyethyl trimellitate anhydride (4-META) facilitate comonomer infiltration into moist

| TABLE 2. SPREADING VELOCITIES OF ADHESIVE SYSTEMS AT DIFFERENT TEMPERATURES TESTED. | | | | | | | | |
|--|--|------------------------|-----------------------------|-----------------------------|--------------------------|-----------------------------|--|--|
| | Means ± SDs of Spreading Velocity (mm/s) | | | | | | | |
| Temperature | Adper Single Bond | Adper Single Bond Plus | Prime & Bond 2.1 | Prime & Bond NT | One Up Bond F | Adper Prompt | | |
| 25°C | $0.78 \pm 0.06^{\text{Ab}}$ | 0.50 ± 0.07^{Aa} | 1.21 ± 0.16^{Ac} | 1.25 ± 0.14^{Ac} | $0.70 \pm 0.03^{\rm Ab}$ | 1.16 ± 0.18^{Ac} | | |
| 37°C | $0.97 \pm 0.15^{\text{Bbc}}$ | 0.52 ± 0.09^{Aa} | $1.57 \pm 0.18^{\text{Bd}}$ | $1.15 \pm 0.11^{\text{Ab}}$ | 0.77 ± 0.11^{Ac} | $0.96 \pm 0.25^{\text{Bb}}$ | | |
| Uppercase letters in the same column and lowercase letters in the same row indicate mean values that are not statistically significant different $(p > .05)$. | | | | | | | | |



Figure 2. Regression analysis between spreading velocity and time at 25°C. ADP = Adper Prompt; OUB = One Up Bond F; SB = Adper Single Bond; SBP = Adper Single Bond Plus.

substrates, yielding a more uniform and less porous hybrid layer.¹⁶ The type and amount of solvents, monomers, and diluents in the mixtures influence viscosity, contact angle, and surface free energy.^{6–10}

The viscosities of BIS-GMA and UDMA at 20°C are, respectively, 1,200 and 23.1 Pa s. According to Silikas and Watts,⁹ when HEMA or TEGDMA are mixed with UDMA, the final viscosity decreases substantially with increases diluent concentrations. It has been found that high TEGDMA dilutions promote smaller contact angles to solid surfaces (glass, enamel, and dentine) than lower dilutions.8 However, TEGDMA concentrations are limited to those that sufficiently reduce the viscosity because this monomer slows the polymerization reaction.17 Besides, high amounts of TEGDMA lead to a higher concentration of double-bond conversion and higher post-gel shrinkage, providing a high ultimate shrinkage stress development.¹⁷ One study demonstrated that as increasing concentrations of solid 4-META were mixed with other monomers such as HEMA/urethane dimethacrylate (UEDMA) or TEGDMA/UEDMA, there was a reduction in contact angles, thus increasing wettability.⁶ These authors observed that both 4-META and HEMA increased the acid component of the monomer mixture, which is known to increase wettability.

The dilution of a viscous monomer decreases viscosity and allows adequate filler loading,⁸ but the importance of this loading is somewhat controversial.¹⁸ The inclusion of nanofillers in the formulation of an adhesive system is intended to increase adhesive viscosity to prevent its overthinning.^{19,20} This also provides a thicker adhesive layer,



Figure 3. Regression analysis between spreading velocity and time at 37° C. ADP = Adper Prompt; OUB = One Up Bond F; SB = Adper Single Bond; SBP = Adper Single Bond Plus.

thus functioning as an elastic buffer.^{21,22} The filler load or percent mass load differs between products according to manufacturers' technology and is not well described in the adhesives' composition. Nevertheless, there is little information about the shrinkage and stiffness of these filled adhesives after polymerization.¹⁹ The addition of nanofiller particles to adhesive systems does not provide a higher bond strength compared with unfilled systems.¹⁸ It has been suggested that filler may reduce adhesive penetration into etched dentin, producing a defective hybrid layer.¹⁸ Nanofiller particles tend to aggregate and are found congested around the tubular orifices but not within interfibrillar spaces.²³ Fillers or nanofillers may change the viscosity of some similar adhesives, as noted in SB and Adper Single Bond Plus (SBP). Nevertheless, this effect could not be observed using acetone-based PB2.1 and Prime & Bond NT (PBNT).

Adhesive systems have different formulations, resulting in different performance characteristics. The content of monomers, even though not specified by manufacturers, makes adhesive system more or less viscous. The choice of solvent of adhesive systems often depends upon the solubility of the adhesive comonomers.²⁴ Moreover, the percentage of solvent is not listed by the manufacturers since the final formulation is a proprietary secret. Solvents decrease the viscosity of the adhesive solution and thus may enhance the penetration of the bonding agent into demineralized dentin.¹³

The acetone-based adhesive systems (PB2.1 and PBNT) showed the greatest spreading velocities. Although acetone has a lower boiling temperature (56.5°C) and a higher vapor pressure (about 200 mm Hg) compared with the other components in the adhesive systems,^{12,25} its high concentration is probably responsible for the higher spreading velocity among the materials tested. Some authors have suggested that PB2.1 contains approximately 81 wt% acetone.¹³ However, as observed in the present study, this solvent has a high vapor pressure that facilitates solvent evaporation, regardless of temperature; both PB2.1 and PBNT quit spreading before 10 seconds.

The solvents of SB and SBP, ethanol and water, have vapor pressures of approximately 43.9 and 17.5 mm Hg.²⁶ These characteristics are quite different of those of acetone and may support the different spreading velocity results.

Although PBNT has nanofillers, its spreading velocity was not statistically different from PB2.1 at 25°C. Some authors have related that nanofillers probably do not increase the viscosity of PBNT, as observed in the present study.¹⁸ However, when the temperature increased to 37°C, the spreading velocity of PB2.1 increased. In contrast, PBNT showed similar spreading velocities at both temperatures tested. We speculate that at 37°C, acetone evaporated more rapidly and the presence of nanofillers increased the viscosity of the solvent-free comonomers, yielding a lower spreading velocity compared with that of PB2.1.

Single Bond Plus yielded a lower spreading velocity compared with SB, regardless of temperature. The addition of nanofillers in the former may explain these results. The spreading velocity of SB at 25°C was statistically lower than that at 37°C. This increase in the spreading velocity at 37°C is probably due to a decrease of monomer viscosity (HEMA and BIS-GMA) induced by the elevated temperature.9 A similar increase in spreading velocity was observed for PB2.1. Silikas and Watts observed that the viscosity of monomer mixtures decreased when temperature increased from 23°C to 60°C in their study of the rheology of UDMA and diluent formulations (HEMA, TEGDMA) at different concentrations.9

The solvent content of SB is approximately 31% of its mass.¹³ The present study showed that the spreading velocities of SB and SBP at 25°C were statistically lower than those for PB2.1 and PBNT, which is probably due to the solvent content in each adhesive system. However, when the temperature increased to 37°C, there were no statistically significant differences between SB and PBNT. Thus, other factors such surface free energy of monomers may have influenced the results.

Upon comparison of the two conventional adhesive systems with nanofillers (SBP and PBNT) at 37°C, it is noted that the spreading velocities were statistically lower than their unfilled counterparts (SB and PB2.1). However, PBNT showed a higher spreading velocity than SBP. Nanofillers and the amount/type of solvent may explain these observations, respectively.

The water-based self-etching adhesive systems showed different spreading velocities at the temperatures tested. One Up Bond F (OUB) presented similar spreading velocities, regardless of temperature. However, the spreading velocity of ADP decreased when temperature increased. OUB demonstrated a lower spreading velocity than did ADP. The presence of fluoramine silicate glass filler particles in OUB is probably responsible for the observed lower spreading velocity.

ADP, SB, and SBP contain BIS-GMA, HEMA, and polyalkenoic acids in their composition, but they spread quite differently at 25°C. It was surprising that when the temperature increased to 37°C, the SB velocity increased whereas that of ADP decreased, but they reached similar spreadings at the final measurements. Whereas the solvents in SB are water and ethanol, ADP has only water. Other components, or their interaction, in ADP may have more influence on the spreading velocity at 25°C than does the amount of solvent of SB.

It was noted that in all adhesive systems tested, the spreading velocities slowed over the 30 seconds, regardless of the temperature (see Figures 2 and 3). Presumably, this is due to a continued evaporation of the solvent content of each adhesive system, increasing the viscosity of the systems and, consequently, decreasing their spreading velocities in each time interval.²⁶

The presence of solvents in adhesive systems during the adhesion procedures is relevant in a variety of ways. Solvent evaporation is necessary prior to light curing of adhesive systems to avoid a negative influence on photopolymerization.²⁷ On the other hand, the solvent has an important function in adhesion, facilitating the removal of water and the infiltration of the adhesive system on the demineralized dentin substrate.²⁸ Thus, solvent evaporation should not occur before the hybridization of dental substrates because if an adhesive system does not infiltrate properly onto a demineralized substrate, it will create a poor-quality hybrid layer that is susceptible to degradation.²⁶

The results of this study suggest that adhesive systems spread differently over a clinically relevant period of time (30 s). If an adhesive spreads rapidly along a cavity wall, it can accumulate on the internal angles of cavity and yield different adhesive thicknesses, which can result in different levels of stress relief during composite shrinkage. On the other hand, if there is a uniformly thick layer of adhesive along the cavity border, although stress relief would be more uniform, there would be more water sorption into thicker hydrophilic resin layers.^{29,30}

CONCLUSIONS

The null hypothesis is rejected. Temperature increased the spreading velocity of adhesive systems for SB and PB2.1 but decreased it for ADP. The influence of temperature on the spreading velocity was material dependent. Knowledge of adhesive systems' composition and physical characteristics, such as viscosity, may be important to understanding their spreading characteristics on substrate surfaces. Further research is needed to observe the influence of solvent evaporation on the spreading velocity onto dental substrates.

DISCLOSURE AND Acknowledgments

The authors do not have any financial interest in the companies whose materials are discussed in this article. This work was partially supported by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) (no. 03/11721-9), Brazil. The authors gratefully acknowledge 3M ESPE, Dentsply, and Tokuyama for donating the adhesive systems studied.

REFERENCES

- Eick JD, Johnson LN, Fromer JR, et al. Surface topography: its influence on wetting and adhesion in a dental adhesive system. J Dent Res 1972;51:780–8.
- Combe EC, Owen BA, Hodges JS. A protocol for determining the surface free energy of dental materials. Dent Mater 2004;20:262–8.
- 3. Ruyter IE. The chemistry of adhesive agents. Oper Dent 1992;5:32–43.
- Van Meerbeek B, Lambrechts P, Ionokoshi S, et al. Factors affecting adhesion to mineralized tissues. Oper Dent 1992;5:111–24.
- Pashley DH, Horner JA, Brewer PD. Interactions of conditioners on the dentin surface. Oper Dent 1992;5:137–50.
- Asmussen E, Peutzfeldt A. Surface characteristics of adhesive monomers. Dent Mater 1998;14:21–8.
- 7. Asmussen E. Penetration of restorative resins into acid etched enamel I. Acta Odontol Scand 1977;35:175–82.
- Davy KWM, Kalachandra S, Pandain MS, Braden M. Relationship between composite matrix molecular structure and properties. Biomaterials 1998;19:2007–14.
- Silikas N, Watts DC. Rheology of urethane dimethacrylate and diluent formulations. Dent Mater 1999;15:257–61.
- Rosales-Leal JI, Osorio R, Holgado-Terriza JA, et al. Dentin wetting by four adhesive systems. Dent Mater 2001;17:526–32.

- Hisamatsu N, Atsuta M, Matsumura H. Effect of silane primers and unfilled resin bonding agents on repair bond of a prosthodontic microfilled composite. J Oral Rehabil 2002;29:644–8.
- Abate PF, Rodriguez VI, Macchi RL. Evaporation of solvent in one-bottle adhesives. J Dent 2000;28:437–40.
- Reis AF, Oliveira MT, Giannini M, et al. The effect of organic solvents on onebottle-adhesives' bond strength to enamel and dentin. Oper Dent 2003;28:700–6.
- Oh W-S, Shen C, Alegre B, Anusavice KJ. Wetting characteristic of ceramic to water and adhesive resin. J Prosthet Dent 2002;88:616–21.
- 15. Hitmi L, Bouter D, Degrange M. Influence of drying HEMA treatment on dentin wettability. Dent Mater 2002;18:503–11.
- Pashley DH, Carvalho RM. Dentine permeability and dentine adhesion. J Dent 1997;25:355–72.
- Feilzer AJ, Dauvillier BS. Effect of TEGDMA/BisGMA ratio on stress development and viscoelastic properties of experimental two-paste composites. J Dent Res 2003;82:824–8.
- Nunes MF, Swift E Jr, Perdigão J. Effects of adhesive composition on microtensile bond strength to human dentin. Am J Dent 2001;14:340–3.
- Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dent Mater 1999;15:128–37.
- Pashley DH, Pashley EL, Carvalho RM, Tay FR. The effects of dentin permeability on restorative dentistry. Dent Clin North Am 2002;46:211–45.
- 21. Montes MAJR, de Goes MF, Cunha MRB, Soares AB. A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats. J Dent 2001;29:435–41.
- Ausiello P, Apicella A, Davidson CL. Effect of adhesive layer properties on stress distribution in composite restorations—a 3D finite element analysis. Dent Mater 2002;18:295–303.

- Tay FR, Moulding KM, Pashley DH. Distribution of nanofillers from a simplifiedstep adhesive in acid-conditioned dentin. J Adhes Dent 1999;1:103–17.
- Kanca J III. Effect of primer dwell time on dentin bond strength. Gen Dent 1998;46: 608–12.
- Reis A, Loguercio AD, Azevedo CLN, et al. Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases. J Adhes Dent 2003;5:183–92.
- Gallo JR, Burgess JO, Xu X. Effect of delayed application on shear bond strength of four fifth-generation bonding systems. Oper Dent 2001;26:48–51.
- 27. Zheng L, Pereira PNR, Nakajima M, et al. Relationship between adhesive thickness and microtensile bond strength. Oper Dent 2001;26:97–104.
- Jacobsen T, Söderholm K-JM. Effect of primer solvent, primer agitation, and dentin dryness on shear bond strength to dentin. Am J Dent 1998;11:225–8.
- 29. Burrow MF, Inokoshi S, Tagami J. Water sorption of several bonding resins. Am J Dent 1999;12:295–8.
- Tanaka J, Ishikawa K, Yatani H, et al. Correlation of dentin bond durability with water sorption. Dent Mater J 1999; 18:11–8.

Reprint requests: Maria Teresa Atta, DDS, MS, PhD, Departamento de Dentística, Endodontia e Materiais Dentários-FOB/USP, Alameda Octávio Pinheiro Brisolla, 9-75, Bauru, SP, Brazil, CEP 17012-901; e-mail: tereatta@usp.br ©2006 BC Decker Inc Copyright of Journal of Esthetic & Restorative Dentistry is the property of Blackwell Publishing Limited and its content may not be copied or emailed to multiple sites or posted to a listserv without the copyright holder's express written permission. However, users may print, download, or email articles for individual use.