Scanning Electron Microscopy Evaluation of the Bonding Mechanism of a Self-etching Primer on Enamel

Julio P. Cal-Neto; José Augusto M. Miguel

Abstract: The aim of this study was to analyze the effect of a self-etching primer (Transbond Plus SEP, 3M Unitek, Monrovia, Calif), developed for orthodontic use, in the regularity and depth of adhesive infiltration in the enamel of human permanent teeth and to compare it with phosphoric acid using scanning electron microscopy (SEM). Thirty premolars were divided in two groups of 15 each: group 1 (control)—phosphoric acid + Transbond XT Primer (3M Unitek) and group 2—Transbond Plus SEP. Transbond XT Adhesive Paste (3M Unitek) was used in both groups for bracket bonding. All products were used according to the manufacturer’s instructions. Dental fragments were decalcified, and for micromorphologic observation of the adhesive penetration in enamel, the resin replicas, remnant at the base of the brackets, were covered with a thin gold layer and examined by SEM. Three calibrated examiners evaluated the photomicrographs and gave scores from 0 without penetration to 2 deep penetration. The Mann Whitney U-test (P < .0001) showed a statistical difference between the two groups. The results demonstrated that the SEP was more conservative and produced a smaller amount of demineralization and less penetration of adhesive in the enamel surfaces when compared with the conventional phosphoric acid system. (Angle Orthod 2006;76:132–136.)

Key Words: Phosphoric acid etching; Scanning electron microscopy; Self-etching primer

INTRODUCTION

The enamel-etching technique presented by Buonocore is commonly used with composite resin when attaching brackets to the enamel surface. Phosphoric acid etching causes dissolution of interprismatic material in the enamel, producing a roughened and porous layer that ranges in depth from five to 50 μm. In the past years, there has been a major research drive to increase bond strength between dental materials and dental hard tissues, although most of the adhesive systems in use have provided clinically acceptable bond strengths. Despite the fact that the acid-etching technique is a useful procedure in the orthodontic field, there is a need to simplify the technique to reduce the number of steps and to improve the bonding procedure in order to maintain clinically useful bond strengths while minimizing the amount of enamel loss.

New bonding systems used in operative dentistry were developed to combine conditioning and priming agents into a single acidic primer for simultaneous use on enamel and dentin, eliminating steps of separate etching, rinsing, and drying. The use of a self-etching primer (SEP) would have the advantage of a faster and simplified application technique, allowing adequate etching and priming of enamel and dentin in only one step. In addition to saving time, fewer steps in the bonding process might translate into fewer procedural errors, minimizing technique sensitivity.

Recently, a new SEP, Transbond Plus Self-Etching Primer (3M Unitek, Monrovia, Calif) was developed especially for orthodontic bonding. It includes methacrylated phosphoric acid esters, which will both etch and prime the enamel surface before bonding. The manufacturers also claim that good results can be achieved, with a more conservative etch pattern thereby reducing the enamel dissolution.

The purpose of this study was to investigate under laboratory conditions the bonding mechanism of an SEP in enamel of human permanent teeth by scanning
electron microscopy (SEM). The null hypothesis was that there would be no difference in the regularity and depth of adhesive penetration between groups, whether a conventional multistep or an SEP system was used.

**MATERIALS AND METHODS**

A total of 30 human premolars, free from caries and fillings, were used. These teeth had been extracted for orthodontic reasons with the informed consent of the patients. The teeth were washed in water and stored in a 0.1% thymol solution for no longer than six weeks before use. The buccal surfaces were cleaned and polished with a rubber cup and slurry with pumice and water, followed by rinsing with a water spray and drying with compressed air.

Orthodontic metal brackets (Dyna Lock Series, 3M Unitek) were used in this study. The teeth were divided into two groups, and brackets were bonded on the buccal surfaces according to the manufacturer’s instructions following one of two protocols.

- **Group I** (control group)—phosphoric acid/Transbond XT primer. The area where the bracket was to be located was etched with a 37% phosphoric acid gel (3M Dental Products, St Paul, Minn) for 15 seconds and then washed with water. After washing, the enamel surface was completely dried with compressed oil-free air. A layer of Transbond XT primer was applied on the tooth. Transbond XT paste was applied to the base of the bracket and pressed firmly onto the tooth. Excess adhesive was removed from around the base of the bracket, and the adhesive was light cured, positioning the light guide of an Ortholux XT Visible light curing unit (3M Unitek) on each interproximal side for 10 seconds.

- **Group II**—Transbond Plus SEP. SEP was gently rubbed onto the surface for approximately three seconds with the disposable applicator supplied with the system. Then, a moisture-free air source was used to deliver a gentle burst of air to the enamel. The bracket was bonded within 15 seconds of priming with Transbond XT adhesive paste as in group I.

Afterward, the crowns were sectioned from the roots with a diamond disc (Superflex 273D, Horico, Berlin, Germany) at the cementalabial enamel junction, and each crown was cut longitudinally in a mesiodistal direction. All samples were stored for one week in distilled water at room temperature.

To observe the adhesive penetration into the enamel, specimens were mounted with brackets in epoxy resin and submitted to demineralization cycles, which promoted complete dissolution of the dental structures. On average, five consecutive cycles were carried out, which were composed of 10% chloridric acid solution for five hours and 5% sodium hypochlorite solution for one hour, and all baths and cycles were intercalated with a five-minute distilled water rinse. After complete dissolution of the dental tissues, the specimens were placed on aluminum stubs, resin replicas remnant at brackets base were sputter-coated with gold (Edwards Coater S150B, Edwards, London, UK) and evaluated under a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan).

To standardize the microscopic observations, the microphotographs of the composite resin replicas were made at 1000× and 3500× for each enamel surface in the center of the samples. The lowest magnifications showed the uniformity of the etch pattern of the enamel, whereas the highest magnifications demonstrated morphological characteristics of resin tags penetrating both substrates. The samples were evaluated double-blind by three different calibrated examiners that gave comparative scores according to the following adhesive penetration on enamel: 0, without penetration; 1, shallow penetration; and 2, deep penetration. Characteristic microphotographs of each score were used previously to perform intra- and interexaminer calibration.

The Mann-Whitney U nonparametric test was used to determine whether significant differences were present in the adhesive penetration between the two groups. The significance level was predetermined at a probability value of 5% or less.

**RESULTS**

The null hypothesis was rejected. The Mann-Whitney U-test indicated that the adhesive penetration when the SEP was used was significantly lower ($P < .001$; mean rank = 26.91) than when phosphoric acid and the primer were used separately with conventional adhesive system (mean rank = 64.09) (Table 1).

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<tr>
<th>TABLE 1. Frequency Distribution of Scores and Mean Ranks of Adhesive Penetration in Enamel</th>
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<tr>
<td>Scores</td>
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<tr>
<td>Phosphoric acid + Transbond XT primer</td>
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<td>Examiner 1</td>
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<td>Examiner 2</td>
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<td>Examiner 3</td>
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<td>Transbond Plus SEP</td>
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* SEP indicates self-etching primer.
* The Mann-Whitney U-test detected significant difference between groups ($P < .001$).
FIGURE 1. (A) Enamel-etch pattern obtained after phosphoric acid treatment (group 1). Uniform formation and deep penetration of resin tags are clearly noted (scanning electron microscopy [SEM] 1000×). (B) Higher magnification of resin-tag formation (SEM 3500×).

FIGURE 2. (A) Resin sample of enamel conditioned with Transbond Plus self-etching primer. The etch pattern was shallower than that observed in Figure 1A, minimizing the enamel demineralization (scanning electron microscopy [SEM] 1000×). (B) The resin tags were short and regularly formed (SEM 3500×).

The intact enamel surfaces etched with different acids had different surface morphologies. A characteristic and uniform etch pattern was observed in the resin samples of the phosphoric acid/Transbond XT primer group, revealing increased roughness and resin tags penetrating the demineralized enamel surface (Figure 1).

The use of Transbond Plus SEP produced a uniform etch pattern that was more conservative and less destructive to the enamel surface. Consequently, a regular resin tags distribution was observed, which showed less magnitude when compared with the control group (Figure 2).

**DISCUSSION**

Etching tooth enamel with phosphoric acid creates surface irregularities and microporosities that can be filled through adhesive penetration. The presence of a hybrid layer formed by cured resin on phosphoric acid–etched enamel has been the major factor responsible for the enamel adhesion of resin-based composite.7,8

Enamel etching with phosphoric acid created an etch pattern characterized by a deep and uniform demineralization area. These demineralized areas were infiltrated by the resin of the priming solution, producing well-formed resin tags penetrating into demineralized surface, which is in agreement with previous studies.4,9,10 The use of phosphoric acid on enamel has been associated with an increase in the superficial roughness, rendering the enamel more retentive and producing a higher bond strength.10 However, this may not be desirable clinically because there are concerns that such bonding levels may be higher than what is required for a successful orthodontic bonding. In addition, Çehreli and Altay11 observed that regardless of
treatment time, etching with 37% phosphoric acid results in irreversible damage of the enamel surface. Currently, there is an increasing preference for milder etching procedures.\textsuperscript{15–14}

SEPs are agents that combine conditioning and priming into a single clinical step. They do not have to be rinsed off with water but just spread gently by applying a stream of air. As the monomers that cause etching are also responsible for bonding, the depth of the demineralized zone corresponds to the depth of penetration of the adhesive to be polymerized. This avoids problems with insufficient penetration depth and improves the quality of hybridization.\textsuperscript{8,15}

Compared with phosphoric acid, Transbond Plus SEP produced a uniform and more conservative etch pattern, with regular adhesive penetration and a less-aggressive enamel demineralization. The resin tags were shorter than those observed in control group. However, in the context of bond strength, the increase of surface area and the rheological properties of the resin may be more significant than the depth of adhesive penetration.\textsuperscript{11,16,17} This can be inferred from previous laboratory investigations that suggested that Transbond Plus SEP can successfully bond orthodontic brackets as well as when phosphoric acid is used with Transbond XT primer.\textsuperscript{6,8,18}

The method used in this study, ie, using SEM and completely decalcified resin samples, is extremely simple and very useful for the evaluation of resin tags and to assess the etching pattern of enamel surfaces.\textsuperscript{19} According to Ferrari et al,\textsuperscript{20} low magnifications show the uniformity of the etch pattern of enamel and the density and real depth of the resin tags, whereas high magnifications demonstrate the morphological characteristics of the resin tags penetrating enamel. Perhaps this is the most adequate technique to evaluate the bonding mechanism of SEPs on enamel, although the evaluations have been essentially subjective regarding the depth of etching. SEPs have demonstrated a shallower etch pattern. This might be simply because of a poorer penetration of the SEP into the enamel porosities or the result of interference from mineral precipitates on enamel surface that mask the etch pattern. Because the acidic primer is not rinsed off, phosphorous and calcium ions released from the hydroxyapatite crystal dissolution remain suspended in the primer solution.\textsuperscript{8} As in this study, if resin replication rather than direct observations of enamel were evaluated, exact information could be obtained about the etching pattern and the adhesive penetration into enamel surface, regardless of the acid-etching technique. This is true because with the SEPs the ionic precipitate remains embedded in the resin after polymerization.

Because of its new properties, SEPs are interesting and promising materials. From a clinical standpoint, the use of SEPs can be desirable because they reduce clinical steps, save chair time, improve the adhesive procedures, and reduce the risk of salivary contamination. Furthermore, the claim that SEP produces a more conservative etch pattern than phosphoric acid, thereby minimizing the enamel loss, was confirmed in this study. However, to recommend the use of this product in a large scale, more studies are required, particularly in vivo studies and clinical trials.

**CONCLUSIONS**

- Transbond Plus SEP produced a more conservative etch pattern and a lower adhesive penetration than when 37% phosphoric acid and a separate primer were used.
- The results of this study indicate that SEP is potentially adequate for orthodontic bonding needs.

**ACKNOWLEDGMENTS**

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