# Phosphoric Acid Incorporated with Acidulated Phosphate Fluoride Gel Etchant Effects on Bracket Bonding

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**Abstract:** The aim of this study was to evaluate the effects of etching with phosphoric acid incorporated in an acidulated phosphate fluoride (APF) gel on the bonding of a bracket and the loss of sound enamel. In the control group, the enamel was etched with 37% phosphoric acid for 30 seconds. In the experimental groups, the enamel was etched for 30 seconds with 37% phosphoric acid blended with 1.23% APF gel at various ratios (25%, 33%, 50%, 67%, and 75% APF gel). The brackets were bonded with Transbond XT light-cured orthodontic adhesive according to the manufacturer's instructions. The specimens were then treated under three different conditions: 37°C for one hour, 37°C for 24 hours, and thermocycling (2500 times) between 5°C and 55°C in deionized water. The shear bond strength of 10 specimens in each condition was measured and the results analyzed using a Tukey multiple comparison test ($P = .05$). The shear bond strength decreased significantly as the fraction of the APF gel increased in the experimental etchant. An apparent increase in the adhesive remnant index score was also observed in the large fraction of the APF gel. To minimize the damage of the sound enamel surface during the etching and debonding procedures, a mixture of phosphoric acid and an APF gel (50% and 67% APF fraction) can be used as an phosphoric acid etchant substitute without loss of the proper bracket bond strength. (*Angle Orthod* 2005;75:678–684.)

**Key Words:** Etchant; Acidulated phosphate fluoride gel; Enamel surface; Shear bond strength; Adhesive remnant index; Surface damage

## INTRODUCTION

Orthodontic brackets are routinely bonded to the teeth using an acid etch technique. Because most orthodontic bonding adhesives are composite resins, the current techniques involve applying 37% phosphoric acid to the tooth enamel for approximately 15–30 seconds.[1] This acid etching causes the dissolution of the interprismatic material in the enamel, producing an irregular enamel surface that facilitates the retention of an orthodontic bracket.[1] Acid etching has been reported to cause from 5 up to 10 μm of enamel loss.[1] The permanent loss of enamel calcium during the acid etching procedure may render the enamel surface more susceptible to demineralization during and after the orthodontic treatment.

The bonding of the brackets to the tooth surfaces is a temporary procedure to the extent that the brackets are removed after the active treatment period. Therefore, greater consideration has been given to the debonding techniques in addition to the effect that these procedures have on the enamel surfaces. Damage to the enamel can be attributed to not only the acid-etching procedure but also to the process of bracket removal and cleaning of the teeth after debonding. Therefore, new bonding techniques other than those with conventional phosphoric acid are needed to minimize the significant enamel loss during orthodontic treatment. Several alternative approaches to bonding have been investigated, such as different enamel preparations[2,3] and adhesives systems.[4–6]

Many studies have demonstrated that a topical fluo-

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**TABLE 1.** The Enamel Conditioner Used in This Study[a]

| Group | APF gel:37% Phosphoric Acid (Weight%) |
|---|---|
| Group 1 | 3A:1P (75% APF) |
| Group 2 | 2A:1P (67% APF) |
| Group 3 | 1A:1P (50% APF) |
| Group 4 | 1A:2P (33% APF) |
| Group 5 | 1A:3P (25% APF) |
| Control | 0A:1P (0% APF) |

[a] APF indicates acidulated phosphate fluoride.

ride is effective in increasing the resistance to dental caries or enamel demineralization.[7–14] Several methods of applying a topical fluoride to increase the resistance of the enamel to caries attack have been suggested.[10,11] These include the topical applications of the fluoride before etching, the incorporation of fluorides in the etching solutions, the topical application of fluorides to the etched enamel surfaces before bonding, and the topical applications of the fluorides after bonding. However, some studies showed controversial results as to the effect of topical applications of fluoride on the bracket bond strength.[7–14]

The null hypothesis tested was that there is a significant decrease in enamel loss, but acceptable bond strength remained after the enamel etching with the etchants that were formulated with phosphoric acid and acidulated phosphate fluoride (APF) gel. The aim of this study was to evaluate the effect of etching with phosphoric acid incorporated with an APF gel on the shear bond strength of the orthodontic brackets and the damage to the enamel surface.

## MATERIALS AND METHODS

A total of 180 extracted human premolars were stored in a 0.12% thymol (Aldrich, St Louis, Mo) solution. All the teeth were examined macroscopically to ensure that the buccal surface was both intact and caries-free. The teeth were divided randomly into six groups of 30 teeth each. They were mounted vertically in an acrylic resin block ($18 \times 18 \times 14$ mm$^3$) with a

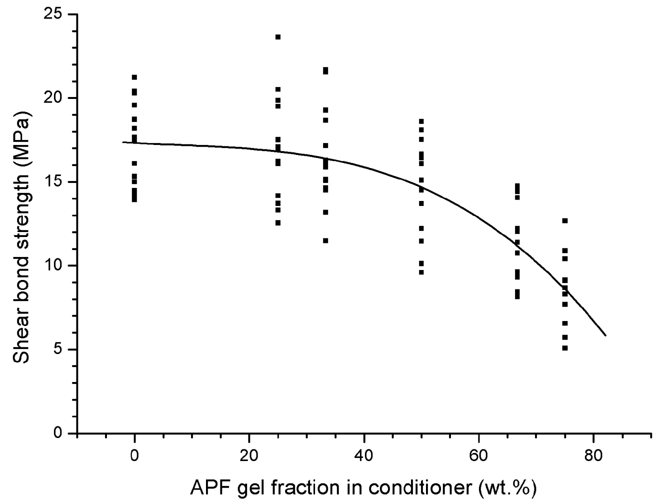


**FIGURE 1.** Shear bond strength of the bracket after 24 hours as function of the acidulated phosphate fluoride (APF) fraction on the etchant.

self-cure acrylic resin. The buccal enamel surfaces of the teeth were cleaned with fluoride-free pumice (Moyco, Philadelphia, Pa) in a rubber cup, sprayed with water, and dried with a compressed oil-free stream for 15 seconds.

In the control group, the teeth were etched with 37% phosphoric acid (Vericom, Anyang, Korea) for 30 seconds. In the experimental groups, the teeth were etched with 37% phosphoric acid blended with a 1.23% APF gel (Pascal, Bellevue, Wash) at various ratios for 30 seconds (Table 1). The etched teeth were washed and dried with oil-free compressed air. Standard metal premolar brackets (Micro-Loc, Tomy, Japan) were bonded according to the manufacturer's instructions with Transbond XT (3M Unitek, Monrovia, Calif), and any excess resin was thoroughly removed using an explorer before the resin was light-cured. A halogen light-curing unit (VIP, Bisco, Schaumburg, Ill) with 11-mm curing light guide was used for curing for a total of 40 seconds from each of the mesial, distal, gingival, and occlusal sides. Light energy density at the end of the light guide was 500 mW/cm$^2$.

After the complete sample preparation, all the spec-

**TABLE 2.** Shear Bond Strength (MPa) of the Specimens Treated at Various Conditions[a]

| Code (APF %) | 1 h | 24 h | Thermocycled |
|---|---|---|---|
| Group 1 (75%) | 7.63 (2.42)$^{A,1}$ | 8.57 (2.27)$^{C,1}$ | 7.26 (1.08)$^{G,1}$ |
| Group 2 (67%) | 8.41 (2.42)$^{A,2}$ | 11.22 (2.32)$^{C,D,3}$ | 13.39 (2.62)$^{H,3}$ |
| Group 3 (50%) | 9.33 (2.63)$^{A,4}$ | 14.62 (3.01)$^{D,E,5}$ | 14.92 (2.22)$^{H,I,5}$ |
| Group 4 (33%) | 13.42 (2.27)$^{B,6}$ | 16.47 (2.64)$^{E,F,7}$ | 15.77 (3.17)$^{H,I,6,7}$ |
| Group 5 (25%) | 13.56 (2.83)$^{B,8}$ | 16.77 (2.65)$^{E,F,9}$ | 17.83 (1.19)$^{I,J,9}$ |
| Control (0%) | 14.05 (3.26)$^{B,10}$ | 20.94 (2.50)$^{F,11}$ | 18.03 (2.66)$^{J,12}$ |

[a] Means within the columns with the same superscript letters are not significantly different ($P > .05$), and means within the rows with the same superscript numbers are not significantly different ($P > .05$).

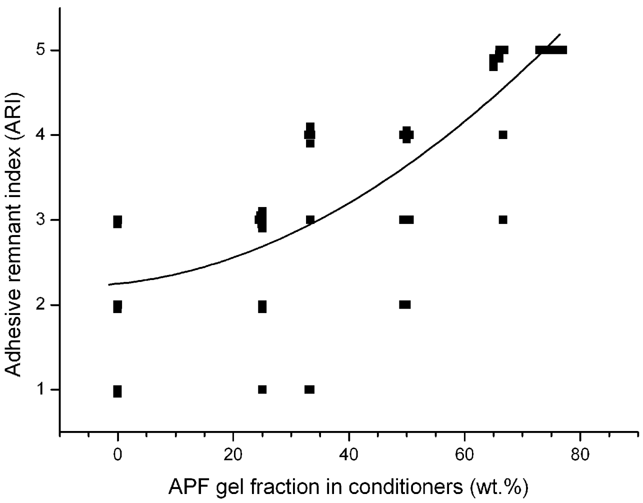


**FIGURE 2.** Adhesive remnant index (ARI) scores after 24 hours as function of the acidulated phosphate fluoride (APF) fraction on the etchant.

imens were kept at room temperature for 10 minutes and stored in either a deionized water bath at 37°C for one hour or 24 hours or 2500 thermocycling steps (Thermal cycling system, Kwangju, Korea) ranging from 5°C to 55°C, with a dwell time of 30 seconds in deionized water.

The embedded specimens were secured in a jig attached to the base plate of a universal testing machine (4465, Instron, Canton, Mass). A chisel-edge plunger was mounted to the movable crosshead of the testing machine and was positioned so that the leading edge was aimed at the enamel-adhesive interface at a crosshead speed of one mm/minute. The bond strength was calculated using the nominal surface area of the bracket (11.488 $mm^2$). The results were analyzed using a Tukey multiple comparison test ($P = .05$).

After the debonding test, the debonded enamel surfaces were examined by the same operator using a stereomicroscope (SMZ-U, Nikon, Tokyo, Japan) with 5× magnification. The amount of adhesive remaining on the enamel surface for each tooth was scored as the adhesive remnant index (ARI),[15] which is a method of visually determining where the bond failure has occurred. The ARI score ranged from 1 to 5: 1 indicates that all the adhesive remains on the enamel, with the impression of the bracket base; 2 indicates that ≥90% of the adhesive remains; 3 indicates that ≥10% but ≤90% of the adhesive remains; 4 indicates ≤10% of the adhesive remains; and 5 indicates that no adhesive remains on the enamel surface.

Scanning electron microscopy (SEM) (JSM-840A, Jeol, Tokyo, Japan) was used to observe the enamel surface conditioned with the different experimental etchants, and energy dispersive X-ray analysis (EDXS, Oxford, Inca, Oxon, UK)
with 15 kV was performed to detect fluoride on the enamel surface. The surface roughness was measured using confocal laser scanning microscopy (LSM5 Pascal, Carl Zeiss, Thornwood, NY).

## RESULTS

The shear bond strength characteristics of the test groups are shown in Table 2. The shear bond strength

**TABLE 3.** Adhesive Remnant Index (ARI) Scores[a]

| Group | ARI Score | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|---|
| 1 (75% APF) | 1 h | 0 | 0 | 0 | 0 | 10 |
| | 24 h | 0 | 0 | 0 | 0 | 10 |
| | Thermocycling | 0 | 0 | 0 | 0 | 10 |
| 2 (67% APF) | 1 h | 0 | 0 | 0 | 2 | 8 |
| | 24 h | 0 | 0 | 1 | 1 | 8 |
| | Thermocycling | 0 | 0 | 0 | 5 | 5 |
| 3 (50% APF) | 1 h | 0 | 0 | 2 | 4 | 4 |
| | 24 h | 0 | 0 | 1 | 3 | 6 |
| | Thermocycling | 0 | 0 | 2 | 3 | 5 |
| 4 (33% APF) | 1 h | 0 | 1 | 2 | 7 | 0 |
| | 24 h | 1 | 1 | 4 | 4 | 0 |
| | Thermocycling | 0 | 2 | 3 | 4 | 1 |
| 5 (25% APF) | 1 h | 0 | 4 | 4 | 2 | 0 |
| | 24 h | 1 | 3 | 6 | 0 | 0 |
| | Thermocycling | 0 | 3 | 6 | 1 | 0 |
| Control (0% APF) | 1 h | 2 | 3 | 3 | 1 | 0 |
| | 24 h | 3 | 3 | 4 | 0 | 0 |
| | Thermocycling | 1 | 3 | 5 | 1 | 0 |

[a] The ARI score used had a range between 5 and 1:[15] 1, indicates that all the adhesive remains on the enamel; 2, less than 90% of the adhesive remains; 3, more than 10% but less than 90% of the adhesive remains; 4, less than 10% of the adhesive remains; and 5, no adhesive remains on the enamel surface.

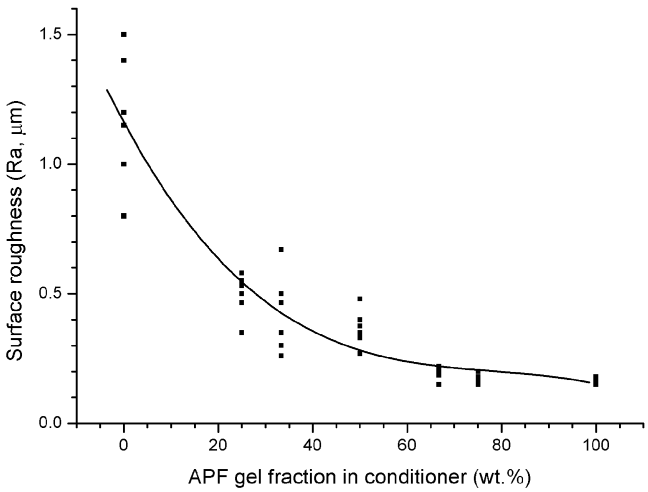


**FIGURE 3.** Surface roughness as function of the acidulated phosphate fluoride (APF) fraction on etchant.

decreased significantly, from 14.05–20.94 MPa to 7.26–8.57 MPa, as the fraction of the APF gel in the etchant increased as shown in Figure 1 ($P < .05$). However, all the different fractions of the APF gel incorporated with the 37% phosphoric acid provided clinically acceptable shear bond strength (6–10 MPa).[1,16–19]

The sites of bond failure in the different treatment groups along with the ARI score are shown in Figure 2. The groups conditioned with phosphoric acid incorporated with the APF gel had less resin remaining on the enamel than the groups conditioned with only phosphoric acid. An apparent increase in the ARI score was also observed with the etchants with a large fraction of the APF gel. The ARI score was 5 for group 1, and mainly 4 or 5 for groups 2 and 3. The enamel/resin interface was the most common failure site for groups 1–3 (Table 3). However, the bracket/resin interface was the most common site of failure in the control group. Spearman's correlation coefficient between the group (APF fraction) and the ARI score was −.763. Pearson' correlation coefficient between the ARI score and the shear bond strength was .597.

From the confocal laser scanning microscopic evaluations, the surface roughness ($R_a$) ranged from 0.80–1.50 μm for the control, 0.35–0.58 μm for group 5, 0.26–0.67 μm for group 4, 0.27–0.48 μm for group 3, 0.15–0.22 μm for group 2, and 0.15–0.20 μm for group 1. In contrast, only the APF gel–treated group and untreated group showed 0.15–0.18 μm and 0.10–0.16 μm, respectively (Figure 3). A three-dimensional trace of the different treatment groups is shown in Figure 4. The surface roughness was highest in the control group and decreased significantly as the fraction of the APF gel in the etchant was increased ($P < .05$). Spearman's correlation coefficient between the group (APF fraction) and the surface roughness was .877. Pearson' correlation coefficient between the surface roughness and the shear bond strength was .600.

The SEM images of the enamel treated with the various etchants are shown in Figure 5. Variations in the etching patterns were observed. The microscopic observations indicate that the enamel etching pattern of the 37% phosphoric acid etching group was rough and the amount of enamel loss decreased apparently as the fraction of the APF gel increased. The EDXS examinations did not detect fluoride on the enamel surface–treated etchants with APF gel.

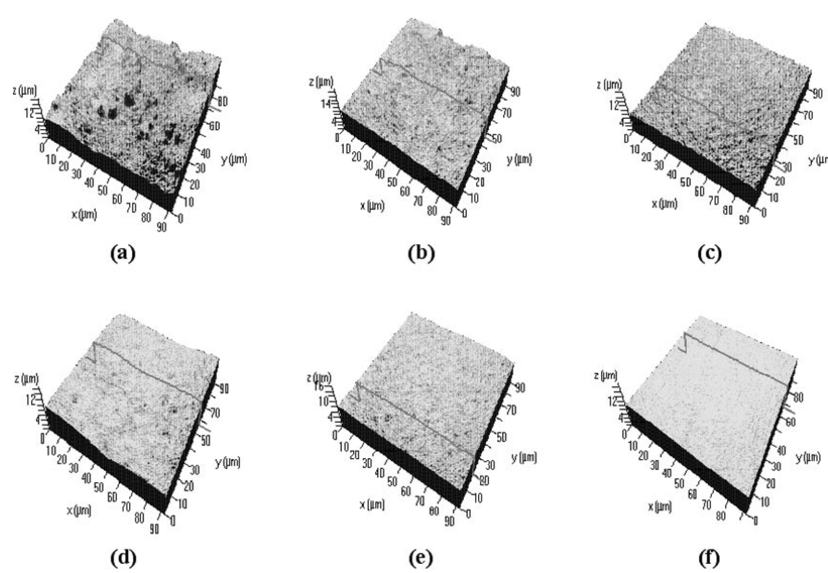


**FIGURE 4.** A three-dimensional trace of the enamel surface with various etchants by confocal laser scanning microscopy (1000×): (a) control group, (b) group 1, (c) group 2, (d) group 3, (e) group 4, and (f) group 5.

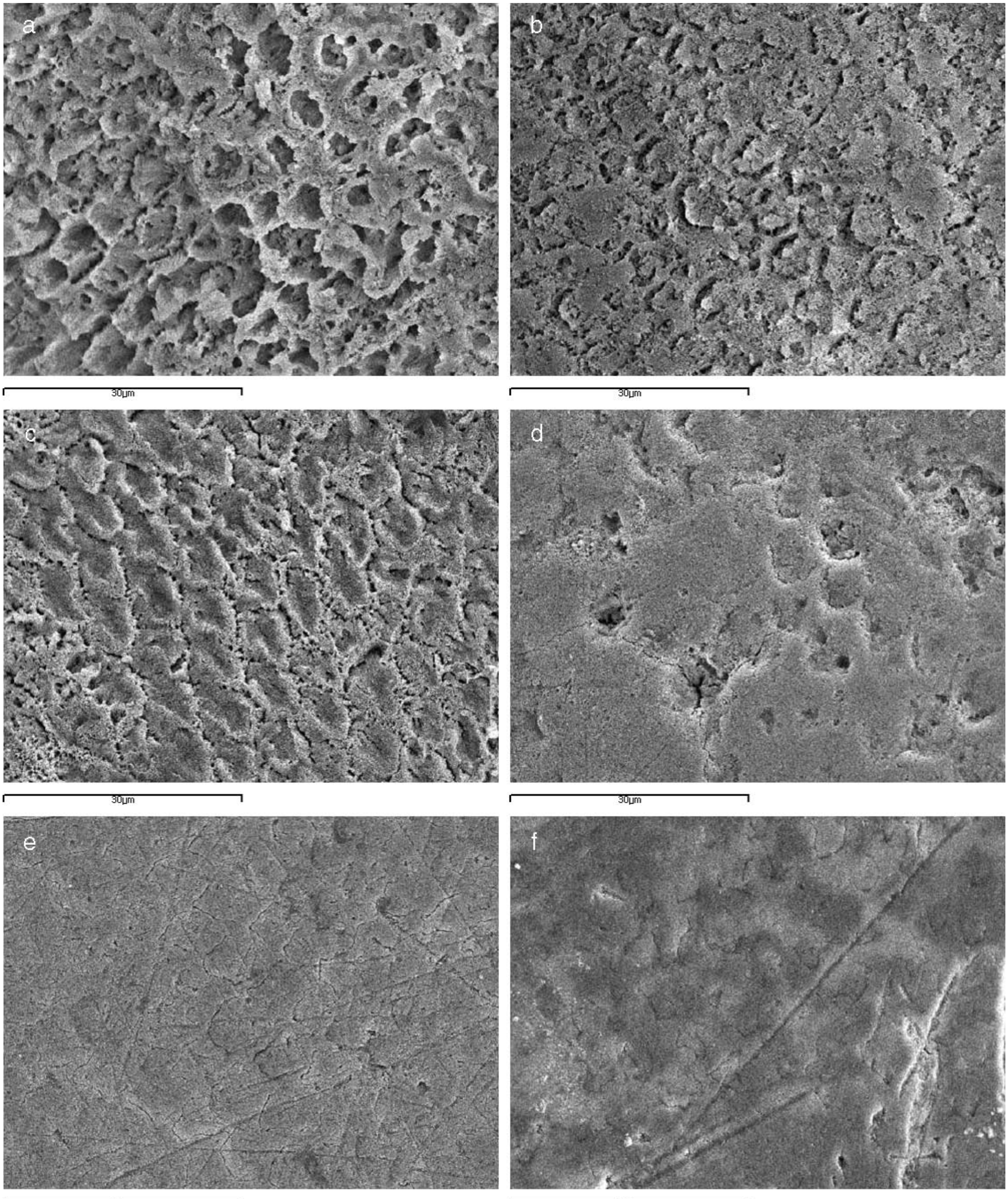


**FIGURE 5.** The scanning electron micrographs of the enamel treated with the various etchants: (a) control group, (b) group 1, (c) group 2, (d) group 3, (e) group 4, and (f) group 5.

## DISCUSSION

Caution should be used when interpreting the results of any in vitro bond strength study, particularly when predicting the clinical performance. Currently, there is no universally accepted minimum clinical bond strength. However, it has been suggested that bond strengths of 6–10 MPa are sufficient for orthodontic bracket bonding.[1,16–19]

Direct bracket bonding to the etched enamel surface has many advantages but also many disadvantages. The main problems are surface enamel loss and demineralization adjacent to the bracket. A strong acid conditioner or a longer etch time can cause surface enamel loss as well as weakening of the subsurface enamel, leading to enamel surface detachment or fracture during debonding. The removal of the residual adhesive or cement on the enamel after debonding may also lead to surface scratches, cracking, and a loss of sound enamel.

Many studies have been undertaken to reduce the adverse effects of the direct bracket bonding technique. Although 32–37% phosphoric acid is routinely used as an enamel conditioner, mild acid conditioners can lead to less enamel loss. Several acid etching systems have been suggested as enamel conditioning for bracket bonding, including 10% phosphoric acid for 30 seconds, 10% salicylic acid for 10 seconds, 10% citric acid for 60 seconds, 10% benzoic acid for 10 seconds, 25% tannic acid for 15–60 seconds, 10% polyacrylic acid for 20 seconds, 2.5% nitric acid for 15 seconds, and 10% maleic acid for 10 seconds.[1,5,6,20–22]

The results of this study demonstrate that adequate bond strengths can be achieved with all experimental etching systems tested. In addition, when assessing the site of bond failure, the percentage of brackets failing at the enamel/resin interface increased as the fraction of the APF gel increased. There was a tendency to have less residual resins remaining on the enamel when an APF gel was incorporated to the etchants.

Damage to the enamel during bracket debonding has been a great concern to clinicians. In this study, experimental groups 1–3 that were subjected to shear stress failed mainly at the etched enamel/bonding resin interface. This would be an advantage in clinical situations because less surface damage will occur at the end of treatment as a result of removal of the retained resin from the enamel surface. The main benefit from using these experimental etchants appeared to be the minimization of sound enamel loss during the orthodontic bracket attachment and detachment.

In previous studies examining the effect of fluoride on the bond strength and fluoride uptake, it was suggested that fluoride be incorporated into the phosphoric acid etchant before bonding the orthodontic brackets.[9–11] The mechanism of fluoride reducing either demineralization or caries is multifunctional: fluoride increases the resistance of the enamel to acid, increases the maturation rate of the enamel, and interferes with the metabolism of microorganisms.[10,13] Fluoride reacts with the enamel forming calcium fluoride and fluoroapatite, which act as slow-releasing agents enhancing the remineralization of the etched enamel and making it more resistant to acid dissolution.[8,10] However, the formation of reaction products on the enamel surface because of the acidic fluoride treatments has been reported to reduce the resin bond strength.[7–10] Some studies showed controversial results as to the effect of topical applications of fluoride on the bracket bond strength.[7–14]

It was reported that a high fluoride concentration (2% NaF) in the etching solution decreased the bond strength, but the bond strength was not affected with a 0.5% NaF concentration.[7] The application of 30% $H_3PO_4$ solution containing 0.02% NaF resulted in an increase in the fluoride content in the enamel surface without decreasing the bond strength.[9] The addition of fluoride to phosphoric acid did not impede the etching effect on the enamel. Moreover, the tensile bond strengths of the enamel surfaces etched with the phosphoric acid containing fluoride and those without fluoride were similar.[11]

The stronger bond strength was obtained with the fluoride containing the etching gel and the larger number of specimens in this group that failed mainly at the resin-bracket interface, whereas most failures occurred at the resin-tooth interface in the nonfluoridated etching gel.[12] There were no significant statistical differences in the tensile bond strength with both the 37% phosphoric acid–etching group and 37% phosphoric acid incorporated with the 1.23% sodium fluoride–etching group.[13]

Fluoride could not be detected on the enamel surface by SEM/EDXS in this study because SEM/EDXS might not be sensitive enough. However, adding an APF gel to the phosphoric acid etchants adversely affected bracket bond strength. The bond strength might be lowered by forming calcium fluoride at high APF fractions in the experimental etchants. At low APF fractions, however, the protective effect of fluoride might be not sufficiently high to prevent the etching of a comparatively conventional phosphoric acid etchant. The etch depth or the amount of surface enamel removed by the experimental etchant was dependent on the APF gel fraction in the phosphoric acid. The APF gel could have acted as a buffering agent, making the phosphoric acid action milder. The bond strengths of the brackets to the enamel surfaces etched with the phosphoric acid containing fluoride were dependent on the fluoride concentration in the etching solution.[9]

It can be suggested that the addition of an APF gel to phosphoric acid provides favorable effects on the enamel surface for bracket bonding.

## CONCLUSIONS

Our results indicated that a mixture of the 37% phosphoric acid and an APF gel (50% and 66% APF fraction) can be used as an etchant to minimize the loss of sound enamel during the etching as well as debonding procedure without compromising the required bracket bond strength.

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