Effects of Multiple Coatings of Two All-in-one Adhesives on Dentin Bonding

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**Purpose:** Simple changes to bonding technique can improve resin-dentin bond strengths. The objective of this study was to evaluate the effects of multiple coatings of two all-in-one adhesive resins on both microtensile bond strength (\(\mu TBS\)) and nanoleakage.

**Materials and Methods:** The mid-coronal occlusal dentin of extracted human molars was used. Two all-in-one adhesives – iBond (Heraeus Kulzer) and Xeno III (Caulk Dentsply) – were applied to 320-grit abraded dentin surfaces. In groups 1 and 3 during bonding, monomer application and solvent evaporation were done 1, 2, 3, 4 or 5 times on the dentin surface before light curing. In groups 2 and 4 after light curing the first layer, the adhesive was re-applied, the solvent evaporated, and the layer light cured. This was repeated from 2 to 5 times, followed by creation of composite buildups. After 24 h storage in 37°C water, the teeth were sectioned perpendicular to the adhesive interface to produce multiple beams of composite-bonded dentin, approximately 0.9 mm\(^2\) in area. These were tested to failure in tension. Data were evaluated by three-way ANOVA (material vs coatings vs light curing) followed by multiple comparisons at \(\alpha = 0.05\). Additionally, nanoleakage of silver uptake and adhesive layer thickness were evaluated using transmission electron microscopy (TEM).

**Results:** The results indicated that bond strengths increased with the number of coatings (\(p < 0.0001\)) with both adhesives, up to 3 layers, especially if each layer was light cured. Nanoleakage of silver tended to decrease with each coat in both adhesive systems.

**Conclusion:** By simply applying more coats of adhesive, the strength and quality of dentin adhesion can be improved.

**Key words:** all-in-one adhesives, dentin bond strength, nanoleakage, multiple coatings.

\(J\) Adhes Dent 2005; 7: 133–141. Submitted for publication: 06.02.04; accepted for publication: 05.08.04.

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\textsuperscript{I} It is well known that bond strength is affected by the extent of resin infiltration into the exposed collagen network.\textsuperscript{3,8} Ideally, adhesive monomers should occupy the interfibrillar spaces around exposed collagen fibrils.\textsuperscript{3,14} Dentin adhesives are currently available as three-step, two-step, and single-step systems, depending on how the three cardinal steps of etching, priming, and bonding to tooth substrates are accomplished or simplified.\textsuperscript{12} Recently introduced all-in-one adhesives further combined...
these three bonding procedures into a single step. However, recent studies have shown that some all-in-one adhesives exhibited relatively low bond strengths when compared with two-step self-etching systems. As these products create very thin coatings, they may be oxygen inhibited and hence poorly polymerized. The manufacturer’s instructions for iBond recommend application of three coats of adhesive prior to evaporation of solvent and light curing. A recent study also reported that bonding of Prompt L-Pop may similarly be improved with an additional layer of adhesive.

Evaluation of silver uptake by transmission electron microscopy (TEM) can provide clues on the extent of resin monomer infiltration or degree of water uptake. Recently, several TEM studies reported incomplete resin infiltration into exposed collagen fibrils. Although there are two reports showing that multiple coats of Prompt L-Pop can improve bond strengths to dentin, this effect using other all-in-one adhesives has not yet been determined.

Using the microtensile test, the purpose of this study was to evaluate the effects on bond strength of multiple applications of two all-in-one adhesives to dentin with and without light curing each layer. Additionally, the effects of multiple coats of all-in-one adhesives on nanoleakage of the hybrid and adhesive layers were evaluated using TEM. The null hypotheses tested were that 1 to 5 applications of all-in-one adhesives have no effect on either bond strength or nanoleakage, and that there was no difference of all-in-one adhesives have no effect on either bond strength or nanoleakage, and that there was no difference of multiple consecutive applications (2, 3, 4, or 5 coats), adhesive application and solvent evaporation steps were done repeatedly (Fig 1). In one subgroup, after applying one coat of A + B liquid and evaporating the solvent, another coat of liquid B only was applied, the solvent evaporated and the layer light cured. In another subgroup, after the first layer was applied and the solvent evaporated, but only light cured after application of the final coat. Group 4 specimens were bonded with iBond but each layer was both evaporated and light cured before application of the next layer.

**Materials and Methods**

Fifty-six extracted noncarious human third molars were used in this study. The teeth were collected after obtaining the patients’ informed consent under a protocol approved by the Medical College of Georgia Institutional Review Board. Flat dentin surfaces were created in mid-coronal dentin perpendicular to the tooth’s longitudinal axis using a slow-speed diamond saw (Isomet, Schaumburg, IL, USA) to remove occlusal enamel and superficial dentin. Each surface of mid-coronal dentin was ground with 320-grit silicon carbide paper under running water for 30 s just prior to bonding.

**Experimental Design**

Two single-step adhesives were used in this study. The compositions of these adhesives, Xeno III (Dentsply Degussa, Hanau, Germany) and iBond ( Heraeus Kulzer, Hanau, Germany), are shown in Table 1.

There were four groups. Group 1 was composed of Xeno III bonded specimens in which the solvent in each coating was evaporated but not light cured until the final coating was applied. Group 2 specimens were bonded with Xeno III, but the solvent was evaporated from each layer and the coating was light cured before applying the next layer. Group 3 specimens were bonded with iBond, and the solvent was evaporated, but only light cured after application of the final coat. Group 4 specimens were bonded with iBond but each layer was both evaporated and light cured before application of the next layer.

**Group 1 (evap)**

For Xeno III, equal volumes of liquids A and B were mixed for approximately 5 s. A generous amount was applied to the dentin surface. After leaving the adhesive undisturbed for 20 s, the adhesive was uniformly spread with a gentle air stream, and the solvent evaporated for 5 s. For multiple consecutive applications (2, 3, 4, or 5 coats), adhesive application and solvent evaporation steps were done repeatedly (Fig 1). In one subgroup, after applying one coat of A + B liquid and evaporating the solvent, another coat of liquid B only was applied, the solvent evaporated and the layer light cured. In another subgroup, after the first layer was applied and the solvent evaporated, an additional layer of Scotchbond Multi-Purpose Plus (3M-ESPE, St Paul, MN, USA) adhesive was applied and light cured for 10 s.

All adhesives were subsequently light cured for 10 s with the light-curing unit (Bisco VIP halogen light, Bisco, Schaumburg, IL, USA) operated at 600 mW/cm².

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**Table 1 Composition of the all-in-one adhesives used**

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Application procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>i-Bond</td>
<td>Heraeus Kulzer, Hanau, Germany</td>
<td>4-META, UDMA, glutaraldehyde, acetone, water</td>
<td>Apply three layers with scrubbing, allow 30 s of dwell time, evaporate solvent, light cure for 10 s.</td>
</tr>
<tr>
<td>SBMP</td>
<td>3M-ESPE, St Paul, MN, USA</td>
<td>Bis-GMA, HEMA, CQ</td>
<td></td>
</tr>
<tr>
<td>Xeno III</td>
<td>Dentsply Degussa, Hanau, Germany</td>
<td>A. Universal: HEMA, aerosil R-947 (fumed silica), BHT (stabilizer), ethanol, water B. Catalyst: Pyro-EMA-SK, PEM-F, UDMA, BHT, CQ, p-dimethyl amine ethyl benzoate (co-initiator)</td>
<td>1. Dispense liquid from Universal and Catalyst bottles and mix thoroughly. 2. Leave on dentin undisturbed for 20 s. Gently air dry. 3. Light activate for 10 s.</td>
</tr>
</tbody>
</table>

Abbreviations: 4-META = 4-methacryloxyethyltrimellitic acid; SBMP = Scotchbond Multi-Purpose Plus adhesive; BHT = 2,6-di-tert-butyl-p-cresol; bis-GMA = (1-methyleneylene)bis(4,1-phenyleneoxy)[2-hydroxy-3,1-propanediyl)] bismethacrylate; CQ = camphorquinone (2,3-bornanedione); HEMA = 2-hydroxyethyl methacrylate; PEM-F = Penta-methacryl-oxy-ethyl-cyclo-phosphazene monofluoride; pyro-EMA-SK = tetra-methacryl-ethyl-pyrophosphate; UDMA = urethane dimethacrylate (1,6-dimethacryl-ethyl-oxy-carbonylamine-2,4,4-trimethyl hexane).
The calculation of mean bond strengths in each group.

Pretest failures were recorded as zero values in failure, to obtain the ultimate tensile strength, calculated in MPa. (Vitrodyne V1000, John Chatillon & Sons, Greensboro, NC, USA) at a crosshead speed of 1.0 mm/min until light curing (10 s) did not occur until the last coat was applied (Fig 1).

After light curing the first layer, a second layer of iBond was applied, the solvent evaporated, and the adhesive light cured for 10 s in a similar manner. These steps were repeated for 3, 4, and 5 coatings.

Following bonding, five 1 mm increments of TPH resin composite (Caulk/Dentsply for Xeno III) and Venus (Heraeus-Kulzer for iBond) were used to create resin composite buildups. Each layer was light cured for 40 s.

A three-factor ANOVA was used to examine differences in the mean microtensile bond strength (μTBS). The three factors were type of adhesive (Xeno III or iBond), light curing (after each layer or after last layer only), and layers (1 to 5 layers, 1 layer + B, 1 layer +MP). All two- and three-factor interactions between the three variables were included in the model. A Bonferroni correction to the alpha level was used to determine post-hoc differences. Statistical significance was assessed using an alpha level of 0.01, and all statistical analyses were performed using SAS 8.2 (SAS Institute, Cary, NC, USA).

Microtensile Bond Testing

After the bonded specimens had been stored in water at 37°C for 24 h, the built-up teeth were sectioned perpendicular to the adhesive interface to remove the peripheral 3 mm from each side of the tooth, yielding a cube-like specimen that represents the center of the tooth. This central cube was cut into 0.9-mm-thick slabs. Each slab, in turn, was sectioned into 1.0-mm-wide beams (adhesive area: approximately 0.9 mm²) with a diamond saw under water cooling/lubrication. Although 9 beams were produced, only the 8 with the greatest dentin thicknesses were selected for testing. Two teeth were used for each of the 7 subgroups (14 teeth) per material. Beams prepared were attached to a testing apparatus with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA). A tensile load was applied with a material tester (Vitrodyne V1000, John Chatillon & Sons, Greensboro, NC, USA) at a crosshead speed of 1.0 mm/min until failure, to obtain the ultimate tensile strength, calculated in MPa. Pretest failures were recorded as zero values in the calculation of mean bond strengths in each group.

Transmission Electron Microscopy (TEM) of Nanoleakage

Four additional teeth with flat dentin surfaces produced in the same manner as for microtensile bond testing were employed for each of the subgroups in Groups 1 to 4 (Table 1). After treating the dentin surfaces with the respective bonding protocol, a microfilled resin composite containing pre-polymerized fillers (EPIC-TMPT, Parkell, Farmingdale, NY, USA) was coupled to the bonded dentin in order to facilitate ultramicroscopy. The bonded specimens were stored in distilled water at 37°C for 24 h. A 1-mm-thick slab was sectioned from each specimen using the slow-speed saw under water cooling. The slabs were immersed in 50 wt% ammoniacal silver nitrate for 24 h, according to a TEM silver tracer technique. Following the reduction of the diamine silver ions into metallic silver grains, the slabs were dehydrated in an ascending ethan-ol series and embedded in epoxy resin in preparation for ultramicroscopy. Ninety- to 100-nm-thick undemineralized sections containing the resin-dentin interfaces were collected on single-slot, carbon- and formvar-coated copper grids. They were examined without further staining using a transmission electron microscope (Philips EM208S, Eindhoven, The Netherlands) operating at 80 kV.

RESULTS

Effects on Bond Strength of Multiple Coats Without Curing each Layer

Groups 1 and 3: Xeno III (evap) and iBond (evap)

Single applications of Xeno III or iBond produced low μTBS (7.2 ± 6.3 and 12.2 ± 7.5 MPa, respectively) that were not statistically different. Four of the 16 beams bonded with one coat of Xeno III in group 1-1 (evap) exhibited pretest failures (Table 2). No other subgroups in groups 1 and 3 showed any pretest failures. The second layer of Xeno III increased the bond strength, although the increase was not statistically significant (Table 2). The effect of multiple applications of Xeno III reached significance after three
layers (p < 0.001) and a plateau after four coats, since there was no statistically significant difference between four and five coats (Table 2). When one coat of Xeno III was covered with a second layer of solution B and then light cured, the µTBS was not different than if a second layer of A + B had been applied (Table 2). Similarly, when one coat of Xeno III was covered with one layer of Scotchbond Multi-Purpose Plus (SBMP) adhesive and then light cured, there was no significant improvement in bond strength over using a second coat of Xeno III.

Using iBond (Group 3), application of two coats did not significantly increase µTBS, although three coats (the manufacturer’s instruction) significantly (p < 0.002) increased bond strength (Table 2). There was no significant effect of a fourth or fifth coat on improving bond strength. Covering one coat of light-cured iBond with SBMP adhesive significantly improved bond strength (p < 0.002) by a factor of five as compared to a one-coat application. Using SBMP adhesive increased the µTBS 3.4 times (p < 0.002) over that produced by a second coating of iBond (Table 2).

**Effects on Bond Strength of Individually-cured Multiple Coats**

**Groups 2 and 4: Xeno III (evap + lc) and iBond (evap + lc)**

When a second layer of adhesive was applied to the already light-cured first layer in Group 2 specimens bonded with Xeno III, the microtensile bond strength increased 6.6-fold (p < 0.002) over the single coat value (compare 7.2 ± 6.3 to 47.3 ± 19.2, Table 2). Adding a third coating significantly (p < 0.002) increased bond strength again to 65.4 MPa. Addition of a fourth and fifth coat did not significantly increase bond strength over that produced by three coats. When one coat of light-cured Xeno III was covered with another coat of bottle B, there was no significant increase in bond strength over that produced by one coat of Xeno III alone. However, when one coat of light-cured Xeno III was covered with one coat of light-cured SBMP adhesive, the bond strengths increased significantly to 64.7 MPa.

When the second layer of adhesive was applied to the already light-cured first layer in Group 4 specimens bonded with iBond, the bond strength increased and was as high as three layers of iBond where only the last layer was light cured (Table 2). Addition of a third, fourth, or fifth light-cured layers produced no further increase in bond strength. When one layer of light-cured iBond was covered with one layer of light-cured SBMP adhesive, the bond strength was significantly (p < 0.002) higher than a second, third, fourth, and fifth coat of light-cured iBond.

**Effects on Nanoleakage of Multiple Coatings not Cured Individually**

TEM of specimens treated with one layer of iBond (not used according to the manufacturer’s directions of applying three layers) revealed direct contact of the resin composite with the surface of dentin (Fig 2a). That is, there was no layer of polymerized adhesive between the top of the hybrid layer and the bottom of the composite. Nanoleakage in the form of heavy silver deposits could be seen within the 1-µm-thick, partially demineralized hybrid layer (Fig 2a). When one layer of Xeno III was used, the hybrid layer was covered by 2 to 3 µm of adhesive (Fig 2b). The amount of nanoleakage was similar when a second coat of adhesive was applied before light curing, which created a 4-µm-thick adhesive layer above a 1-µm-thick hybrid layer (not shown). Both all-in-one adhesives removed most of the smear layer, although some smear plugs remained in some tubule orifices.

With the application of three consecutive coats of iBond prior to light curing, the thickness of the adhesive layer increased to 7 to 8 µm (Fig 2c). The increase in thickness of the adhesive layer was also accompanied by an increase in the propensity to form linear and spherical

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**Table 2 Effects of multiple XIII and iBond adhesive coatings on microtensile bond strengths to dentin**

<table>
<thead>
<tr>
<th>Groups</th>
<th>Layers</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>1 + B</th>
<th>1 + SBMP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 X (evap)</td>
<td>12.2 ± 7.5a</td>
<td>22.6 ± 9.2ab</td>
<td>30.0 ± 9.9b</td>
<td>43.5 ± 7.7c</td>
<td>41.4 ± 8.0c</td>
<td>28.4 ± 9.3b</td>
<td>27.8 ± 5.9b</td>
<td></td>
</tr>
<tr>
<td>2 X (evap + lc)</td>
<td>7.2 ± 6.3a</td>
<td>47.3 ± 19.2b</td>
<td>65.4 ± 20.4d</td>
<td>81.8 ± 20.8d</td>
<td>68.9 ± 22.6d</td>
<td>24.4 ± 15.2b</td>
<td>64.7 ± 21.2d</td>
<td></td>
</tr>
<tr>
<td>3 iB (evap)</td>
<td>12.2 ± 7.5a</td>
<td>18.5 ± 6.2a</td>
<td>30.6 ± 7.0b</td>
<td>34.2 ± 6.0bc</td>
<td>51.6 ± 14.8bc</td>
<td>62.3 ± 23.0c</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 iB (evap + lc)</td>
<td>12.2 ± 7.5a</td>
<td>39.6 ± 15.2b</td>
<td>42.7 ± 12.3bc</td>
<td>36.6 ± 7.7a</td>
<td>35.8 ± 13.4a</td>
<td>58.0 ± 23.5c</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Values are mean ± SD (n = 16). In groups 1 and 2, 4 out of the 16 failed prematurely. Zero values were used for those 4 specimens in each group. (evap) = solvent evaporation between coatings but no light curing; (evap + lc) = solvent evaporation and light curing between each coating. Superscript letters designate statistically different (p < 0.01) groups.

1 + B = after applying the first coating, a second coating of solution B was applied. 1 + MP = after applying the first layer, an additional layer of Scotchbond Multi-Purpose Plus adhesive was applied and light cured.
silver-filled structures within the iBond adhesive layer (Fig 2c). In addition, numerous isolated silver grains could be identified throughout the entire adhesive layer (Fig 2c). Extensive nanoleakage could be seen within the hybrid layer. Three coats of Xeno III exhibited less silver uptake into the hybrid layer compared to three coats of iBond (compare Figs 2c vs 2d), although the Xeno III adhesive showed similar isolated spot-like silver grains.

A further increase in the number of adhesive coats to five layers did not necessarily result in an overall increase in the thickness of the hybrid layer or the polymerized adhesive layers (Figs 2e and 2f), but it did decrease the
amount of nanoleakage in the hybrid layers produced by both adhesives.

**Effect on Nanoleakage of Multiple Coatings Cured Individually**

Figure 3a is an overview of the resin-dentin interface that was bonded using three coats of iBond that were light cured separately, creating an adhesive layer that was about 17 to 20 µm thick. The amount of nanoleakage in the adhesive layer was less than that observed in the comparable 3-coat group, where the layers were not light cured (compare Fig 2c vs Fig 3a). Although the thickness of the adhesive layer was similar to that achieved previously with the use of three consecutive coats of adhesive (Fig 2c), there was a complete absence of linear distributions of silver within with the adhesive when these coats were light cured separately (Fig 3a). This did not, however, eliminate the heavy silver staining within the hybrid layer. The absence of linear silver-filled channels in the adhesive of iBond (Fig 3a) and the gradual reduction in the size of the isolated silver grains (Fig 3c) became more prominent with the use of five coats of separately light-cured iBond adhesives, which generated a 35-µm-thick adhesive layer between the composite and dentin, although nanoleakage in the hybrid layer was severe. Similar results were obtained using three vs five coats of Xeno III (Figs 3b and 3d), although the amount of nanoleakage in the hybrid layer was much less than was seen in hybrid layers created by iBond.

The difference between the use of multiple coatings of iBond or Xeno III and the use of the Scotchbond Multi-Purpose resin over a single layer of these cured adhesives is shown in Figs 3d and 3f. Although the thickness of the hybrid layers and the nanoleakage within the hybrid layer were not very different from those previously described, there was a complete absence of silver uptake within the top of the 15-µm-thick adhesive layer (Figs 3d and 3f). The most distinctive features, however, were the dramatic reduction in the size of the isolated silver grains within the intermixed zone between the all-in-one adhesives and the Scotchbond Multi-Purpose resin (Fig 3e), followed by the total elimination of these silver grains in the latter (Fig 3f).

**DISCUSSION**

The results of this study require rejection of the null hypothesis that multiple coatings of these two all-in-one adhesives do not increase µTBS. The results also require rejection of the second null hypothesis that there is no difference in µTBS regardless of whether the multiple layers were light cured.

The TEM observations indicate that one layer of iBond (not used according to the manufacturer’s recommendation) is insufficient to adequately create a hybrid layer covered by an adhesive layer that will provide sufficient coupling to subsequently applied resin composite. However, one coating of Xeno III which contains nanofillers was enough to create a hybrid layer covered by an adhesive layer. Although two or more coatings with either material dramatically increased µTBS when both layers were light cured, there was a further significant increase in µTBS (Table 2). This was especially evident using Xeno III. Using three layers of Xeno III without light curing

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**Fig 2e** TEM micrographs of specimen bonded with five coats of iBond that had the solvent evaporated prior to application of the next layer but no light-curing until application of the final layer. All specimens were undemineralized and were immersed in AgNO₃ prior to embedding to reveal nanoleakage. C: composite; D: intertubular dentin; P: peritubular dentin. Five coats of iBond produced a 1-2 µm thick hybrid layer (H) with only sporadic silver uptake. The adhesive layer (A) was only 7-8 µm thick but was almost free of silver nanoleakage that was limited to spot-like deposits (open arrowheads). Silver deposits were seen both in the bottom of the hybrid layer (white arrow) and in the top of the hybrid layer (pointer).

**Fig 2f** TEM micrographs of specimen bonded with five coats of Xeno III that had the solvent evaporated prior to application of the next layer but no light-curing until application of the final layer. All specimens were undemineralized and were immersed in AgNO3 prior to embedding to reveal nanoleakage. C: composite; D: intertubular dentin. Five coats of Xeno III produced a 1 µm thick hybrid layer (H between two open arrows) that showed minimal silver uptake, and a 10 µm thick adhesive layer (A) that showed only spot-like silver deposits.
between layers (but with solvent evaporation at each step) produced a mean µTBS of 30.0 MPa. However, if each layer was light cured, three layers gave a µTBS of 65.4 MPa (p < 0.05 compared to three coats, of which only the last one was light cured).

iBond contains water and acetone as solvents. The evaporation of acetone is much faster than that of water. After acetone is gone, water must evaporate by itself, albeit at a much lower vapor pressure. Thus, multiple applications may tend to permit the accumulation of water on the surface. Such water should be evaporated to avoid low bond strengths.11

Solution B in Xeno III contains all of the comonomers, initiators, and accelerators except HEMA (Table 1). It contains no solvent. Solution A contains HEMA and a water-ethanol mixture. It was thought that adding an
additional coating of B (without A) to the first coating of A + B would enhance the $\mu$TBS of Xeno III because it would be less hydrophilic. This was unsuccessful (Table 2), regardless of whether the first layer was light cured or not prior to the application of material from solution B (that was subsequently light cured).

When one layer of iBond or Xeno III was covered with SBMP adhesive, there was a significant increase ($p < 0.05$) in $\mu$TBS (Table 2) regardless of whether the first coat was light cured or not. The adhesive of SBMP is basically a blend of bis-GMA/HEMA without any volatile solvent. This adhesive layer may have increased the mechanical properties of the bond due to improved polymerization. The more hydrophobic SBMP adhesive may have also sealed the surface of the more hydrophilic iBond. Indeed, Figs 3e and 3f shows that almost no silver uptake was seen in the SBMP adhesive layer (R), unlike the substantial silver uptake seen in the underlying iBond and Xeno III adhesives.

It is likely that the increased bond strength seen in both all-in-one adhesives with multiple coatings is due to several mechanisms operating simultaneously. As the first layer of adhesive begins to etch dentin, it is probably rapidly buffered, so that additional layers of unpolymerized comonomers may improve the etching ability of adhesives. Although the hybrid layer thickness of iBond specimens increased from 0.8 to 1.5 $\mu$m when one vs three layers was applied, the values were not statistically significantly different. When using multiple coatings of adhesives, there are several techniques that can be employed. If the solvent is evaporated between each coating, the concentration of comonomers that exists after each coating should increase, thereby facilitating comonomer infiltration with minimal increase in the thickness of the adhesive layer.

These solvated adhesives only contain 25 to 30 wt% comonomers and 70 to 85% solvents (preliminary gravimetric analysis). The presence of the solvent facilitates wetting and spreading of the solvated comonomers and may assist in preserving the width of interfibrillar spaces during infiltration. This may facilitate inward diffusion of the comonomers into the matrix. When most of the solvent is evaporated, the concentration of the adhesives must increase dramatically, although the thickness of the layer may be extremely thin. The cumulative concentration of these constituents must rise in the adhesive layer with each application. We speculate that this contributes to improved polymerization of the final mixture. Although the thickness of the resin layer increases enough to prevent oxygen inhibition of its entire thickness, we believe that the final adhesive layer has an improved quality (ie, better cross linking, improved interpenetration of polymer chains, improved percent conversion). We speculate that the bond strength reached a plateau following sequential coatings when a limiting critical variable had been reached (eg, film thickness, cross-linking density, concentration of photoinitiators, accelerators, etc).
In contrast, when each layer was light cured following solvent evaporation, the improved bond strengths may have been due to mechanical rather than chemical factors. After the first layer was cured, it would be unlikely that there could be any more resin infiltration into the matrix. However, it might be possible to improve the quality of each layer of adhesive. That is, since each layer was polymerized, only the oxygen-inhibited portion of the layer would be available to mix with the newly applied layer. However, the oxygen-inhibited layer in iBond can be 8 to 10 µm thick and can easily mix with subsequently applied layers. This might improve its quality. One layer of iBond produced an adhesive layer thickness that ranged from 0 to 3 µm. When three layers were applied and each one was light cured, the adhesive layer was about 15 to 17 µm thick. Five layers of iBond (each cured individually) produced a 34-µm-thick adhesive layer (Fig 3c), while five layers of individually cured Xeno III produced a 26-µm-thick adhesive layer. We speculate that the major improvement in bond strength with successive layers in group 2 and 4 specimens was due to increased adhesive layer thickness. Multiple layers of adhesive tend to create thicker adhesive layers that increase the compliance of the joint. This may lower local stress concentrations during tensile testing, resulting in higher yield strengths.

Regarding the mechanism(s) responsible for increased bond strength, the need for multiple applications increases the bonding time of these ‘simplified’ adhesives. Although we did not attempt to optimize the solvent evaporation time, we used 5 s. Five seconds for each of 4 layers adds 20 s to the procedure. However, adding these 20 s, plus light curing each of the four layers for 10 s (40 s cumulative curing time plus 20 s evaporation time), requires more time than is stated in current manufacturer’s instructions.

These results confirm how important bonding technology is to producing optimal resin-dentin bonds. Simple changes in bonding technique, such as applying more layers of all-in-one adhesives, can lead to large increases in initial bond strength. Whether this improves the long-term durability of resin-dentin bonds remains to be determined.

ACKNOWLEDGMENTS

We thank Amy Wong of the Electron Microscopy Unit, The University of Hong Kong for technical assistance and Michelle Barnes of the Medical College of Georgia for secretarial support. This work was supported, in part, by grants DE 014911 and DE 015306 (to DHP) from the National Institute of Dental and Craniofacial Research. The materials (iBond/Heraeus Kulzer, Hanau, Germany and Xeno III/Caulk Dentsply, Germany) used in this study were generously supplied by each manufacturer. The testing device used for microtensile bond testing (Ciucchi’s jig) was supplied by Kuraray Co, Ltd, Osaka, Japan.

REFERENCES


Clinical relevance: By simply applying more coats of adhesive, the strength and quality of dentin adhesion can be improved.