IMMEDIATE DENTIN BOND STRENGTH OF SELF-ETCH DENTINE ADHESIVES

ABSTRACT

AIM: The purpose of this study was to evaluate the immediate bond strength of two sixth generation and two seventh generation dentin bonding agents to superficial dentin. MATERIAL AND METHODS: Specimens were prepared to exposed superficial dentin and either Clearfil SE, Clearfil S3, Adper Prompt-l-pop of G-bond was applied over the dentin surface and light cured. Then composite resin was applied to the treated surface and light-cured in two increments. Specimens were tested 15 minutes after they were made for shear bond strength at 0.01 cm/min. RESULTS: Clearfil SE had statistically higher bond strength than the other three adhesives evaluated (42.9 MPa). There was no statistical difference among Clearfil S3, Adper Prompt-l-pop and G-Bond. CONCLUSION: The dentin adhesive with an application of an acidic primer before the application of the adhesive showed better immediate bond strength.

KEYWORDS

INTRODUCTION

Dentin bonding agents are substances that produce a mechanical interlocking with the demineralized tooth structure, and have a chemical bonding through primary bonds with the composite resin. Through history, these materials have change from multiple components to only one that doesn’t need acid etching to be performed separately. It has evolved from the use of hydrophobic agents with an insufficient penetration to a bi-functional agent with both hydrophobic and hydrophilic properties.\(^1\) These type of materials have acidic primers that produce both: etching of the surface and priming of the dental surface for the bonding, all in one step.\(^1\) This simplifies the bonding process and eliminates the more sensitive steps we had before. The need for a separate acid etching with its washing and drying is eliminated. The SE bonding systems work by conditioning the smear layer and demineralizing the underlying dentin at the same time that in penetrate the dentin with its resin monomers.\(^2\) One problem in the storage of these systems is that they need water as part of they ingredients, but water tends to neutralize the etching effect over time, and the shelf-life of the material is reduced.

Normal acid etching has a lot of advantages and it has been studied for over 40 years, showing that it is needed to produce a reliable bonding, provided that when it is applied to dentin, after it is washed, the dentin must remain moist. Acid etching produces a much greater bond strength when precedes the bonding agent. Acid etching provides a bond to enamel, which some authors have regarded as low, that can benefit from acid etching it before applying the self-etch adhesive.\(^3\)\(^5\) Some studies have found,\(^6\) that there is no statistical difference among self-etching bonding systems compared with total-etch systems, but some of the later do produced a higher bond strength. Some authors reported this bond strength to be low in un-cut enamel, but not so in cut or ground enamel.\(^7\)\(^10\)

As far as dentin, there is a trend to move from total-etch to self-etch adhesive to simplify the application of the adhesive and the application steps. Self-etch adhesives are not necessarily better than total-etch.\(^11\) Some authors have stated that an active application of Self-etch adhesive and a second coat increases bond strength,\(^12\)\(^13\) because, as Walshaw and others found, an insufficient thickness of it was associated with low fracture toughness.\(^14\) Thick films between 60 and 250 are effectively sealing the dentin surface despite moderate fracture toughness results.\(^14\) Etching the dentin before the application of the SE adhesive does not improve the bond strength to dentin as it does to enamel.\(^15\) And though SE adhesives may have high bond
strength at early stages, many have found that, unlike total-etch, it decreases with time.\textsuperscript{16-19}

The purpose of this study was to evaluate the immediate bond strength of two sixth generation and two seventh generation dentin bonding agents to superficial dentin, to establish if they have enough strength to withstand the occlusal forces at early stages of the restoration.

\textbf{MATERIAL AND METHODS}

40 recently extracted human molars, caries and restoration free, stored following ISO 11405-2003, were embedded in acrylic resin and polished under water with 600 grit SiC paper, until deep dentin was exposed. Specimens were divided into four groups (n=10). Each group received a different sixth or seventh generation dentine adhesive. The adhesives (Table 1) used in this study were: G-bond from GC America (Chicago, Illinois), Adper Prompt-l-pop from 3M ESPE (St Paul, Minnesota) and Clearfil S\textsuperscript{3} Bond and Clearfil SE Bond both from Kuraray (Tokyo, Japan).

\textbf{CLEARFIL S\textsuperscript{3} BOND:}

SE Primer was applied to moist dentin rubbing it on the surface for 10 seconds, allowing it to penetrate for 20 seconds before being air-thinned. Immediately, SE Bond was again rubbed on the surface for 10 seconds, allowed to penetrate for another 10 seconds, air-thinned, and light-cured for 20 seconds (\textit{HiLUX Ledmax, Benlioglu Dental}) at 1200 mW/cm\textsuperscript{2}. A second coat of SE bond is applied over the surface by brushing it for 5 seconds, air-thinned, and light cured for 20 seconds.

\textbf{CLEARFIL SE BOND:}

Since this is a seventh generation dentin adhesive, there is only one bottle, it was applied to moist dentin rubbing it on the surface for 10 seconds, allowing it to penetrate for 20 before being air-thinned, and light cured for 20 seconds, a second coat was brushed over the surface for 5 seconds, air-thinned, light cured for 20 seconds.

\textbf{ADPER PROMPT-l-POP:}

The seal is broken and components are mixed following manufacturer's instructions, one coat of adhesive is rubbed over the dentin surface for 10 seconds, then another coat of adhesive is applied the same way, the adhesive is allowed to penetrate for 10 seconds before being air-thinned. Adhesive is light-cured for 20 seconds. A third coat of adhesive is applied over the dentin surface, air thinned and light-cured again for 20 seconds.

\textbf{G-BOND:}

The adhesive was applied rubbing it to the surface for 10 seconds, allowed to penetrate for 20 seconds, air-thinned, and light-cured for 20 seconds, a second coat is
brushed for 5 seconds then air-thinned, and light cured for 20 seconds.

Z-100 composite was cured over the surface of all specimens in two increments, using a Teflon mold of 2 mm in diameter. The first increment was 1 mm deep and light-cured for 40 seconds; the second increment was 2 mm deep, also light cured for 40 seconds. All this would form a composite cylinder of 2 mm diameter and 3 mm tall over the dentine surface.

Table 1. Dentine adhesives used in this study.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Manufacturer</th>
<th>Code</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil SE Bond</td>
<td>Tokuyama</td>
<td>SE</td>
<td>Tokyo, Japan</td>
</tr>
<tr>
<td>G-bond</td>
<td>GC America</td>
<td>GB</td>
<td>Illinois, USA</td>
</tr>
<tr>
<td>Prompt-l-pop</td>
<td>3M ESPE</td>
<td>LP</td>
<td>Minnesota, USA</td>
</tr>
<tr>
<td>Clearfil S³ Bond</td>
<td>Tokuyama</td>
<td>CS</td>
<td>Tokyo, Japan</td>
</tr>
</tbody>
</table>

Table 2. Mean bond strength of dentin bonding agents in this study. Standard deviation is shown in parentheses.

<table>
<thead>
<tr>
<th></th>
<th>Clearfil SE</th>
<th>Prompt-l-pop</th>
<th>G-Bond</th>
<th>Clearfil S³</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPa</td>
<td>42.9 (6.5)</td>
<td>31.5 (10.0)</td>
<td>27.2 (4.3)</td>
<td>27.9 (6.1)</td>
</tr>
</tbody>
</table>

Table 3. Analysis of variance for dependant variable: Bond Strength.

<table>
<thead>
<tr>
<th>Source</th>
<th>df</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding</td>
<td>3</td>
<td>736.203</td>
<td>245.401</td>
<td>4.946</td>
<td>.0099</td>
</tr>
<tr>
<td>Residual</td>
<td>20</td>
<td>992.232</td>
<td>49.612</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Dependent: Bond strength, MPa.

RESULTS

Specimens were made so after 15 minutes of having the seconds layer of composite resin cured over the surface, they were tested for shear bond strength on the universal testing machine (H10K-S, Tinius Olsen, Philadelphia, USA) until break at a crosshead speed of 0.5 cm/min.

Data were stored and shear bond strength was calculated in MPa. Results were analyzed using a one-way ANOVA calculated at a 0.05 significance level; means were compared using the Tukey-Kramer interval, also calculated at a 0.05 significance level.

Results for immediate bond strength are stated in table 2 and interfaces shown on figure 1. It was observed that both sixth generation dentin adhesives had higher values of immediate dentin bond strength than the two seventh generation adhesives tested in this research. The bond strength of both seventh generation adhesives was almost identical, although CS had a higher variation than GB. The adhesive with the higher bond strength value was SE with 43 MPa, and the lower was GB with 27 MPa. ANOVA (Table 3) showed that there was a statistical difference.
among adhesives (p=0.009). Tukey-Kramer interval for comparison of means was 11.3 calculated at a 0.05 significance level, which means that SE was statistically higher that the other three adhesives tested in this research. The other adhesives: GB, LP and CS were not statistically different among them.

Figure 1. SEM pictures of bonding interface of bonding systems used in study. A: Clearfil SE Bond, B: Prompt-l-pop, C: G-Bond, D: Clearfil S3.

DISCUSSION

Good bond strength, right after the final composite is finished is important to prevent post-operative sensitivity and early fracture of the restoration, immediate bond strength is therefore as important as long term bond strength. Many studies describe the performance of self-etch adhesives as far as sensitivity, and longevity of the bond, since they tend not to hydrolyze with water, preventing the collagen fibers from getting loose without their inorganic support. In this study, Clearfil SE Bond had the highest bond strength value of all bonding systems used, it has also been proven to have excellent clinical performance in five years clinical trials. Some studies found that Clearfil SE lower its bond strength after aging and other have found no negative effect of long term water storage. In this study, the two-step self-etch adhesive had the highest bond strength among all systems evaluated. Many authors have also found that Clearfil SE produce better bond strength than one-bottle self-etch systems.

The other two one-bottle SE systems evaluated in this study had the same bond strength, unlike a previous study by Salza and Bock where Clearfil S3 had higher bond strength than G-bond of more than 30%. Prompt-l-pop is considered an aggressive low-pH adhesive (pH=0.9-1.0) and Clearfil S3 and G-Bond are moderate (pH >1.5) this produces a thicker hybrid layer but with some tendencies to dissolve over time and allow nanoleakage, resulting in lowering of bond strength. Clearfil S3 has been found to increase its bond strength after 6 or 12 months storage.
CONCLUSION

Self-etch primers that are applied separately for the adhesive, showed higher bond strength values than those who were combined in one application.

REFERENCES


16. Armstrong SR, Vargas MA, Fang Q Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-


