Fluoride-releasing materials for orthodontic appliances

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CHAPTER 3

Effect of enamel pretreatments on bond strength of compomer

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Abstract

Objectives. The objective of this study was to evaluate the effect of various enamel surface treatments on the bond strength of a compomer to enamel.

Methods. Ground bovine enamel specimens were divided into four groups. A compomer (F2000, 3M) was bonded to the specimens using different enamel surface treatments. Two groups examined the effect of application of the F2000 self-etching primer/adhesive (3M) with respect to static or dynamic priming. A third examined use of the primer/adhesive after phosphoric acid etching, and the fourth (control) group provided bond strength of the compomer to phosphoric acid etched enamel with a resin bonding system (Single Bond, 3M). Shear bond strengths for the specimens were measured after 24 hours storage in water at 37°C. Effects of the various surface treatments on enamel were examined by SEM.

Results. Significant differences in bond strength of compomer to enamel were found that were related to the various surface pretreatments. Dynamic priming resulted in higher enamel bond strengths than static priming, and the best bond strengths were obtained when the enamel was etched with phosphoric acid. SEM analysis showed that depth of etch and resin penetration was also directly related to the bond strengths measured.

Significance. Bond strength of compomer to enamel is significantly affected by the method of pretreatment of the enamel.

Key words: enamel, bond strength, compomer, self-etching systems

Introduction

The success of the acid-etch technique for bonding resin-based restorative
materials to enamel is well supported by numerous studies and many years of clinical experience. Traditionally, enamel etching has been with phosphoric acid at concentrations ranging from 30-50%, and for 15-60 seconds. This procedure results in substantial penetration of the resin into microporosities created by the etch and is believed responsible for the strength and durability of the bond. With the recent introduction of self-etching bonding systems, there is less confidence that the same durable micromechanical bond to enamel will be achieved, due to the reduced acidity of many of these systems. Even for phosphoric acid, it has been found that with a concentration of 3% by weight, and a 30 second etch time, the bond strength to enamel was reduced by about 40% compared to etchant concentrations of 10-65% by weight [1]. The observed etch patterns and tag formation for the 3% by weight phosphoric acid-etched samples were minimal, while higher acid concentrations produced well developed etch patterns, and substantial resin penetration.

Relatively few published studies have looked at the ability of self-etching systems to adequately etch enamel and provide strong, durable bonds. Several studies of acidic primer/adhesives used with composites found enamel bond strengths that ranged from 29 to 75% of the bond strengths achieved when the same systems were used, but with a phosphoric acid etch of the enamel [2-5].

A mixture of results was found for the self-etching adhesive system, Clearfil Liner Bond 2, when tested with composite on ground enamel. Two studies found bond strengths equivalent to those obtained when the enamel was etched with phosphoric acid [6,7], but a third study found that when the self-etching primer was used, the bond strength was about 66% of the enamel bond strength achieved when bonding after phosphoric acid etching [8]. When used on unground enamel, this material produced a bond strength that was about 68% of that found for the phosphoric acid etched control [7].

Durability of bonds was studied using thermocycling in water between 5 and 60°C for up to 30,000 cycles as a means of fatiguing the bonds [8]. In this study, the two systems that showed the greatest fatigue effect on enamel bond strength were self-etching systems and were about 55% of their initial bond strength. The best results were shown by two composites bonded to acid-etched enamel which were
relatively unaffected by thermocycling and retained about 96% of their initial bond strength. These results show that the self-etching systems were more prone to fatiguing effects, suggesting that the durability may be compromised.

The effect of fatigue on self-etching systems has also been examined in microleakage studies using thermocycling of Class V restorations, where etching with phosphoric acid or using increased etching time with a self-etching primer resulted in better sealing in Class V restorations [9,10].

These reductions in enamel bond strength and increases in microleakage might be presumed to be a result of reduced etching of enamel by the self-etching systems. Two different acidic primer/adhesives were examined for their enamel etching ability [11,12]. For each of these materials, the investigators found a lack of the characteristic etch pattern and resin tag formation. For the most part, investigations of the self-etching systems indicate a reduced ability to etch the enamel surface, and lower bond strengths than those typically achieved when phosphoric acid etching is used.

The purpose of the present study was to examine the effect of several surface treatments on the bond strength of a compomer (F2000) to enamel. This compomer is recommended for use with either a self-etching primer/adhesive, or with the resin bonding system, Single Bond, which utilizes a phosphoric acid etch.

**Materials and methods**

The commercially available materials used in this investigation are listed and described briefly in Table 1.

**Shear bond strength**

Forty extracted bovine teeth, which had been stored in deionized water at about 5°C for less than 4 months, were embedded in self-curing acrylic, (Quickmount, Fulton Metallurgical Products Corp., Saxonburg, PA, USA) and an area of flattened enamel was exposed using 120, 320 and 600 grit silicon carbide papers. The specimens were randomly divided into four groups of ten teeth each, and pretreatment of the enamel was performed for each group as follows:
Table 1
Materials investigated in bond strength study.

<table>
<thead>
<tr>
<th>Material (abbreviation)</th>
<th>Manufacturer</th>
<th>Type</th>
<th>Batch Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>3M™ F2000 Compomer Primer/Adhesive (F2000 Primer/Adhesive)</td>
<td>3M Dental Products St. Paul, MN, USA</td>
<td>Self-etching primer/adhesive</td>
<td>Lot ACAD 0799</td>
</tr>
<tr>
<td>3M™ Single Bond Adhesive System (SB)</td>
<td>3M Dental Products St. Paul, MN USA</td>
<td>Resin bonding system</td>
<td>7AD</td>
</tr>
</tbody>
</table>

Table 2.
Mean bond strength and failure mode for the test groups.

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean Bond Strength, MPa (sd)</th>
<th>Adhesive</th>
<th>Cohesive (in compomer)</th>
<th>Cohesive (in enamel)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>11.0 (1.6) a</td>
<td>99</td>
<td>1</td>
<td>--</td>
</tr>
<tr>
<td>B</td>
<td>16.8 (2.3) b</td>
<td>96</td>
<td>4</td>
<td>--</td>
</tr>
<tr>
<td>C</td>
<td>24.2 (2.8) c</td>
<td>68</td>
<td>32</td>
<td>--</td>
</tr>
<tr>
<td>D</td>
<td>26.9 (4.1) c</td>
<td>24</td>
<td>63</td>
<td>13</td>
</tr>
</tbody>
</table>

Groups identified with different superscript letters are significantly different (p<0.01)
Group A - Static Priming: F2000 Primer/Adhesive was applied to the enamel surface with a brush, left undisturbed for 30 seconds, then air-dried gently for 10 seconds, followed immediately by light curing with a Visilux™ 2 Visible Light Curing Unit (3M Dental Products, St. Paul, MN) at 650 mW/cm² for 10 seconds.

Group B – Dynamic Priming: F2000 Primer/Adhesive was applied to the enamel surface, reapplied several times with gentle agitation during the 30 second application time, and then air-dried gently for 10 seconds. This was followed immediately by light curing for 10 seconds.

Group C – Phosphoric acid etch/Prime: Phosphoric acid gel etchant (35% wt/wt) was applied to the enamel and left undisturbed for 15 seconds, then rinsed and dried gently. Two consecutive coats of F2000 Primer/Adhesive were applied and immediately air-dried gently, followed by a 10 second light cure.

Group D – Phosphoric acid etch/Singl e Bond: The enamel was etched with phosphoric acid gel etchant for 15 seconds, rinsed and dried as in Group C. Two consecutive coats of Single Bond Adhesive were applied and immediately air dried gently, followed by a 10 second light cure.

After the specified enamel pretreatment, a 2 mm thick Teflon mold with a hole 4 mm in diameter was clamped over the treated area and filled with the F2000 compomer prior to light curing for 40 seconds. The samples were stored in deionized water at 37 °C for 24 hours, after which the shear bond strength between the F2000 and the enamel was measured using a wire loop on an Instron universal testing machine at a crosshead speed of 2 mm/min. Statistical differences between groups were determined by ANOVA with Tukey-Kramer comparisons of groups at a p=0.01 level.

SEM analysis of etch pattern
Scanning electron microscopy was used to visualize the effect on bovine teeth, of the various surface treatments utilized in the bonding study. Areas of flattened enamel were exposed using 120, 320 and 600 grit silicon carbide papers, with four specimens prepared using each of the following surface treatments: A) 30 second, static application of F2000 Primer/Adhesive; specimens were rinsed with deionized water for 15 seconds, with acetone for 10 seconds, to remove primer, and then air dried gently. B) 30 second, dynamic application of F2000 Primer/Adhesive, with gentle agitation and replenishment of primer/adhesive;
specimens were rinsed with deionized water for 15 seconds, with acetone for 10 seconds, and then gently air dried. C) 15 second, static application of 35% phosphoric acid gel; specimens were then rinsed with deionized water for 15 seconds and gently air dried. All specimens were affixed to SEM stubs, sputter coated with gold-palladium and examined on a JEOL 820 scanning electron microscope. Representative images were captured digitally and stored in computer files.

SEM analysis of resin penetration
The resin and enamel interfaces showing resin penetration were also examined. Four bonded specimens were prepared similarly to each group that was tested in the bonding study. Each was cross-sectioned and polished on 600 grit silicon carbide papers. The sections were etched for 60 seconds with 50% (v/v) nitric acid, rinsed thoroughly with deionized water and gently air dried. Specimens were mounted on SEM stubs with the cross-sectioned surfaces exposed for examination by SEM. Representative images were saved.

Failure mode analysis
Visual examination of failure mode of the bonding specimens was accomplished by viewing all of the debonded samples under a light microscope at about 12x. Failure was identified as “adhesive” if no compomer remained on the enamel surface, “cohesive in compomer” if remnants of the compomer remained in the bonded area and “cohesive in enamel” if a portion of the tooth was removed in the debonding of the material. Failure mode was estimated to the nearest 5%.

Results

Shear bond strength
Mean shear bond strengths to enamel for the four treatment groups are shown in Table 2. In Groups C and D, where phosphoric acid etching was used, mean enamel bond strengths of 24.2 MPa and 26.9 MPa respectively were obtained and were not statistically different (p=0.05). Both were statistically greater than the bond strengths of Groups A and B (p<0.01). Additionally, in Group B, where dynamic priming was employed, a mean bond strength of 16.8 MPa was found and this was significantly greater (p<0.01) than the mean bond strength of 11.0
MPa for Group A, where static priming was used.

**SEM analysis of etch pattern**
Representative enamel etch patterns obtained by applying the self-etching primer in static and dynamic modes as used in bonding Groups A and B are shown in Fig.1a and 1b respectively. Figure 1c shows the etch pattern for phosphoric acid etching as used in Groups C and D.

**SEM analysis of resin penetration**
Representative photomicrographs of resin penetration resulting from the four enamel surface treatments are shown in Fig. 2. Figure 2a shows minimal resin penetration when static priming was used (Group A), while Fig. 2b shows greater resin penetration after dynamic priming, as used in bonding Group B. Figure 2c shows penetration of the self-etching primer/adhesive into phosphoric acid etched enamel, while Fig. 2d shows penetration of Single Bond adhesive, again into phosphoric acid etched enamel.

**Failure mode analysis**
The mode of failure for the four bonding test groups, as determined by observation under optical microscope, is shown in Table 2. Where the self-etching primer/adhesive was used, there was a progressive increase from Group A to Group C in the amount of cohesive failure in the compomer material, and a corresponding decrease in adhesive failure. Group D had a higher percentage of cohesive failure in compomer and 13% cohesive failure in the enamel.

The category “adhesive failure”, for the optical observations above, showed variations from glossy to a slightly matte appearance, suggesting subtle differences in the microscopic failure at that interface. Figure 3 shows representative SEM micrographs of the variations in the enamel surface classified as “adhesive failure” by optical analysis. Figure 3a shows the typical appearance of the surface categorized as having “adhesive failure” for Group A, while Groups B and C were primarily characterized by surfaces as shown in Fig. 3b. Finally, Fig. 3c shows the typical surface for an “adhesive failure” area of Group D. These figures demonstrate an increasing amount of microscopic enamel fracturing at the interface which accounts for the increasing amount of matte surface observed.
Figure 1a. SEM view of the enamel etch pattern after 30 s static application of the self-etching primer/adhesive to flat-ground enamel. No retentive surface can be seen.

Figure 1b. Enamel etch pattern after 30 s dynamic application of the self-etching primer/adhesive. The enamel rod structure has become apparent in the shallow etch.

Figure 1c. Enamel etch pattern after phosphoric acid etching for 15 seconds. A characteristic retentive etch pattern showing enamel rods and deep interrod porosity is exposed.
Figure 2a. SEM showing minimal resin penetration into enamel following 30 s static application of the self-etching primer/adhesive.

Figure 2b. SEM showing somewhat greater resin penetration into enamel following dynamic application of self-etching primer/adhesive compared to static priming as shown in Fig. 2a.

Figure 2c. SEM showing resin penetration of the self-etching primer/adhesive into phosphoric acid etched enamel. Note the more extended tags that are reflective of the deeper etch pattern.

Figure 2d. SEM showing penetration of Single Bond adhesive into phosphoric acid etched enamel. Extended tags reflect the deeper etch.
Figure 3a. SEM micrograph of debonded area representative of "adhesive failure" for Group A. There is a lack of microscopic fracturing of enamel.

Figure 3b. SEM of debonded area characteristic of "adhesive failure" for Groups B and C. Shows increased microscopic fracturing compared to Group A.

Figure 3c. SEM micrograph of debonded area representative of "adhesive failure" for Group D. Note greater microscopic fracturing compared to all other groups.
Discussion

One requirement for achieving a good bond to enamel is the creation of adequate microporosity by means of an acidic conditioner or primer. For the compomer used in this study, preconditioning of the enamel is recommended with either an enamel conditioner (35% phosphoric acid) or with an acidic primer/adhesive, which contains approximately 2.5% (wt/wt) maleic acid. The purpose of this study was to contrast these two methods of enamel surface conditioning and their effect on the bond of F2000 compomer to enamel.

For Group A and B, where the primer/adhesive was used, a statistically greater bond strength was achieved when it was applied dynamically (Table 2). In Figs. 1 and 2, it is clear that the degree of etching and monomer penetration that occurred with dynamic priming was greater than with static priming. This increased bond strength can presumably be attributed to the greater degree of etching. This is not surprising, since agitation and replenishment of the primer/adhesive assures that the enamel surface is exposed to fresh acid, and the etching by-products are removed more efficiently.

Group C and D both used a 35% phosphoric acid treatment, but in Group C, the acidic primer/adhesive was subsequently applied to the etched enamel, while in Group D, the resin adhesive, Single Bond was applied. This latter technique is one of the recommended procedures for the F2000 compomer. There was no statistical difference in enamel bond strength between the two groups even when tested at p=0.05, suggesting that either the F2000 primer/adhesive or Single Bond could be used with F2000 compomer when coupled with phosphoric acid etching. The resin penetration demonstrated in Fig. 2c and 2d appears to be equivalent for the two treatments. The bond strengths of these groups were both statistically greater than Groups A and B. Since Group C used the same primer/adhesive system as Groups A and B, the increased bond strength presumably is due to the increased etching and resin penetration afforded by phosphoric acid etching.

Despite the increased bond strength for Group B, there was only a very small increase in the cohesive failure in compomer recorded by visual examination compared to Group A. However, SEM observations of the regions of adhesive
failure showed a relatively undamaged enamel surface for Group A as depicted in Fig. 3a, whereas Group B showed a trend toward some regions with microscopic fracture of enamel prisms as seen in Fig. 3b. With the deeper etch in Group C, the extent of cohesive failure in compomer increased substantially. Examination of the “adhesive failure” regions of the debonded specimens indicated that virtually the entire area was characterized by microscopic fracture of enamel prisms as seen in Fig. 3b. When the Single Bond adhesive was used (Group D) on phosphoric acid etched enamel, even greater amounts of cohesive failure occurred in the compomer, and there was additionally some cohesive failure of enamel. The microscopic fracture of enamel in the regions judged as “adhesive failure” was substantially greater than for the other groups, as seen in Fig. 3c. The greater microscopic and macroscopic fracturing of enamel, as seen in Group D, may be attributed to the fact that the Single Bond adhesive is composed of several dimethacrylate monomers, which can result in a highly crosslinked adhesive, unlike the F2000 Primer/Adhesive which has no dimethacrylate functionality. The greater strength that might be achieved with the Single Bond adhesive could account for the visual and SEM observations seen in failure mode analysis. Though obvious microscopic differences were observed between Groups C and D, the bond strength was not statistically different.

The study data support the idea that the degree of microporosity created by pretreatment of the enamel surface is an important factor in the bond strength achieved. Similar observations have been made when contrasting the use of PSA Primer/Adhesive or phosphoric acid to pretreat enamel when bonding Dyract [2-4]. The enamel bond strengths with the PSA Primer/Adhesive ranged from 29 to 54% of the bond strengths achieved when the same system was used, but with a phosphoric acid enamel etch. In a similar study, Prime and Bond 2.0 Adhesive used with Dyract, and Syntac Single Component used with Compoglass, produced enamel bond strengths which were approximately 75% of those produced when the enamel was first etched with phosphoric acid [5]. The above studies did not examine degree of etch, or resin penetration into enamel.

The above results are consistent with those from a microleakage study that compared pretreatment with Prime and Bond 2.1 [9]. The leakage at both the enamel and dentin margins of Class V cavities was greater when the primer was
used alone compared to when phosphoric acid etching was used. In addition, SEM analyses of Prime and Bond 2.1, Prime and Bond NT and Syntac Sprint primer/adhesives found little evidence of etch patterns or resin tags compared to when phosphoric acid etching was used [11,12]. The data from these studies collectively suggest that the self-etching primer/adhesives used were not able to provide an adequate level of enamel demineralization to achieve optimum bonding to enamel.

Two studies of the Clearfil Liner Bond 2 system, which utilizes a self-etching primer with a separate adhesive component, have found bond strengths to enamel that were equivalent to those found with phosphoric acid etching, even though SEM observation showed minimal etch patterns on enamel [6,7]. However, one of these studies compared bond strengths on ground and intact enamel for the Clearfil Liner Bond 2 and MacBond II systems, and on the latter type of enamel, the bond strengths for both materials were about 68% of those achieved when composites were bonded and phosphoric acid etching was used. The etch pattern of Liner Bond 2 primer, particularly on intact enamel, was extremely weak [7]. In another study, the bond strength to ground enamel for Clearfil Liner Bond 2 was about 66% of composites bonded to enamel where the enamel was etched with phosphoric acid [8]. Further, a microleakage study found that two successive 30 second applications of Clearfil Liner Bond 2 primer resulted in significantly lower leakage than when the recommended single 30 second application was utilized [10].

The results of the present study are consistent with those cited above. Bond strengths for Groups A and B were about 40 to 70% of those for Groups C and D, where phosphoric acid was used to etch the enamel surface. The combined results of the present study and those cited above suggest that the use of self-etching primers may provide inadequate demineralization of enamel for optimum bonds to be achieved.

Not only is the initial bond strength important, but the durability of the bond under fatiguing conditions is important to provide an indication of how the bonds may perform under clinical conditions. One study examined the effect of thermocycling bonded specimens in water for up to 30,000 cycles between 5 and
Three self-etching adhesive systems and four bonding systems that use phosphoric acid etching were used to bond composite to bovine enamel. The self-etching systems produced initial enamel bond strengths that were generally lower than the initial bond strengths for the standard bonding systems. Under fatiguing conditions of thermocycling, the bond strength for the groups using the self-etching systems were reduced to 53 to 72% of their initial bond strengths, while the standard bonding systems had post-thermocycling bond strengths of 79 to 96% of their initial bonds. Not only were initial bond strengths lower for the self-etching systems, but they were also more susceptible to thermocycling effects, with resultant loss of bond strength. For example, the Clearfil Liner Bond 2 system had an initial enamel bond strength of 16.4 MPa, which dropped to 8.7 MPa after 30,000 cycles. However, when a composite was bonded to phosphoric acid etched enamel with Single Bond, an initial bond strength of 24.4 MPa was seen, and was only somewhat reduced to 23.3 MPa after similar thermocycling. This particular study suggests that the bonds from self-etching systems are more susceptible to fatiguing effects. Additional fatigue studies are needed to determine if all self-etching systems are prone to this effect.

The bond strength of F2000 to enamel was found to be highly dependent on the pretreatment of the enamel surface. These results have potential implications in the clinical use of the F2000 compomer material, but other self-etching systems could face similar issues. Because placement techniques by individual practitioners may vary anywhere from static placement of the primer/adhesive, to a more dynamic placement, where the primer/adhesive is agitated and replenished, it might be expected that the ability to adhere and seal a restoration could be directly affected. Furthermore, cavities can have vertical as well as horizontal enamel surfaces, the former requiring a dynamic primer application to ensure adequate exposure to the primer. Even with dynamic priming, the bond strengths measured in this study were less than those achieved when phosphoric acid etching was used. Whether this reduction in enamel bond strength has clinical significance would best be determined through clinical investigations.
References


