Effect of simplified ethanol-wet bonding on microtensile bond strengths of dentin adhesive agents with different solvents

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Abstract  Background/目的: Available knowledge about the effect of solvent-type etch-and-rinse adhesives on dentin bond strengths achieved with ethanol-wet bonding is limited. Therefore, this study was conducted to determine 24-hour bond strengths of etch-and-rinse adhesives with different solvents to acid-etched dentin saturated with either water-wet bonding or ethanol-wet-bonding techniques.

Materials and methods: Sixteen bovine incisors were divided into the following four groups based on the bonding techniques and adhesives used: Group I, water-wet bonding + Single Bond 2 (water/ethanol-based adhesive); Group II, water-wet bonding + Prime & Bond NT (acetone-based adhesive); Group III, ethanol-wet bonding + Single Bond 2 (water/ethanol-based adhesive); and Group IV, ethanol-wet bonding + Prime & Bond NT (acetone-based adhesive). After etching and rinsing, dentin surfaces were either left moist with water or immersed in ethanol. Following adhesive application and composite buildups, bonded teeth were sectioned into resin-dentin sticks for microtensile bond strength testing, which was conducted after storing the sticks in water for 24 hours. Data were analyzed by two-way analysis of variance and Tukey test (P < 0.05).

Results: Mean bond strength values (MPa) and standard deviations at 24 hours were as follows: Group I, 34.41 (12.6); Group II, 41.62 (11.8); Group III, 43.52 (13.8); and Group IV, 41.68 (9.1). No significant difference in bond strength was observed between different bonding techniques for both adhesives (P > 0.05).

Conclusion: Simplified ethanol-wet bonding exhibited similar 24-hour bond strength mean values for both ethanol/water-based and acetone-based etch-and-rinse adhesives. Therefore, solvent content may not interfere with bond strength to ethanol-saturated dentin.

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Introduction

Resin composite restoratives are able to bond to enamel and dentin through the use of dentin adhesive agents. Dentin adhesive agents are basically a blend of hydrophobic and hydrophilic resin monomers, solvents, cosolvents, photoinitiators, and inhibitors. Bonding mechanism of the current etch-and-rinse adhesive agents to dentin is supposed generally as a mechanical interlocking. Acid etching of smear layer-covered dentin surfaces results in the removal of smear layers and demineralized dentin matrices in which the mineral content of dentin is replaced with water. When adhesive blends containing resin monomers with both hydrophilic and hydrophobic groups are applied onto acid-etched dentin surfaces, resin monomers infiltrate into demineralized dentin matrices, impregnate interfibrillar spaces around collagen fibrils, encapsulate collagen fibrils, displace water, polymerize, and create interlocking with acid-etched dentin, respectively. Consequently, this process—dentin hybridization—provides mechanical retention for resin composite restoratives that chemically bond to adhesive resin.

It is accepted that achieving ideal dentin hybridization depends on preventing the collapse of demineralized dentin matrices, or in other words, keeping interfibrillar spaces open as these play a role as infiltration pathways for adhesive resin monomers. When demineralized dentin matrices are saturated with water, maximum openings in interfibrillar spaces are obtained. However, adhesive resin monomers have limited capability to displace water from interfibrillar spaces, and therefore, different solvents (e.g., ethanol and acetone) are used as ingredients in adhesive formulations to effectively displace water from resin—dentin interfaces prior to polymerization of adhesive resin. Nevertheless, under clinical conditions, the current solvent-type etch-and-rinse adhesives are not able to displace enough water from interfaces, which is necessary to achieve ideal dentin hybridization.

Residual water within interfaces reduces durability of the resin—dentin bonding due to hydrolytic and enzymatic degradation of dentin hybrid layer components over time, and thus jeopardizes longevity of adhesive restoration.

A novel wet-bonding technique called "ethanol-wet bonding" was recently introduced to improve the durability of the resin—dentin bonding. Unlike the conventional water-wet-bonding technique, the ethanol-wet-bonding method uses ethanol instead of water to saturate and prevent the collapse of demineralized dentin matrices prior to resin application. This means that water is replaced from interfibrillar spaces with ethanol during saturation of demineralized dentin matrices with ethanol prior to the application of the adhesive agent. The rationale behind this is that miscibility of adhesive resin monomers in the ethanol-saturated dentin matrices is better than those in the water-saturated dentin matrices, and thus ethanol as a saturation solvent for demineralized dentin matrices may allow intimate encapsulations of collagen fibrils with adhesive resin monomers. Consequently, this technique can provide more ideal hybrid layers. Therefore, ethanol-wet bonding may diminish enzymatic degradation of collagen fibrils and improve durability of resin—dentin bonds.

The current etch-and-rinse adhesive systems are formulated to be compatible with the water-wet-bonding technique. Hence, they contain both hydrophobic (i.e., bisphenol A—glycidyl methacrylate) and hydrophilic monomers (i.e., hydroxyethyl methacrylate), which are well miscible in ethanol than in water. Therefore, it is possible that the current adhesive resins may provide improved penetration with ethanol-wet bonding. However, available knowledge on the effect of different solvent contents of current etch-and-rinse adhesives on bonding strength achieved with ethanol-wet bonding is scarce in the literature.

This study was conducted to determine the effects of different solvent contents of two etch-and-rinse adhesives [Single Bond 2 (3M ESPE, St. Paul, MN, USA), a water/ethanol-based adhesive, and Prime & Bond NT (DENTSPLY De Trey, Konstanz, Germany), an acetone-based adhesive] on microtensile bond strengths (μTBSs) to dentin with ethanol-wet bonding and compare the results obtained using these agents with water-wet bonding. The null hypothesis tested was that different solvent contents will not affect the bond strength of tested adhesives to dentin with ethanol-wet bonding.

Materials and methods

Sample preparation

In this study, 16 bovine incisors, collected from bovines that were at least 2 years old, were used. Teeth were stored in 0.02% sodium azide solution at 4°C for a maximum period of 6 months prior to use. Soft tissues around teeth were scalped and roots were removed using low-speed diamond disk under water. Crowns were embedded into self-cure acrylic blocks using a double-sided adhesive band. Enamel surfaces were ground using 320-grit silicon carbide papers to obtain flattened surfaces. The box-shaped cavities were prepared on exposed enamel surfaces using a coarse diamond bur with high-speed turbine. Prepared samples were randomly divided into the following four groups (n = 4) based on the adhesives and bonding techniques used: Group I: Single Bond 2 (water/ethanol-based adhesive) + water-wet bonding; Group II: Prime & Bond NT (acetone-based adhesive) + water-wet bonding; Group III: Single Bond 2 (water/ethanol-based adhesive) + ethanol-wet bonding; and Group IV: Prime & Bond NT (acetone-based adhesive) + ethanol-wet bonding.

Bonding procedures

Dental adhesives were used according to the manufacturer’s instructions in the water-wet-bonding groups (Groups I and II; Table 1). In the ethanol-wet-bonding groups (Groups III and IV), the simplified ethanol-wet-bonding technique was used. This technique presented a simplified way of ethanol dehydration of demineralized dentin matrices. The method involves saturation of acid-etched dentin surfaces with absolute ethanol using a needle for 1 minute. Surfaces should be kept visibly wet with ethanol during this period and adhesives should then be applied to these ethanol-saturated surfaces according to
the manufacturer’s instructions (Table 1). Following the application of adhesives, composite buildups were done (Valux Plus; 3M ESPE) in four 1-mm-thick increments in all groups.

### μTBS test and statistical analysis

Bonded specimens were immersed in distilled water and incubated (E-420P; Megaterm, Istanbul, Turkey) at 37 °C for 24 hours prior to μTBS testing. Then, the resin—dentin μTBS specimens with approximately 0.7 mm² dimensions were obtained using a diamond saw under copious water irrigation (MICRACUT 125; Metkon Instruments Inc., Bursa, Turkey) at 300 rpm. Five μTBS specimens of each tooth were randomly selected, and a total 20 μTBS specimens/group were used for the μTBS test. The numbers of prematurely debonded resin—dentin sticks during specimen preparation and fixing to jig were recorded. The μTBS specimens were fixed to the jig with cyanoacrylate glue (Pattex; Henkel, Düsseldorf, Germany) and forced in tension at a crosshead speed of 1 mm/minute using Bisco microtensile testing machine. The μTBS was derived by dividing the enforced force at the time of fracture by the bond area (mm²). The mode of failure was determined with a stereo microscope (Meade Bresser BioluX; Meade Bresser, Rhede, Germany), and recorded as adhesive (failure at the dentin—resin interface), cohesive (failure entirely within the dentin substrate or resin composite), or mix (failure at the dentin—resin interface including cohesive failure of one of the substrates).

A two-way analysis of variance (ANOVA) with Tukey honest significant difference tests was performed for determining the effects of independent variables (adhesive resins and wet-bonding techniques) on dependent variable (μTBS). After the two-way ANOVA, one-way ANOVAs and/or pairwise comparisons were performed. Statistical significance was set in advance at P = 0.05.

### Results

The bond strength means (MPa) of all the groups are presented in Table 2. The bond strength means vary from 34.41 MPa to 43.52 MPa. The lowest bond strength mean was obtained for the group of Single Bond 2 with water-wet bonding, whereas the highest bond strength mean was obtained for the group of Single Bond 2 with ethanol-wet bonding. However, results of Tukey test showed no significant difference between the bond strength means for water- and ethanol-wet-bonding techniques for all adhesive systems tested (P > 0.05). The ANOVA result shows that the interaction between the adhesive agent and the technique applied is not significant (P = 0.091). During the specimen preparation and μTBS testing, no prematurely debonded resin—dentin stick was observed. The mode of failure was determined to be adhesive, that is, failure at the dentin—resin interface.

### Discussion

The null hypothesis tested, that is, different solvent contents will not affect the bond strength of dentin adhesive agents with ethanol-wet bonding, was accepted because our study findings revealed that μTBS mean values of the dentin adhesive agents were not affected by either solvent differences or wet-bonding techniques used.

<table>
<thead>
<tr>
<th>Groups (adhesive/bonding technique)</th>
<th>μTBS (MPa), (SD)</th>
<th>Failure modes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Adhesive</td>
<td>Mix</td>
</tr>
<tr>
<td>Group I (Single Bond with water-wet bonding)</td>
<td>34.41 (12.6)*</td>
<td>80</td>
</tr>
<tr>
<td>Group II (Prime &amp; Bond NT with water-wet bonding)</td>
<td>41.62 (11.8)*</td>
<td>55</td>
</tr>
<tr>
<td>Group III (Single Bond with ethanol-wet bonding)</td>
<td>43.52 (13.8)*</td>
<td>50</td>
</tr>
<tr>
<td>Group IV (Prime &amp; Bond NT with ethanol-wet bonding)</td>
<td>41.68 (9.1)*</td>
<td>65</td>
</tr>
</tbody>
</table>

*Significantly different (P < 0.05).
SD = standard deviation; μTBS = microtensile bond strength.
Resin—dentin bonding using ethanol-wet bonding

Previous studies have demonstrated that bond strength is dependent on the type of solvent used with either commercial dentin adhesive agent with the same monomer contents\(^1\) or experimental model dentin adhesive agents\(^2\) when the water-wet-bonding technique was adopted; however, another study demonstrated that the bond strength upon rewetting depends on both the type of solvent in the bonding system and rewetting time.\(^3\) The common explanation for this effect is the positive correlation between bond strength and maintaining interfibrillar spaces; in other words, avoiding the collapse of demineralized dentin matrices during and after adhesive resin infiltration and solvent evaporation.\(^3,12\)

The hydrogen bonding capacity (\(\delta_h\)) is Hoy’s solubility parameters for hydrogen bonding forces of solvents determines the amount of solvent that can remove residual water from demineralized dentin matrices while simultaneously maintaining interfibrillar spaces (i.e., preventing their collapse).\(^4\) Water with high hydrogen bonding capacity \(\delta_h = 40.4\) (J/cm\(^3\))\(^{1/2}\)) can easily break down hydrogen bonds between collagen fibrils while it is collapsing, and create new ones with collagen fibrils, thereby re-expanding collapsed matrices. However, this process softens matrices dramatically, so that these matrices collapse again during evaporation and displacement of water with solvents during and after monomer infiltration. By contrast, ethanol \(\delta_h = 20\) (J/cm\(^3\))\(^{1/2}\)) and acetone \(\delta_h = 11\) (J/cm\(^3\))\(^{1/2}\)) have lower hydrogen bonding capacities than water and can be used for this purpose.\(^1\)

It is reported that once demineralized dentin matrices are saturated with water and interfibrillar spaces are maintained by applying the water-wet bonding technique, the hydrogen bonding capacity of acetone as a saturation solvent will not be able to prevent the formation of new hydrogen bonds between collagen fibrils during displacement of water with acetone, and therefore, the matrices collapse again. By contrast, ethanol as a saturation solvent is able to prevent collapse of matrices to a certain extent and maintain interfibrillar spaces.\(^3\) Principles of ethanol-wet-bonding concept are based on these interactions between dentin matrices, solvents, and solvated resins. When water-saturated demineralized dentin matrices are converted to ethanol-saturated matrices, matrices maintain some stiffness and in such cases infiltration of neat and solvated adhesive resin monomers into these matrices is enhanced.\(^3,10\) It should be noted that conversion of water-saturated dentin to ethanol-saturated dentin refers to the chemical dehydration of acid-etched moist dentin with ethanol. This is achieved by several techniques in vitro. For example, in the "full-dehydration protocol", ethanol-wet bonding is accomplished with a series of increasing concentrations of ethanol (50%, 70%, 80%, 95%, and 100% for three times for 30 seconds each). This technique imitates epoxy resin tissue embedding in transmission electron microscopy.\(^5\) Another technique called "simplified protocol" accomplishes ethanol-wet bonding using 100% ethanol only once for just 60 seconds.\(^10,16\) Although the first technique defies the principles of user friendliness and technique simplification, it is able to replace adequate water unlike the second one.\(^17,18\) Nevertheless, the current more hydrophilic etch-and-rinse adhesive agents are more tolerant to the presence of residual water after undergoing the simplified ethanol-wet-bonding technique. Thus, this technique shows the potential for use in clinical practice.

Seeing interactions among saturation solvents and demineralized dentin matrices, a mixture of residual water and ethanol within matrices after simplified ethanol dehydration protocol might soften matrices again. This may explain why different solvents of dentin adhesive agents had no influence on bond strength to dentin with wet-bonding techniques in the current study, as the stiffness of the ethanol/water mixture-saturated matrices, which might be similar to water-saturated matrices, may not be able to avoid collapse during evaporation of primer solvents with low hydrogen bonding capacity such as ethanol/water (combined) and acetone, respectively.

One issue that should be addressed is that high values for standard deviation in the present study might not allow for assessing treatment effects on bond strength. This might be explained by the technique sensitivity of water-wet\(^9\) and ethanol-wet-bonding methods.\(^7\) Although it is well-known that water-wet bonding is a technique-sensitive bonding procedure that results in high discrepancy in bond strength values, there is limited information about technique sensitivity of ethanol-wet bonding. However, according to Osorio et al.,\(^17\) simplified ethanol-wet bonding is sensitive to water contamination. Therefore, it may yield high standard deviation in bond strength values.

The current study demonstrated that etch-and-rinse adhesive agents with different solvents exhibited similar dentin bond strengths with simplified ethanol-wet bonding. The stiffness of ethanol/residual water mixture-saturated demineralized dentin matrices might not be able to avoid collapse during evaporation of different primer solvents. Therefore, solvent content might not influence bond strength to dentin saturated with simplified ethanol-wet-bonding technique.

Conflicts of interest

The author has no conflicts of interest relevant to this article.

References


