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Effects of Resin Hydrophilicity on Dentin Bond Strength

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Abstract

The purpose of this study was to determine if hydrophobic resins can be coaxed into dentin wet with ethanol instead of water. The test hypothesis was that dentin wet with ethanol would produce higher bond strengths for hydrophobic resins than would dentin wet with water. This study examined the microtensile bond strength of 5 experimental adhesives (50 wt% ethanol/50% comonomers) of various degrees of hydrophilicity to acid-etched dentin that was left moist with water, moist with ethanol, or air-dried. Following composite buildups, hourglass-shaped slabs were prepared from the bonded teeth for microtensile testing. For all 3 types of dentin surfaces, higher bond strengths were achieved with increased resin hydrophilicity. The lowest bond strengths were obtained on dried dentin, while the highest bond strengths were achieved when dentin was bonded moist with ethanol. Wet-bonding with ethanol achieved higher bond strengths with hydrophobic resins than were possible with water-saturated matrices.

Keywords

dentin bonding; hydrophilic resins; wet-bonding; ethanol

INTRODUCTION

The current generation of dentin adhesives has been criticized as being too hydrophilic (Tay and Pashley, 2003) and absorbing too much water, which lowers their stiffness when compared with that of more hydrophobic resins (Ito *et al.*, 2005). Hydrophilic all-in-one self-etching adhesives yield lower bond strengths than do more hydrophobic etch-and-rinse adhesives (De Munck *et al.*, 2005). The bond strengths of self-etching hydrophilic adhesives can be significantly increased when they are covered with a solvent-free, more hydrophobic adhesive (Brackett *et al.*, 2005; King *et al.*, 2005). Presumably, if dentin is bonded with more hydrophobic resins, they would absorb less water, plasticize less (Ito *et al.*, 2005), and produce more durable bonds.

The challenge is how to coax hydrophobic monomers into a hydrophilic matrix without inducing phase changes (Spencer and Wang, 2002). Most of the "hydrophilicity" of acid-etched dentin matrices is due to the presence of water. After dentin is acid-etched, water replaces the volume of dentin previously occupied by mineral (*ca.* 50 vol%; Kinney *et al.*, 2003). Since this water surrounds and wets the collagen fibrils, adhesive monomers must displace water from collagen if they are to develop an intimate contact with collagen fibrils. However, because the molecular weight of dental monomers ranges from 100–580 g/mol, their molar

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concentrations (0.3–5 mol/L) are but a fraction of the molar concentration of water (55.6 mol/L). Thus, even hydrophilic monomers such as hydroxyethyl methacrylate (HEMA) cannot displace much water from collagen fibrils. Hydrophobic dimethacrylates that create strong polymer networks by cross-linking polymer chains have very low solubilities in water. These problems may be avoided if ethanol (17.1 mol/L) is exchanged for the water within acid-etched dentin. Most dental monomers, including dimethacrylates like BisGMA, are soluble in ethanol. With ethanol filling the interfibrillar spaces, the dental matrix becomes much more hydrophobic. As ethanol replaces water, some interpeptide hydrogen bonding (H-bonding) develops, within collagen, that stiffens the matrix enough to minimize its shrinkage (Eddleston *et al.*, 2003; Garcia *et al.*, 2005; Becker *et al.*, 2006). This should lead to better infiltration of hydrophobic dimethacrylates into ethanol-saturated matrices, than into water-saturated matrices. The more resin (both mono- and dimethacrylates) that infiltrates acid-etched matrices, the higher are the resin-dentin bond strengths.

The purpose of this study was to compare resin-dentin bond strengths of hydrophobic vs. hydrophilic resins bonded to acid-etched dry dentin or dentin wet with water or ethanol. The test null-hypotheses were that resin hydrophilicity has no effect on microtensile bond strengths, and that bonds made to dentin wet with alcohol are no different from those made to dentin wet with water.

MATERIALS & METHODS

Sixty extracted, unerupted human third molars—obtained with informed patient consent under a protocol approved by the Human Assurance Committee of the Medical College of Georgia—were stored in 0.9% NaCl containing 0.02% sodium azide at 4°C until used. Using a diamond-impregnated copper disk, we removed the occlusal enamel and superficial dentin with water irrigation (Isomet saw, Buehler Ltd., Lake Bluff, IL, USA). The tooth surface was ground with #320-grit silicon carbide paper, creating a standardized smear layer. The study design consisted of 5 experimental resins $\times$ 3 surface conditions $\times$ 4 teeth *per* group.

The use of Hoy's solubility parameters to define the degree of hydrophilicity or hydrophobicity of solvated comonomer blends (Miller *et al.*, 1998; Chappelow *et al.*, 2000; Pashley *et al.*, 2001, 2002; Eddleston *et al.*, 2003) provides a useful method for ranking dentin adhesive hydrophilicity. Since manufacturers refuse to reveal the exact composition of their products, the Hoy's solubility parameters of commercially available dentin adhesives cannot be calculated. In this study, 5 comonomer blends were formulated based on known concentrations of all ingredients, including the solvents, so that their Hoy's solubility parameters could be calculated. The Hoy's solubility parameters of 50 wt% ethanol/resin mixtures were also calculated. Similar calculations were made for Hoy's solubility parameters for dentin matrices saturated with water or ethanol that occupied 30% of the volume of the matrix, with collagen occupying the remaining 70%. The details of how Hoy's solubility parameters for collagen were calculated have been previously published (Agee *et al.*, 2006; Becker *et al.*, 2006).

Experimental Resins

The compositions of the 5 experimental resins, together with the Hoy's solubility parameters of these comonomers solvated in 50 wt% ethanol, are given in Table 1. We calculated the latter by summing the group molar attraction constants of their structures (Fig. 1) according to the method of Hoy, using commercially available software (Computer Chemistry Consultancy, www.compchemconsul.com). We used Hoy's solubility parameters to rank the degree of hydrophilicity of solvent/monomer mixtures. Resins 1 and 2 represent non-solvated hydrophobic resins used in the final step of three-step, etch-and-rinse, and two-step, self-etching adhesives. Resin 3 represents the formulation of typical two-step, etch-and-rinse

adhesives, while resins 4 and 5 correspond to very hydrophilic one-step, self-etching adhesives containing carboxylic- or phosphate-substituted methacrylates, respectively.

Bonding Procedures

We formulated experimental adhesives 1–5 by combining 50 wt% comonomer mixtures 1–5 with 50 wt% ethanol (Table 1). All dentin surfaces were acid-etched with 37% phosphoric acid gel (Etch 37, Bisco Inc., Schaumburg, IL, USA) for 15 sec, then were rinsed for 15 sec with water. The acid-etched teeth were randomly divided into 3 surface-treatment groups of 20 teeth each. Four teeth in each group were bonded with 1 of the 5 experimental resins. In Group 1, the teeth were blot-dried to leave a water-moist surface just before being bonded. In Group 2, teeth were treated with 100% ethanol delivered from a squeeze bottle, to replace rinsing water with ethanol. After 20 sec, the excess ethanol was removed with the use of absorbent pellets just before teeth were bonded. The ethanol-saturated dentin surface appeared moist with ethanol just prior to the application of ethanol-solvated comonomer mixtures. In Group 3, the teeth were strongly dried with an air-syringe for 15 sec just prior to the application of ethanol-solvated blends. There were 5 resin subgroups within the 3 bonding-surface groups.

During bonding, a generous amount of each experimental adhesive was applied to the dentin, by means of a microbrush, with agitation for 15 sec. A second application of fresh adhesive was made, giving a total application time of 30 sec. Excess solvent was evaporated with a gentle air stream for 10 sec at a distance of 15 cm, and then the adhesive was light-cured for 40 sec by means of an Optilux 500 halogen light-curing unit (Demetron/Kerr, Danbury, CT, USA) with a power output of 600 mW/cm². Resin composite build-ups were made with three 1.5-mm increments of AP-X composite (Kuraray Medical Inc., Tokyo, Japan) that were individually light-cured for 40 sec. All bonded teeth were incubated in 37°C water for 24 hrs.

Tensile Testing

Using the Isomet saw, we vertically sectioned the bonded teeth into 0.7-mm-thick serial slabs. The central region of each slab, in turn, was trimmed into hourglass-shaped specimens (0.49 mm² cross-section). Only the central 4 slabs were used from each tooth. Thus, there were 4 slabs × 4 teeth = 16 specimens in each subgroup. Modes of failure were classified as adhesive (A), cohesive in resin (C), or mixed (M) failures when the failed bonds were examined at 30X by stereoscopic microscopy.

All tensile testing was performed with the use of a Bisco testing jig mounted in a Vitrodyne V-1000 universal tester at a cross-head speed of 1 mm/min. The specimens were glued to the testing jig by cyanoacrylate (Zapit, Dental Ventures of America, Corona, CA, USA).

Statistical Analyses

We used a two-way ANOVA design with the general linear model for examining the effects of dentin surface condition and resin hydrophilicity on microtensile bond strengths (μ TBS). The least-squares means (LSM) analysis was used, due to significant interactions between the 2 factors. Multiple comparisons of the LSM were performed by the Holm-Sidak method. LSM are the expected value of group or subgroup means that one expects for a balanced design involving the group variable, with all covariates at the mean value. The variances in LSM values are given in standard error of the mean (SEM) instead of standard deviation (SD). With an $\alpha = 0.05$, the power of the performed test was 1.00 for resins, 1.00 for surface conditions, and 1.00 for their interaction. Correlations between bond strengths and solubility parameters were done by regression analysis. Statistical significance was set in advance at $\alpha = 0.05$.

RESULTS

The results of the microtensile bond tests, reported as least-squares means, are summarized in Table 2. Highly significant differences were observed among the surface treatment ($p < 0.001$) groups and resins ($p < 0.001$). There was a significant interaction ($p < 0.01$) between resins and surfaces. Group 1 specimens (*i.e.*, moist water bonding) gave μ TBS values of only 3.1 and 6.4 MPa (Table 2) for the hydrophobic resins 1 and 2, respectively. These values were significantly ($p < 0.05$) lower than those of specimens bonded with resin 3 (27.9 ± 2.0 MPa, Table 2). The bond strengths produced by resins 4 and 5 (36.7 ± 2.0 MPa and 35.5 ± 2.2 , respectively, Table 2) were not significantly different from each other, but were significantly higher than the others ($p < 0.05$).

In group 2 (*i.e.*, moist ethanol bonding), all resins produced significantly higher bond strengths ($p < 0.05$) when compared with the same resins in group 1 specimens (Table 2). Within group 2, the bond strength of resin 1 was significantly lower ($p < 0.05$) than those made with resins 2–5, which were not different from each other (Table 2).

Resins 1 and 2 in group 3 (*i.e.*, dry bonding) yielded bond strengths that were similar to those in group 1 (Table 2). However, resin 3 in group 3 gave significantly lower ($p < 0.05$) bond strengths to dry dentin, compared with dentin wet with water (Table 2). Similarly, resins 4 and 5 in group 3 gave lower bond strengths than did the same resin in group 1 (Table 2).

Failure modes were predominantly adhesive when μ TBS were less than 15 MPa, mixed when they were between 15 and 30 MPa, and largely cohesive when the bond strengths exceeded 36 MPa (Table 2).

When the mean μ TBS of the 5 resins in group 1 (moist water bonding) were plotted against their Hoy's solubility parameters for polar forces (δ_p), a highly significant ($R^2 = 0.85$, $p < 0.05$) positive exponential relationship was obtained (Fig. 2A). Similar correlations with δ_h and δ_d were not significant ($R^2 = 0.41$ and 0.36 , respectively). When the tensile bond strengths of resins 1–5 in group 2 (moist ethanol bonding) were plotted against δ_p values (Hoy's solubility parameter for polar forces) of the polymers, a higher correlation was found ($R^2 = 0.90$, $p < 0.05$). Similar plots of δ_h or δ_d were not significant ($R^2 = 0.40$ and 0.36 , respectively) (Fig. 2B). In group 3 (dry bonding), significant ($R^2 = 0.86$, $p < 0.05$) positive correlation was found between the Hoy's solubility parameters for polar forces (δ_p) (Fig. 2C). Also in group 3, lower R^2 values were obtained with δ_d and δ_h (0.19 and 0.50 , respectively).

DISCUSSION

The results of this study confirm those of previous reports that resin-dentin bond strengths are related to the solubility parameters of dentin adhesives (Asmussen *et al.*, 1991; Asmussen and Uno, 1992; Finger *et al.*, 1994; Miller *et al.*, 1998). This study extends previous work by comparing acid-etched surfaces bonded dry or moist with water *vs.* ethanol. Wet-bonding with 100% ethanol clearly increased microtensile bond strengths of all the experimental adhesives tested. These results warrant rejection of the null hypothesis, that bonds made to dentin wet with ethanol are no different from those made to dentin wet with water. Since bonds made with hydrophilic resins (#4 and #5) were generally significantly higher than bonds made with more hydrophobic resin (#1), regardless of the surface condition, the second null hypothesis—that hydrophilicity of resins has no effect on bond strength—must also be rejected.

Clearly, the condition of the acid-etched dentin surface exerts a strong influence on the μ TBS produced by the tested experimental resins. In group 3 (*i.e.*, dry bonding), the collagen fibrils are known to collapse and to associate so closely that there are inadequate interfibrillar spaces available for resin infiltration (Pashley *et al.*, 1993; Tay *et al.*, 1996, 1998; Kanca and Sandrik,

1998; Nakaoki *et al.*, 2000). This is due to the development of interfibrillar hydrogen bonding (H-bonds) between collagen peptides that develops a calculated attractive force of $14.8 \text{ (J/cm}^3\text{)}^{1/2}$ (Agee *et al.*, 2006). For solvated comonomer mixtures to break these H-bonds, the solution must have a Hoy's solubility parameter for H-bonding forces (δ_h) that is close to $14.8 \text{ (J/cm}^3\text{)}^{1/2}$. If not, little infiltration of solvated resins can occur within the intertubular dentin (Agee *et al.*, 2006), although they can flow down open tubules to form resin tags. Thus, the low bond strengths produced by resins 1 and 2 in group 3 are probably due only to resin tag formation. The modest bond strengths produced by resins 3–5 may have been attributed to partial infiltration of intertubular dentin, since resins 3 and 5 have respective δ_h values of 14.3 and $15.5 \text{ (J/cm}^3\text{)}^{1/2}$.

In group 1, the etched dentin was saturated with water [$\delta_h = 40.4 \text{ (J/cm}^3\text{)}^{1/2}$], which breaks interpeptide H-bonds and allows for expansion of the dentin matrix (Pashley *et al.*, 2001; Nakajima *et al.*, 2002; Eddleston *et al.*, 2003). This is the mechanism responsible for the success of the wet-bonding technique with hydrophilic resins (Kanca and Sandrik, 1998). However, resins 1 and 2 gave very low μTBS values. We speculate that ethoxylated BisPhenol A dimethacrylate (resin 1) and BisGMA (resin 2) underwent phase changes when applied to water-saturated dentin, thereby weakening the hybrid and adhesive layers (Spencer and Wang, 2002). Use of the same resins in a different model system produced milky phase changes in resins 1 and 2, but not in resins 3–5. These phase changes were never seen in ethanol-saturated matrices (Becker *et al.*, 2006).

In group 2, water-saturated dentin was treated with a large excess of ethanol, to create ethanol-saturated acid-etched dentin. In expanded acid-etched dentin, where collagen fibrils were never allowed to collapse and form H-bonds, μTBS correlated best with the δ_p values of the solvated resins. While the δ_p for water-saturated matrices was $15.3 \text{ (J/cm}^3\text{)}^{1/2}$, that of ethanol-saturated matrices was only 12.5, which is closer to the δ_p values of the resins. The closer the solubility parameters of monomers are to polymers, the better they are at swelling or wetting the polymers (Barton, 1991). Treatment of acid-etched dentin with 100% ethanol causes a 15% shrinkage in the matrix (Becker *et al.*, 2006), by allowing for the formation of some matrix interpeptide H-bonds. This stiffens the matrix (Carvalho *et al.*, 2003; Garcia *et al.*, 2005) and prevents any further shrinkage during resin infiltration. When the experimental adhesives were applied to ethanol-saturated dentin in the current study, the bond strengths produced by resins 1 and 2 increased about six- to seven-fold, respectively, when compared with the same resins in group 1. Smaller increases were seen with resins 3 and 4, and no significant increase was seen with resin 5. Clearly, increases in the bond strengths of hydrophobic comonomer blends can be produced by the use of moist-bonding with ethanol instead of water. Saturation of the matrix with ethanol brings the δ_p of the matrix closer to those of the ethanol-solvated resins. We speculate that optimal wetting of collagen fibrils by these solvated resins occurs when the polar surface-free energy components are similar (Barton, 1991). Thus, the significant relationships between resin hydrophilicity and μTBS may be due to the degree of wetting and penetration of acid-etched ethanol-saturated dentin by the resins (Rosales *et al.*, 1999; Asmussen and Peutzfeldt, 2005).

The results of this study indicate that wet-bonding with ethanol instead of water facilitates higher bond strengths with the use of relatively hydrophobic resins. This new wet-bonding concept may successfully coax hydrophobic monomers into the dentin matrix, thereby creating more hydrophobic hybrid and adhesive layers that absorb less water over time (Ito *et al.*, 2005). More research is required to determine if this strategy will lead to more durable resin-dentin bonds.

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Figure 1.
Chemical structures of the monomers used in the solvated comonomer blends listed in Table 1. Definition of abbreviations is in the Table 1 footnote. $n = 3$ for E-BisADM.

Figure 2.

Relationships between least-squares means of microtensile bond strengths and Hoy's solubility parameters for polar forces (δ_p) of resins 1–5 (numbers in parentheses indicate the N upon which the mean value is based). **(A)** Least-squares means bond strengths of 50% ethanol-solvated resins 1–5 to acid-etched dentin saturated with water. **(B)** Least-squares means bond strengths of 50% ethanol-solvated resins 1–5 to acid-etched dentin saturated with ethanol. **(C)** Least-squares means bond strengths of 50% ethanol-solvated resins 1–5 to dry acid-etched dentin.

Compositions of Experimental Resins 1–5 and Hoy's Solubility Parameters for the Comonomers and for Demineralized Dentin (collagen)

Resin #	Resin Composition ^b	Hoy's Solubility Parameters ^a (J/cm ³) ^{1/2}		
		δ _d	δ _p	δ _t
1	35 wt% E-BisADM; 14.4% TEGDMA; 0.5% EDMAB; 0.13% CQ; 50% ethanol	13.8	10.7	13.3
2	35% BisGMA; 14.4% TEGDMA; 0.5% EDMAB; 0.13% CQ; 50% ethanol	14.3	11.8	13.3
3	35% BisGMA; 14.4% HEMA; 0.5% EDMAB; 0.13% CQ; 50% ethanol	14.1	12.1	14.3
4	20% BisGMA; 15% TCDM; 14.4% TEGDMA; 0.5% EDMAB; 0.13% CQ; 50% ethanol	14.6	12.0	13.5
5	20% BisGMA; 15% BisMP; 14.4% HEMA; 0.5% EDMAB; 0.13% CQ; 50% ethanol	13.9	12.3	15.5
Collagen	30% water, 70% peptides	11.8	15.3	22.5
Collagen	30% EtOH, 70% peptides	12.0	12.5	18.1
Collagen	0% water, 100% peptides	11.7	12.1	14.8
Ethanol		12.6	11.2	26.1
Water		12.2	22.8	40.4

^a All Hoy's solubility parameters were calculated with the use of commercially available software (Computer Chemistry Consultancy, <www.compchemconsul.com>). δ_d - Hoy's solubility parameter for dispersive forces; δ_p - Hoy's solubility parameter for polar forces; δ_t - Hoy's solubility parameter for hydrogen-bonding forces; δ_t - Hoy's solubility parameter for the total cohesive forces, that is, equivalent to Hildebrand's solubility parameter, $\delta_t = \sqrt{\delta_d^2 + \delta_p^2 + \delta_h^2}$.

^b Abbreviations: BisGMA = 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]-phenyl propane; TEGDMA = triethylene-glycol dimethacrylate; EDMAB = 2-ethyl-4-aminobenzoate; CQ = camphorquinone; E-BisADM = ethoxylated BisPhenol A dimethacrylate; HEMA = hydroxyethyl methacrylate; TCDM = di(hydroxyethyl methacrylate)ester of 5-(2,5-dioxotetrahydrofurfuryl)-3-methyl-3-cyclohexenyl-1,2'-dicarboxylic acid, and other stereo-isomers; BisMP = Bis[2-(methacryloyloxy) ethyl] phosphate.

Microtensile Bond Strengths* of Experimental Resins to Acid-etched Dry Dentin or Dentin Wet with Water or Ethanol

Table 2

50 wt% Ethanol- solvated Resins	Group 1, Water-saturated Dentin			Group 2, EtOH-saturated Dentin			Group 3, Air-dried Dentin		
	μ TBS (MPa)	Pre- test Failures	Failures A/ C/M	μ TBS (MPa)	Pre- test Failures	Failures A/ C/M	μ TBS (MPa)	Pre- test Failures	Failures A/ C/M
Resin 1	3.1 $\pm$ 2.0 ^{aA}	(6/16)	91/0/9	18.5 $\pm$ 2.3 ^{aB}	(11/16)	17/21/62	5.3 $\pm$ 2.3 ^{aA}	(4/16)	89/0/11
Resin 2	6.4 $\pm$ 2.4 ^{aA}	(10/16)	88/0/12	43.7 $\pm$ 2.2 ^{bB}	(16/16)	11/19/70	7.9 $\pm$ 2.5 ^{aA}	(4/16)	91/0/9
Resin 3	27.9 $\pm$ 2.0 ^{bA}	(16/16)	22/36/42	37.1 $\pm$ 2.0 ^{bB}	(16/16)	9/41/50	9.6 $\pm$ 2.4 ^{aA}	(11/16)	83/0/17
Resin 4	36.7 $\pm$ 2.0 ^{cA}	(15/16)	29/38/33	43.1 $\pm$ 2.2 ^{bB}	(16/16)	11/32/57	14.2 $\pm$ 2.3 ^{aC}	(16/16)	88/0/12
Resin 5	35.5 $\pm$ 2.2 ^{cA}	(15/16)	0/93/7	44.9 $\pm$ 2.2 ^{bB}	(10/16)	0/74/33	15.2 $\pm$ 2.2 ^{bC}	(9/16)	81/0/11

* Values are least-squares means $\pm$ SEMs. Within any vertical column, values identified by different lower-case letters are significantly different ($p < 0.05$) by the Holm-Sidak test. Within any horizontal row, values identified by different upper-case letters are significantly different by the Holm-Sidak test ($p < 0.05$). Numbers in parentheses indicate the number of specimens that could be successfully trimmed and tested, out of 16 specimens per group. A = adhesive failure; M = mixed adhesive and cohesive failure; C = cohesive failure in resin composite or adhesive. Values are the modes of failures (in %).