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Effects of acid etch parameters on enamel topography and composite resin — enamel bond strength

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Abstract

The objective of this study was to evaluate the effects of methods of acid application, wash times, and saliva contamination on the tensile bond strength of a composite resin to enamel. Ninety human maxillary central incisors were used and the tensile bond strength was determined for the composite resin to enamel surfaces subjected to the following acid etch parameters. In the control group (A) the acid was applied by dabbing followed by a 30-second wash. The acid was similarly applied for 30 seconds (B) and 120 seconds (C), rubbed for 60 seconds (D), not agitated for 60 seconds (E) followed by 30 seconds wash respectively. Wash times of 5 seconds (F) and 10 seconds (G) were used after 60 seconds of acid application by dabbing. Control procedures were followed by 30 seconds of saliva contamination and 5 seconds wash (H) or 5 seconds wash, 10 seconds re-etch and 30 seconds wash (I) were finally used. Incisors treated with one of each of the procedures were viewed in the SEM. Method of acid application by dabbing, rubbing, or no agitation resulted in bond strengths that were not significantly different, a 5-second wash after acid etching was sufficient to maintain bond strength and saliva contamination was eliminated by a 5-second wash. The effect of acid etch parameters on enamel topography was difficult to evaluate because of variable etch patterns.

The acid etch procedure is essential for the establishment of the mechanical bond between composite dental resins and enamel.[1] It consists of several steps which may affect the bond strength of the composite resin to etched enamel.

The first step of the acid etch technique involves the application of the orthophosphoric acid (H_3PO_4) etching solution to the enamel surface. Rubbing the enamel during conditioning adversely affects the bonding of composite resins to these surfaces.[2] Hormati and coworkers[3] reported, however, that the shear bond strength of a composite resin to enamel surfaces which were etched by a dabbing or a rubbing action were not significantly different despite the marked differences in etch patterns as observed by scanning electron microscopy (SEM).

The duration of etch and subsequent wash times are also important factors which may affect the bond strength of composite resins to etched enamel. It has been reported that with a short etch time of 10 seconds, the bond strengths increased as the wash time increased.[4] Other studies showed that the bond strengths of a composite resin to enamel surfaces etched with 50% H_3PO_4 for five seconds or 60 seconds were not significantly different if the enamel was washed with at least two ml of water after etching.[5,6] Washing with quantities of water greater than this volume provided no additional benefit. On the other hand, it was reported that the tensile bond strength increased with a thorough 60-second wash time as opposed to a 15-second wash.[7]

Bond strength may also be adversely affected by saliva contamination of the etched enamel surface. Hormati and coworkers[3] reported that etched enamel surfaces which were air dried after a 60-second saliva contamination significantly reduced the shear bond strength of a composite resin when compared with bond strengths obtained on enamel surfaces which were re-etched, rewashed, and redried after similar saliva contamination.

The objective of this investigation was to study further several of the parameters which may influence the bond strength of composite resins to etched enamel. Scanning electron microscopy (SEM) was used to determine the effects of the various procedures on the etching pattern of enamel.

Methods and Materials

Ninety noncarious, extracted human maxillary central incisors were used to study the tensile bond strength of a composite resin to etched enamel surfaces. The test method developed by Kemper and Kilian[8] was used in this investigation. Subsurface

Table 1. Acid etch procedures.

Group	Etch (sec)	Application Method	Wash (sec)	Contamination Saliva (sec)	Wash (sec)	Re-etch (sec)	Rewash (sec)
A	60	dabbing	30	—	—	—	—
B	30	dabbing	30	—	—	—	—
C	120	dabbing	30	—	—	—	—
D	60	rubbing	30	—	—	—	—
E	60	no agitation	30	—	—	—	—
F	60	dabbing	5	—	—	—	—
G	60	dabbing	10	—	—	—	—
H	60	dabbing	30	30	5	—	—
I	60	dabbing	30	30	5	10	30

enamel was exposed by lapping the facial surfaces of the crowns of the teeth on 600 grit silicon carbide paper in a polishing machine[a] under a continuous stream of water. The crowns were embedded in epoxy resin in the tooth specimen cups with the flat surfaces parallel to and projecting just above the lip of the cup. The epoxy resin was allowed to cure at room temperature for 24 hours and the embedded specimens were then stored in water at 37°C for at least 24 hours. Immediately prior to the preparation of the bonded test specimens the crown surfaces were lightly polished by placing the tooth cup in a polishing block on 600 grit silcon carbide paper in the polishing machine. This provided a planar enamel surface perpendicular to the direction of force application during the bond strength testing procedure. The embedded teeth were subjected to one of the procedures outlined in Table 1. A random distribution table was computer processed to determine the order in which the test specimens were to be prepared.

The tooth surfaces were acid etched with 37% H_3PO_4, washed and dried with an air-water syringe. Water was collected in a graduated cylinder to determine the volume used in each washing procedure. Each timed collection was repeated 10 times and the average volume of water determined. A two paste composite resin[b] was mixed as directed, placed in the composite cup and assembled in the bonding alignment block. A low viscosity bonding resin was not applied to the etched enamel surfaces. The composite cup was lowered to contact the flat enamel surface. The composite resin was allowed to cure against the enamel surface under a load of one pound for 15 minutes at room temperature, after which the bonded specimens were carefully removed and stored in water at 37°C for 24 hours. Test specimens were mounted in the measurement alignment block and suspended in the jaws of an Instron[c] testing machine. The tensile load was applied at a rate of 0.02 inch/min until the bonded specimens failed. Tensile bond strengths were calculated and expressed in $MN.m.^{-2}$ Statistical comparisons of the data were made by an analysis of variance at the 0.05 level of significance.

Etch patterns for each treatment group were determined by subjecting two maxillary central incisor crowns to one of the procedures listed in Table 1. The etched crowns were coated with gold/palladium in a high vacuum evaporator[d] and examined in a Cambridge Mark 2A[e] scanning electron microscope operated at 20 kV.

Results

The mean (± SD) of the volumes of water used in each wash regimen were as follows: five seconds, 16.3 ± 1.0 ml; 10 seconds, 33.8 ± 2.0 ml; 30 seconds, 95.3 ± 1.6 ml.

The Mean (± SD) of the tensile bond strengths of the composite resin to enamel subjected to the different etching procedures are given in Table 2. Evaluation of the data by an analysis of variance showed that there were no statistically significant differences among the tensile bond strengths.

When the acid was applied with a dabbing action for 60 seconds, typical Type 1 (Figure 1) Type 2 (Figure 2) and Type 3 (Figure 3) etching patterns were observed on the same tooth surface with the Type 3 pattern predominant in the cervical third of the crown. At a lower magnification all three etching patterns were frequently observed in the same field of vision (Figure 4). The etching produced by the application of the acid for 30 seconds could not be distinguished from that of a 60-second etch while a more pronounced etching effect was produced by a 120-second etch. When the acid was applied with a rubbing action, the prism peripheries were obscured resulting in a more uniform etching (Figure 5). The

[a]Buehler Ltd., 2120 Greenwood St., Evanston, IL.
[b]Concise, 3 M Co., St. Paul, MN.
[c]Instron Engineering Corp., 2500 Washington St., Canton, MA.
[d]Hitachi Ltd., Tokyo, Japan.
[e]Cambridge Scientific Instruments Ltd., Cambridge, England.

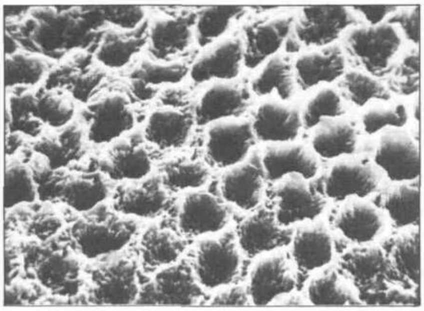

Figure 1. Etching solution was applied for 60 seconds with a dabbing action. Type 1 etching pattern (2,500x).

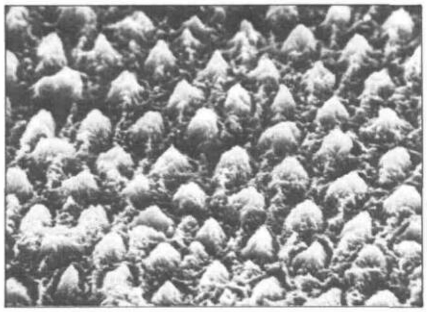

Figure 2. Etching solution was applied for 60 seconds with a dabbing action. Type 2 etching pattern (2,500x).

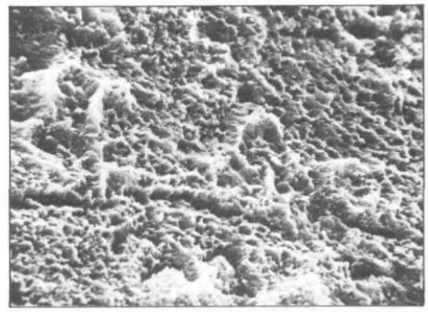

Figure 3. Etching solution was applied for 60 seconds with a dabbing action. Type 3 etching pattern (2,500x).

etching effect of nonagitated acid could not be distinguished from that produced by acid applied with a dabbing action. The reaction products of a 60-second etching were not completely removed from the etched surfaces by a 5-second wash (Figure 6). The etched surfaces obtained after washing for 10 seconds were similar to those described for 30-second wash times. Salivary contamination of 30 seconds was effectively removed by a 5-second wash, and re-etching of these surfaces for 10 seconds produced a similar etching effect.

Discussion

Under the experimental conditions defined in this *in vitro* study, it would appear that it is not necessary to adhere strictly to some of the generally accepted parameters associated with the acid etch technique in order to obtain comparable bond strengths of composite resins to etched enamel surfaces. The tensile bond strengths of test specimens prepared on enamel surfaces to which the acid was applied by a rubbing action were not significantly different from those in which the acid was applied by dabbing as usually recommended. Similar findings were previously reported.[3] Although the etching pattern on enamel surfaces exposed to acid applied by a dabbing action varied greatly from one tooth to another and even in adjacent areas of the same tooth, it resulted in clear delineation of the enamel prisms in the Type 1 and Type 2 etching patterns (Figures 1 and 2). When the acid was applied by a rubbing action, the prism peripheries were obscured and the etched surface appeared more uniform (Figure 5). One would expect that this procedure would limit the extent of resin penetration into the etched enamel surfaces and thus adversely affect bond strength. This was not observed in this study however. A direct relationship between resin tag length and bond strength has not yet been demonstrated. Intimate interfacial contact between a dental resin and relatively smooth enamel surface without deep resin penetration may result in adequate bond strengths.[9]

Table 2. Mean and SD of the tensile bond strengths of the composite resin to enamel subjected to the various etching procedures.

Procedure	Mean and SD of Tensile Bond Strength $MN.m^{-2}$	Coefficient of Variation %
A	16.8 ± 5.8	34.5
B	17.8 ± 8.2	46.2
C	16.1 ± 6.6	41.1
D	20.7 ± 7.6	36.8
E	19.8 ± 8.1	41.2
F	13.8 ± 5.6	40.5
G	19.6 ± 5.6	28.5
H	16.4 ± 5.3	32.5
I	15.4 ± 6.8	44.2

Means are not significantly different.

The various parameters investigated in this study had no significant influence on tensile bond strengths. The high coefficients of variation in the tensile bond strengths ranging from 28% to 46% in the present study, are usual in this type of test.[10] If the number of specimens in each group had been appreciably larger, some of the nonsignificant differences may have been statistically significant. The test method used in this investigation reduces the introduction of forces other than tensile during the testing procedure. It is, however, impossible to eliminate these forces altogether. A slight malalignment of the test specimens in the bonding or measurement alignment blocks may introduce a shear component at the resin-enamel interface during the application of the tensile load. Examination of fractured test specimens by SEM revealed that the majority of test specimens failed partly within the resin and partly at the resin-enamel interface, and that air bubbles incorporated into the resin during

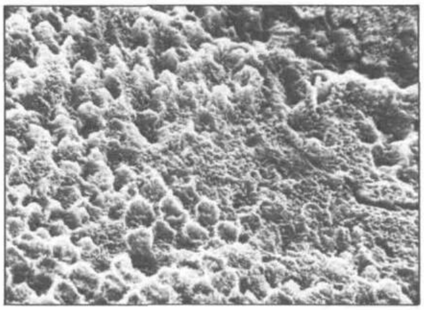

Figure 4. Etching solution was applied for 60 seconds with a dabbing action. Mixed etching pattern (1,250x).

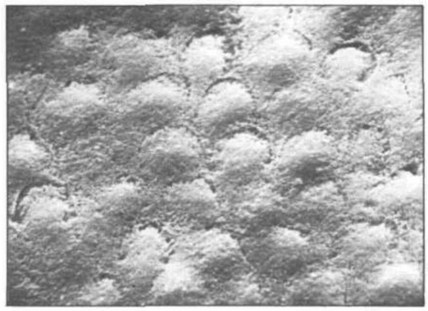

Figure 5. Etching solution was applied for 60 seconds with a rubbing action (2,500x).

Figure 6. Enamel surface etched by dabbing action for 60 seconds and washed for 5 seconds (2,500x).

the mixing procedure were distributed within the resin along the fracture lines.[11] Stress concentration may arise at these sites (which develop during polymerization of the resin) and fractures may be propagated along the defects during the application of the tensile load. Test specimens may fail prematurely as a result of the well-known zipper effect.[12]

The phosphoric acid solutions used as etching agents in clinical dentistry contain more than 27% H_3PO_4. Hydroxyapatite dissolution during phosphoric acid application proceeds rapidly initially, but is eventually impeded by the precipitation of monocalcium phosphate monohydrate $[Ca(H_2PO_4)_2.H_2O]$ on the enamel surface.[13] This may be another reason for the recommendation of acid application by dabbing. It is believed that the agitation resulting from the dabbing action will disrupt the precipitate and continually expose the underlying enamel to dissolution. The test specimens which were prepared on enamel surfaces to which the acid was applied but not agitated were expected to yield lower bond strengths than those specimens prepared on enamel surfaces to which the acid was applied by a dabbing action. This however, was not observed in the present study and may be explained by the fact that nonagitated acid also produced a well-defined etching pattern.

Unlike Soetopo and his colleagues,[7] no increases were found in bond strengths as wash times after etching were increased. Beech[5] and Beech and Jalaly[6] reported that two ml were adequate for washing etched enamel surfaces. The volume of water used with a 5-second wash in the present study (16.3 ml) exceeded this volume greatly. Monocalcium phosphate monohydrate is readily soluble in water and would be completely washed away in the clinical situation.[13] Although some precipitate may remain in isolated areas as shown in Figure 6, this did not affect bond strength adversely.

Contamination of etched enamel surfaces by saliva can readily occur in the clinical situation — particularly if rubber dam is not used. The results of the present study indicated that tensile bond strengths were maintained by washing salivary contaminated etched enamel surfaces for 5 seconds with an air-water spray and that it was not necessary to re-etch contaminated surfaces. It is not possible to extrapolate the results of this *in vitro* study directly to the clinical situation. The authors believe that salivary contamination of etched enamel surfaces prior to the application of composite resins should be avoided at all costs. This can readily be achieved by the use of rubber dam.

The great variation in etching patterns on enamel surfaces etched with H_3PO_4 has previously been demonstrated.[14-16] The reason for this is not known but the Type 1 and Type 2 etching patterns may be due to variations in crystallite orientation in the enamel prisms.[17] The Type 3 etching pattern is produced on prismless enamel, that is enamel surfaces in which the enamel prisms do not reach the surface.[18]

No significant differences were observed in tensile bond strengths of test specimens prepared on enamel surfaces etched with 37% phosphoric acid for 30, 60, or 120 seconds respectively. The tensile bond strengths were determined on enamel surfaces in which the superficial enamel had been removed by the polishing procedure prior to the preparation of the test specimens. Enamel fluoride concentration is greatest in superficial enamel.[19] Etching times longer than 30 seconds may be required in the clinical situation as high fluoride concentrations have an adverse effect on bond strength[20] and decrease the surface free energy of enamel.[21]

Conclusions

The results of this *in vitro* study indicate that the method of acid application by dabbing, rubbing, and no agitation had no significant effect on the tensile bond strength of a composite resin to etched enamel. The tensile bond strengths to enamel surfaces etched for 30, 60, and 120 seconds were not significantly different. A 5-second wash after acid etching was adequate to maintain bond strength. Saliva contamination of etched enamel surfaces was effectively eliminated by a 5-second wash without resorting to re-etching of the enamel surface.

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Quotable Quote

Sometimes give your services for nothing, calling to mind a previous benefaction or present satisfaction. And if there be an opportunity of serving one who is a stranger in financial straits, give full assistance to all such. For where there is love of man, there is also love of the art. For some patients, though conscious that their condition is perilous, recover their health simply through their contentment with the goodness of the physician. And it is well to superintend the sick to make them well, to care for the healthy to keep them well, but also to care for one's own self, so as to observe what is seemly.

From: Hippocratic Collection, Precepts, Chapter 6.