Bond strength and micro morphology of a self-etching primer versus a standard adhesive system with varying etching times in primary teeth

J.R. BOJ, A.M. MARTÍN, E. ESPASA, O. CORTÉS

Abstract. Aims These were to firstly evaluate the shear bond strength of a composite resin to primary dental enamel treated with a standard adhesive system but with varying phosphoric acid etching times along with a self-etching prime, secondly to analyse the etching patterns using SEM. Methods Forty primary molars were used. In the first three groups, following acid etching, a layer of Prime & Bond NT adhesive was employed, and in group 4 the self-etching adhesive system (Prompt-L-Pop) was used. A composite cylinder (Spectrum, Denstply, Germany) was then applied. After processing of the samples, the teeth were subjected to shear stress using an Instrom universal machine. The study of primary enamel morphology after etching for different time periods and using different etching systems was carried out with a scanning electron microscope. Statistics Data were analysed using one way ANOVA, followed by Scheffé test to determine differences between groups. Results In the groups subjected to acid etching for 5 and 15 seconds significant differences were recorded versus the self-etching group (p<0.0001). Regarding the SEM study of primary enamel morphology, there was predominately a clear and marked type 2 etching pattern. Conclusions Longer acid etching times for primary enamel do not appear necessary, as 15 seconds suffice to obtain retentive etching patterns, and the bonding strengths obtained with the traditional etching technique followed by two-step adhesive application are sufficient to ensure good composite resin bonding to the enamel surface. However, the self-etching adhesive system employed yields less than optimum bonding strength to primary enamel surfaces.

Keywords: Bond strength, Enamel patterns, Self-etching system.

Introduction
The use of resin based restorative materials has increased in recent years thanks to improvements in the adhesive systems used. However, their application to primary teeth may represent a problem, as primary and permanent teeth show differences in enamel and dentine morphology and composition. One of the problems with the use of these materials in paediatric dentistry is the working time needed. Early studies indicated that 2 minutes of etching time were needed in primary enamel to ensure good enamel patterns. This time has been changed in subsequent studies over the years by different authors. Nowadays many reports state that between 15 and 30 seconds of etching time would be enough for both permanent and primary enamel [Mazzeo et al., 1995; Megid and Salama, 1997; Jumlongras and White, 1997]. An alternative to reduce the total working time and the chance of introducing errors during the dental treatment is the use of self-etching primers, which avoid the rinsing and the drying steps in the adhesion process [Fritz et al., 2001; Oberländer et al., 2001; Inoue et al., 2003].

In 1955 Buonocore introduced the acid etching technique, involving the application of 30% phosphoric acid for 30 seconds, thereby generating a rough enamel surface with microporosities that can be penetrated by the applied adhesive resins [Perdigao et al., 1997; Kugel and Ferrari, 2000; Rosa and Perdigao, 2000]. However, this technique, applied to primary teeth, may not produce the same effects, due to the greater thickness of the prismatic layer in which the crystalline components of the enamel are distributed in parallel and perpendicular to the surface.
Buonocore [1955] also noted that primary enamel is more resistant to acid etching, and in view of its characteristics could contribute to limited resin penetration, thereby reducing the strength of restorative adhesive material bonding to the primary tooth. At that time, two minutes of acid etching of the enamel were recommended in such cases. However, recent studies show few significant differences in the strength of adhesion between primary and permanent teeth. Thus, 15 seconds of acid etching seem to suffice for both primary and permanent enamel [Swift et al., 1995; Cehreli et al., 2000; Hosoya et al., 2000].

Acid etching induces important morphological changes in the outer enamel layers. The selective loss of tissue produces a variety of prismatic patterns including the preferential elimination of organic and inorganic material from the center of the prisms and their periphery. Galil and Wright [1979] described 5 acid etching patterns following the exposure of human dental enamel to acid solutions:
- Type 1 - etching pattern involving preferential dissolution at the center of the prisms;
- Type 2 - etching pattern involving preferential dissolution at the periphery of the prisms;
- Type 3 - etching pattern representing a combination of the previous two patterns;
- Type 4 - etching pattern in which the enamel shows jigsaw, map-like or network irregularities;
- Type 5 - etching pattern in which the enamel surface appears smooth and level.

The conventional adhesive systems use three different agents: an enamel and dentine conditioning acid, a primer, and an adhesive resin. An alternative is offered by recently introduced self-etching adhesive systems. These simplify the adhesion process, eliminating the cleansing and drying step (critical for the demineralized matrix), and moreover shortening operating time and reducing the possibility of operating field contamination [Ferrari et al., 1997; Kanca, 1997; Perdigao et al., 1999].

In addition to the conventional monomers such as 2-hydroxyethyl methacrylate (HEMA), these systems contain acidic monomers capable of etching and penetrating the enamel simultaneously. They afford an aqueous solution of phosphoric acid esters that dissolve the superficial enamel and dentinal zone, forming a continuum between the treated dental surface and the adhesive material [Bishara et al., 2001; Tay and Pashley, 2001; Van Meerbeek et al., 2001].

The aim of the present study was, therefore, to evaluate the strength of adhesion of a composite restorative material to primary enamel subjected to etching for different times (5, 15 and 30 seconds) and involving different etching and adhesive systems: etching with 36% phosphoric acid followed by the application of Prime & Bond NT adhesive (Dentsply, Germany) and the new self-etching systems, Prompt L-Pop (ESPE, Germany) with an analysis of the etching patterns thus obtained.

**Materials and methods**

**Adhesion study.** Forty primary molars without clinically detectable structural alterations were used. The lingual and vestibular surfaces of each tooth were polished with discs (average grain 5/8 and fine grain 3/4) to reduce their convexity and leave a flat enamel area to be etched. The series was divided into four groups of 20 working surfaces each:
- Group 1 - etching with 36% orthophosphoric acid for 30 seconds;
- Group 2 - etching with 36% orthophosphoric acid for 15 seconds;
- Group 3 - etching with 36% orthophosphoric acid for 5 seconds;
- Group 4 - single-step self-etching adhesive system for 15 seconds, following the instructions of the manufacturer.

Following acid etching, the samples in the first three groups were washed for 10 seconds and pressure air dried for another 10 seconds. A layer of Prime & Bond NT adhesive was applied, followed by a 30 seconds pause, then air was gently blown to dry and the material polymerized for 10 seconds. A composite (Spectrum, Dentsply, Germany) cylinder measuring 4 mm in diameter and 3 mm in height was then applied.

In the samples belonging to Group 4 the self-etching adhesive system (Prompt-L-Pop) was applied for 15 seconds, followed by drying until a fine film was obtained, and photopolymerized for 10 seconds. A composite (Spectrum) cylinder measuring 4 mm in diameter and 3 mm in height was then applied.

In order to simulate oral conditions, the prepared specimens were stored in distilled water when not being prepared or tested and thermocycled for 500 cycles at 5°C and 55°C with a 30 seconds pause time. After processing of the samples, artificial roots were prepared to allow inclusion in plaster blocks, leaving only the crown with the treated surfaces exposed. Shear bond loading test of each composite resin cylinder until failure was accomplished using a chisel shaped blade, lined perpendicularly with the cylinder of resin in an Instrom, model 1011HIGH machine at a cross speed of 5 mm/minute. Shear bond strength was calculated as the ratio of fracture load and bonding area expressed in Megapascals (Mpa).
**SEM study.** This was carried out with a scanning electron microscope, employing 8 caries-free primary canines lacking clinically detectable structural alterations. Each canine was sectioned mesiodistally and longitudinally, leaving a lingual and a vestibular surface. These surfaces were in turn polished with discs (average grain 5/8 and fine grain 3/4). In order to evaluate the etching patterns, the samples were divided into four groups of four working surfaces each, followed by distribution of the groups as described previously.

After acid etching, the samples of the first three groups were washed with water for 10 seconds, and pressure air dried for another 10 seconds. In Group 4 the self-etching adhesive system was applied for 15 seconds, followed by alcohol irrigation to clean the surface. An acetone layer was then applied for 30 seconds to eliminate the adhesive, followed by pressure air drying to allow visualization of the etching pattern created by the acid monomers.

The samples were coated with an electron-conducting colloidal silver solution, dried and vacuum sprayed with gold. The specimens were then examined under a Hitachi 2300 scanning electron microscope, evaluating the resulting etching patterns according to the classification of Galil and Wright [1979].

**Statistics.** Data were analyzed using ANOVA one way, followed by Scheffé test to determine differences between groups (p<0.05).

**Results**

Table 1 summarizes the shear bond strengths (MPa) obtained for primary enamel treatment with 36% orthophosphoric acid at each of the specified time periods and using the self-etching adhesive system. The greatest strength corresponded to the group subjected to acid etching for 5 seconds with 36% orthophosphoric acid (26.4 MPa), while the lowest value corresponded to the self-etching system (11.6 MPa).

The results considering the different 36% phosphoric acid enamel etching times were analyzed by ANOVA one way test and no statistically significant differences between groups were noted. Thus, the mean adhesion strength obtained in the group subjected to acid etching for 30 seconds was 19.25 MPa, versus 24.85 MPa for the 15 second groups, and 26.47 MPa for the group subjected to etching for 5 seconds.

Taking into account the acid etching system employed, it was found that the difference in adhesion strength between the group subjected to etching for 30 seconds and the self-etching was not significant. However, in the groups subjected to acid etching for 15 and 5 seconds significant differences were recorded versus the self-etching group.

Regarding the SEM of primary enamel morphology, the samples subjected to 36% orthophosphoric acid etching showed Galil and Wright [1979] type 1, 2 and 3 etching patterns, in the same way as the samples treated with the self-etching adhesive system (Fig. 1). However, in this latter case the resulting patterns were more irregular and of lesser depth in some cases. The most frequently identified pattern corresponded to type 1. On the other hand, on considering the influence of etching time, Figure 2 (corresponding to surfaces etched for 30 seconds) shows a clear and marked type 2 etching pattern at different magnifications, with clear dissolution of the periphery of the enamel prisms. The Galil and Wright type 2 pattern likewise predominated in the specimens subjected to 36% orthophosphoric acid etching for 15 seconds, though in some cases type 1 images were seen either alone or in combination with type 5 images (Fig. 3). Lastly, Figure 4 shows the pattern generated by 36% phosphoric acid applied for 5 seconds. The most frequently observed pattern again corresponded to type 2, though we also observed types 1 and 3, and even some aprismatic pattern images.

<table>
<thead>
<tr>
<th>Group</th>
<th>N</th>
<th>Minimum</th>
<th>Maximum</th>
<th>Mean ±SD</th>
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<tr>
<td>Group 4</td>
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<td>37.37</td>
<td>11.63</td>
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</table>

**Table 1** - Results of the adhesion strengths for different groups of primary teeth using self-etching or standard adhesive systems.

Group 1 = 36% orthophosphoric acid - 30 seconds
Group 2 = 36% orthophosphoric acid - 15 seconds
Group 3 = 36% orthophosphoric acid - 5 seconds
Group 4 = single step etching - 15 seconds
To study the shear bond strength of a resin to enamel, the buccal surface of a premolar is usually used [Nör et al., 1997; El-Kalla and García-Godoy, 1998]. As the present investigation aimed to study the shear bond strength to primary enamel, primary molars were used. Due to the difficulty in obtaining sufficient molars, it was decided to use both buccal and lingual surfaces of first and second, maxillary and mandibular, primary molars. The enamel surfaces were polished to leave them flat and for morphological conditions to be as similar as possible among different surfaces.

It is generally accepted that bonding strengths of between 17 and 24 MPa are required for permanent teeth to resist the forces generated by polymerization contraction or shrinkage. In our study the samples treated with 36% orthophosphoric acid for 5, 15, and 30 seconds yielded adhesion strengths within the range considered adequate for permanent teeth. In the same way, García-Godoy and Gwinnett [1991; 1992] conducted adhesion tests with primary teeth, etching the enamel surface for periods of between 15 and 120 seconds. No differences were observed in the results obtained not even on comparing them with the results afforded by permanent teeth [Costa et al., 1998]. In turn, some authors published similar results, no differences being observed between phosphoric acid application for 5 and 30 seconds. Others likewise reported no differences between 60 and 240 seconds of etching [García-Godoy and Gwinnett, 1991; Gwinnett and García-Godoy, 1992].

Hosoya and Goto, [1992] observed that samples etched for 10 seconds afforded greater adhesion strengths than those etched for 20 seconds, though in contrast the specimens subjected to acid etching for 30 seconds exhibited the greatest bonding strengths. In this study the samples processed with the self-etching adhesion system failed to yield the minimum bonding strength required in the case of permanent teeth, the mean strength being 11.63 MPa (±7.73). Indeed, some of these teeth were rejected on the grounds that the
composite cylinders detached spontaneously without the application of forces of any kind. Our results coincide with those of other authors who found the self-etching systems to afford poor performance compared with the conventional adhesion systems used for bonding resin-type composites and compomer in both primary and permanent teeth [da Silva Telles et al., 2001; Agostini et al., 2001; Pashley and Tay, 2001].

However, according to other studies these self-etching adhesive systems represent a good alternative, as they reduce the number of required treatment steps versus the traditional systems that involve separate acid etching and adhesive application procedures [Hannig et al., 1999; Perdigao et al., 1999; Kaaden et al., 2002].

On the other hand, the required duration of acid etching of primary teeth is subject to controversy. A number of studies have used different etching times in an attempt to produce adequate etching patterns in primary enamel, varying from 240 to 15 seconds [García-Godoy and Gwinnett, 1991].

In this study the 5 acid etching patterns described by Galil and Wright [1979] were identified and recorded more or less defined porosity according to the type of treatment used. The type 2 pattern predominated in the samples treated with 35% orthophosphoric acid for 5, 15 and 30 seconds, with prior polishing of the enamel surface, in coincidence with the observations of Costa et al. [1998], in which peripheral enamel prism dissolution was the most frequent pattern associated with acid etching for 10, 20 and 60 seconds. Likewise, Hosoya and Goto [1992] found the type 2 pattern to be the most prevalent in primary teeth, regardless of the duration of acid etching or the tooth area treated. Other authors, however, have reported a predominance of the type 1 pattern after 30, 60, and 120 seconds of acid etching, while the type 2 pattern has been fundamentally associated with 15 seconds of acid treatment [Tandon et al., 1989].

In the case of the samples treated with the self-etching adhesive system, type 1 and type 2 etching patterns were recorded, though the results were less uniform than in

**Fig. 3** - Primary teeth samples treated with 36% orthophosphoric acid for 15 seconds. a) Type 2 pattern, magnification 1,500X; b) combination of type 1 pattern, with clear dissolution of the prism centres, exhibiting smooth zones that may correspond of a type 5 etching pattern, magnification 1,500X.

**Fig. 4** - Primary teeth samples treated with 36% orthophosphoric acid for 5 seconds. a) Clear type 2 pattern, with peripheral dissolution of the enamel prisms, magnification 1,500X; b) scantly defined type 1 etching pattern, with peripheral dissolution of the enamel prism centres, magnification 1,500X.
the case of acid etching with 36% orthophosphoric acid. Shimada et al. [2002] found that although the enamel patterns obtained with the self-etching system are not as evident as those afforded by orthophosphoric acid treatment, the resulting retention performance is sufficient for both primary and permanent teeth.

Conclusions

Longer acid etching times for primary enamel do not appear necessary, as 15 seconds suffice to obtain retentive etching patterns. The bonding strengths obtained with the traditional etching technique followed by two-step adhesive application are sufficient to ensure good bonding to the enamel surface, but yield less than optimum bonding strength to primary enamel surfaces.

References


