



EVALUATION OF ETCH PATTERNS, PENETRATION DEPTH, AND BOND STRENGTH BETWEEN VARIOUS ETCHING TECHNIQUES IN ORTHODONTICS

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Abstract :

Objective: To compare etch patterns, penetration depth, and bond strength between conventional phosphoric acid etching, crystal growth technique and self-etching primer.

Material and Methods: 60 freshly extracted maxillary first premolar teeth were collected for the study and the samples were divided into three groups for each etchant (20 samples per group). 10 samples from each group were tested for enamel penetration depth and tensile bond strength and remaining 10 samples from each group were used for assessing etch pattern using Scanning Electron Microscope.

Results: Conventional 37% phosphoric acid produced sufficient bond strength with deeper enamel penetration. Self-etching primer produced clinically acceptable bond strength values but lower than phosphoric acid etching with moderate etch pattern. Etch pattern produced by both of these etchants were similar in appearance. Crystal growth technique by 20% polyacrylic acid failed to achieve clinically acceptable bond strength values.

Conclusion: The self-etching primer can be used as an alternative to phosphoric acid etching. Etching with poly acrylic acid failed to achieve clinically acceptable bond strength even though it results in less penetration into the enamel.

Key words: Acid etching, crystal growth, Self-etching primer, Etch pattern, penetration depth, tensile bond strength

I. INTRODUCTION

Since Buonocore¹ first introduced the use of phosphoric acid for etching the enamel surface of teeth, several other alternative acids and chelating agents have been studied. None of these methods have proved superior to the conventional phosphoric acid. Most of them resulted in poorer etch pattern, lower bond strength, and excessive enamel dissolution. Iatrogenic effects of conventional phosphoric acid etching include loss of enamel while debonding, increased plaque retention, loss of fluoride rich enamel with aging etc. In this perspective, the discovery of crystal growth pattern as by Smith and Cartz² in 1973 and the introduction of self-etching primers (SEP) by Bishara³, that combined the conditioner and primer into a single acidic solution receives attention. The crystal growth phenomenon by using sulphated polyacrylic acid on enamel surface serves as an additional retentive mechanism for the resin to attach the orthodontic attachment to the teeth. This way, extensive penetration into the enamel is avoided. The enamel surface creates micromechanical interlocking, which removes the majority of the aforementioned restrictions. Newer self-etching adhesive systems have been developed to reduce the number of steps involved in bonding, thus considerably reducing the chair side time. It has the benefit of achieving a gentler etch configuration as compared to 37% phosphoric acid. The aim of this study is to evaluate the effect of crystal growth, conventional acid etching and self-etching primer on etch pattern, penetration depth, and tensile bond strength.

II. MATERIALS AND METHODS

In this invitro study 60 permanent Maxillary first premolar teeth extracted as part of orthodontic treatment were collected and stored in 0.1 percent(weight/volume) thymol solution. The selection criterion for teeth included having completely erupted teeth free of restorations, caries, or other gross morphological abnormalities.

Materials

- Group I : 37% phosphoric acid (D -Tech Etching Gel XT3G)
- Group II : 20% sulphated polyacrylic acid
- Group III : Self etching primer (3M ESPE Adper prompt L-Pop)
- Orthodontic stainless steel pre-adjusted edgewise bracket with standard bracket slot .022"x.028" (Ormco Minidiamond) and bracket base area of 9.32mm²
- Bonding agent : (3M Unitek Transbond XT)
- Light cure adhesive : 3M Unitek Transbond XT
- Curing unit : 3M ESPE Elipar with intensity 1300Mw/cm² and wave 450nm

Methods

To assess these parameters, the samples were randomly divided into groups of 20 teeth for each group, of which 10 samples from each group taken for SEM analysis and remaining 10 samples from each group were used for assessment of tensile bond strength followed by evaluation of etchant penetration depth.

Group I: Phosphoric acid etching: Buccal surface of the teeth was polished with pumice paste prior to etching. Ten samples were etched with 37% phosphoric acid for 30 seconds, rinsed and air-dried until a frosted whitish appearance was seen on the enamel surface. After etching Transbond XT primer (3M Unitek) was applied to the etched enamel surface in the form of a thin layer. A small amount of adhesive paste of Transbond XT (3M Unitek), was applied on the base of the bracket. Immediately after placing the adhesive the attachment was lightly placed on the tooth surface and pressed firmly after adjusting the position. Excess resin material around the bracket was removed and curing of the adhesive done for a total of 30 seconds with 3M ESPE Elipar curing unit with intensity of 1300Mw/cm² and wavelength of 450nm.

Group II: Sulphated polyacrylic acid: Twenty percent polyacrylic acid was sulphated using 0.3M K₂SO₄ in the Department of Biochemistry. Ten samples were etched with 20% sulphated polyacrylic acid for 30 seconds, rinsed and air-dried until a uniform chalky white appearance was seen on the enamel surface. After etching Transbond XT primer (3M Unitek) was applied to the etched enamel surface in the form of a thin layer. A small amount of adhesive paste of Transbond XT (3M Unitek), was applied on the base of the bracket. Immediately after placing the adhesive the attachment was lightly placed on the tooth surface and pressed firmly after adjusting the position. Excess resin material was removed around the bracket and curing of the adhesive done for a total of 30 seconds.

Group III Self-etching primer: Buccal surface of ten samples polished with pumice paste prior to etching. Self-etching primer Prompt L-Pop*(Fig.1), available as a blister pack was activated by emptying the liquid from the first reservoir into the second reservoir. The activated liquid mixture was then emptied into the green section of the pack and then applied to the enamel surfaces using the pre-packaged, disposable applicator, followed by dried lightly using oil free compressed air recommended by manufacture. Composite resin (Transbond XT) was then applied to the bracket base and the bracket positioned firmly on the tooth surface. Light curing of the adhesive done for 30 seconds.



Fig.1 Self-etching primer Adper Prompt L-Pop

The study was split into three parts:

1. Assessment of tensile bond strength -using Universal Testing Machine
2. Assessment of penetration depth –using Profilometer
3. Evaluation of etch patterns – using scanning electron microscope (SEM)

ASSESSMENT OF TENSILE BOND STRENGTH

Ten samples of bonded tooth from each group mounted on acrylic block (Fig.2), were used to assess the tensile bond strength. Each acrylic block was mounted firmly to the lower jaw of the universal testing machine (Instron) (Fig.3). The test configuration was then loaded in tension, with the generation of tensile forces perpendicular to bracket at a cross-head speed of 1mm/min until failure occurred. The load required to break the bond is recorded in kilogram. The tensile bond strength was calculated by dividing the force by the bracket base area.

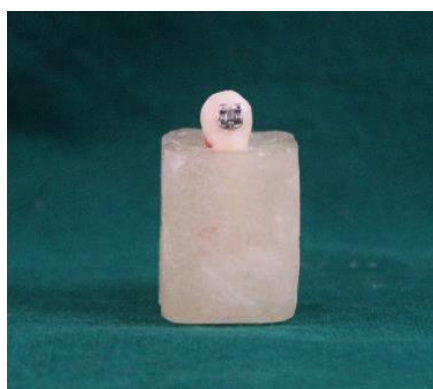


Fig.2. bonded tooth mounted on acrylic



Fig.3-Universal testing machine

Assessment of etchant penetration depth

Ten samples of deboned tooth after tensile bond testing were used to assess the penetration depths. The samples were mounted over the measuring table of the profilometer Mitutoyo sj-410. The etchant penetration depth of these specimens was recorded with a stylus of radius 0.5 μm . The mean maximum peak to height velocity is measured. The mean values obtained were in micron meters (μm).

Assessment of etch pattern by sem.

The remaining thirty teeth had their root sectioned out and their crowns cleaned and pumice prophylaxis was done prior to etching. The samples were randomly divided into group of 10 teeth for each group and etching was performed. The etched surfaces were air dried and kept in a desiccator overnight and then observed under JEOL JSM-6390LA analytical scanning electron microscope. Representative photomicrographs were taken for each sample at magnifications 1000X and 2000X

Statistical analysis

Statistical package for social science (SPSS) software (version 16. SPSS Inc., Chicago, Illinois USA) was used for statistical analysis. Descriptive statistics of penetration depth and tensile bond strength between three groups were calculated. The level of significance (P-Value) was set as $P < 0.05$.

III.Results

The Scanning Electron Microscopic evaluation shows different patterns of etching on the enamel surface with the three etchants studied. Etching with 37% phosphoric acid showed mostly the type I etch pattern characterised by hollowing of prism centres with relatively intact peripheral regions (Fig.4). The SEM photograph of the 20% sulphated polyacrylic acid etch was unique. It showed a mildly etched enamel surface with characteristic spherulitic crystals throughout the etched surface (Fig.5). self-etching primer shows etch pattern resembling to that of phosphoric acid etching, but it is more consistent, shallower, and less damaging to the enamel surface (Fig.6).

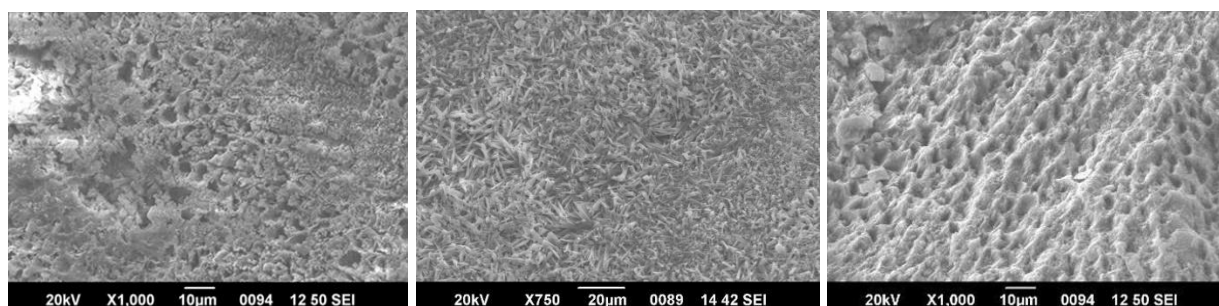


Fig.4-Enamel surface etched with 37% Phosphoric acid (SEM1000X), Fig.5-Enamel surface etched with 20% sulfated polyacrylic acid (SEM 750X), Fig.6-Enamel surface etched with self-etching primer (SEM 1000X)

Table 1 and 2 shows statistical comparison of tensile bond strength of three groups. Phosphoric acid etching produces the highest tensile strength (11.043 MPa), suggesting it provides the strongest bonding. Self-etching primer shows moderate tensile strength (8.943 MPa), but lower than phosphoric acid etching. Polyacrylic acid etching results in the lowest tensile strength (4.54 MPa), indicating it is less effective in enhancing adhesion.

Table 1: Descriptive statistics for comparison of Tensile bond strength between three groups

	Number of samples	Mean	Std. Deviation	Std. Error
Phosphoric acid etching	10	11.043	1.4501	0.45856
Polyacrylic acid etching	10	4.54	1.08288	0.34244
Self-etching primer	10	8.942	1.83981	0.5818
Total	30	8.175	3.10845	0.56752

Table 2: Comparison of mean tensile bond strength between three groups

ANOVA					
Tensile Strength					
	Sum of Squares	Degree of freedom	Mean Square	F	Significance.
Between Groups	220.269	2	110.135	49.608	0.00
Within Groups	59.943	27	2.22		
Total	280.212	29			

ANOVA is used to assess the statistical significance of the differences in etchant penetration depth among three groups. Deepest penetration depth of 16.612 was obtained with 37% phosphoric acid with the range of 10.37-21.32 μm (SD = 3.21) suggesting more enamel loss and the least was with 20% sulfated polyacrylic acid in the range of 5.23-9.37 μm with a mean of 7.038 μm (SD = 1.48). Self-etching primer shows moderate penetration in the range of 5.98-11.41 μm with a mean of 8.096 μm (SD = 1.64) but is significantly lower than phosphoric acid etching (Table 3).

Table 3: Descriptive statistics for comparison of Penetration Depth between three groups

Penetration Depth				
	Samples N	Mean	Std. Deviation	Std. Error
Phosphoric acid etching	10	16.612	3.2097	1.015
Polyacrylic acid etching	10	7.038	1.47558	0.46662
Self-etching primer	10	8.096	1.64111	0.51896
Total	30	10.582	4.8692	0.88899

IV.DISCUSSION

Enamel loss on etching and finishing has been well documented in various literature. About 10–20 μm of outer enamel is lost during acid etching⁴, and another 6–50 μm is lost during debonding, depending on the operator and technique⁵. Therefore, an attachment mechanism which produces sufficient bond strength without significantly damaging the enamel would be more advantageous.

Maijer and smith^{2,6} introduced a new method of bonding that involves crystal growth on the enamel surface. This system involves the use of polyacrylic acid with residual sulphate ion that react with the calcium in the enamel surface to form a dense growth of small, needle shaped crystals. The crystal buildup serves as an additional retentive mechanism for the resin that bond the orthodontic bracket to teeth. They claim that the crystal buildup will provide sufficient strength to withstand tensile forces up to 11kg. From their further research on crystal growth technique, they reported that, crystal morphology can be changed by changing the concentration of sulfate ion thereby a better retentive surface can be created into which an unfilled resin could flow. They found that the new formulation's tensile bond strength was comparable to that of traditional phosphoric acid etching.

Another alternative to phosphoric acid etching which is most commonly used now in practice is the self-etching primer. They are based on hydrophilic acid monomers, do not require the rinsing step and are capable of acting as bonding agents to both enamel and dentine¹⁴. Despite being demonstrated to provide modest time saving the use of self-etching primers has increased because of its simplicity.

This study compared the etch patterns, penetration depth, and bond strength between conventional phosphoric acid etching, crystal growth technique and self-etching primer in order to determine the most suitable technique which will offer clinically acceptable bond strength with minimal enamel damage.

The etch pattern obtained with conventional 37% phosphoric acid are in good agreement with those reported in the literature^{7,8,9}. It showed mostly the type I etch pattern characterised by hollowing of prism centres with relatively intact peripheral regions and it is considered to be the desirable etched surface for an adequate bonding to enamel. Etching with 20% sulphated polyacrylic acid showed crystal formation as described by Maijer and Smith². They were small, needle like crystals which was found spread all over the etched tooth surface. The etch pattern produced by self-etching primer is resembling to that of phosphoric acid etching, but it is more consistent, shallower, and less penetrating to the enamel surface. Bishara¹⁰, Oliveira¹¹ and Julio P. Cal-Neto¹² reported similar etch pattern with self-etching primer. The first persons to characterise the resin tags inside the enamel as filamentous resin projections were Gwinnett⁴ and Buonocore¹. These tags are roughly 10–20 μm long. Resin tags are the factors responsible for adhesive retention. Buonocore¹ was the first to investigate the depth of resin penetration. According to him, attaching brackets to enamel that had been etched with 35% phosphoric acid produced thick resin tags that were 10–20 μm long and comparatively deep in the enamel. Similar outcomes were found in this study, where etching with 37% phosphoric acid for 30 seconds shows penetration depth of 16.612 μm . This value is similar to the results reported by Retief¹³, Caio

G. Zanet¹⁴, dos Santos¹⁵. However, the resin penetration depth for phosphoric acid etching in a couple of investigations, including Arakawa¹⁶ (50 μm) and Julio P. Cal-Neto¹² (64.09 μm), was significantly more than in our investigation. Prism orientation and variations in phosphoric acid concentration and duration of etching could be the cause of the variation in tag length caused by phosphoric acid etching in these studies.

Crystal growth technique shows less etchant penetration than both conventional etching and SEP with the mean value of 7.038 μm . This is consistent with the findings of Maijer and Smith⁶, Jon Artun¹⁷ and Pizarro¹⁸. Most of the studies in the literature came to the conclusion that the polyacrylic acid etching cause minimal enamel penetration this study also came to the same conclusion. According to research by Bishara et al¹⁰ and dos Santos¹⁵, SEP had a shallower penetration depth than phosphoric acid. With a mean value of 8.096 μm , this study also confirms that SEP has a lower penetration depth than phosphoric acid ($p = 0.00$) (Table 3). With conservative enamel penetration it produced bond strength that was clinically acceptable, with lower rates of bond failure^{19,20}.

In 1967, the International Association for Dental, Oral and Craniofacial Research (IADR) subcommittee on standard techniques for dental materials recommended that the tensile test should be used in preference to other tests. Therefore, this study tested Tensile Bond strength instead of shear bond strength for evaluating the efficiency of three etchants. The results obtained from this study is similar to the other studies in literature. Phosphoric acid etching produces the highest tensile strength (11.043 MPa), suggesting it provides the strongest bonding followed by self-etching primer and crystal growth technique. The tensile bond strength obtained by polyacrylic acid is considerably lower than phosphoric acid etching indicating it is less effective in enhancing adhesion of orthodontic attachments. The mean tensile bond strength value obtained in this study is 4.54 MPa which is much lower than those reported by Maijer and Smith⁶ who suggested that this solution might generate bond strengths which were 80 percent of the those obtained with the phosphoric acid etching technique. According to Reynolds²¹ the minimum tensile strength required for clinical reliability is 4.2-5.6 MPa. The result obtained is low and very close to the critical level. The findings by Jon Artun¹⁷, Russell Bert Farquhar²², A. J. Maskeroni²³, and Knox and Jones²⁴ are in strong agreement with this study. Self-etching primer shows moderate tensile strength (8.942 MPa), but lower than phosphoric acid etching. Even though the bond strength obtained from SEP is lower compared to that from phosphoric acid etching, it met the clinically acceptable range of bond strength. The results reported by Jason C. Dorminey²⁵, Aljubouri²⁶, and Amit et al²⁷ are in strong agreement with this study. These findings seem to support previous research by Bishara³, who found that lower but clinically acceptable shear bond forces (mean, 7.1 ± 4.4 MPa) were created when orthodontic brackets were bonded to the enamel surface using self-etch primer. However, with fewer steps in the bonding process, clinicians can save time and lower the risk of procedural error and contamination. On the other hand, Ulrike²⁸ and Gladys²⁹ reported no statistical difference between conventional phosphoric acid etching and self etching primer indicating similar clinical efficiency between both adhesive systems.

In summary the phosphoric acid etching produces high bond strength but deeper enamel penetration. Even though it causes considerable loss of surface enamel, it is widely accepted as the etchant of choice in bonding purposes because of its superior efficiency. SEP can be considered as an alternative to phosphoric acid etching since it produces clinically acceptable bond strength with moderate enamel penetration. The only published information in the applicability of crystal growth technique in orthodontics is the work done by Maijer and Smith². Further studies on crystal growth technique shows inadequate bond strength values. This could be due to different methods of bond strength evaluation, concentration of the acid and sulphate ion used, the age of the conditioning solution which is now extremely difficult to obtain and which had to be freshly prepared in the laboratory or most likely due to lack of robustness of the crystal growth technique in general.

V.CONCLUSION

The conclusions drawn from the present study were:

- Scanning electron micrographs revealed that phosphoric acid etching cause mostly the type I etch pattern characterised by hollowing of prism centres with relatively intact peripheral regions. Etching with 20% sulphated polyacrylic acid shows crystal growth formation throughout the etched enamel surface. The etch pattern produced by self-etching primer is resembling to that of phosphoric acid etching, but it is more consistent, shallower, and less damaging to the enamel surface.
- Assessment of penetration depth shows that 37% phosphoric acid dissolves more enamel than polyacrylic acid and self-etching primer. It produces adequate bond strength by deeper penetration into the enamel. Crystal growth technique penetrates very minimal depth into the enamel but give weaker bond strength values to be used as an etchant. Self-etching primer produced moderate enamel

dissolution and have clinically acceptable bond strength values, so it can be considered as an alternative to conventional acid etching.

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