

## In vitro comparison of primary incisor enamel surfaces etched with an acid solution or acid gel

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### Abstract

*This study compared the effectiveness of an acid solution and an acid gel in etching the facial surfaces of primary anterior teeth. Comparisons included: (1) microscopic examination of the quality of etched enamel surfaces, (2) microscopic examination of the formation and penetration of resin tags into the etched surface, and (3) mechanical tests of the shear strength of the enamel-resin bond.*

*The acid solution yielded a more uniform etch. Depth of resin tag penetration was about the same on surfaces prepared with either etchant; however, surfaces treated with acid solution exhibited a greater number of tags that were distributed more uniformly along the entire resin surface. Shear strength of the enamel-resin bond was the same on surfaces prepared with either etchant.*

**P**revious research on acid etching has focused primarily on acid solutions. Over the past several years, gels for etching enamel have appeared on the market and have replaced acid solutions as the etchant of choice of many clinicians.

Some of the newer gels are thixotropic in nature. This property permits the gel to become less viscous when subjected to forces encountered during the application of the gel to the tooth surface and to return to its original viscosity upon standing. Because of this property, gels allow for increased control in placement and thus offer a distinct technical advantage to the clinician in terms of convenience. With gels it is possible to reduce or eliminate acid overflow to gingival tissues and to avoid accidental etching of hard tissues adjacent to the desired site.

Despite these apparent advantages, there has been contradictory documentation in the literature on whether gels are as effective as acid solutions. Some have noted that due to the viscous nature of gels, their ability to wet the enamel surface effectively may be reduced.<sup>1</sup> Others have stated that by-products from the etching reaction form at the enamel-gel interface

and reduce the effect of the acid; they contend that these by-products are eliminated only when fresh acid is applied continuously.<sup>2</sup>

It also has been stated that a longer wash time is necessary to remove gel residues from the etched enamel surface.<sup>1,3</sup> However, reported data on wash times for gels are inconclusive. Some have reported that microscopic remnants and by-products were left on the surface even after copious amounts of water were used.<sup>1</sup> Another study found that a 2-second wash left acid residue but a 5-second wash provided a clean surface.<sup>4</sup>

Laboratory studies comparing solution and gel etchants have been contradictory. When Brannstrom et al.<sup>5</sup> etched the buccal surfaces of young permanent teeth for 60 seconds with either 50% phosphoric acid gel or 37% phosphoric acid solution, resulting etched enamel surfaces were similar. In a later study, they found that a 15-second etch with 50% phosphoric acid gel provided a more retentive surface on young permanent teeth than a 60-second etch using the same gel.<sup>6</sup> Yet, Diedrich<sup>4</sup> found that etching permanent teeth with 50% phosphoric acid gel for 30 seconds left the surface relatively unaffected; in his tests, a 60- or 120-second etch produced the most desirable etching pattern. In a clinical study by Hardison,<sup>7</sup> sealant retention on the occlusal surfaces of permanent molars was compared when the etchant was either a 33% phosphoric acid gel or 35% phosphoric acid solution; the results showed no significant difference in retention after six months.

In summary, only a few studies have compared the etching abilities of acid gels for conditioning permanent tooth enamel and the findings are inconclusive. Moreover, because there is clear evidence that primary and permanent enamel respond differently to acid etching,<sup>8,9</sup> findings for etching characteristics of permanent teeth cannot be generalized to include primary teeth. To date no studies have been reported that compared acid gels and acid solutions for etching

primary tooth enamel. Therefore, the present study was undertaken to compare the effectiveness of an acid gel and an acid solution for etching primary teeth.

## Methods and Materials

This study consisted of three parts:

1. Evaluation of etched enamel surfaces with a scanning electron microscope (SEM)
2. Measurement of tag length as an indication of etch depth
3. Measurement of shear strength of the enamel-resin bond.

## General Procedures

The acid gel selected for this investigation was ESPE Gel,<sup>a</sup> a 33% unbuffered phosphoric acid in an alginic acid base. The acid solution selected was a 33% solution of unbuffered phosphoric acid. To insure that both preparations were fresh and uncontaminated, the gel was obtained directly from the manufacturer and the solution was prepared freshly in the Dental Research Center at the University of North Carolina at Chapel Hill.

The primary anterior teeth selected for this study were collected immediately after exfoliation or extraction. They were donated by dentists in the Chapel Hill community. To prevent desiccation, the teeth were stored in airtight containers with a preparation of thymol and distilled water. Thirty teeth with blemish-free facial surfaces were selected.

Each tooth carefully was sectioned longitudinally in a buccolingual direction using a water-cooled diamond disk. Thus, there were 30 matched pairs of teeth, a total of 60 samples. Each sample was sectioned at the cemento-enamel junction, and the root fragments were discarded. At all times, the matched pairs of halves were stored together to avoid confusing the samples.

The 30 matched pairs of teeth then were separated randomly into three groups of 10 pairs each, one group for each part of the study.

### Part 1: Evaluation of the Etched Enamel Surface

The facial surface of each specimen was polished lightly for 10 seconds using a rubber cup in a slow-speed handpiece with a fine-grain pumice slurry. The specimen surface then was washed carefully and dried. Each specimen was glued to an aluminum stud. One half of each matched pair was assigned randomly for etching with the acid gel and the other was assigned for etching with the acid solution. This approach was used to control the effect of extraneous variables such as age and previous fluoride exposure.

<sup>a</sup> ESPE, Premier Sales Corp., Norristown, PA.

The etch time was 2 minutes. The etching gel was applied with a plastic instrument and was left undisturbed on the tooth according to the manufacturer's recommendations. The etching solution was applied with a sable-hair brush, gauge 00. The acid solution was replenished continuously and agitated gently over the enamel for the entire two minutes. After exposure to the gel or solution, the surface was washed with a water spray from an air-water syringe for 20 seconds and then dried thoroughly with compressed air uncontaminated with oil or water vapor.

On completion of the etching procedure, the specimens were desiccated in a vacuum oven for 24 hours. The specimens then were vacuum-coated with gold-palladium in preparation for examination with an ETEC Autoscan U-1 SEM.<sup>b</sup> Six photomicrographs were obtained of each specimen at a magnification of 800×: two in the incisal third of the specimen, two in the middle third, and two in the cervical third. Photomicrographs were obtained at random within each of the three sites.

The photomicrographs were examined by two evaluators in a manner such that the etching agent and the etching site were unknown. Each photomicrograph was scored according to the degree of surface etching observed. A scale of 1-3 was used: 1 represented a relatively smooth surface with little or no evidence of surface porosity, and 3 represented a surface that was etched uniformly with a high degree of porosity. A score of 2 was given to surfaces that appeared definitely porous, whereas surrounding areas were relatively smooth. Disagreements in scoring between evaluators were discussed immediately and a consensus was reached for each photomicrograph.

The photomicrographs were examined twice with one week separating the evaluation sessions. Scores from the two evaluations were compared to ensure reliability of the evaluation system; agreement was 95% confirming the reliability of the scoring system.

For statistical analysis, the two scores for each region of each sample specimen were averaged to yield a single sample value. The Sign test was used to analyze these data; level of significance was set at  $\alpha = 0.05$ .

### Part 2: Measurement of Tag Length

The facial surface of each specimen was polished and etched using the same methods as in Part 1. One-half of each matched pair was assigned randomly for acid gel etching and the other was assigned for acid solution etching. Prisma Bond<sup>c</sup> then was applied with a brush to the etched surface, followed by a veneer of Prisma Fil<sup>c</sup> to produce a composite layer 2 mm in

<sup>b</sup> Perkin Elmer Electron Beam Technical Division, Hayward, CA.

<sup>c</sup> L. D. Caulk Co., Division of Dentsply International Inc., Milford, DE.

depth. This composite resin layer then was polymerized with visible light according to manufacturer's instructions.

In preparation for light microscopy, each specimen was mounted on a thin-sectioning machine. The specimen was sectioned longitudinally in a buccolingual direction, producing a cut section approximately 1 mm thick. Each section then was thinned with wet, 600-grit silicon carbide grinding paper until a final thickness of 90  $\mu\text{m}$  was obtained. The section was placed on a glass slide and covered with a glass coverslip. The remaining enamel was removed by demineralization using 10% hydrochloric acid solution, placed under the coverslip and replenished as necessary.

During the demineralization process, the entire resin surface from incisal to cervical was scanned under the light microscope at a magnification of 250 $\times$ . A subjective assessment of the quantity and distribution of tags was made. A single photomicrograph was taken of the resin surface in an area where the tags appeared to be of average length for that given specimen.

Average tag length on each photomicrograph was measured, and these measurements were used for statistical analysis. The Wilcoxon matched-pairs test was used to analyze the data and the level of significance was set at  $\alpha = 0.05$ .

### Part 3. Shear Strength of the Enamel-Resin Bond

Prior to etching, the specimens were embedded in a block of self-curing acrylic resin so that the flattest portion of the facial surface was parallel to and just adjacent to the top surface of the block. The top surface then was rubbed carefully across wet, 600-grit silicon grinding paper to obtain a flat enamel surface. Because of the convexity of the primary anterior teeth, the flattened area generally was located between the incisal and middle third of the facial surface on each specimen. One-half of each matched pair randomly was assigned for acid gel etching and the other was assigned for acid solution etching. Etching procedures were the same as in Parts 1 and 2.

To produce a known surface area for bonding, self-adhesive tape, punched with a circular hole 1.78 mm in diameter, was used to mask off all areas of each specimen except the enamel exposed through the punched hole. The exposed enamel was painted with Prisma Bond. A stainless steel tube approximately 4 mm long with an inside diameter slightly larger than the diameter of the exposed enamel was luted perpendicular to the embedded specimen, with the center of the exposed enamel at the center of the tube. The tube was filled with Prisma Fil in increments and light cured. The luting material then was removed.

The embedded specimen with the bonded resin was

aligned and held in the upper jaw of an Instron testing machine TTC-M.<sup>d</sup> A metal bar with a hole that allowed the bonded tube to slide through was attached to the lower jaw of the testing machine.<sup>d</sup> A shearing force was applied to the bond. A crosshead speed of 0.05 cm/min was delivered until the bond was broken, and the load at this point was recorded. The shear strength of the enamel-resin bond was calculated and expressed in MN/m<sup>2</sup>. These measurements were analyzed statistically by a Wilcoxon matched-pairs test; level of significance was placed at  $\alpha = 0.05$ .

## Results

### Part 1: Evaluation of the Etched Enamel Surface

Scanning electron photomicrographs representative of those obtained in the study are illustrated in Figure 1 (acid solution) and Figure 2 (acid gel). Results of the statistical analysis for etch quality are presented in Table 1. Comparison within site for treatment was not possible because of the small sample size and the large proportion of zero differences.

Visual inspection indicated that the relationship between the solution and gel was the same regardless of site. Site specification was therefore ignored. The statistical analysis showed that the acid solution was significantly more effective than the acid gel for etching the facial surface of primary anterior teeth ( $p < 0.001$ ).

### Part 2: Measurement of Tag Length

A typical photomicrograph of resin tags is illustrated in Figure 3. Results of the statistical analysis for tag length are presented in Table 2. No significant difference in depth of resin penetration was found between the two treatment groups.

The subjective assessment of quantity of tags observed between treatments revealed that though extensive tag formation was found after etching with either acid solution or acid gel, surfaces treated with the acid solution generally yielded a greater number of tags that were more uniformly distributed. Gel-treated surfaces showed a greater tendency toward dense concentrations of tags with neighboring areas of sparse concentration.

### Part 3: Shear Strength of the Enamel-Resin Bond

Results of the statistical analysis for shear strength are presented in Table 3. No significant difference in shear strength of enamel-resin bond was found for surfaces treated with the acid solution or acid gel.

## Discussion

Results from Part 1 of the study showed the acid solution produced superior etching on the facial sur-

<sup>d</sup> Instron Corp., Canton, MA.

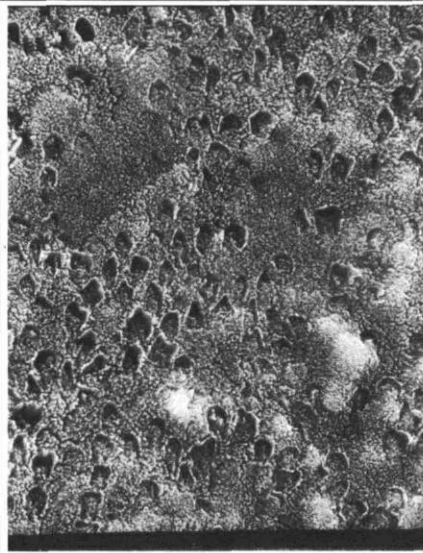


FIGURE 1. Scanning electron photomicrograph of primary incisor enamel etched with 33% phosphoric acid solution (800×).

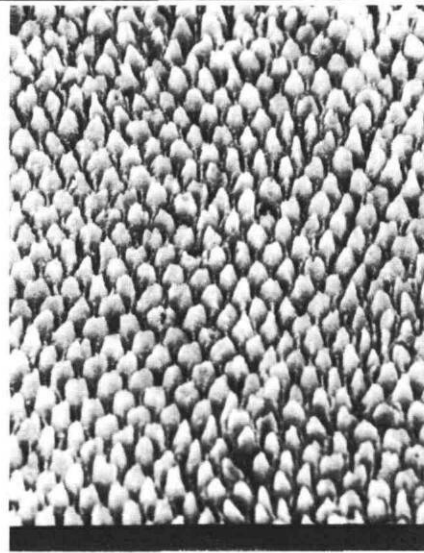


FIGURE 2. Scanning electron photomicrograph of primary incisor enamel etched with an acid gel containing 33% phosphoric acid (800×).

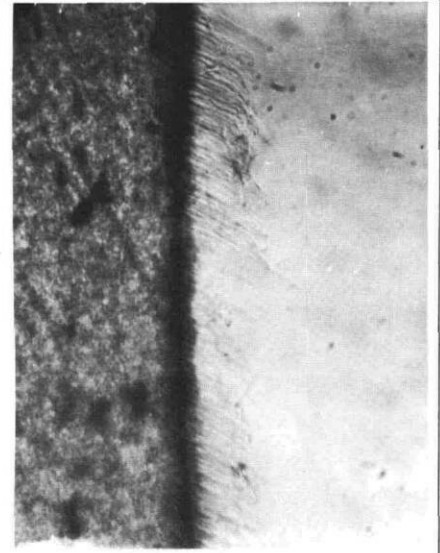


FIGURE 3. Photomicrograph of resin tags after removal of enamel by demineralization (250×).

TABLE 1. Comparison of Etch Quality Between the Acid Solution and the Acid Gel

Solution vs. Gel	(0)	Sign (+)	(-)
	Matched Pairs With no Difference	Matched Pairs with Solution Superior to Gel	Matched Pairs with Solution Inferior to Gel
Incisal	5	5	0
Middle	2	7	1
Cervical	2	8	0
Sites combined	9	20	1

p < 0.001

faces of primary anterior teeth. The majority of areas treated with the acid solution appeared to be etched evenly and showed a great deal of surface porosity (Figure 1). The gel-treated surfaces generally appeared less evenly etched; although these surfaces exhibited areas of definite surface porosity, they often were surrounded by areas that appeared smooth and prismless in nature (Figure 2).

Several factors may have contributed to the poorer etch observed on gel-treated surfaces. The viscous nature of the gels may inhibit their ability to wet the enamel surface uniformly, which would result in uneven etching.<sup>1</sup> Also, the acid gel simply was applied and left undisturbed for two minutes; prior evidence that continuous renewal and agitation of acid solutions produces more even etching suggests that the

TABLE 2. Comparison of Tag Length Between the Acid Solution and the Acid Gel

Tooth	Tag Length Solution	Tag Length Gel	Difference	Rank
1	55 μm	44 μm	11	6
2	64	72	-8	-3.5
3	73	40	33	10
4	42	50	-8	-3.5
5	32	53	-21	-9
6	72	55	17	8
7	37	48	-9	-5
8	55	52	3	1
9	44	40	4	2

Σ T- = 28; Σ T+ = 27; p > 0.05.

gel application process may have impeded dissolution or allowed the acid to become neutralized.<sup>2,10,11</sup> A third cause of the poorer etch may have been that the wash time was inadequate to remove all traces of the gel.<sup>1,3</sup> Because the heavy water spray delivered to each specimen for 20 seconds left no detectable surface residue when examined under the SEM, this explanation seems unlikely.

The formation and penetration of resin tags within the enamel surface is greatly dependent upon the ability of the acid to create a porous surface. Because Part 1 of the study found that the acid solution rendered a more uniform etch than the acid gel, it was not surprising to discover that the acid solution produced a greater quantity and more even distribution

**TABLE 3.** Comparison of Shear Strength Between the Acid Solution and Acid Gel

Tooth	Shear Strength Solution	Shear Strength Gel	Difference	Rank
1	19.49 MN/m <sup>2</sup>	16.37 MN/m <sup>2</sup>	3.12	3
2	8.56	19.49	-10.93	-10
3	12.47	14.03	-1.56	-2
4	18.32	10.91	7.41	7.5
5	18.71	15.20	3.51	4.5
6	19.10	13.64	5.46	6
7	7.80	8.18	-.38	-1
8	11.69	16.49	-7.80	-9
9	13.64	10.13	3.51	4.5
10	9.36	16.76	-7.40	-7.5

$\Sigma T^- = 29.5$ ;  $\Sigma T^+ = 25.5$ ;  $p > 0.05$ .

of tags. Though numerous tags were seen in specimens from both treatment groups, tags in gel-treated enamel were often fewer in number and less evenly distributed across the resin surface. Despite these discrepancies in quantity and distribution of tags, the depth of tag penetration was similar in both groups. Tags averaging slightly longer than 50  $\mu\text{m}$  were observed routinely (Figure 3). These findings on tag length agree with those reported in other studies using similar etching and investigative procedures.<sup>8,12</sup>

Shear strength values were the same for both treatment groups. Because the solution-treated surfaces had exhibited a more uniform etch and more numerous tag formation, it was expected that tests of shear strength would show that specimens treated with acid solution had a stronger enamel-resin bond than those treated with acid gel. This somewhat surprising result may be due to the lack of sensitivity in or logistical requirements of the shear strength test. To produce a flat working surface, preparations for the test involved abrading as much as 40-80  $\mu\text{m}$  of enamel from each specimen. At that depth of exposure, enamel may be histologically different from true "surface" enamel and maybe equally susceptible to the acid solution and the acid gel conditioners. This explanation is consistent with findings for the other parts of the study and with prior studies that found no significant differences in etch quality on permanent teeth when comparing acid gels and acid solutions.<sup>5,6</sup>

## Conclusions

This study of the facial surface of primary anterior teeth led to the following conclusions.

1. The acid solution yielded significantly better etch quality than the acid gel.
2. There was no significant difference in the depth of resin tag penetration on surfaces etched with acid solution as compared with acid gel. However, the acid solution yielded a greater number of more uniformly distributed tags.
3. There was no significant difference in shear strength of the enamel-resin bond found on surfaces treated with acid solution or acid gel.

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