Effect of Repeated Container Lid Opening on Dentin Shear Bond Strength of Two Dentin Adhesive Systems

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Abstract:

Objective: Comparing the effect of repeated opening of the container lid of two dentin adhesive systems, Prime&Bond NT (P&B NT) and iBond (iB), on shear bond strength.

Materials and Methods: Intact bovine lower incisors (n=60), fixed in acrylic were randomly divided into six groups (n=10). Groups I and II were set as control groups. P&B NT and iB were applied on the samples after five days a week, three times a day for two weeks of use in groups III and VI; and after four weeks of use in groups V and VI. The samples were evaluated by a universal testing-machine (Instron, cross-head speed 1 mm/min) and stereomicroscope.

Results: There was no significant difference between the bond strengths in any of the three P&B NT. The mean amount of the shear bond strength for iB after 60 times of use (15.31 MPa) was significantly lower than that at the baseline (23.51 MPa). There was no significant difference between iB at the baseline and after 30 times of use (19.26 Mpa), and also between iB after 30 times of use and after 60 times of use. All P&B NT groups showed significantly higher shear bond strengths when compared with their similar iB groups in iB.

Conclusion: Repeated use (60 times) of the all-in-one adhesive container seems to reduce dentin shear bond strength. Therefore, containers with a lower content of the same adhesive or a single-dose of the adhesive are preferred.

Key Words: Acetone, Dentin-Bonding Agents; Prime and Bond NT; Dental Bonding Shear Strength

INTRODUCTION

Effective bonding to dental substrates has certainly been one of the major and important goals in restorative dentistry. With acid etching, enamel shows favorable and high bond strength; however, the development of a strong, lasting bond to dentin still seems to be remaining a challenge. Many factors account for this difference between the two substrates. Their different structural composition is the primary factor; micromechanical retention within demineralized dentin surface through a resin-reinforced hybrid layer has generally been accepted to be the mechanism for bonding adhesive resin to dentin [1,2].

In enamel-dentin adhesive systems introduced throughout the past decade, the stages of application have been tried to be simplified. The fifth generation of adhesives (one-bottle) is used by combining primer and adhesive resin in a single bottle on moist dentin in one step (wet-bonding). In most of these systems, resin monomers are solved in an organic solvent (acetone or ethanol) with a water chasing ef-
ffect by lowering the surface tension of water and replacing it within the collagen network of demineralized dentin resulting in enhancement of water removal from collagen surface and ultimately being exchanged for the adhesive resin by increasing the vapor pressure of water. In addition, these solvents may help in the penetration of the bonding agents into the demineralized, collagen rich dentin surface by lowering the viscosity of the solution [1,3,4].

Acetone-based adhesive systems can generate a continuous, thick hybrid layer and close contact with restorative material, adhesive and dentin [5]. These adhesives should not be used on completely dried dentin and some moisture should exist in the collagen network after etching and rinsing of the dentin. Proper wetness of the dentin and the technique sensitivity of these adhesive should always taken into consideration [1,2,6].

Thus, to simplify the bonding steps, self-etch adhesives were brought forward. In some of them, the so called all-in-one, three steps of etching, priming and resin bonding are combined together. Application of acidic primer simultaneously provides us with the demineralization of dentin and penetration into the adhesive. Water is the main component of these adhesives as it generates acidic H⁺ and, in some products, ethanol and/or acetone have been added to water to promote the solubility of resin monomers [2,7]. Ethanol (boiling temperature 78.3 °C) and specially acetone (boiling temperature 65.5 °C) possess relatively high vapor pressures. In some studies, the effects of solvent evaporation (specially acetone) from the containing bottles have been taken into account and a decline in the bond strength of acetone-based adhesives after repeated opening of the container has been reported [8,9].

The present study aims to evaluate the effect of repeated opening of adhesive bottles during clinical use on shear dentin bond strength in two simplified adhesive systems: fifth generation (one-bottle) Prime&Bond NT (P&B NT) and seventh generation (single-component all-in-one) iBond (iB) [10].

MATERIALS AND METHODS
In this experimental study, flat middle dentin surfaces were prepared on 60 extracted intact bovine lower incisors with 600-grit silicon carbide paper. The specimens were mounted in acrylic molds and randomly divided into six groups (n=10).

In groups I and II (control groups), P&B NT and iB were applied at the baseline with their respective composites, Spectrum TPH microhybrid (Dentsply De Trey, Germany) and Charisma microhybrid (Kulzer, Germany) respectively according to the manufacturers’ instructions. P&B NT and iB were applied on the samples after five days a week, three times a day for two weeks of use in groups III and IV (30 times); after four weeks of use in groups V and VI (60 times); and in groups V and VI, similar to groups I and II. In each turn of use, the containers were kept opened for one minute. All specimens were stored in distilled water for 24 hours at room temperature and thermo-cycled for 500 cycles at 5 °C and 55 °C. Dwell time for each bath was 20 seconds and with a 10 second transfer time. The specimens were loaded to failure in a universal testing machine (Instron model 4302, Germany) with a cross-head speed of 1 mm/min. Shear bond strength (SBS) was recorded in MPa and the data were analyzed with Kruskal-Wallis and Dunn tests. Finally, stereomicroscopic (Ziess, Germany) observation (20X) determined the mode of failures occurred during debonding.

RESULTS
Mean amounts of SBS values were obtained and recorded (Table 1, Fig 1).

There was no significant difference between SBSs of each P&B NT group (P>0.05), while
SBS of iB after 60 times of use (15.31 MPa) was found to be significantly lower than its control group at baseline (23.51 MPa) (P<0.05). There was no significant difference either between SBSs of iB at baseline and that after 30 times of use (19.26 MPa), and SBSs of iB after 30 and 60 times of use (P>0.05). All P&B NT groups showed significantly higher SBSs compared with their similar iB groups in iB (P<0.05).

Stereomicroscopic observation revealed four fracture types at the debonding sites (Table 1):

I) Adhesive
II) Cohesive in dentin
III) Cohesive in composite
IV) Mixed (adhesive & cohesive)

**DISCUSSION**

In the present study, by application of P&B NT at baseline, after two weeks (30 times of use), and also after four weeks (60 times of use) the bond strength to dentin did not show any significant change.

Considering the important role of acetone in the development of dentin bonding and its high evaporation rate, some believe that in one-bottle system, due to evaporation of the solvent penetration of the adhesive into the demineralized dentin decreases [11]. Reis et al [12] reported a significant reduction in dentin bond strength following the elimination of the organic solvent (acetone or ethanol) in two bonding systems (P&B 2.1, single bond) attributed to incomplete penetration of monomers into demineralized dentin and improper water displacement resulting into dilution of water-soluble components of the resin and reduction of the degree of polymerization and bond strength [12]. However, Cho and Dickness reported an increase in bond strength to dentin by lowering the acetone content in an experimental adhesive. They anticipated that lower acetone concentration which may occur due to solvent evaporation during clinical use can improve the integrity of dentin/adhesive bond [13].

Perdigao et al [9] reported a significant decrease comparing the shear bond strength to dentin at baseline and after three weeks (30 times) in one-step system (with acetone as the solvent), although, they didn’t observe any difference in bond strength with similar conditions in the other three adhesive systems with ethanol, ethanol + water, and water as the solvents. They attributed this difference to the evaporation of the acetone solvent during the repeated opening of the container [9]. The reason for the difference between that and our

**Table 1.** Mean (Standard deviation) of shear bond strengths (MPa) and types of fracture for the tested groups.

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean (SD)</th>
<th>Maximum</th>
<th>Minimum</th>
<th>Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>P&amp;B NT at baseline</td>
<td>31.94 (4.68)</td>
<td>41.2</td>
<td>23.09</td>
</tr>
<tr>
<td>II</td>
<td>iB at baseline</td>
<td>23.51 (4.85)</td>
<td>34.7</td>
<td>15.37</td>
</tr>
<tr>
<td>III</td>
<td>P&amp;B NT after 30 times</td>
<td>31.89 (6.5)</td>
<td>40.8</td>
<td>23.12</td>
</tr>
<tr>
<td>IV</td>
<td>iB, after 30 times</td>
<td>19.26 (6.99)</td>
<td>32.47</td>
<td>11.49</td>
</tr>
<tr>
<td>V</td>
<td>P&amp;B NT after 60 times</td>
<td>26.11 (6.75)</td>
<td>39.7</td>
<td>18.02</td>
</tr>
<tr>
<td>VI</td>
<td>iB after 60 times</td>
<td>15.31 (4.14)</td>
<td>22.35</td>
<td>46.49</td>
</tr>
</tbody>
</table>

P&B NT=Prime&Bond NT; iB=iBond; A=Adhesive; Cd=Cohesive in dentin; Cc=Cohesive in composite; M=Mixed
study, may be related to the difference in the simulated way the repetitive clinical use of the adhesive, as in study conducted by Perdigão et al [9] during 30 times of opening container lid, the bottle was squeezed a little so that the adhesive was only evident at the orifice during the one-minute period. In our study, to imitate the clinical conditions more properly, during the one minute period of opening the container lid, the adhesive was picked just to the estimated amount of one time clinical use. Thus, in case of evaporation, the decrease in adhesive content inside the bottle occurred simultaneously. Consequently, the proportion of the solvent to other components has not changed that much to negatively affect the bond strength. On the other hand, the smart design of the adhesive container bottle resulted in minimum evaporation of the solvent during use. Reis et al [12] reported little weight reduction in P&B 2.1 adhesive with acetone solvent by the amounts of 0.08%, 0.15% and 0.22% after the lid remaining open for one minute, two, and 48 hours at 22°C respectively. This shows that in spite of the lid remaining open, much evaporation has not occurred, whereas, by measuring the evaporation rate through the variation of the adhesive weight (little drops of adhesive) as a function of time, after 5.5 minutes, the P&B 2.1 adhesive solution lost 81% of its mass, and single bond solution underwent a 31% reduction after 11.5 minutes [12].

Another point in our study was shaking the bottle before opening the lid to prevent the phase-separation. This is particularly important to P&B NT which contains filler particles. Nicholls depicted that as the liquid contents in the bottles have different densities and are not chemically bond to one another, phase separation can occur in layers within the bottle, so that in one to two hours the solvent is separated from the resin. On the basis of this fact, he recommends shaking the adhesive bottle before use [14]. Accordingly, by shaking the bottle before use, we prevented the mentioned separation and thus, the evaporation of acetone during opening of the bottle's lid was decreased.

Gallo et al [15] did not report any significant decrease in shear dentin bond strength by delayed application (10 minutes after dispensing of adhesive) of P&B 2.1; however, it showed a trend towards lower bond strength. According to testing conditions in a closed environment, 22 °C of temperature and 60% (SD=5%) of humidity, they notified that increased air-flow could also affect the bond strength of the adhesive by increasing the evaporation of the solvent [15]. Our study was carried out in conditions similar to that described by Gallo et al [15], so the mentioned factors may apply here as well and once the testing conditions are altered (increased air-flow or temperature) it may lead to lower bond strength when the number of using times increase.

No significant difference in iB dentin bond strength was observed 30 times of clinical use. However, by doubling the times of use, the bond strength decreased significantly. This might be as well due to the smart container design, picking up the adhesive by the amount of one time clinical use only, and shaking the bottle before use leading to less evaporation of acetone during the first 30 times of use. However, by doubling times of adhesive use and remaining the lid open, even a little evaporation of acetone can decrease the bond strength. This difference, not observed for P&B NT, may be related to the difference in function and bonding procedure of iB system. IB is a one-step self-etching system (all-in-one) in which etching, priming and adhesive resin are performed with the application of three layers of one single solution and certainly even a little change in the components of this single solution can alter its bonding abilities. Van Lnduyl et al [16] named these compositions difficult mixtures comprised of a complex of hydrophilic and hydrophobic monomers with
relatively high concentrations of solvent and water to keep them soluble.

By the elimination of HEMA (having the ability of maintaining water) from iB formulation, water was separated from the other components during the evaporation of acetone and, therefore, this phase separation can play a role in weakening the efficiency of this bonding system [16,17].

On the other hand, same thing may occur in the adhesive inside the bottle during the evaporation of acetone from the iB container before its application on dentin leading to a decrease in the bond strength after 60 times of clinical use. Primary bond strength of P&B NT was also found to be significantly higher than that of iB which is in agreement with the results of many previous studies [18-21].

Generally, one-step self-etch system or the so-called all-in-one, has weaker bonding abilities in comparison with other bonding systems (like two-step etch & rinse) which seems to be related to the following factors:
1) Acidic, hydrophilic and hydrophobic monomers, the organic solvent and water are all placed in one or two bottles and this adversely affects the function and the efficiency of these components.
2) High concentrations of the solvent
3) Insufficient thicknesses of the adhesive layer (due to the high water content and low viscosity), all of which may include an oxygen inhibited layer during light curing.
4) Possibility of the remaining solvent (water) and its interference with resin polymerization.
5) High hydrophilicity after polymerization resulting in acting as permeable membrane.
6) During solvent evaporation, monomer-water ratio may change which results in phase separation and formation of water blisters [18,22-25], whereas P&B NT can be effective in protecting the hybrid layer integrity and resistance against polymerization shrinkage stress by forming the appropriate adhesive layer and having filler particles which consequently functions as a stress absorbing layer, improving the bond strength [13,26,27].

CONCLUSION
The results of this study showed that repeated use of the one-bottle system did not affect the bonding efficiency, however when using the all-in-one adhesive system, by increasing the times of use (60 times) regardless of the container lid remaining open, even a little evaporation of acetone can lead to a decrease in the dentin bond strength. According to the relatively low primary bond strength of this adhesive, this decrease will be more critical and, thus, needs more attention to prevent acetone evaporation during the clinical use. Therefore, it may be better if all-in-one adhesives containing acetone solvent are manufactured in smaller packages, containing less content and/or even single dose.

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REFERENCES


