Effect of Delayed Light-Cured Activation on Bond-Strengths between Composites and Adhesives

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ABSTRACT

The objective of this study was to evaluate and compare the effect of times elapsed on the application of a single-step adhesive system and activation of light cured subsequently placed composite to the bond strength between composites and adhesives. This was an experimental study using fifteen teeth extracted human premolars, which were caries and pathology free. Sample of teeth were embedded up to cemento-enamel junction on box of acrylic resin. All teeth were cut to obtain flat occlusal dentin surfaces. The specimens were randomly selected and divided into three groups with five teeth each. All the teeth were assigned to single-step bonding system AQ Bond (Sun Medical Co. Ltd., Shiga, Japan) according to manufacturer’s direction. Group 1 was left for 45 seconds, group 2 for 2 minutes, while group 3 was 5 minutes before putting subsequently restorative material. Composite resin was applied in 3 mm diameter and 6 mm height of cylinder mold and cured according to manufacturer’s direction on the dentin surface. Shear bond strengths were measured using a testing system (Instron 8874, Instron Corp., Canton, MA, USA) at a crosshead speed of 2 mm/min with cell load capacity of 25 KN. The bond-strengths between single step adhesive system (AQ Bond) and light cured composites were tested. Mann-Whitney test showed no significant differences in bond-strength between Group 1 and Group 2. However, there were significantly differences (p<0.01) in decreased of bond-strength between Group 2 and Group 3. As conclusion, the bond strength between composites and single-step adhesive system was reduced after 5 minutes delayed curing of composites.

Key words: single step adhesives, shear bond strength, delayed activation

INTRODUCTION

Adhesive dentistry originated from a simple clinical experiment and the vision and dedication of a unique dental scientist (Nakabayashi and Pashley, 1998). Phosphoric acid was the agent of choice and was applied to the enamel surface for 30 seconds at a concentration of 85%. The first clinical trial of the method was concerned with the sealing of pits and fissures of molar teeth to prevent their decay. This approach to caries prevention has become an important adjunct to fluoride therapy (Retief et al., 1986). Acid etching by far provides the best technique for achieving both chemical and mechanical adhesion between resin and enamel. The placement of a low-viscosity an adhesive bonding resin before the restorative resin material readily wets tooth surface and allows flow of material into the micro pores created by the etching process (Retief et al., 1986).

The new single step adhesive technique uses acid etching liquid, primer and bonding resin in combination and thereby shortens the etching-bonding procedure. These are simple one step light-curing dentin-enamel bonding resin for resin-based restorations that do not require any additional priming or acid etching (Hewlett, 2003). It contains 4-META (4-methacryloxy ethyl trimellitate anhydride); an adhesive monomer that decalcifies tooth substrate and penetrates through the smear layer to form a hybrid layer creating excellent bond values to dentin and enamel. Recent clinical studies claimed that 4-META has a 90% success level in clinical evaluation (Wilder and Swift, 2000). In vitro study shows that restoration with 4-META reduces the amount of trans-dentinal antigenic challenge through a tight marginal seal, which may have special implications for its capacity to protect the dentin-pulp complex (Kamal et al., 2000). Another investigation confirms that 4-META adhesive liners can be effectively used as a bonding resin between amalgam restorations and tooth structure when it is placed prior to amalgam setting (Abraham et al., 1999).

Composites resin is used as fillings in the anterior teeth more for esthetic reason. Nowadays, new brand of composites are merging up with the used of class I, and class II filling on posterior teeth which requires better strength. Although such esthetics and strength some are
good in achievement, clinicians still facing the same old problem when comes to composite build-up which it may delay curing composite until proper anatomy is achieve. Delay of light activation while removing excess cement may allow diffusion of dentinal fluid into the adhesive/cement interface and result in a diminished bond between these layers (Suh, 2004; Christensen, 2001). Careful implementation of each stage has achieved a gap free attachment under clinical conditions, but the procedure is considered technique-sensitive (Bouillaguet et al., 2001).

In one step adhesive system, etching and priming of the dentin occur simultaneously by infiltrating the smear-covered dentin with acidic resins. Thus critical procedures like rinsing of the etchant and priming of the hydrated collagen fibers are eliminated (Bouillaguet et al., 2001). Results with these newer formulations have been conflicting. In some reports, shear bond strength was found to be inferior to conventional three-step system, while other single bottle system have matched, or exceeded the shear bond strength of current bonding agents. However, recent studies have suggested that combining the primer and adhesive resins into single application step may reduce hybridization effectiveness. Some components within the oxygen inhibitors layers of the all-in-one adhesives are adversely affecting the polymerization of light cured composites, and the magnitude of the interaction increases with time (Nakabayashi and Pashley, 1998). Adverse interaction between acidic monomers in adhesive and photo-sensitizer (tertiary-amine) in light cured composite normally does not occur due to the rapid rate of free radical generation in the photochemical reaction, however such interaction can occur with cured acidic all-in-one adhesives in prolonged contact with light curable composites. Bond failure occurs between adhesive and composite interface, and not between adhesive and dentin (Suh, 2004).

The hypothesis tested in this study is to present that composites bond strengths to dental adhesive using single-step adhesive system is different, depending on different time elapsed between applications of adhesives and curing subsequently placed composites.

**MATERIALS AND METHODS**

Fifteen extracted human single rooted upper premolars caries free and free from any pathology were selected from the Dental Clinic of Universiti Sains Malaysia (USM) hospital. The teeth were randomly selected and divided into 3 groups, which contain 5 teeth per group. Using a circular cutting disc, the coronal portions are sectioned off slightly, just so as to expose the Dentino Enamel Junction (DEJ) of the crowns. Teeth were cut occlusally reaching 1 mm down to DEJ and all teeth were embedded up to CEJ in 7 mm thick acrylic plates, which are 40 mm long and 15 mm wide. The extruded part of the composite to be tested is 4 mm with a diameter of 3 mm. This is designed so that the acrylic plates embedding the teeth can fit directly into the base ‘grip’ that usually holds the lower chamber of an interlinked two chamber Jig assembly which houses a specifically designed template that holds a clamp. For this experiment however, as the servo-hydraulic testing system (Instron model 8874, Instron Corp., Canton, MA, USA) can administer a direct compressive force, the load force will be directly applied on the samples.

Occlusal surface of each tooth was ground using a wet grinder to expose a flat dentin surface. The teeth were checked to make sure no enamel is left on the dentin surface using stereo microscope examination. Tooth surfaces was ground using No. 600 grit-wet sandpaper in a circular motion to remove a thin layer of dentin. Teeth were rinsed and flash dried. Dentin surfaces were applied with single-step adhesive: ‘AQ Bond’ (Sun Medical Co. Ltd., Japan). Hybrid type light cured ‘SDI Rok’ composite resin (SDI Ltd., Australia), was used in this study. The dentin surfaces were primed and bonded following the manufacturer’s instructions. The base liquid is stirred with the AQ sponge for a few seconds. The entire dentin surface applied with 2 successive coatings for 20 seconds, during which the surface is kept wet with the base liquid. A very gentle air blow applied to evaporate the
solvent. The second coating applied to the dentin surface using the AQ sponge. The coat was blown gently until it dried and then polymerizes for 10 seconds with a visible light-curing unit. Metal cylinder mold (diameter = 3 mm and height = 4 mm) was used to obtain same size of composites. The mold were applied on top of the flatten dentin surface as described in Fig. 1. After bonding, the SDI Rok composites were then packed in to fill the mold, and composites then bonded onto each tooth in each group. The delayed light curing time were 45 seconds for the first group, two minutes for the second and five minutes for the third group before light activation.

Figure 1. Sample preparation for shear bond test.

The upper and lower grips were checked to see the objects were clamped tight enough and the alignment for force loading was in correct position. Computer programming and the Servo-hydraulic compression mode of the Instron 8874 were activated (Fig.2). All specimens were prepared for shear bond testing using Instron 8874 machine at a crosshead speed of 2 mm/min. and load cell capacity of maximum 25KN (Fig. 3) as described by Schneider et al. (2000).

RESULTS

The bond-strengths between single step adhesive system (AQ Bond) and light cured composites were tested in 45 seconds (Group 1); 2 minutes (Group 2) and 5 minutes (Group 3). Figure 4 showed the effect of delayed Light-cured on shear bond strength from all groups tested. A Kruskal-Wallis test that used for comparing numerical variables between Group 1; Group 2 and Group 3 showed significant differences (p<0.01) between all groups as given in Table 1.

A Mann-Whitney test statistical analysis showed no significant differences (p>0.05) in bond-strengths between Group 1 (45 seconds delayed) and Group 2 (2 minutes delayed). But, there were significantly differences (p<0.01) of bond-strengths between Group 2 (2 minutes delayed) and Group 3 (5 minutes delayed); whereas Group 3 showed reduced of bond-strengths compared to Group 2 and Group 1 as shown in Table 2 and 3.

Figure 2. Instron 8874 machine with sample to be tested.

Figure 3. Instron 8874 setup for shear bond test.
DISCUSSION

Bond strengths of single-step adhesive systems to the composites tested in this study ranged between 13.69 to 26.72 MPa in 45 seconds light activation delayed group; from 11.81 to 26.97 MPa in 2 minutes delayed group and decreased to ranged between 7.82 to 8.52 MPa in 5 minutes delayed. The bond strength was reduced to the minimum level after 5 minutes delayed curing of composites.

This result is similar to the study on effects of single bottle adhesives on shear bond strength, which resulted in low bond strength when light activation was delayed 10 minutes after application of adhesives (Schiltz et al., 2000). This means that a 10 minutes delay of light activation will destroy some part of photo initiator systems of composite resins. This is an indication that the oxygen inhibited layer that is left in direct contact with the composites in the highly acidic all-in-one or single-step adhesives will interfere with the bonding subsequently applied composites.

Tay et al. (2004) reported in SEM study that adverse interactions between acidic resin monomer in single-step adhesives and tertiary amines in light-cured composites normally do not occur because of the fast rate of free radical generation in photochemical red-ox reactions. However such interactions can occur in single-step adhesives on prolonged contact (delayed) of light cured composites with the cured adhesive layer.

Table 1. Comparing numerical variables between Group1 (45 seconds); Group 2 (2 minutes) and Group 3 (5 minutes).

<table>
<thead>
<tr>
<th>Variables (Mpa)</th>
<th>Time elapsed</th>
<th>n</th>
<th>Median (IQR)</th>
<th>x² statistic (df²)¹</th>
<th>p value²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding strength</td>
<td>45 seconds</td>
<td>5</td>
<td>164 (80)</td>
<td>9.380 (2)</td>
<td>0.009</td>
</tr>
<tr>
<td></td>
<td>2 minutes</td>
<td>5</td>
<td>165 (79)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5 minutes</td>
<td>5</td>
<td>58 (3)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

¹ Kruskal-Wallis test

Table 2. Comparing numerical variables between Group 1 (45 seconds) and Group 2 (2 minutes).

<table>
<thead>
<tr>
<th>Variables (Mpa)</th>
<th>Group 1 Median (IQR)</th>
<th>Group 2 Median (IQR)</th>
<th>Z statistic³</th>
<th>p value³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding strength</td>
<td>23.20 (11.45)</td>
<td>18.50 (12.20)</td>
<td>-0.210</td>
<td>0.834</td>
</tr>
</tbody>
</table>

³ Mann-Whitney test
Table 3. Comparing numerical variables between Group 2 (2 minutes) and Group 3 (5 minutes).

<table>
<thead>
<tr>
<th>Variables</th>
<th>Group 1 Median (IQR)</th>
<th>Group 2 Median (IQR)</th>
<th>Z statistic&lt;sup&gt;c&lt;/sup&gt;</th>
<th>p value&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding strength (Mpa)</td>
<td>18.50 (12.20)</td>
<td>8.27 (0.54)</td>
<td>-2.61</td>
<td>0.009</td>
</tr>
</tbody>
</table>

<sup>c</sup> Mann-Whitney test

Barghi et al. (2002) reported that the adverse effect of time elapsed between the application of single-step adhesives and curing of composite resin varied in different type/brand of adhesives. Micro tensile bond strength (µTBS) in ‘Prime & Bond’ and ‘Clearfil Liner Bond 2V’ single-step adhesives showed significantly lower bond strength after 2 to 5 minutes of delayed curing. This indicates that some components within the oxygen inhibition layers of the single step adhesives are adversely affecting the polymerization of light cured composites, and that the magnitude of the interaction increases with time.

In ultra structural examination of fractured and intact interfaces by SEM and TEM reported by Suh (2004) and Tay et al. (2004) showed micrographs, voids or honeycomb-like fractured interface were consistently observed along the adhesive-composite interfaces, localized to the ‘intermixed zone’ in the 10 and 20 minutes delayed groups. The interfacial abnormalities reduce the bond strengths of ‘intermixed zone’ and lead to low bond strength.

From clinical perspective, clinicians should always be aware of the potential drop in bond strength on prolonged contact of contemporary all in one / single step adhesives with some light-cured composites before light activation. Although clinicians are unlikely to leave a light-cured composites inactivated for more than 5 minutes, clinicians may delay curing composites until proper anatomy is achieved. We can no longer assume that bond failures occur between adhesives and dentin, not between adhesives and composites. With the acidic single-step adhesives, bond failures occur between adhesives and composites (Tay et al., 2004).

**Principal findings**
Composites bond strengths to the dental adhesive by using single-step adhesive system are different, depend on different time elapsed between applications of adhesives and curing subsequently placed composites. Bond strength between composites and single-step adhesive system was reduced after 5 minutes delayed curing of composites.

**Recommendation**
Clinicians should always be aware of the potential drop in bond strength on prolonged contact of contemporary single step adhesives with some light-cured composites before light activation.

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**REFERENCES**


Kamal AMM, Okiji T and Suda H (2000). Responses of class I molecule expressing cells and macrophages to cavity preparation and restoration
with 4-META/MMA-TBB resin. *Intern Endo J*, **33**: 367-373.


