



## Microleakage and Bond Strength of Sealant to Primary Enamel Comparing Air Abrasion and Acid Etch Techniques

Lisa A. Knobloch, DDS, MS<sup>1</sup> Tyra Meyer, DDS, MS<sup>2</sup> Ronald E. Kerby, DDS<sup>1</sup> William Johnston, PhD<sup>3</sup>

### Abstract

**Purpose:** The objective of this study was to compare the effect of air abrasion (KCP 2000), acid etching (37% phosphoric acid), and the combination of both procedures on the shear bond strength and microleakage of a light-cured pit-and-fissure sealant to the enamel of human primary molar teeth.

**Methods:** Noncarious extracted human primary molars were randomly divided into 4 groups in preparation for enamel bonding. The enamel surface was treated as follows for each group: (1) group 1 (control group); (2) group 2 (acid etch group); (3) group 3 (KCP [Kinetic Cavity Preparation System] group); and (4) group 4 (KCP and acid etch group). Delton, a light-cured pit-and-fissure sealant, was then applied to the occlusal surface after conditioning. The bonded specimens were maintained in distilled water at 37°C±2°C for 7 days, after which they were subjected to thermocycling followed by shear bond testing. Microleakage was determined by immersing the prepared teeth in 50% silver nitrate dye followed by sectioning and calculation of dye penetration.

**Results:** The mean shear bond strength of the KCP+acid etch group exhibited nearly 50% higher bond strength than the acid etch group ( $P<.01$ ). In addition, specimens bonded to enamel conditioned only with acid etch exhibited bond strengths that were nearly twice that of those conditioned with the KCP system alone. No significant difference was noted between the air abrasion and control groups.

**Conclusions:** In primary teeth, air abrasion combined with acid etching appears to provide the best conditions for enamel treatment prior to sealant placement. (Pediatr Dent 2005;27:463-469)

**KEYWORDS:** AIR ABRASION, PHOSPHORIC ACID, SEALANT, MICROLEAKAGE

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The prevention of pit-and-fissure caries through the use of occlusal sealants continues to be a focus in the pediatric population. Over the past 2 decades, sealants have proven to be an effective procedure for reducing the incidence of occlusal caries.<sup>1-7</sup> Buonocore introduced the acid etch technique to alter the existing enamel in order to improve the retention of acrylic restorative materials.<sup>8</sup> Initial clinical tests of pit-and-fissure sealants utilized cyanoacrylates that were found to be biodegradable and later replaced with dimethacrylate resins.<sup>9</sup> Today, most commercial sealants are Bis-GMA dimethacrylate- or urethane dimethacrylate-based products. Silverstone concluded that the most even distribution of etched enamel was produced with

a solution of 30% phosphoric acid.<sup>10</sup> This particular acid and concentration resulted in a porous region below the enamel surface and an ideal loss of surface contour. The porous region was regarded as the key to the retention of resinous materials.<sup>11</sup> Most commercial manufacturers supply phosphoric acid for etching enamel in the range of 30% to 40%.

Air abrasion was developed to improve the patient experience by minimizing noise during preparation of the teeth and by reducing vibration, pressure, and heat.<sup>12</sup> In a recent study comparing air abrasion to conventional rotary preparation, all of the patients preferred air abrasion over conventional rotary preparation when removing fissure caries in mandibular premolars.<sup>13</sup> The air abrasion process utilizes a fine stream of compressed air into which an abrasive agent is introduced. The air abrasion technique converts the motor's mechanical energy into the kinetic energy of the particles as they pass through the handpiece to promote the cutting action. With the improved restorative materials available today, a more conservative cavity preparation is acceptable, making air abrasion a potential alternative to conventional handpieces.<sup>12</sup>

<sup>1</sup>Drs. Knobloch and Kerby are associate professors, and <sup>3</sup>Dr. Johnston is professor, Section of Restorative and Prosthetic Dentistry, The Ohio State University College of Dentistry, Columbus, Ohio; <sup>2</sup>Dr. Meyer is in private practice, Columbus, Ohio.  
Correspond with Dr. Knobloch at knobloch.3@osu.edu

Shear bond strength tests have been used to compare the bond strength between materials, such as composite, to enamel and dentin.<sup>14-16</sup> Low shear bond strength is associated with inadequate bonding and wider gaps between restoration and tooth. These gaps may allow: (1) bacterial infiltration; (2) postoperative pulpal inflammation; and (3) microleakage.<sup>17</sup> Microleakage is defined as the ingress of fluids and/or microorganisms into the space between tooth structure and restorative materials.<sup>18</sup> The success of a sealant is related to its ability to act as a physical barrier between oral fluids and bacteria and the pits/fissures of occlusal surfaces.

Because of the different coefficients of thermal expansion of restorative materials and tooth structure, repeated expansion and contraction of material from thermal changes may lead to an increase in leakage as the 2 materials separate. The retention rate of a sealant is directly related to the micromechanical bond between composite and enamel.<sup>19</sup> Therefore, the performance of a sealant will be affected by: (1) bond strength; and (2) amount of microleakage that occurs. If microleakage occurs at the sealant-enamel interface, there is a potential for failure due to: (1) recurrent caries; (2) postoperative sensitivity; (3) adverse pulpal response; or (4) loss of the restoration.<sup>20</sup> Therefore, any technique used to condition enamel prior to sealant placement should:

1. be examined for its ability to provide an adequate seal at the interface;
2. provide sufficient bond strength to retain the resin to the enamel.

If the properties are sufficient, the use of air abrasion as a means to condition teeth prior to sealant placement may improve the delivery and outcome of this treatment for the pediatric dental patient. Air abrasion has shown to be a promising technique for conditioning the enamel surface prior to resin placement.<sup>12,21,22</sup> Studies have compared bond strengths and microleakage of pit-and-fissure sealants to the enamel and dentin of permanent teeth conditioned by air abrasion and acid etch. No studies, however, have examined the properties of primary enamel when conditioned in this manner.<sup>12,23</sup>

Therefore, the purpose of this study was to compare the effect of air abrasion (Kinetic Cavity Preparation System [KCP 2000], American Medical Technologies, Corpus Christi, Tex), acid etching (37% phosphoric acid), and the combination of both procedures on the shear bond strength and microleakage of a light-cured, pit-and-fissure sealant to the enamel of human primary molar teeth.

## Methods

### Shear bond strength

Forty noncarious extracted human primary molars were: (1) debrided; (2) cleaned with pumice and water; and (3) stored at 23°C±2°C in chloramine T solution. All teeth were then randomly divided into 4 groups of 10 in preparation for enamel bonding. Prior to embedding, root

resection and the placement of retentive undercuts were performed. The extracted teeth's buccal surfaces were pressed into irreversible hydrocolloid impression material (Jeltrate, Dentsply International, York, Pa) and mounted in a plastic ring mold (30 mm [diameter] ×12 mm). The ring mold was then filled with a slow-setting epoxy resin (Leco Epoxy Resin, Leco Corporation, St. Joseph, Mich) so that the buccal surface was exposed after removal from the mold. The molds with the embedded teeth were placed in a pan containing water 2 cm deep during the exothermic setting reaction to minimize heat generation. After removal from the plastic mold, the exposed enamel surface was sequentially fine sanded with 320- and 400-grit silicon carbide paper and water to produce a flat enamel surface. The final ground surfaces were visually inspected under low-power magnification (×60) to ensure that the dentinoenamel junction had not been penetrated.

Prior to bonding, each tooth was: (1) cleaned with an aqueous solution of pumice; (2) rinsed; and (3) air dried. Artists' tape (325M, 3M Corporation, Maplewood, Minn) with a 3.9-mm hole was placed on the surface to control the area of the bond. The flat enamel surface was treated as follows for each group:

1. Group 1 (control group): No conditioning was performed to the tooth surface after the prophylaxis with pumice and water.
2. Group 2 (enamel etch group): The flat surfaces were passively etched with 37% phosphoric acid gel (Ultra-etch, Ultradent Products Inc, South Jordan, Utah) for 30 seconds. The surfaces were then rinsed with water for 20 seconds and air dried. Teeth were visually inspected to demonstrate a uniform, frosty appearance.
3. Group 3 (KCP group): The teeth were prepared using the Kinetic Cavity Preparation System (KCP 2000, American Medical Technologies, Corpus Christi, Tex) as follows:
  - a. Using the 50-µm size particles and an air pressure setting of 120 psi, the teeth were prepared with the nozzle tip held 2.5 mm from the tooth surface and slightly offset from perpendicular. The operator used a quick, steady, sweeping motion along the surface to achieve a uniform, frosty appearance.
  - b. The excess particles were then removed with a moisture-free air stream, and the surface was not rinsed.
4. Group 4 (KCP and acid etch group): Teeth in this group were treated exactly as group 3 teeth and then passively etched like group 2 with 37% phosphoric acid for 30 seconds.

Clear plastic cylindrical tubes (3.9 mm [inside diameter] ×6 mm) were placed on the flat enamel surface over the exposed area of the adhesive tape. The tubes were then filled in 1-mm increments with Delton opaque light curing pit-and-fissure sealant (Dentsply International, York, Pa). Each 1-mm increment was cured for 20 seconds with a visible light curing unit (model No. 100, Dentsply International, York, Pa). The bonded specimens were then maintained in dis-

tilled water at 37°C±2°C for 7 days, after which they were subjected to thermocycling (2,500 cycles) in water baths at 5°C and 55°C for a dwell time of 1 minute per bath.

The testing of samples was performed randomly using computer-generated random numbers to avoid sequence bias. In addition, each sample was assigned a number so that the evaluator was unaware of the group being tested. At the time of testing, each specimen was tightly inserted into a custom-made stainless steel jig. To control the location of the load application during shear testing, a 0.1-mm aluminum spacer was inserted between the shear probe and the tooth surface. The specimens were tested using a cross-head displacement rate of 0.5 mm per minute on an Instron Mechanical Testing Machine (model No. 4202, Instron Corporation, Canton, Mass) using a stainless steel shear probe. The minimum load required to produce bond failure was determined from the first load drop on the load deflection plot. Shear bond values were calculated in MPa using the following equation:

$$\text{Bond strength} = \frac{F \text{ (kg)}}{3.1415 \cdot d^2/4}$$

where: d=diameter of cylinder in cm (0.39 cm); F=load at failure in kilograms. The type of bond failure (adhesive, cohesive, or mixed [adhesive/cohesive]) was recorded using a low-power light microscope (×20). The following criteria were used to categorize the type of bond failure:

1. adhesive=<20% resin remaining at interfacial bond area;
2. cohesive>= 80% resin remaining at interfacial bond area;
3. mixed adhesive/cohesive: 20% to 80% resin remaining at interfacial bond area.

Statistical analysis of the shear bond strength values was completed utilizing a 1-way analysis of variance (ANOVA) followed by Tukey's multiple range test.

### Microleakage

Forty noncarious extracted human primary molars were debrided with pumice and water and then stored at 23°C±2°C in chloramine T solution. All teeth were embedded in slow-setting epoxy, as previously described, with the occlusal surface exposed. The occlusal surfaces were then conditioned, as described in the previous section. Delton, a light-cured pit-and-fissure sealant was then applied and light polymerized for 20 seconds. The bonded specimens were maintained in distilled water at 37°C±2°C for 7 days prior to testing. Specimens were subjected to thermocycling (2,500 cycles) in water baths at 5°C and 55°C for a dwell time per bath of 1 minute. Following thermocycling, the prepared teeth were immersed in 50% silver nitrate dye for 2 hours in a dark environment. They were then placed into a radiographic developer solution for 8 hours under fluorescent light to precipitate the silver nitrate. This allowed visualization of the penetration pattern of the silver ions along the enamel-sealant interface. After removal from the developing solution, the teeth were washed in distilled water to remove the excess surface dye.

Specimens were serially sectioned at 1.5-mm intervals longitudinally with a 5-inch diamond blade on a vari-cut

sectioning machine (Series 15 LC Diamond, Buehler Corp, Lake Bluff, Ill). One examiner was utilized in the microleakage study to minimize variation during evaluation. Specimens were assigned a random number for evaluation to ensure that the examiner was blinded to the enamel preparation. Each section was then examined for leakage using a digital image analyzer system (Image-Pro Analyzer, Fryar Co, Cincinnati, Ohio). This system utilizes a light microscope (Nikon SMZ-2T, Nikon Corp, Melville, NY) to view the sections at magnifications up to ×60. The images were captured at ×10 to allow the examiner to view the entire section.

The curvilinear depth of dye penetration was assessed and measured in pixels using the Image Pro Plus software program (Media Cybernetics, Silver Spring, Md). The leakage for each section was recorded as a percentage of dye penetration related to the total length of the sealant from the margins on both buccal and lingual cuspal inclines. Each tooth's penetration was the average percentage of dye penetration of the 2 inclines. The dye penetration data was tested for normality using a variety of tests (Shapiro-Wilk, Kolmogorov-Smirnov, Cramer-von Mises, Anderson-Darling), followed by a Kruskal-Wallis 1-way ANOVA by ranks. Post hoc pairwise comparisons were made using the Wilcoxon rank sum test adjusted by the Bonferroni correction ( $\alpha=0.05$ ).

## Results

### Shear bond strength

The ANOVA ( $P<.001$ ) and Tukey's multiple range test ( $P<.01$ ) showed significant differences between the mean shear bond strengths of the 4 groups tested (Table 1). A group of 10 specimens were prepared as a control. All specimens failed, however, during thermocycling. Shear bond testing of these specimens was not possible. Therefore, only the remaining 3 groups were analyzed. Shear bond strength values, with standard deviations for each of the different groups, are reported in Figure 1. The mean shear bond strength of group 4 (KCP+acid etch) at 7.14 MPa exhibited nearly 50% higher bond strength than group 2 (acid etch). In addition, specimens bonded to enamel conditioned with acid etch (group 2) exhibited bond strengths (4.82 MPa) that were nearly twice that of those conditioned with the KCP system (group 3) at 2.34 MPa.

The types of bond failure characterized as adhesive, cohesive, or mixed adhesive/cohesive are summarized in Table 2. All control specimens exhibited adhesive failures at the

Table 1. Analysis of Variance Procedure for Shear Bond Strength

Source	DF	Mean square	F value	P
Model	2	57.64	85.02	<.001
Error	27	0.67		
Corrected total	29			



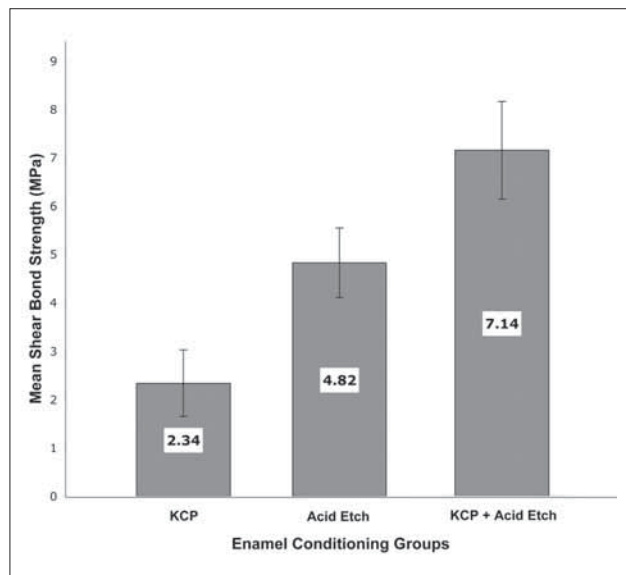


Figure 1. Mean shear bond strength in MPa for the enamel conditioning groups tested. Control group not included due to bond failure during thermocycling.

enamel-resin interface. The majority of failures (70%) exhibited by the KCP group were also adhesive. The acid etch group exhibited 50% adhesive failures and 50% mixed adhesive/cohesive failures. Finally, the majority of failures (70%) for the KCP+acid etch group were either mixed adhesive/cohesive or cohesive.

Scanning electron photomicrographs (Quanta 200, FEI Corporation, Hillsboro, Ore) were made of representative specimens. A representative scanning electron photomicrograph (SEP) of a sample of a specimen conditioned with 37% phosphoric acid is shown in Figure 2. The enamel prisms and micropores are apparent in this SEP viewed at  $\times 800$  magnification. Figure 3 is a representative SEP of a specimen conditioned with air abrasion at 120-psi and 50- $\mu\text{m}$  particle size at  $\times 800$  magnification. Morphological changes of the enamel surface appear to be more at a mac-

Table 2. Characterization of Bond Failure (Adhesive, Cohesive, or Mixed Adhesive/Cohesive)\*

Specimen	Etch	KCP	KCP+etch	Control
1	A/C	A	A/C	A
2	A/C	A/C	A	A
3	A/C	A	A	A
4	A/C	A	A/C	A
5	A	A	A/C	A
6	A	A	C	A
7	A	C	A/C	A
8	A	A/C	A/C	A
9	A	A	A	A
10	A/C	A	A/C	A

\*A=adhesive failure at the enamel-resin interface; C=cohesive failure within the resin; A/C=mixed adhesive/cohesive failure.

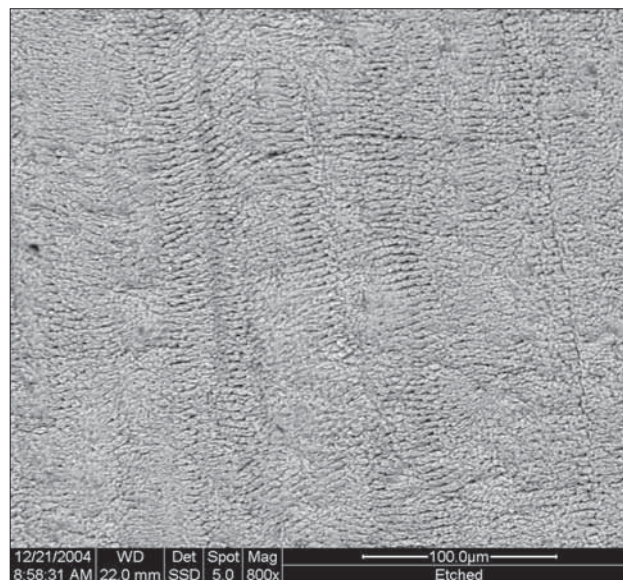


Figure 2. Scanning electron photomicrograph of enamel surface acid etched with 37% phosphoric acid for 30 seconds ( $\times 800$ ).

rosopic level. Figure 4 is a representative SEP of a specimen conditioned with both acid etch (37% phosphoric acid) and air abrasion (120 psi, 50  $\mu\text{m}$ ). The increased surface area and contours created at the macroscopic level by air abrasion, along with the micropores created by acid etching, are apparent in this SEP viewed at  $\times 400$  magnification.

### Microleakage

In at least one group, all tests showed lack of normality. The mean standard deviation and median values for each group and indicators of significant differences are shown in Table 3. Groups 1 (control) and 2 (KCP) as well as groups 3 (etch) and 4 mean standard deviation and median values for each group and indicators of significant differences are shown in Table 3. Groups 1 (control) and 2 (KCP) as well as groups 3 (etch) and 4 (KCP+etch) were not significantly different from each other ( $P < .001$ ). The ranking of percent dye penetration as a function of occlusal conditioning from highest to lowest was KCP, control, etch, and KCP+etch.

### Discussion

Bonding studies have been described in the literature. Experimental designs and reported bond strength values, however, may vary.<sup>12,14</sup> Such variations in testing methodology make any comparison of data between studies difficult. In response to such difficulties, van Noort suggested that the methodology for determination of bond strength should be standardized and controlled.<sup>24</sup> This suggestion was based on the fact that stresses at the interface between the material and tooth structure are nonuniform and highly dependent on the configuration of the applied load and on the test's parameters.<sup>24</sup>

This study's experimental design, including specimen configuration and testing methodology, followed the recommendations suggested by van Noort.<sup>24,25</sup>

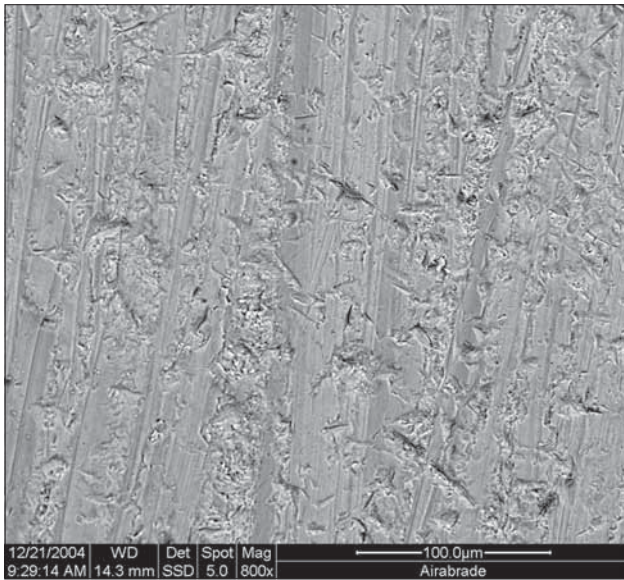


Figure 3. Scanning electron photomicrograph of enamel surface conditioned with air abrasion at 120-psi and 50-µm sized particles (×800).

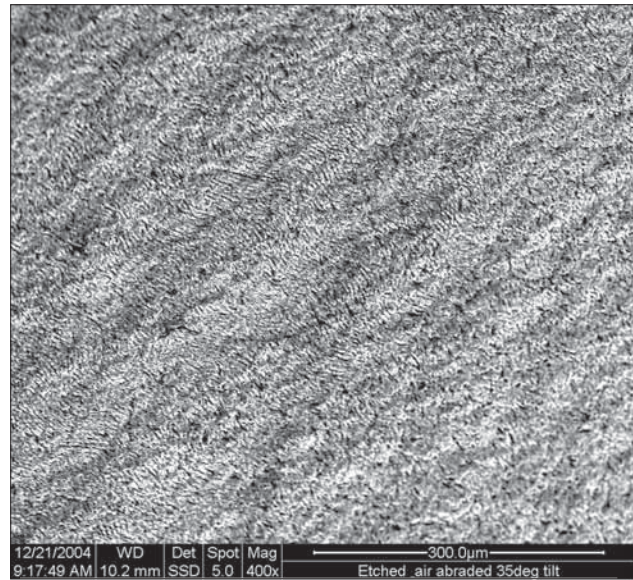


Figure 4. Scanning electron photomicrograph of enamel surface conditioned with air abrasion at 120-psi and 50-µm sized particles and acid etched with 37% phosphoric acid for 30 seconds (×400).

Although studies testing air abrasion as a means of conditioning primary teeth have not been reported, this study's results are comparable to those in previous reports testing permanent teeth.<sup>12,21,22</sup> Ellis et al concluded that, while air abrasion may be an effective pre-etch surface treatment prior to sealant placement, acid etch treatment significantly enhanced the long-term bond of sealant to permanent enamel.<sup>21</sup> Brown and Barkmeier concluded that the highest bond strengths were obtained when a combination of air abrasion followed by phosphoric acid conditioning was used.<sup>22</sup> Roeder et al also concluded that the highest bond strengths were achieved for permanent enamel surfaces that were air abraded and acid etched.<sup>12</sup> In addition, they found that the lowest bond strengths were demonstrated for enamel surfaces that were air abraded only. Although these studies evaluated bond strength to permanent enamel, the trends found are comparable to those in the present study which utilized primary enamel.

The mechanical bonding of resin to enamel plays an important role in the retention of restorations after enamel conditioning with acid etch. Studies have reported that after acid etching an enamel surface, the adhesive resin penetrates into surface irregularities, thereby producing retentive tags.<sup>26,27</sup> Silverstone reported the observed patterns of etch in which enamel prism cores were removed, enamel prism peripheries were removed, or a combination of both patterns were present.<sup>27</sup> In any case, enamel prisms and micropores are evident after etching (Figure 2). Tandon et al observed no differences on enamel surfaces between primary and permanent teeth conditioned with 37% phosphoric acid. Early

studies by Gwinnett, however, suggest that the outermost enamel of primary teeth often show prismless features, which may decrease the penetration of resin after etching.<sup>28,29</sup> In the present investigation, the prismless layer of enamel should not significantly influence the retention results since the enamel was ground flat prior to testing, thus removing part or all of the prismless layer.

Studies have also been conducted to evaluate the enamel surface after conditioning with air abrasion.<sup>30,31</sup> Peruchi et al evaluated cutting patterns produced by an air abrasion system in primary teeth.<sup>30</sup> They reported that:

1. the removal of enamel in primary teeth was most effectively accomplished when a tip (0.38-mm inner diameter) was held at a 2-mm distance from the tooth;
2. application time did not influence the cuts.<sup>30</sup>

This investigation utilized a 2.5-mm distance. Laurell et al found that air abrasion of the surface revealed a uniform roughness of the enamel and that the enamel prisms and dentinal tubules were not identifiable.<sup>31</sup> This is in agreement with the SEPs obtained in this study (Figure 3). The presence of "hills and valleys" can be seen after conditioning the enamel surface with air abrasion. In addition, enamel rod prisms were not evident after air abrasion.

Table 3. Mean, Median, and Standard Deviations (SD) of Percent Dye Penetration as a Function of Occlusal Conditioning

Group	Mean % significant dye penetration	±SD	Median % dye penetration	Significant difference*
Control	92	±11	97	A
KCP	94	±6	96	A
Acid etch	6	±6	5	B
KCP+acid etch	6	±7	4	B

\*Groups denoted by the same letter are not statistically different ( $\alpha=0.05$ ).



Ellis et al reported that enamel surfaces conditioned with a combination of air abrasion and acid etch (Figure 4) revealed a more detailed retentive pattern than surfaces treated with either treatment alone.<sup>21</sup> The enamel's uniform roughness is evident with the presence of "hills and valleys" as well as numerous micropores. The increased surface area and contours created at the macroscopic level by air abrasion, along with the micropores created by acid etching, may explain the increased bond strength observed when both of these surface conditioners are used together. Although air abrasion produces an increase in surface contour at the macroscopic level, the lack of micromechanical retention from resin tags may explain the lower shear bond strength values when used alone. The micropores formed in the enamel surface after acid etching may create resin tags, thereby resisting shear forces during specimen testing that would result in increased shear bond values.

A number of different methods have been utilized to study microleakage including radioactive tracers, dye penetration, bacterial penetration, silver nitrate staining, and scanning electron microscopy.<sup>32-35</sup> This study utilized computer imaging software to determine the extent of dye penetration along the sealant/enamel interface from the sealant margin in millimeters. The ratio of dye penetration compared to the sealant extension was then converted into a percentage dye penetration. In the present investigation, no significant difference was found between the combined use of air abrasion and acid etch and the acid etch only group. These 2 groups, however, exhibited significantly less mean percent dye penetration than air abrasion alone. Setien et al investigated the effect of 5 different cavity preparation devices, including air abrasion, on microleakage of composite restorations.<sup>36</sup> They reported that microleakage did not occur in enamel for any cavity preparation method tested as long as the enamel was acid etched prior to application of the adhesive.

Gungor et al evaluated the microleakage of a flowable composite on air abraded teeth.<sup>37</sup> The results showed that air abrasion alone resulted in significantly greater microleakage when compared to surfaces treated with air abrasion in combination with acid etching and a bonding agent.

Lupi-Pegurier et al evaluated the microleakage of a pit-and-fissure sealant after acid etching, air abrasion, and conventional rotary preparation.<sup>38</sup> They found that the microleakage of the sealants prepared with air abrasion alone showed the highest microleakage when compared to either acid etching or cavity preparation in combination with acid etching.

In all of the aforementioned investigations, air abrasion combined with acid etch produced less microleakage than air abrasion alone. The results obtained from the microleakage portion of this study are also in agreement with these previous investigations.<sup>36-39</sup>

This study's results have important clinical implications. The significant difference in dye penetration between this study's acid etch group (with and without air abrasion) and

the KCP 2000 group suggests a poor marginal seal when conditioned with air abrasions alone. A poor marginal seal may lead to microleakage, which, in turn, may lead to staining, postoperative sensitivity, adverse pulpal response, recurrent caries, and loss of restoration.<sup>18</sup> Therefore, any technique used to condition a tooth surface prior to resin placement should be examined for its effectiveness in creating an adequate marginal seal.

This study's findings indicate that the use of air abrasion as an alternative method to replace acid etching prior to sealant placement on primary teeth cannot be recommended due to the significant marginal leakage and decreased bond strength demonstrated. The use of air abrasion systems such as the KCP 2000 to remove decay and stains and widen grooves prior to sealant placement remains advantageous to the clinician. Acid etching prior to resin placement, however, remains a vital step after the use of air abrasion to decrease microleakage and increase shear bond strength.

## Conclusions

Based on this study's results, the following conclusions can be made:

1. In primary teeth, acid etch enamel treatment is the minimum requirement for sealant retention. Air abrasion complements the performance of the sealant in shear bond strength and microleakage.
2. Shear bond strengths between composite and human enamel were significantly higher following air abrasion and acid etching when compared to acid etch or air abrasion alone.
3. Microleakage around sealants may be decreased when the enamel surface is treated with acid etching or air abrasion and acid etching.

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