# Comparison of Morphological Variation and Shear Bond Strength Between Conventional Acid Etchant at Different Etch Times and Self Etching Primer - An *in vitro* Study

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***Abstract*—The bonding of orthodontic attachments to acid-etched enamel is an accepted clinical procedure. Phosphoric acid is the commonly used acid for etching before bonding. This in vitro study compared the enamel etch patterns achieved on the orthodontic bonding area of extracted premolars treated with 37% wt/wt phosphoric acid applied for 15, 30seconds and self etching primer. The etch patterns were viewed with a scanning electron microscope and assessed. The statistical analysis indicates that the self etching primer and 37% phosphoric acid at 15 & 30 seconds etched tooth produced variable etching pattern. Application of 37% phosphoric acid was more effective at producing a good etch pattern at 30 seconds than 15 seconds. Shear bond strength study shows that the 30 seconds etched tooth surface with 37% wt/wt phosphoric acid shows relatively higher bond strength when compared with 15 seconds etched tooth. While comparing with the 30 seconds of 37% wt/wt phosphoric acid and self etching primer group comparison shows there was no significant difference in bond strength, and comparison of 15 seconds of etching with phosphoric acid and self etching shows that self etching primer etching was effective than the 15 seconds etching with phosphoric acid.**

***Index Terms*—orthodontic bonding, acid etching, etching pattern, self-etching primer, shear bond strength.**

## I. Introduction

The introduction of the acid etching technique by Buonocore in 1955, have greatly influenced and revolutionized orthodontic practice. A key factor in bonding is the enamel to composite interface.

For application of orthodontic appliances to a dental structure surface, the etchants, primers, and adhesives are typically applied in a step-wise fashion. Often between such steps, one or more rinsing and drying steps are used. As a result the application of orthodontic appliances typically involves multi-step procedures[2, 5, 14].

To simplify orthodontic procedures, it would be desirable to provide a single composition that accomplishes both etching and priming self-etching primer was introduced, for improved bonding of adhesives to a substrate surface which eliminated the conventional post-etching rinsing and drying steps and also helps in prevention of contamination of the etched surface[2, 5, 7].

## II. Materials & Methods

This study was performed in the Department of Orthodontics and Dento-Facial Orthopedics, R.M.D.C&H and in collaboration with Sophisticated Test and Instrumentation Centre (STIC) Cochin University, Kochi.

Seventy eight freshly extracted teeth for orthodontic purpose (maxillary and mandibular premolars) were collected for the study. The teeth were clinically sound. Following extraction, residue on the teeth was removed and washed away with tap water. They were then stored in normal saline at room temperature to prevent dehydration and bacterial growth.

### *A. Materials Used*

- Maxillary and mandibular premolar stainless steel brackets (American Orthodontics®).
- 37% phosphoric acid gel.
- Transbond XT light cure adhesive & primer (3M Unitek).

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- Transbond TM plus Self Etching Primer (3M Unitek).
- Universal testing machine (Lloyd Universal testing machine –Model No. L.R. 100K)
- Scanning electron microscope (JOEL Model No-JSM 6390LA)

### B. Methodology

The buccal surface of the teeth were cleaned with a pumice and water with the use of rotary brush in a dental hand piece, they were rinsed with water for 30 seconds and dried with oil and water free compressed air for 30 seconds following which they were mounted on acrylic blocks such that the roots were completely embedded into the acrylic up to the cemento-enamel junction leaving the crown exposed.

The teeth were randomly divided into three groups. Group 1, 2 & 3 respectively. Each group contains 20 teeth.

*Group 1* etched *with 37% phosphoric acid for 15 seconds.*

*Group 2* etched *with 37% phosphoric acid for 30 seconds.*

*Group 3* etched *with self etching primer.*

The blocks were color coded for easy identification. Acid etching was done on the buccal surface of the teeth with 37% phosphoric acid gel for 15 & 30 seconds. The teeth was again washed and dried with oil and water free compressed air. A thin coat of primer was applied to the acid-etched enamel.

The self-etch primer which contains both the acid and the primer, For activation, the 2 components are squeezed together, and the resulting mix can be applied directly on the tooth surface etchant placed on the enamel of 20 teeth for 5 seconds and gently evaporated with oil and moisture free air for 1-2 seconds, Following the enamel conditioning the teeth were bonded with premolar brackets (American Orthodontics®) using Transbond XT light cure adhesive (3M Unitek) the excess material was removed using sickle scaler and cured for 40 seconds using a visible light cure unit.

### C. Bond Strength Testing

Bond strength testing was performed with universal testing machine (Lloyd Universal testing machine – Model No. L.R. 100K) at a crosshead speed of 1mm/min. The shear force was applied with chisel shaped rod from the occlusal side parallel to the bracket surface. The embedded teeth and brackets were aligned in the testing apparatus to ensure consistency for the point of force application and direction of the debonding force for all samples. The load at failure was recorded in a computer in terms of Newtons. This was converted into Mega Pascal as the ratio of debond force to the surface area of the bracket.

$$\text{Bond strength MPa} = \text{Force (Newton)} / \text{Surface area of the bracket (mm)}^2$$

## III. SEM Analysis

### A. Sample Preparation

Eighteen teeth were used for the analysis and randomly divided into three groups contains six teeth each.

*Group 1* etched with 37% phosphoric acid for 15 seconds.

*Group 2* etched *with 37% phosphoric acid for 30 seconds.*

*Group 3* etched *with self etching primer.*

The crown part of the premolars were sectioned at the cemento-enamel junction and they were mounted on the acrylic blocks the crowns of the premolars were oriented with their buccal surfaces facing uppermost and the samples were etched and washed they were prepared for scanning electron microscopy (SEM) the teeth was gold sputtered and examined in the SEM microscope at 10kv and 2000x magnification. Photograph was recorded from the central region of each etched area with a standardized orientation technique to ensure uniformity between specimens the etch pattern was compared using the following 3-grade scale[12].

- Poor etch pattern;
- Moderate etch pattern;
- Good etch pattern.

1) Poor etch pattern. Smooth or amorphous surface with no evidence of "Type 1" etch pattern (preferential dissolution of enamel prism cores) or "Type 2" pattern (dissolution of prism boundaries) in the area examined.
2) Moderate etch pattern. At least 50% of the examined samples exhibiting “Type 1” or “Type 2” etching.
3) Good etch pattern. “Type 1” or “Type 2” etch pattern in nearly all the area examined.

## IV. Statistical Analysis

The descriptive statistics, including the mean, standard deviation and the probability values were calculated for the groups tested. The statistical interactions of the shear bond strength between and within the groups were analyzed with one–way analysis of variance (ANOVA) test. Statistical significance level was established at $p < 0.05$.

TABLE I

| Groups | N | Mean | Std. Deviation | F | Sig. |
|---|---|---|---|---|---|
| Group 1 | 20 | 9.4035* | .06319 | 105.850 | .000 |
| Group 2 | 20 | 9.6305 | .04673 | | |
| Group 3 | 20 | 9.4130* | .05630 | | |
| Total | 60 | 9.4823 | .11911 | | |

*Shear bond strength results*

*comparison of group 1 and group 3 were not significant statistically.

## V. Results

The aim of the present study was to compare the shear bond strength between self etching etching primer and 37% phosphoric acid at different etch times. Also, to evaluate variations in acid etch patterns with 37% phosphoric acid at different etch times and self etching primer. (Table I)

### A. Shear Bond Strength Results

This study found that, there was a significant difference between shear bond strength of the three groups, with the group treated with 37% phosphoric for 30 seconds showing higher bond strength when compared to the other two groups.

In accordance with the present study the results of the work done by Morten Fjeld & Bjorn Ogaard[11] have shown that most of the self-etching primers did not etch enamel as deeply as the phosphoric acid etchants did and the shallow etching pattern could compromise the bonding to enamel.

Reports of Pashley & Tay[13] have shown that the efficiency of self-etching primers in intact enamel does not depend solely upon their etching aggressiveness, but also on monomeric composition of each material.

*1) Description*

The mean shear bond strength of the brackets etched with 37% Phosphoric acid gel for 15 sec (Group 1) 9.403 ± 0.063MPa and for 37% Phosphoric acid gel for 30 sec (Group 2) 9.630 ± .04673 and Self etching primer for (Group 3) 9.4130 ±05630. P value lesser than 0.05 shows there is significant difference in shear strength between groups. (Table II and Table III)

TABLE II

| Groups | Etch pattern observed | | |
|---|---|---|---|
| | Good Etch Pattern | Moderate Etch Pattern | Poor Etch Pattern |
| 15 Seconds Etched 37% $H_3PO_4$ | 0 | 2 | 4 |
| 30 Seconds Etched 37% $H_3PO_4$ | 4 | 2 | 0 |
| Self Etching Primer | 2 | 4 | 0 |

*SEM results of individual groups etch pattern between groups 1, 2 & 3*

TABLE III

| Groups | N | Mean | Std. Deviation | F | Sig. |
|---|---|---|---|---|---|
| Group 1 | 6 | 1.3333 | .51640 | 10.833 | .001 |
| Group 2 | 6 | 2.6667 | .51640 | | |
| Group 3 | 6 | 2.3333 | .51640 | | |
| Total | 18 | 2.1111 | .75840 | | |

*SEM results*

### B. SEM Results

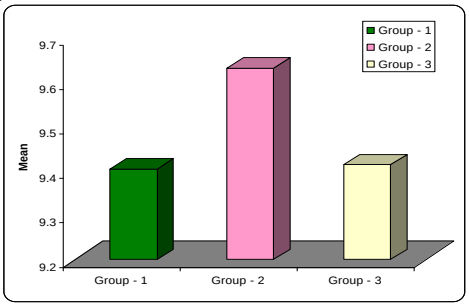
Results of the present study have shown that etching with 37% phosphoric acid at different etch times and the self etching primer produced variable type of etching patterns. The etch patterns were graded by using a 3-grade etch scale[10].

Brannstorm and Nordenval9 found little difference in the microscopic appearance of enamel prepared for resin restoration after etching for 15 seconds and 120 seconds with 37% phosphoric acid solution. In contrast, the present study has shown that there is change in the etching pattern of enamel while changing the time of exposure from 15 seconds to 30 seconds with a 37% phosphoric acid.

*1) Description*

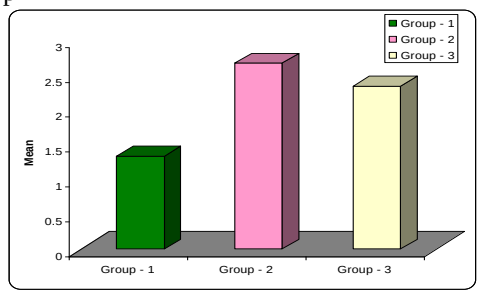
The SEM graded photographs were compared between 37% Phosphoric acid gel for 15 sec (Group 1) 1.3333 ± .51640 and for 37% Phosphoric acid gel for 30 sec(Group 2) 2.6667 ± .51640 and Self etching primer for (Group 3) 2.3333 ± .51640 p value lesser than .05 shows there is significant difference among the groups.

Graph 1




*Shear bond strength result*

Graph 2




*SEM Result*

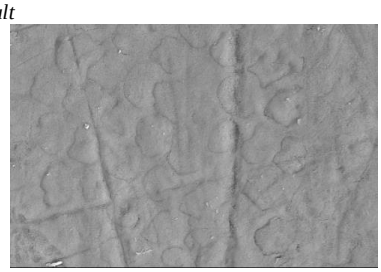


Figure 1. Poor etch pattern.

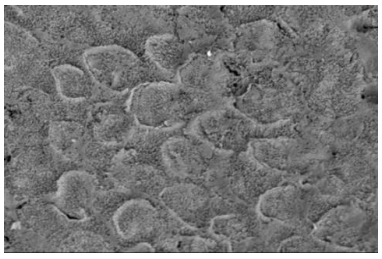


Figure 2. Moderate etch pattern.

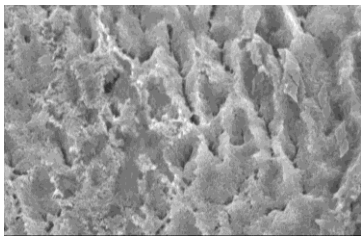


Figure 3. Good etch pattern.

## VI. Conclusions from the Present Study

### A. SEM Study:

1) Self etching primer and 37% phosphoric acid produced variable etching pattern.
2) Application of 37% phosphoric acid produced a good etch pattern at 30 seconds.

### B. Shear Bond Strength Study:

1) A 30 second etched tooth surface with 37% $H_3PO_4$ shows a relatively higher bond strength when compared with 15 second etched tooth surface using 37% $H_3PO_4$ and self etching primer.
2) There was no significant difference in bond strength between self etching primer and 15 second etched 37% $H_3PO_4$.

Phosphoric acid etching causes dissolution of interprismatic material in enamel, producing a roughened and porous layer that ranges in depth from five to 50 μm2,8. 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[1, 3, 6].

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[4, 5, 7]. The use of self-etching primer (SEP) would have the advantage of a faster and simplified application technique, allowing adequate etching and priming of enamel in only one step[15].

From a clinical standpoint, more studies are required to compare the variations in etch patterns as well as bond strengths, among the conventional multistep etching procedure and single step self-etching primers, particularly in-vivo studies and clinical trials.

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