©Operative Dentistry, 2005, **30-3,** 376-381

Effect of Solvent Type on Microtensile Bond Strength of a Total-etch One-bottle Adhesive System to Moist, Dry Dentin

PC Cardoso • GC Lopes
LCC Vieira • LN Baratieri

Clinical Relevance

In this study, the type of organic solvent and dentin moisture had an influence on bond strength to dentin. The results showed that the application of a total-etch, ethanol-based adhesive system to moist dentin results in higher bond strengths.

SUMMARY

This study evaluated the effect of organic solvent (acetone or ethanol) on the microtensile bond strengths (MTBS) of an adhesive system applied to dry and moist dentin. Sixteen extracted human third molars were ground to expose a flat occlusal dentin surface and acid etched for 20 seconds (20% phosphoric acid gel, Gluma Etch 20 Gel, Heraeus/Kulzer). After rinsing the acid etchant, an ethanol-based one-bottle adhesive system was applied to the mesial half of the occlusal dentin surface. An acetone-based, one-bottle adhesive system was applied to the distal half of the ground dentin surface. The teeth were randomly assigned to groups. In Group 1, the etched dentin was thoroughly air dried and an ethanol-based one-bottle adhesive system was applied (Gluma Comfort Bond, Heraeus/Kulzer) (GCB). In Group 2, the etched dentin was thoroughly air dried and an acetone-based one-bottle adhesive system was applied (Gluma One Bond, Heraeus/Kulzer)(GOB). In Group 3 (Gluma Comfort Bond), excess moisture was removed after acid etching, leaving a moist dentin surface and a one-bottle ethanol-based adhesive was applied. In Group 4 (Gluma One Bond), excess moisture was removed after acid etching, leaving a moist dentin surface and an acetone-based adhesive was applied. A hybrid resin composite (Venus, Heraeus/Kulzer) was applied to the bonded surface in four 1-mm increments and light cured according to manufacturer's directions. The specimens were then sectioned with a slow-

Paula de Carvalho Cardoso, DDS, MS, graduate student, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

*Guilherme Carpena Lopes, DDS, MS, PhD, clinical instructor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

Luiz Clovis Cardoso Vieira, DDS, MS, PhD, professor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

Luiz Narciso Baratieri, DDS, MS, PhD, professor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

*Reprint request: Rua Laurindo Januário da Silveira n° 947, Apto 34A, Lagoa da Conceição, Florianópolis, Santa Catarina, Brazil; e-mail: guilherme_lopes@ig.com.br

speed diamond saw in two perpendicular directions to obtain sticks with a cross-section of 0.5 ± 0.05 mm^2. The microtensile bond strength (MTBS) test was performed with a Bencor device in an Instron machine at a crosshead speed of 0.5 mm/minute. The data were subjected to a two-way ANOVA and Scheffé *Post hoc* test (superscript letters, *p*<0.05). The experimental MTBS measured for dry dentin were Group 1=37.0 ± 10.6^b and Group 2=34.7 ± 9.0^b in MPa (mean ±SD); and on moist dentin, Group 3=50.7 ± 11.0^a and Group 4=38.5 ± 10.5^b in MPa (mean ±SD). The ethanol based adhesives resulted in higher MTBS than acetone-based adhesive (*p*<0.008) and bonding to moist dentin resulted in higher MTBS (*p*<0.001). GCB applied on moist dentin resulted in statistically higher bond strengths than the other groups. The highest MTBS were achieved with the use of an ethanol-based adhesive to moist dentin.

INTRODUCTION

Recent advances in aesthetic restorations have driven significant improvements in dental adhesive systems. The objectives are to establish an effective adhesion to dental structure (Bowen, Cobb & Rapson, 1982). Bonding to enamel is well known as being clinically very reliable. Since its introduction in 1955, the acid-etch-technique has provided an ideal surface morphology, because of phosphoric acid (Buonocore, 1955; Swift, Perdigão & Heymann, 1995; Lopes & others, 2002). Although this technique has revolutionized dentistry over the last 20 years, dentin bonding is still a challenge due to the wet tubular ultra structure and heterogeneous, partially organic composition of dentin substrate (Pashley, 1989; Pashley, 1991; Erickson, 1992; Eick & others, 1993).

For contemporary adhesive systems, dentin bonding requires the removal or modification of the smear layer and superficial demineralization through the application of an acid etchant (Gwinnett, 1994). Although chemical reactions between chemical bonding agents and dentin have been reported, it is generally accepted that dentin bonding relies primarily on micro-mechanical interaction similar to enamel bonding, mediated by the permeation of resin monomers into acid-etched dentin (Fusayama & others, 1979; Nakabayashi, Kojima & Masuhara, 1982). The entanglement of polymerized adhesive resin with collagen fibrils and residual hydroxyapatite crystals generates an interfacial structure called the “hybrid layer” or “resin-dentin inter-diffusion zone” (Nakabayashi & others, 1982).

Exposure of the collagen fiber network by acid etching creates favorable conditions for micro-mechanical retention of an adhesive system, but the collagen network can collapse on itself due to the loss of structural support (Pashley, Horner & Brewer, 1992). Furthermore, if the exposed collagen is air dried before the bonding procedure, it may collapse over the underlying unaffected dentin (Pashley & others, 1992; Swift & others, 1999). These two phenomena create a layer of residual, denatured and collapsed collagen on top of the demineralized dentin, which monomers may be unable to penetrate and diffuse into because of limited porosity (Pashley & others, 1992; Gwinnett, 1992). Thus, when demineralized collagen is kept moist, the fibrils are observed as being upright and separated by wide interfibrillar spaces, resulting in better opportunities for resin infiltration and higher bond strengths when compared with those that had been briefly air dried (Nakabayashi & others, 1982; Kanca, 1992a).

The efficacy of dentin adhesives seems to be improved by the addition of high vapor pressure organic solvents to the adhesives’ chemical formulations. Acetone and ethanol are commonly used solvents found in the majority of current bonding systems. These chemical agents function as “water-chasers” and solubilize resin components. They increase the wettability of the dentin substrate by the bonding systems and help to replace water in the acid-etched, rinsed dentin surface with hydrophilic resin monomers (Kanca, 1992a). High bond strengths are obtained with bonding agents dissolved in high vapor-pressure solvents such as acetone or ethanol when acid-etched dentin is left visibly moist (Kanca, 1992a,b; Perdigão, Swift & Cloe, 1993). The moisture dependence of dentin is related to the type of solvent in the adhesives chemical formulations. Ethanol-based adhesive systems seem less sensitive to the amount of moisture in dentin (Jacobsen & Söderholm, 1998; Finger & Balkenhol, 1999; Reis & others, 2003). However, this reduction in sensitivity has not been tested with adhesives with the same monomer composition.

This study evaluated the effect of an organic solvent (acetone or ethanol) on the microtensile bond strengths (MTBS) of an adhesive system applied to dry and moist dentin. The null hypothesis tested was that: 1) bonding strength would not be dependent on dentin moisture after acid etching and 2) the type of solvent would not have an influence on bond strength to dentin.

METHODS AND MATERIALS

The specimens utilized in this study were 16 extracted human third molars that were stored in saline solution at room temperature. The occlusal enamel and roots were removed and the occlusal surface was ground under running water to expose a flat dentin surface parallel to the occlusal surface. The specimens were inspected with magnification glasses for enamel remnants. The exposed dentin surface was polished with a series of wet silicon carbide paper (220, 360 and 600-grid). The specimens were divided in the middle using a round diamond bur (KG Sorensen, Barueri, Brazil) in

Table 1: *Chemical Composition of Adhesive Systems Tested*

Bonding Agent	Manufacturer	Etching Gel	Chemical Composition	Solvent Type
Gluma One Bond (GOB)	Heraeus-Kulzer	20% phosphoric acid	UDMA, HEMA, 4-META	Acetone
Gluma Comfort Bond (GCB)	Heraeus-Kulzer	20% Phosphoric acid polyacrylic and dicarboxylic acids	UDMA, HEMA, 4-META,	Ethanol/water

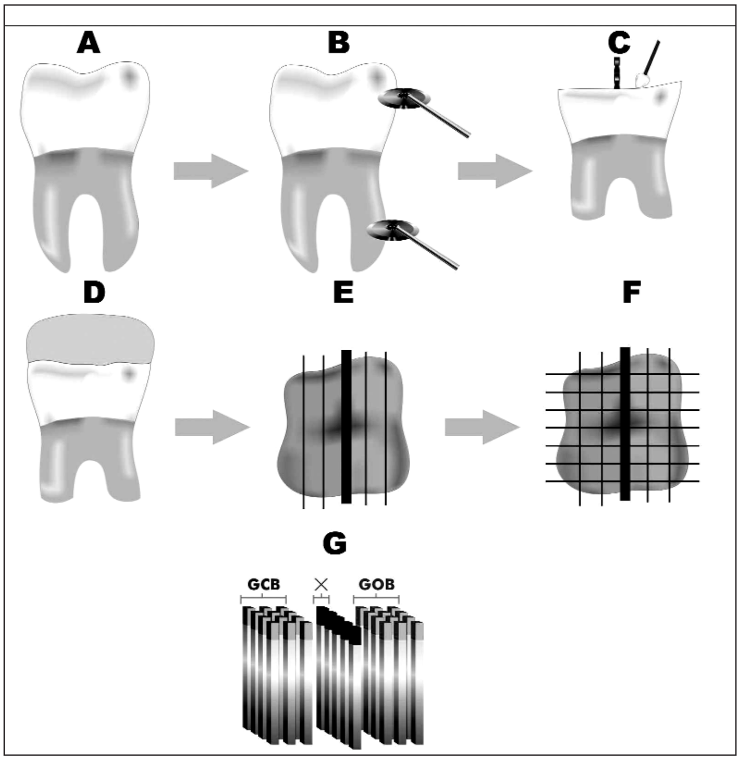

Figure 1. *Schematic illustrating the essence of the microtensile bond testing technique. A, Human third molar. B, the occlusal enamel, and roots were removed. C, Tooth was divided in two parts for a matrix, each half, corresponding to the type of adhesive system. D, Resin composite (Venus, shade A3) was applied in four layers to a height of 4 to 5 mm. E, Restored tooth was sectioned into buccal-lingual parallel slabs 0.7-mm thick. F, New sections of 0.7-mm thick were performed in each slice perpendicular o the first section and the slabs converted into sticks. G, Sticks representative of adhesive system were reserved for testing and sticks with black base were discarded.*

a high-speed handpiece under continuous water cooling. A metallic matrix (Microdont, São Paulo, Brazil) was positioned on the sectioned specimen and the adhesive procedures were carried out. After rinsing the acid etchant, an ethanol-based one-bottle adhesive system was applied to the mesial half of the occlusal dentin surface. On the distal half, an acetone-based one-bottle adhesive system was applied. The teeth were randomly assigned to the experimental groups. Two commercially available dentin bonding systems were used (see Table 1 for a description of materials):

Group 1–Gluma Comfort Bond (GCB), Dry: 20% phosphoric acid gel (Gluma Etch 20 Gel; Heraeus/Kulzer) was applied for 20 seconds to a dry dentin surface and rinsed thoroughly with an air-water syringe for 10 seconds. The etched dentin was dried for 10 seconds with an air syringe at a distance approximately 2 cm from the surface (dry technique). GCB, an ethanol-based one-bottle bonding agent, was applied in two consecutive coats with a saturated disposable brush lightly dried for two seconds to evaporate the solvent and light cured for 20 seconds.

Group 2–Gluma One Bond (GOB), Dry: Specimens were treated in the same manner as Group 1, except that an acetone-based one bottle bonding agent was used.

Group 3–Gluma Comfort Bond (GCB), Moist: 20% phosphoric acid gel (Gluma Etch 20 Gel; Heraeus/Kulzer) was applied for 20 seconds to a dry dentin surface and rinsed thoroughly with an air-water syringe for 10 seconds at a distance approximately 2 cm from the surface. The etched dentin was partially dried by blotting with a small piece of absorbent paper, leaving a visibly moist surface (wet technique). GCB, an ethanol-based one-bottle bonding agent, was applied in two consecutive coats with a saturated disposable brush, lightly dried for two seconds to evaporate the solvent and light cured for 20 seconds.

Group 4–Gluma One Bond (GOB), Moist: Specimens were treated in the same manner as Group 3, except that an acetone-based one-bottle bonding agent was used.

A hybrid resin composite (Venus, shade A3, Heraeus Kulzer) was applied in four layers to a height of 4 to 5 mm. Each layer was light cured for 20 seconds with a light-curing unit (Optilux 400, Kerr/Demetron, Danbury, CT, USA). The light intensity was measured with a radiometer (Demetron/Kerr) and resulted in 500 mW/cm^2.

The specimens were stored in water at room temperature for 24 hours before being sectioned. They were bonded with a cyanoacrylate-based adhesive (Zap-It, DVA, Corona, CA, USA) to acrylic cylinders for easier handling during cutting operations. The restored teeth were attached to a cutting machine (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA), where a diamond disc (South Bay Technology, San Clemente, CA, USA) running at a low speed with water coolant was used to cut the specimens into approximately 0.7-mm thick slices running in a buccal-lingual direction. The slices were then trimmed with a diamond bur to obtain a surface area of 0.5±0.05 mm^2. A total of 12 stick specimens were prepared for each group. These were stored in water for 24 hours at room temperature (Figure 1) before taking the MTBS measurements.

In preparation for the tensile test, the beams were bonded with a quick polymerizing cianoacrylate-based adhesive (Zap-It, DVA) in a special jig for microtensile tests (Bencor Multi-T, Danville Engineering, San Ramon, CA, USA). Then, an Instron Universal testing machine (Model 4444, Instron Corp, Canton, MA, USA) operating at a speed of 0.5 mm/minute was used and the MTBS data were recorded. Before the test, the area next to the adhesive interface of the sticks specimens was computed using the Series IX Software System (Instron Corp).

The data were subjected to two-way ANOVA (Independent variable: solvent type and dentin moisture; outcome variable: MTBS) and *Post hoc* test (Scheffé test) ($p<0.05$). The statistical analysis was carried out with the SPSS 10.0 (SPSS Inc, Chicago, IL, USA) software package.

Table 2: *Microtensile Bond Strength to Dentin and SD*

Group	MTBS (Mean ± SD)
Gluma Comfort Bond, Dry	37.0±10.6[B]
Gluma One Bond, Dry	34.7±9.0[B]
Gluma Comfort Bond, Moist	50.7 ± 11[A]
Gluma One Bond, Moist	38.5±10.5[B]

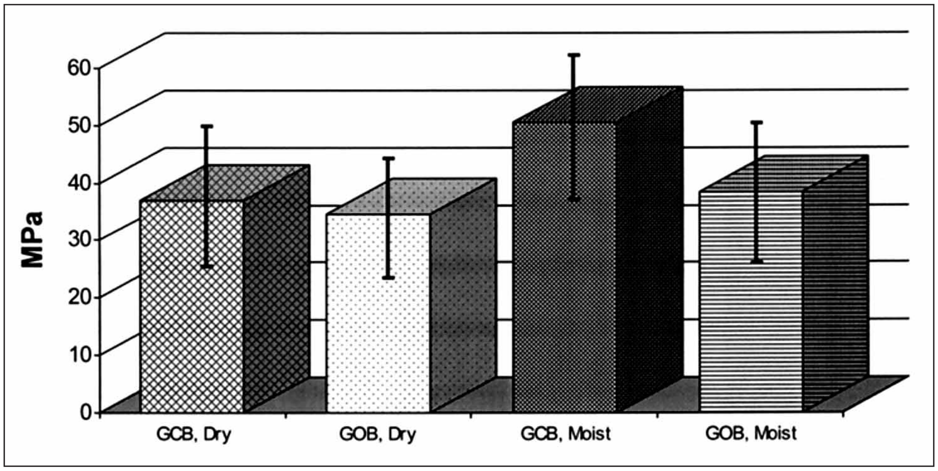

Figure 2. *Mean microtensile bond strengths to dentin (MPa).*

RESULTS

The mean MTBS in MPa, standard deviation, are shown in the Table 2 and Figure 2. The analysis of variance revealed a statistically significant difference between pairs of means for the factor "solvent type," with the ethanol based adhesives higher than the acetone-based ($p<0.008$) adhesives. The *post hoc* test (Scheffé test) ranked this difference in two (Table 2). Gluma Comfort Bond, ethanol-based and applied on moist dentin, resulted in a significantly greater mean MTBS (50.7 ± 11.0 MPa) when compared with either Gluma Comfort Bond (GCB), dry (37.0 ± 10.6 MPa) or Gluma One Bond, moist (38.5 ± 10.5 MPa) and dry (34.7± 9.08 MPa). Bonding to moist dentin resulted in higher MTBS ($p<0.001$). There were no statistically significant interactions between the independent variables "solvent type" and "dentin moisture" ($p<0.069$).

DISCUSSION

The first null hypothesis was rejected. Bonding to wet dentin resulted in statistically higher bond strengths than to dry dentin. This result suggests that the moist bonding technique promotes better infiltration into demineralized dentin than the dry bonding technique. Such results are confirmed by other *in vitro* studies (Gwinnett & Kanca, 1992; Swift & Triolo, 1992; Perdigão & others, 1993) which concluded that bonding to moist dentin is more effective in terms of bond strength than techniques that air-dry the acid-etched dentin prior to priming. However, at least one researcher reported that the dry and moist techniques did not affect bond strengths (Miears, Charlton & Hermesch, 1995). It is interesting to note that the higher bond strength to wet dentin obtained in this study does not necessarily hold in microleakage tests, where at least two dye-penetration studies showed that microleakage around the dentin margins of composite restorations was similar, irrespective of the wet or dry bonding technique (Vargas & Swift, 1994; Santini & Mitchell, 1998).

The second null hypothesis was rejected; the ethanol–based bonding system (GCB) resulted in higher MTBS than the acetone-based system tested (GOB). According to Lopes and others (2004) and Ritter, Bertoli and Swift (2001), the ethanol-based adhesive had higher bond strengths when compared to all other acetone-base systems, confirming the results of this study. Moreover, other characteristics of acetone, such as its high volatilization (Van Meerbeek, Vargas & Inoue, 2001) and quick evaporation from an open bottle (Perdigão, Swift & Lopes, 1999) makes it a less favorable solvent option.

The application of a total-etch ethanol-based adhesive system to moist dentin results in higher bond strength than to dry dentin. For the acetone-based adhesive system, MTBS was not affected by the degree of dentin wetness after acid etching, resulting in similar bond strength to moist or dry dentin. These results are contradicted in the literature data by Jacobsen and Söderholm (1998), Finger and Balkenhol (1999) and Reis and others (2003), who affirm that acetone-based adhesive systems are more dependent on an accurate wet bonding technique than ethanol-based adhesives. However, these studies compared adhesive systems with different solvent and monomer compositions. In this study, the only difference between the adhesives was the solvent type. Another possible explanation for this disagreement could be differences between the monomer composition of the Gluma systems and the other adhesives tested in these cited studies. GOB and GCB are based on a 4-META monomer. This monomer was not present in the composition of other adhesive systems tested in the cited studies, which used adhesives such as Single Bond, One-Step and Syntac Single Component. Therefore, either the difference in monomer composition or the solvent concentrations could be the reason for the results obtained.

The microtensile test (MTBS) produces a more consistent measurement of bond strengths than the conventional shear bond test (Sano & others, 1994). The microtensile test also allows for the measurement of bond strengths using bonding surfaces with a cross-sectional area in the range of 0.5 to 1.5 mm^2. Other advantages over conventional shear and tensile bond strength tests include the use of only one tooth to make several bonded interfaces, testing distinct substrates, such as sclerotic dentin and carious dentin and testing regional differences in bond strengths within the same tooth (Pashley & others, 1995). According to Pashley and others (1999), microtensile bond testing methods offer versatility that cannot be achieved by conventional methods. It is more labor-intensive than conventional testing but holds great potential for providing insight into the strength of adhesion of restorative materials used in dentistry. In conclusion, due to the use of MTBS in the current work, the variation coefficient was reduced and permitted the use of two different adhesive systems on the same dentin substrate.

Under the limitations of this *in vitro* study, further clinical studies should test the same dentin adhesives. Also, additional research, where the bonding systems are applied *in vivo* to reproduce clinical conditions such as a positive intra-pulpal pressure and the presence of the fluid in the dentinal tubules, is suggested to verify its possible influence on bond strength to moist and dry dentin.

CONCLUSIONS

Based on results of this study, the use of an ethanol-based adhesive system to moist dentin resulted in higher MTBS.

(Received 2 February 2004)

References

Bowen RL, Cobb EN & Rapson JE (1982) Adhesive bonding of various materials to hard tooth tissues: Improvement in bond strength to dentin *Journal of Dental Research* **61(9)** 1070-1076.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.

Erickson RL (1992) Surface interactions of dentin adhesive materials *Operative Dentistry* **Supplement 5** 81-94.

Eick JD, Robinson SJ, Chappell RP, Cobb CM & Spencer P (1993) The dentinal surface: Its influence on dentinal adhesion Part III *Quintessence International* **24(8)** 571-582.

Finger WJ & Balkenhol M (1999) Practitioner variability effects on dentin bonding with an acetone-based one-bottle adhesive *Journal of Adhesive Dentistry* **1(4)** 311-314.

Fusayama T, Nakamura M, Kurosaki N & Iwaku M (1979) Non-pressure adhesion of new adhesive restorative resin *Journal of Dental Research* **58(4)** 1364-1370.

Gwinnett AJ & Kanca JA 3rd (1992) Micromorphology of the bonded dentin interface and its relationship to bond strength *American Journal of Dentistry* **5(2)** 73-77.

Gwinnett AJ (1992) Moist versus dry dentin: Its effect on shear bond strength *American Journal of Dentistry* **5(3)** 127-129.

Gwinnett AJ (1994) Altered tissue contribution to interface bond strength with acid conditioned dentin *American Journal of Dentistry* **7(5)** 243-246.

Jacobsen T & Söderholm KJ (1998) Effect of primer solvent, primer agitation and dentin dryness on shear bond strength to dentin *American Journal of Dentistry* **11(5)** 225-228.

Kanca J 3rd (1992a) Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin *American Journal of Dentistry* **5(4)** 213-215.

Kanca J 3rd (1992b) Improving bond strength through acid etching of dentin and bonding to wet dentin surfaces *Journal of the American Dental Association* **123(9)** 35-43.

Lopes GC, Baratieri LN, de Andrada MA & Vieira LC (2002) Dental adhesion: Present state of the art and future perspectives *Quintessence International* **33(3)** 213-224.

Lopes GC, Cardoso PC, Vieira LCC, Baratieri LN, Rampinelli K & Costa G (2004) Shear bond strengths of acetone-based one-bottle adhesive systems *Brazilian Dental Journal* (accepted for publication).

Miears JR Jr, Charlton DG & Hermesch CB (1995) Effect of dentin moisture and storage time on resin boding *American Journal of Dentistry* **8(2)** 80-82.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal Biomedical Materials Research* **16(3)** 265-273.

Pashley DH (1989) Dentin: A dynamic substrate—a review *Scanning Electron Microscopy* **3(1)** 161-176.

Pashley DH (1991) Dentin bonding: Overview of the substrate with respect to adhesive material *Journal of Esthetic and Restorative Dentistry* **3(2)** 46-50.

Pashley DH, Horner JA & Brewer PD (1992) Interaction of conditioners on dentin surface *Operative Dentistry* **Supplement 5** 137-150.

Pashley DH, Sano H, Ciucchi B, Yoshiyama M & Carvalho RM (1995) Adhesion testing of dentin bonding agents: A review *Dental Materials* **11(2)** 117-125.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Perdigão J, Swift EJ Jr & Cloe BC (1993) Effects of etchants, surface moisture, and resin composite on dentin bond strengths *American Journal of Dentistry* **6(2)** 61-64.

Perdigão J, Swift EJ Jr & Lopes GC (1999) Effects of repeated use on bond strengths of one-bottle adhesives *Quintessence International* **30(12)** 819-823.

Reis A, Loguercio AD, Azevedo UN, Singer JM, Carvalho RM & Grande RH (2003) Moisture spectrum of demineralized dentin for adhesive systems wtih different solvent-bases *Journal of Adhesive Dentistry* **5(3)** 183-192.

Ritter AV, Bertoli C & Swift EJ Jr (2001) Dentin bond strengths as a function of solvent and glutaraldehyde content *American Journal of Dentistry* **14(4)** 221-226.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho RM & Pashley DH (1994) Relationship between surface area for adhesion and tensile bond strength—Evaluation of a microtensile bond test *Dental Materials* **10(4)** 236-240.

Santini A & Mitchell S (1998) Microleakage of composite restorations bonded with three new dentin bonding agents *Journal of Esthetic Dentistry* **10(6)** 296-304.

Swift EJ Jr, Perdigão J & Heymann HO (1995) Bonding to enamel and dentin. A brief history and state of the art *Quintessence International* **26(2)** 95-110.

Swift EJ Jr, Perdigão J, Heymann HO & Ritter AV (1999) Shear bond strengths of one-bottle adhesives to moist enamel *Journal of Esthetic Dentistry* **11(2)** 103-107.

Swift EJ Jr & Triolo PT Jr (1992) Bond strengths of Scotchbond Multi-purpose to moist dentin and enamel *American Journal of Dentistry* **5(6)** 318-320.

Van Meerbeek B, Vargas M & Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **Supplement 6** 119-144.

Vargas MA & Swift EJ Jr (1994) Microleakage of resin composites with wet versus dry bonding *American Journal of Dentistry* **7(4)** 187-189.