Esthetic and bonding enhancements of tooth colored indirect restorations
El-Zohairy, A.A.

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CHAPTER 5

Effect of Conditioning Time of Self-etching Primers on Dentin Bond Strength of Three Adhesive Resin Cements.

5.1 Abstract

*Objective.* To evaluate the effect of increasing the dentin conditioning time with self-etching primers of different aggressiveness (pH) on the microtensile bond strength (μTBS) of three resin cements.

*Materials and methods.* Pre-cured composite blocks were cemented with M-Bond, Bistite II DC, or Panavia F to flat occlusal dentin surfaces of human third molars, which were conditioned with M-Bond or Bistite II primer for 30, 60 or 180 s or with Panavia primer for 60 or 180 s. Each dentin-composite block assembly (four in each group) was longitudinally sectioned to obtain 1x1 mm microbars and tested for the μTBS. The morphology of the conditioned dentin surface and the hybrid layer was examined with SEM.

*Results.* Extending the primer conditioning time for Bistite II DC from 30 to 60 s significantly increased the μTBS (p<0.001), but did not further increase from 60 s to 180 s. For M-Bond there was only a significant increase from 30 to 180 s (p<0.05). For Panavia F the primer-conditioning time had no influence on the μTBS. SEM observations of conditioned dentin showed that the Bistite II DC and M-Bond self-etching primers with the lowest pH completely dissolved the dentin smear layer and plugs and formed well-defined hybrid layers. This was not found for Panavia F.

*Conclusions.* Dentin bond strength of resin cements using more aggressive self-etching primers is improved by increasing the conditioning time. To enable resin infiltration of highly filled resin cements, sufficient smear layer dissolution is necessary.
Successful bonding systems typically accomplish resin-dentin adhesion in three steps: etching, priming, and bonding resin application.[1,2] Etching of dentin using strong acidic etchants followed by water rinsing is the first necessary step to remove the smear layer and to expose the collagen fibers of the dentinal matrix.[3] Subsequently, the hydrophilic primer is applied to increase dentin surface energy and to facilitate the penetration of the bonding resin monomer, generating a mixed zone of resin-entangled collagen fibrils, known as the hybrid layer [4] or the 'resin-dentin interdiffusion zone'.[5]

It has been reported that the quality of resin-dentin adhesion can greatly be influenced by the duration of the etching process, and by the amount of dentin surface humidity following rinsing of the acid and prior to resin infiltration.[6,7] Therefore, much of the current research and development in dentin adhesion aims to simplify the bonding procedures and to eliminate all possible technical sensitivities by reducing the number of bonding steps. Historically these developments started when the primer and bonding resin were combined in one bottle. This was followed by the development of a so-called self-etching primer that can etch and prime in one step. Finally, the one-step self-etch, or so-called all-in-one adhesive was introduced, combining the conditioning, priming, and bonding resin in a single step.

Besides the development of adhesive systems for bonding direct restorations, several adhesive resin cements have been recently introduced that utilize self-etching primers for bonding indirectly fabricated restorations. Generally, the formulation of the self-etching primers to combine with these cements includes an aqueous mixture of acidic monomers, such as a phosphate ester or a carboxylic acid; and hydrophilic monomers such as hydroxyethyl methacrylate (HEMA).[8,9] Due to their intrinsic acidity, these primers can simultaneously condition and prime the hard tooth tissues, using the smear layer as an intermediate bonding substrate.[10]

The variation in composition and concentration of the acidic resin monomers in self-etching primers gives rise to variation in the primer acidity. On the basis of the acidity of the primer and its ability to dissolve and/or penetrate dentin smear layers, these systems can be classified into mild, moderate and aggressive.[11] In addition, by virtue of the filler loading and composition of resin monomers in these systems, adhesive resin cements vary in their viscosity and mode of polymerization.

Previous studies on conventional total-etch adhesive systems have reported a reduction in resin-dentin bond strength when the phosphoric acid conditioning time exceeded the recommended 15-30 s.[12,13] This drop in bond strength was attributed to the inability of the
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resin to fully penetrate and impregnate the collagen network to the depth of the demineralized dentin. In the absence of adjunctive acid etching, resin-infiltrated dentin that is formed by using self-etching primers should not exhibit areas of incomplete infiltration, as dentin is simultaneously demineralized and infiltrated by the same resin component.[14] However, there is only limited information concerning the influence of the conditioning time of self-etching primers on bond strength. In addition, self-etching primers of different aggressiveness may produce different conditioning effects on the surface of the smear layer covered-dentin. A relatively mild self-etching primer may not be able to adequately demineralize the smear layer in a way that allows sufficient infiltration and hybridization by the filled adhesive resin cement subsequently applied.

The aims of this study were; 1) to evaluate the microtensile bond strength (µTBS) to dentin of three contemporary adhesive resin cements that use self-etching primers of different acidity; and 2) to examine the effect on bond strength of increasing the conditioning time of these primers from 30 to 60 to 180 s. It was hypothesized that unlike the total-etch adhesive systems, prolonged conditioning times of dentin with self-etching primers would improve the bond strength. To explain and support the results a scanning electron microscopy (SEM) study was performed on fractured surfaces of specimens from the bond strength test and also on intact resin-dentin interfaces and dentin surfaces that were treated using the various treatment times.

5.3 Materials and methods

Preparation of dentin surface

Caries-free freshly extracted human third molars were used in this study to evaluate the bond strength to dentin. Flat dentin surfaces were created by removing the coronal enamel by wet grinding the occlusal surfaces perpendicular to the long axis of the teeth on a polishing machine with 240 grit SiC paper (Buehler Ecomet V, Buehler Ltd, Lake Bluff, IL, USA). To create as much as possible a standardized smear layer the dentine surfaces were finished for one minute by wet grinding with grit 600 SiC paper.

Preparation of composite blocks to bond to dentin

Z100 composite was placed in a Teflon mould (height x diameter = 5 x 10 mm) and cured with an Elipar Highlight (3M ESPE, St. Paul, MN, USA, 800 mW/cm²) each side for 40 s. After light curing the composite cylindrical block was removed and one of the flat surfaces (to be used as the bonding area) lightly ground, followed by sandblasting (50 µm Alumina). The block was
then ultrasonically cleaned in distilled water for 3 min and dried. Finally a mixture of ceramic primer A and B of Bistite II DC (Tokuyama Dental Corp., Tokyo, Japan) was applied in a thin layer to the bonding surface of the composite block and blown dry after 10 s.

**Table 5.1 Resin cements used in this study.**

<table>
<thead>
<tr>
<th>M-Bond self cure unfilled resin cement</th>
<th>Batch X760322 (Tokuyama, Tokyo, Japan)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder (clear)</td>
<td>Polymethyl methacrylate, benzoyl peroxide</td>
</tr>
<tr>
<td>Liquid</td>
<td>Methyl methacrylate, MAC-10*, amine</td>
</tr>
<tr>
<td>Primer (A/B)</td>
<td>Methacryloxyalkyl acid phosphate, acetone Water, acetone, borate catalyst pH = 1.6* (Primer mixture)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Bistite II DC dual cure filled resin cement</th>
<th>Batch EB44442 (Tokuyama, Tokyo, Japan)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paste (A/B)</td>
<td>Silica, zirconia, MAC 10, dimethacrylate, initiator</td>
</tr>
<tr>
<td>Primer 1 (A/B)</td>
<td>Methacryloxyalkyl acid phosphate, acetone, alcohol, water, and initiator pH = 1.8* (Primer mixture)</td>
</tr>
<tr>
<td>Primer 2</td>
<td>HEMA*, acetone, initiator</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Panavia F dual cure filled resin cement</th>
<th>Batch 41194 (Kuraray, Osaka, Japan)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Universal Paste/Catalyst</td>
<td>Silanated barium glass, silanated silica, surface treated</td>
</tr>
<tr>
<td>ED primer A</td>
<td>HEMA, MDP, 5-NMSA, water, accelerator pH = 3.0* (Primer mixture)</td>
</tr>
<tr>
<td>ED primer B</td>
<td>5-NMSA*, accelerator, water, sodium benzene sulphonate</td>
</tr>
</tbody>
</table>

* HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MAC-10, Methacryloxyundecane dicarboxylic acid; 5-NMSA, N-Methacryloyl 5-aminosalicylic acid.

* pH values according to the manufacturers.

**Bonding procedure**

Three adhesive resin cements M-Bond, Bistite II DC, and Panavia F were used in this study for bonding the composite blocks to the prepared flat dentin surfaces. The general composition and batch numbers of the cements and their self-etching primers are listed in Table 5.1. The manufacturers instructions for using the self-etching primers are summarized in Table 5.2. The experiments were divided into three main groups, according to the resin cement used (M-Bond, Bistite II DC, and Panavia F). The M-Bond and Bistite II DC groups were further divided into three subgroups based upon the time of treatment of the self-etching primer: a subgroup with a 30 s treatment time (according to the manufacturer) and two subgroups for which the treatment times were extended, one to 60 s and one to 180 s. Group Panavia F was divided into two
subgroups: a subgroup with 60 s treatment time (according to the manufacturer) and a subgroup for which the treatment time was extended to 180 s. A 30 s treatment for Panavia F was not evaluated, as the minimum time specified by the manufacturer is 60 s. Each subgroup (eight in total) consisted of four dentin samples.

During the cementation procedure the cement thickness was kept constant at a thickness of 100 μm by spacers, while applying a load of 10 N on the composite block. The cements of groups Bistite II DC and Panavia F were light cured for 20 s evenly from all sides.

Table 5.2 Bonding procedures for M-Bond, Bistite II DC, and Panavia F according to the manufacturer’s instructions.

<table>
<thead>
<tr>
<th>Group</th>
<th>Dentin conditioning</th>
<th>Luting procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-Bond</td>
<td>Mix primer A and primer B (one drop each), apply undisturbed for 30 s, air-dry for 5 s</td>
<td>Mix 3 drops of Liquid and 1 scoop of clear powder and apply by bulk-mix technique</td>
</tr>
<tr>
<td>Bistite II DC</td>
<td>Mix primer 1A and primer 1B (one drop each), apply undisturbed for 30 s, air-dry for 5 s. Apply primer 2 for 20 s, air-dry</td>
<td>Mix Bistite paste A and B, apply and light cure 20 s</td>
</tr>
<tr>
<td>Panavia F</td>
<td>Mix ED primer A and B (one drop each), apply undisturbed for 60 s, air-dry</td>
<td>Mix Panavia F paste A and B, apply and light cure 20 s</td>
</tr>
</tbody>
</table>

μTBS evaluation

After a storage period of 24 h in distilled water at 37 °C each dentin-composite block assembly was sectioned into slabs of 1 mm thickness perpendicular to the bonded interface with a low speed cutting saw (Buehler Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). The block of slabs was then rotated 90° and again cut perpendicular to the bonded interface to gain microbars of 1.0 ± 0.1 mm². During this second cutting procedure none of the microbars showed premature debonding of the composite from the dentin. After measuring the cross-sectional area of each microbar with a digital caliper (Mitutoyo Co., Japan) they were fixed with a dental adhesive (Clearfil SE Bond, Kuraray Co., Japan) to a modified micro-tensile testing device (Figure 3.1).

The specimens were tested under tension using a universal testing machine (Model no. 6022; Instron, High Wycombe, Bucks, U.K.) at a crosshead speed of 1 mm/min. To determine the mode of failure, all specimens were immediately examined after fracturing under a stereomicroscope (Olympus, Tokyo, Japan) at a magnification of 20x.

Failure modes were categorized as: a) adhesive failure along the cement-dentin interface; (b) adhesive failure along the composite–cement interface; (c) mixed failure involving cohesive failure of the cement and adhesive failure along a, or b; (d) cohesive failure of the composite, and (e) cohesive failure of the dentin. Representative resin-dentin interface fractures were
additionally examined by SEM (Phillips SEM XL 20, Eindhoven, Holland) to confirm the stereomicroscope observations. Prior to the SEM observations the specimens were air-dried and sputter-coated with gold.

**Statistical analysis**

Bond strength data obtained for the eight subgroups were analyzed using SPSS statistical software package 9.01 (SPSS Inc., Chicago, IL, USA). A two-way ANOVA test was used to examine the effects of resin cement type, treatment times, and the interaction of these two parameters on the bond strength. One-way ANOVA and Bonferroni Post Hoc test were used to identify statistical differences between pairs of means. Statistical significance was set at $\alpha = 0.05$ for all tests.

**SEM study of cement-dentin interface and primed dentin surfaces**

From sixteen additional third molars flat dentin discs were cut with a thickness of approximately 1 to 1.5 mm perpendicular to the long axis of the teeth from the mid-coronal part. A standard smear layer was created on the occlusal surfaces by wet grinding in a similar way as was done for the samples in the $\mu$TBS test. The 16 disks were divided into two sets of eight disks of which each set received the primer pretreatments according to the eight subgroups. One set of eight disks was covered with the corresponding resin cements in a layer of 1 mm thickness and cured, then cut in half and stored in distilled water. This set was used for micro morphological examination of the intact resin cement-dentin interfaces of each subgroup. After 24 h the disk halves were fixed in 2.5% glutaraldehyde, dehydrated in ascending concentration of ethanol (25-50 - 70 - 95 - 100%) and immersed in hexamethyldisilazane (HMDS, Sigma-Aldrich GmbH, Steinheim, Germany) for 10 minutes. The interfaces were polished with wet silicon carbide paper of decreasing abrasiveness (up to 1200 grit) and soft tissue with increasing fine diamond suspension (Buehler Ltd, Lake Bluff, IL, USA) to particle size of 1 $\mu$m. Finally the specimens were demineralized with 6N hydrochloric acid for 45 s and deproteinized by a 10 minute immersion in 2.5% sodium hypochlorite.[15] The specimens were then mounted on aluminum stubs and gold sputter-coated for examination in the scanning electron microscope. The thickness of the hybrid layer was measured from the micrographs. Three measurements were performed on each specimen. No statistical analysis was performed due to the limited number of the specimens and because this study was designed to be qualitative.

The other set of eight disks were used for SEM examination of the morphological characteristics of the conditioned dentin surfaces. After priming the surfaces the specimens were
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rinsed with ethanol for 5 min to remove the primer[16], then fixed, and dehydrated as previously described. The discs were longitudinally fractured, by using a scalpel blade that was inserted into a pre-cut groove in the disc base. One half of each disc was mounted on an aluminum stub to examine dentinal tubules in cross-section and the other half to examine dentinal tubules longitudinally.

5.4 Results

*Microtensile bond strength (μTBS)*

The mean μTBS and standard deviations for the three adhesive resin cements M-Bond, Bistite II DC, and Panavia F with treatment times of 30, 60, and 180 s with the self-etching primers are presented in Table 5.3. For Panavia F a treatment time of 30 s was not evaluated, as the minimum time specified by the manufacturer is 60 s. Two-way ANOVA revealed that the effect of cement type, treatment times and their interaction were statistically significant (*P < 0.001*).

Table 5.3 Mean microtensile bond strength (μTBS) and standard deviations of M-Bond, Bistite II DC, and Panavia F* for 30, 60 or 180 s application times of their corresponding self-etching primer.

<table>
<thead>
<tr>
<th>Group</th>
<th>μTBS of subgroups (MPa)</th>
<th>30 s priming</th>
<th>n</th>
<th>60 s priming</th>
<th>n</th>
<th>180 s priming</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bistite II DC</td>
<td>29.9 (8.4)*</td>
<td>39</td>
<td></td>
<td>54.0 (10.0)b</td>
<td>34</td>
<td>54.4 (10.1)b</td>
<td>32</td>
</tr>
<tr>
<td>Panavia F</td>
<td>*</td>
<td>*</td>
<td></td>
<td>19.0 (6.0)c</td>
<td>32</td>
<td>18.1 (8.6)c</td>
<td>32</td>
</tr>
<tr>
<td>M-Bond</td>
<td>19.6 (6.2)f</td>
<td>37</td>
<td></td>
<td>24.8 (5.3)c</td>
<td>42</td>
<td>29.7 (5.6)f</td>
<td>36</td>
</tr>
</tbody>
</table>

n: Denotes the number of microbars tested for each subgroup.
* Subgroup Panavia F with 30 s application times was not tested, as 60 s is the minimum time specified by the manufacturer. Subgroups with the same letters are not significantly different (*P > 0.05*)

When the treatment times of the self-etching primers were according to the manufacturers recommendations (30 s for Bistite II DC and M-Bond and 60 s for Panavia F), Bistite II DC exhibited the highest μTBS (*P < 0.001*). M-Bond and Panavia F were not significantly different from one another.

Extending the treatment time for Bistite II DC from 30 to 60 s significantly increased the μTBS (*P < 0.001*). However, there was no significant difference between 60 s and 180 s. For M-Bond extending the treatment time from 30 to 60 s had no significant influence on the μTBS. Only when the treatment time was extended to 180 s a significant increase was found in comparison to the 30 s treatment (*P < 0.001*).
Increasing the treatment time for Panavia F from 60 s to 180 s had no significant influence on the µTBS.

Fracture mode

Figure 5.1 shows the distribution of failure modes in percentage for the 284 microbars tested.

For Bistite II DC, the predominant mode of failure after 30 s treatment was adhesive between the resin cement and the dentin (84 %). When the primer treatment time was extended to 60 s there was an increase in the percentage of the adhesive failures at the cement-composite interface (40.5 %).

For M-Bond the majority of failures for the 30 and 60 s treatment times were in the interface of either the cement and dentin or the cement and composite. However, when the treatment time was extended to 180 s a substantial increase of mixed failures occurred, which involved bulk fracture of the cement and fractures in the interfaces.

For Panavia F the predominant mode of failure was adhesive at the resin cement-dentin interface for both treatment times, 60 s and 180 s.

SEM examination of the fractured interfaces

The SEM of representative specimens confirmed the fracture mode recorded by a 20x magnification with the stereomicroscope. Figures 5.2a and 5.2b show the dentin side of fractured microbars from the Bistite II DC and Panavia F subgroups treated according to the manufacturers. These specimens are representative for these subgroups in which the predominant mode of failure is adhesive at the resin cement-dentin interface.

The dentin sides of fractured microbars from the M-Bond subgroups treated for 30 and 60 s are shown in Figures 5.2c and d. The fractured interfaces are dominated by numerous voids along the fractured resin cement-dentin interface (Figure 5.2c) or along the resin cement-composite interface (Figure 5.2d). Failures in the M-Bond subgroup treated for 180 s were predominantly mixed type of failures, while the bulk of the resin cement layer was completely void-free (Figure 5.2e).

SEM examination of the conditioned dentin surfaces

The SEM examinations of the conditioned dentin surfaces after 30 s application of the selfetching primers of Bistite II or M-Bond for 30 s showed that both primers had a similar effect on the dentin surface. Both primers were able to dissolve the smear layer and plugs (Figures 5.3a and b) and widen the dentinal tubule openings (Figure 5.4a).
In contrast, the ED primer of the Panavia F cement partially dissolved the smear layer and plugs after 60 s treatment time (Figure 5.3c), leaving residual smear plugs partially obliterating the dentinal tubules (Figure 5.4c). The longitudinal views of the conditioned dentin samples showed that there were increases in the depth of peritubular dentin demineralization by extending the priming time of Bistite II and M-Bond self-etching primer (Figure 5.4b). However, the SEM micrographs of the ED primer treated samples revealed no change in the depth of peritubular dentin demineralization by extending the priming time.

**SEM examination of the intact resin cement-dentin interfaces**

SEM evaluation of intact resin-dentin interfaces produced by the M-Bond and Bistite II cements after 30 s treatment time with the self-etching primers showed that both cements produced a uniform and well formed hybrid layer that were 1.6-2 \( \mu \) thick. The resin tags were partially hybridized and had some lateral branches (Figure 5.5a and b). On the other hand no hybrid layer could be clearly observed when the interfaces produced by Panavia F cement after 60 s treatment time were examine (Figure 5.5c). In addition, resin tags were few and short. When the treatment times were extended for the three tested resin cements, no obvious differences were found in the resin cement-dentin interfaces in comparison to the interfaces produced by the manufacturers recommendation times.
Figure 5.1 Distribution of failure modes in percentage for Bistite II DC, and M-Bond with primer conditioning time of 30 s, 60 s and 180 s, and Panavia F with primer time; 60 s and 180 s.
**Figure 5.2** SEM micrographs (x1000) of fractured specimens. (a) Dentin side of Bistite II treated for 30 s, showing cohesive failure within the hybrid layer and at the top of the hybrid layer where most of the resin tags remained firmly embedded in the tubules. (b) Dentin side of Panavia F treated for 60 s, showing failure at the top of the hybrid layer where few tubules remained sealed. (c) Dentin side of MB treated for 30 s, showing numerous formations of voids at the resin cement-dentin interface. (d) Failure at the resin cement-composite interface of MB subgroup treated for 60 s, showing the porous structure of the resin cement caused by formation of voids (SEM x2000). (e) Dentin side of MB subgroup treated for 180 s. Voids were absent from the cement and fracture occurred at the top of the hybrid layer.
Figure 5.3 SEM micrographs (x4000) of the dentin surfaces treated with the three self-etching primers. (a) Bistite II primer applied for 30 s, showing complete removal of the smear plugs, exposed outer peritubular collagen fibers (white arrow) and small traces of the smear layer (black arrow). (b) M-bond primer applied for 30 s. The smear layer and the smear plugs were removed with formation of peritubular dentin cuffs (arrows). (c) Panavia F primer applied for 60 s, showing residual smear layer and smear plug partially obliterating the dentinal tubules orifices.
**Figure 5.4** SEM micrographs (x5000), showing lateral views of the conditioned dentin samples. (a) Bistite II primer applied for 30 s, showing widening of the dentinal tubules opening and exposed outer peritubular collagen. (b) Bistite II primer applied for 180 s, showing deeper demineralization of peritubular dentin matrix. (c) Panavia F primer applied for 60 s, showing residual smear plug (arrow) obliterating the dentinal tubule opening.
Figure 5.5 (a) Resin-dentin interface produced by Bistite II cement after 30s treatment time with the self-etching primer (manufacturers recommendation time). Hybrid layer (HL) thickness is 1.6-2 μ. Numerous funnel-shaped resin tags (T) and their lateral branches are observed. (b) Resin-dentin interface produced by M-Bond cement after 30s treatment time with the self-etching primer (manufacturers recommendation time). The hybrid layer is well formed with thickness of 1.8-2μ. Numerous long resin tags could be observed. (c) Resin-dentin interface produced by Panavia F cement after 60s treatment time with the self-etching primer (manufacturer recommendation time). No distinct hybrid layer formation could be observed. The resin tags are short and few (SEM x4000).
5.5 Discussion

The basic mechanism of bonding in restorative dentistry is based on micro-mechanical interlocking derived from an exchange process that involves replacement of minerals from the hard dental tissue by resin monomers.[9] During this exchange process the smear layer that covers the cut tooth tissues acts as a barrier to resin infiltration. There are two approaches for treating the smear layer: (1) by complete removal of the smear layer through rinsing after application of a mineral acidic etchant, such as 30-40% phosphoric acid, or (2) by demineralization of the smear layer through using a self-etching primer, which is a mixture of a hydrophilic resin and acidic monomers. The etching kinetics of the latter approach depend upon the concentration of the acidic component of the primer, the viscosity of the solution, its pH and pKa, the application time, and the thickness and packing density of the smear layer.[10,16,17]

When the self-etching primer is applied to the dentin surface, its acidic component either completely dissolves the smear layer, or creates diffusion channels through the smear layer where the resin can diffuse a short distance into the underlying dentin.[10] The bonding process is completed by the application of a low viscosity bonding agent that co-polymerizes with the resin of the primer, and results in the formation of a thin hybrid layer with a strong resin-dentin bond.[18]

The difference between bonding systems using self-etching primers for direct composite restorations and those for adhesive cements is that the bonding agent of the latter is the resin cement itself. Because the filler loading of the resin cements is higher than that of the bonding agent, the presence of remnants of smear layer and smear plugs after conditioning with the self-etching primers could complicate the infiltration process of the cement into the underlying dentin. This may explain the lower dentin bond strength found for Panavia F than for Bisilit II when the primers were applied according to the time specified by the manufacturers. The ED primer of Panavia F, being the least aggressive among the three self-etching primers tested may not have been able to completely dissolve the smear layer and smear plugs (Figure 5.3c). Thus, demineralization of the underlying dentin and further penetration of the filled cement was limited, resulting in unclear hybrid layer formation and short resin tags (Figure 5.3c). It was reported that the hybridized complex created by a mild self-etching primer consists of a top hybridized smear layer and a subsurface zone of thin hybridized intertubular dentin.[8,11] Such hybrid layers produce interfaces that are too thin (400-500 nm) to be seen by SEM.[19]

In contrast, the self-etching primer of Bisilit II, which has a pH of 1.8, did react more aggressively with the smear layer. The acidic primer dissolved most of the smear layer and
plugs, giving the applied cement the chance to fully penetrate the demineralized dentin substrate (Figure 5.3a). In addition, the acetone in the primer with its water displacing properties may have facilitated deeper infiltration of the resin monomers, resulting in long resin tags and many lateral branches (Figure 5.5a).

When the primer treatment time of Bistite II was increased to 60 s, there was a dramatic increase in dentin bond strength over that achieved after a treatment time of 30 s. Although SEM examination of the resin-dentin interface showed no difference in the hybrid layer thickness between the different treatments times, deeper peritubular dentin demineralization was more evident in samples treated for 60 s and 180 s (Figure 5.4b). This is caused by the higher etching rate for the more mineralized peritubular dentin in comparison to that of intertubular dentin.[20] The continuation of demineralization after the 30 s application time recommended by the manufacturer indicated that the acids in the self-etching primer were not yet neutralized, and were still active. The opportunity for prolonged demineralization with deeper resin infiltration and penetration explains a great deal of the increased bond strength.[21,22] In addition, the increased time between primer application and air-drying could have allowed more solvent to evaporate from the primer. Solvent surplus can weaken bond integrity, or may affect polymerization of the infiltrated monomers.[2] When the treatment time of Bistite II was extended to 180 s, there was no further increase in bond strength over the 60 s treatment. Together with the increased percentage of failures between the cement and the composite substrate for the 60 and 180 s treatments, this finding suggests that a value of 54 MPa is close to the maximum bond strength of Bistite II cement to composite, and cannot become higher.

Unlike Bistite II, Panavia F showed no improvement in bond strength when the primer treatment time was increased, neither did the mode of failure change. Consistently the predominant mode of failure remained adhesive at the resin cement-dentin interface. These results are supported by the SEM observations that there were no differences between the 60 and 180 s treatment with the Panavia F primer. Unlike Bistite II primer, the weaker acids in the ED primer may have been neutralized by the smear layer and the underlying intact dentin already at 60 s, such that prolonging the application time did not cause further demineralization.[23] However, despite the fact that lower dentin bond strengths are produced by cements that utilize less aggressive self-etching primers such as the ED primer, a good seal might be achieved by these systems, as the smear plugs are usually left intact.[22]

For M-Bond, the SEM observations of the conditioned dentin samples after different treatment times with the acidic primer were similar to those of Bistite II. This can be explained by the similarity in chemical compositions of the M-Bond and Bistite II self-etching primers.
Effect of Self-etching Time on Bond strength to Dentin

Like the SEM photomicrographs of Bistite II, the resin-dentin interface of M-Bond also showed the formation of a well-defined hybrid layer (Figure 5.5b). However, the recorded bond strengths for the M-Bond subgroups (30, 60, and 120 s) were significantly lower than those of the corresponding Bistite II subgroups. This may have been caused by the fact that M-Bond is an unfilled cement consisting of linear polymer chains of PMMA, these cements are generally weaker than the Bis-GMA type filled cements, and may thus lower the resin-dentin bond strength.[24]

Also another effect may have played a role, similar to that observed with chemically activated resin composites combined with acidic dentin adhesives. Several studies have reported the occurrence of a low dentin bond strength for such combinations [25,26], which has been ascribed to an acid-base reaction between the acidic resin monomers in the adhesive, and the basic tertiary amine activator in the auto-polymerizing composite. This inactivates the amine. The same may have occurred for the primer/cement combination of M-Bond, resulting in insufficient polymerization of the resin at the interface.[25] This adverse chemical interaction is less likely to happen with light-activated resin systems, which have a higher rate of polymerization and a shorter pre-gel phase.

Although M-Bond cement has a novel initiating system based on an aryl borate catalyst that improves its bonding capability under acidic conditions [27], we speculate that this acidic environment may still have a negative influence on the bond strength of the self-cured resin cement.

The low pH of the M-Bond primer may also have been responsible for the numerous voids seen along the fractured interfaces of the M-Bond subgroups treated for 30 and 60 s (Figures 5.2b and c). As the voids were so great in number, they were probably generated by carbon dioxide gas produced by the reaction between the acidic primer and the carbonate in the dentin apatite.[20] Some may have originated from the production of carbon dioxide gas as a result of decomposition of the benzoyl peroxide initiator in the cement.[26] The fractured specimens of the M-Bond subgroup treated for 180 s did not show voids, but predominantly the mixed type of failure (Figure 5.2d). The extended application time of the M-Bond primer beyond 60 s allowed neutralization of the acidic primer and the escape of the carbon dioxide bubbles from the primer, resulting in a smoother fracture at the interface. Although there was no significant difference in bond strength going from 30 s to 60 s to 180 s treatment times, the difference between 30 and 180 s was significant. This indicates that there was a tendency for the bond strength to be improved when the time of treatment with the primer was extended.
The present study showed that filled adhesive cements that utilize low pH (i.e. moderately aggressive) self-etching primers resulted in higher bond strengths to dentin than those that utilize mild self-etching primers. We also found that extending the treatment time with the more aggressive self-etching primers increased the cement-dentin bond strength; extending the treatment time with the mild self-etching primers had no influence on the bond strength. Thus, the results partially support the hypothesis that was tested.

5.6 References

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