Influence of Curing Rate of Resin Composite on the Bond Strength to Dentin

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Clinical Relevance
When deciding on a specific curing protocol, dental professionals should be aware of the advantages and limitations of each curing mode.

SUMMARY
This study determined whether the strength with which resin composite bonds to dentin is influenced by variations in the curing rate of resin composites. Resin composites were bonded to the dentin of extracted human molars. Adhesive (AdheSE, Ivoclar Vivadent) was applied and cured (10 seconds @ 1000 mW/cm²) for all groups. A split Teflon mold was clamped to the treated dentin surface and filled with resin composite. The rate of cure was varied, using one of four LED-curing units of different power densities. The rate of cure was also varied using the continuous or pulse-delay mode. In continuous curing mode, in order to give an energy density totaling 16 J/cm², the power densities (1000, 720, 550, 200 mW/cm²) emitted by the various curing units were compensated for by the light curing period (16, 22, 29 or 80 seconds). In the pulse-delay curing mode, two seconds of light curing at one of the four power densities was followed by a one-minute interval, after which light cure was completed (14, 29, 27 or 78 seconds), likewise, giving a total energy density of 16 J/cm². The specimens produced for each of the eight curing protocols and two resin composites (Tetric EvoCeram, Ivoclar Vivadent; Filtek Supreme XT, 3M ESPE) were stored in water at 37°C for seven days. The specimens were then either immediately subjected to shear bond strength testing or subjected to artificial aging (6,000 cycles between 5°C and 55°C baths) prior to testing. Failure modes were also assessed. The shear bond strengths were submitted to factorial analysis of variance, and the failure modes were submitted to a Chi-square test (α=0.05). All but power den-
ty (curing mode, resin composite material and mode of aging) significantly affected shear bond strength. The curing mode and resin composite material also influenced the failure mode. At the selected constant energy density, pulse-delay curing reduced bonding of the resin composite to dentin.

INTRODUCTION
The degree of cure of a given polymeric material is directly related to the energy density received by the material: the higher the energy density, the higher the degree of cure, strength and stiffness of a resin composite. However, a given energy density level may be obtained by different combinations of curing time and power density. There is evidence that increasing the power density, while maintaining constant energy density, decreases the degree of cure. Furthermore, power density and cure temperature dramatically influence the kinetics of polymerization.

The initial curing rate of a light-curing resin composite is influenced by the power density of the curing unit: a reduction in power density implies a reduction in the curing rate. For a given energy density level, the curing reaction may also be slowed down by using so-called soft-start curing modes, which include step-curing and pulse-delay curing. Among these, in particular, the pulse-delay curing mode has been found to be effective in reducing the rate of cure as compared to continuous cure.

The curing rate is related to stress formation in resin composites. The remaining stresses in the tooth-restoration complex result in a higher risk of failure during function. It was with the aim of minimizing the shrinkage stresses produced during curing of resin composites that soft-start curing modes were introduced. Slowing down the curing reaction by varying the curing mode has been found to minimize damage at the tooth-restoration interface. Studies show that pulse-delay curing minimizes shrinkage stresses at the cavosurface interface, diminishes post-gel polymerization shrinkage and reduces contraction force during the initial phase of polymerization. Since reducing the curing rate results in smaller remaining stresses in resin composites, one might speculate that reducing the curing rate may result in stronger bonds.

Only a few studies have investigated the effect of curing modes on the shear bond strength of resin composite to dentin. Price and others found that varying curing modes by changing them from continuous to step-cure reduced the bonding of resin composite to dentin. Caldwell, Kulkarni and Titeley, however, did not observe significant differences between step-cure and continuous cure regarding bond strength. It should be noted that the energy density was not kept constant in either study.

While in service, the adhesive strength of restorations can be also challenged by thermal and fatigue stresses. Thermocycling is a widely accepted artificial mode of aging used to simulate alternating in vivo temperatures. Although thermocycling does not always influence the mechanical properties of resin composite materials, it may result in the decreased bonding of resin composite to enamel or dentin, particularly when increasing the number of cycles. Since the curing rate and curing mode may affect the polymer structure, thermocycling may have an influence on shear bond strength.

Therefore, the aim of this study was to determine whether the strength with which resin composite bonds to dentin is influenced by variations in the curing rate of the resin composite. The curing rate was varied: 1) by use of curing units operating at different power densities and 2) by the use of different curing modes. The null hypothesis for this experiment states that, at a given level of energy density, the curing rate does not influence the strength with which resin composite bonds to dentin.

METHODS AND MATERIALS
Shear Bond Strength
Human molars kept in 0.5% chloramine aqueous solution were embedded in epoxy resin. After setting, the mesial or distal surfaces were ground using sequential silicon carbide paper up to #1000 grit to expose a flat dentin surface. The dentin was treated with a self-etch adhesive (AdheSE, Ivoclar Vivadent, Schaan, Liechtenstein) following the manufacturer’s instructions. The primer was applied for 30 seconds, the solvent evaporated with a strong stream of air and the adhesive was subsequently applied and light cured for 10 seconds (Bluephase, Ivoclar Vivadent, 1000 mW/cm²).

A split Teflon mold (diameter 3.6 mm; height 2.5 mm) was clamped to the treated dentin surface and filled with resin composite (Tetric EvoCeram A3, Ivoclar Vivadent; Filtek Supreme XT A3, 3M ESPE, St Paul, MN, USA). Continuous or pulse-delay curing was performed with one of four LED-curing units (Bluephase, Ivoclar Vivadent; LEDemetron 1, sds Kerr, Orange, CA, USA; DioPower, CMS–Dental ApS, Copenhagen, Denmark; Elipar Freelight, 3M ESPE) and is described in Table 1. The power densities emitted by the various curing units, verified by using a radiometer (LED Radiometer, Demetron, sds Kerr), were compensated for by the curing period to give an energy density totaling 16 J/cm². For the pulse-delay technique, two seconds of light curing was followed by a one-minute interval, after which light curing was completed. For each of the eight curing protocols, 16 specimens were produced with each resin composite.
Ten minutes after completion of the light curing, the bonded specimens were freed from their molds and stored in water at 37°C for seven days. The teeth were then equally divided into two groups. Half of the specimens were immediately submitted to shear bond strength testing. The other half was thermocycled for 6,000 cycles between 5°C and 55°C baths before shear bond strength testing. Shear bond strength testing was performed in a universal testing machine (Instron, High Wycombe, United Kingdom) at a crosshead speed of 1 mm/minute. Mean values and standard deviation were calculated from the eight specimens in each group.

### Analysis of the Failure Modes

The failure modes were evaluated at 18x magnification under a stereomicroscope. Failure was characterized as primarily being adhesive (A), primarily being cohesive within dentin (CD), primarily being cohesive within resin composite (CR) or mixed (M).

### Statistical Methods

Shear bond strength data were submitted to factorial analysis of variance. Failure mode data were grouped within curing mode, power density, resin composite material or mode of aging before testing for significant effects. A Chi-square test was used to test for the number of adhesive failures against the combination of the other types of failure. The level of significance was defined as \( \alpha = 0.05 \).

### RESULTS

Table 1 shows the mean values and standard deviations of bond strength for the different conditions investigated.

Four factorial analysis of variance were used to investigate influence of the mode of curing (continuous or pulse-delay), power density, resin composite material and mode of aging before testing for significant effects. A Chi-square test was used to test for the number of adhesive failures against the combination of the other types of failure. The level of significance was defined as \( \alpha = 0.05 \).

### Table 1: Mean Shear Bond Strength (MPa) and Standard Deviation for Tetric EvoCeram and Filtek Supreme XT According to the Curing Mode and Mode of Aging

<table>
<thead>
<tr>
<th>Curing Unit</th>
<th>Power Density</th>
<th>Time</th>
<th>Tetric No-Thermocycling</th>
<th>Filtek No-Thermocycling</th>
<th>Tetric Thermocycling</th>
<th>Filtek Thermocycling</th>
</tr>
</thead>
<tbody>
<tr>
<td>Continuous</td>
<td>Bluephase</td>
<td>1000 mW/cm²</td>
<td>16 seconds</td>
<td>22.0 ± 2.8</td>
<td>24.4 ± 4.0</td>
<td>23.3 ± 4.4</td>
</tr>
<tr>
<td></td>
<td>LEDemotron 1</td>
<td>720 mW/cm²</td>
<td>22 seconds</td>
<td>19.2 ± 4.4</td>
<td>26.2 ± 4.3</td>
<td>24.8 ± 6.3</td>
</tr>
<tr>
<td></td>
<td>DioPower</td>
<td>550 mW/cm²</td>
<td>29 seconds</td>
<td>22.2 ± 5.5</td>
<td>22.8 ± 2.6</td>
<td>27.7 ± 6.1</td>
</tr>
<tr>
<td></td>
<td>Freelight</td>
<td>200 mW/cm²</td>
<td>80 seconds</td>
<td>20.4 ± 6.6</td>
<td>25.5 ± 4.4</td>
<td>21.0 ± 4.3</td>
</tr>
<tr>
<td>Pulse-delay</td>
<td>Bluephase</td>
<td>1000 mW/cm²</td>
<td>14 seconds</td>
<td>18.0 ± 5.7</td>
<td>24.0 ± 2.7</td>
<td>24.1 ± 6.4</td>
</tr>
<tr>
<td>- two seconds-</td>
<td>LEDemotron 1</td>
<td>720 mW/cm²</td>
<td>20 seconds</td>
<td>19.5 ± 2.5</td>
<td>19.8 ± 4.3</td>
<td>21.9 ± 5.3</td>
</tr>
<tr>
<td></td>
<td>DioPower</td>
<td>550 mW/cm²</td>
<td>27 seconds</td>
<td>18.4 ± 4.2</td>
<td>21.7 ± 5.8</td>
<td>21.9 ± 8.0</td>
</tr>
<tr>
<td></td>
<td>Freelight</td>
<td>200 mW/cm²</td>
<td>78 seconds</td>
<td>16.0 ± 3.9</td>
<td>21.3 ± 7.1</td>
<td>25.0 ± 4.7</td>
</tr>
</tbody>
</table>

*For the pulse-delay groups, two additional seconds should be added to the exposure durations in the time column, corresponding to the initial pulse activation.

<table>
<thead>
<tr>
<th>Effect</th>
<th>df</th>
<th>ss</th>
<th>ms</th>
<th>F</th>
<th>p</th>
</tr>
</thead>
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<tr>
<td>Mode of Curing (A)</td>
<td>1</td>
<td>279.39</td>
<td>279.39</td>
<td>9.17</td>
<td>0.0027*</td>
</tr>
<tr>
<td>Power Density (B)</td>
<td>3</td>
<td>139.19</td>
<td>46.40</td>
<td>1.52</td>
<td>0.2093</td>
</tr>
<tr>
<td>Resin Composite (C)</td>
<td>1</td>
<td>539.23</td>
<td>539.23</td>
<td>17.70</td>
<td>&lt;0.0001*</td>
</tr>
<tr>
<td>Mode of Aging (D)</td>
<td>1</td>
<td>489.29</td>
<td>489.29</td>
<td>16.06</td>
<td>&lt;0.0001*</td>
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<tr>
<td>A*B</td>
<td>3</td>
<td>22.42</td>
<td>7.47</td>
<td>0.25</td>
<td>0.8646</td>
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<tr>
<td>A*C</td>
<td>1</td>
<td>35.37</td>
<td>35.37</td>
<td>1.16</td>
<td>0.2824</td>
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<tr>
<td>A*D</td>
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<td>53.60</td>
<td>53.60</td>
<td>1.76</td>
<td>0.1860</td>
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<tr>
<td>B*C</td>
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<td>24.56</td>
<td>8.20</td>
<td>0.27</td>
<td>0.8477</td>
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<tr>
<td>B*D</td>
<td>3</td>
<td>23.19</td>
<td>7.73</td>
<td>0.25</td>
<td>0.8586</td>
</tr>
<tr>
<td>C*D</td>
<td>1</td>
<td>46.58</td>
<td>46.58</td>
<td>1.53</td>
<td>0.2175</td>
</tr>
<tr>
<td>A<em>B</em>C</td>
<td>3</td>
<td>33.22</td>
<td>11.07</td>
<td>0.36</td>
<td>0.7795</td>
</tr>
<tr>
<td>A<em>B</em>D</td>
<td>3</td>
<td>96.08</td>
<td>32.03</td>
<td>1.05</td>
<td>0.3707</td>
</tr>
<tr>
<td>A<em>C</em>D</td>
<td>1</td>
<td>30.26</td>
<td>30.26</td>
<td>0.99</td>
<td>0.3200</td>
</tr>
<tr>
<td>B<em>C</em>D</td>
<td>3</td>
<td>146.84</td>
<td>48.95</td>
<td>1.61</td>
<td>0.1886</td>
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<tr>
<td>Error</td>
<td>227</td>
<td>6915.46</td>
<td>30.46</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Significant effect
DISCUSSION

Varying the rate of cure by varying the power density did not affect shear bond strength. This is apparently in contrast with previous works by Uno and Asmussen and Miyasaki and others, in which bond strength to dentin decreased as power density decreased. However, as the energy density was not kept constant in these studies and, in fact, it decreased along with power density, the decline in bond strength caused by reduced power density may have reflected a reduced degree of cure instead of a reduced rate of cure.

Varying the rate of cure by varying the curing mode significantly influenced shear bond strength, which leads to partial rejection of the null hypothesis. Pulse-delay curing resulted in lower shear bond strength of resin composite to dentin when compared to continuous curing. Although this was not expected, it is possible that the more linear polymer structure, previously reported to result from this curing mode, affected the mechanical properties of resin composite materials to such an extent that it influenced bond strength. It has also been observed that, at a given energy density, pulse-delay curing may result in a lower degree of cure at 2-mm depths when compared to continuous irradiation. This observation constitutes a further possible explanation for the findings in this study, including the observation that pulse-delay curing resulted in more adhesive failures than did continuous cure. The effect of curing mode on bond strength has been previously investigated. Earlier studies compared the effect of step-cure and continuous cure, with divergent results being observed. In agreement with the results of the current study, Price and others found that changing from continuous to step-cure reduced the bonding of resin composite to dentin. Caldwell, Kulkarni and Titley, on the other hand, did not register significant differences between step-cure and continuous cure regarding bond strength. However, as opposed to the current work, there was no standardization of energy density emitted for the different groups, nor was the power density systematically varied in previous publications.

Filtek Supreme XT was found to give rise to higher bond strengths than Tetric EvoCeram. The mechanical properties of resin composites are reflected in bond strength. Filtek Supreme XT has been found to have higher modulus of elasticity as compared to Tetric EvoCeram (Asmussen & Peutzfeldt, 2006, unpublished results), which may explain why Filtek Supreme resulted in significantly higher bond strengths and significantly fewer adhesive failures than Tetric EvoCeram.

Thermocycling increased the bonding of resin composite to dentin. It is possible that the increase in temperature caused by the hot bath (55°C) has improved the degree of conversion of the resin composite and that this was reflected in shear bond strength. Thus, when resin composite materials were exposed to heat after light irradiation, an increase in the degree of conversion was observed. However, ranking of the groups was maintained after thermocycling and has been likewise observed in other publications. Thermocycling may still be considered a valuable means to stress the adhesive interface. It must be noted, however, that the influence of thermocycling on bond strength may depend on the specific test set-up or adhesive used.

CONCLUSIONS

Compared to continuous curing, pulse-delay curing reduced the bonding of resin composite to dentin at a constant energy density. Therefore, when deciding on a specific curing protocol, clinicians are advised to be aware of the advantages and limitations of each curing mode.

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References


