Original Research

Effect of Acid Etching on Surface Characteristics Of Enamel

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A R T I C L E   I N F O

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A B S T R A C T

Background: The objective was to perform a comparative analysis of effect of different etching time on surface roughness and surface loss of enamel.

Materials and method: The sample was composed of 40 teeth divided into four groups: Group I - Etching time 10 seconds, Group II - Etching time 20 seconds, Group III - Etching time 30 seconds and Group IV - Etching time 40 seconds. After etching for respective duration the teeth were dried and subjected to Profilometer and Profile projector to find change in surface roughness and surface loss of enamel respectively. The average value of surface roughness and surface loss was recorded for each tooth before and after etching at different time inetrval. The increase in surface roughness and surface loss caused by etching was calculated and compared between groups.

Results: The mean increase in surface roughness caused by etching was group I (0.331); group II (0.370); group III (0.606); group IV (0.573). There was a significant difference between the groups but on comparison between group III and group IV the difference was insignificant. Mean increase in surface loss caused by etching was group I (0.006); group II (0.012); group III (0.016); group IV (0.023). There was a significant increase in the surface loss of enamel between each group with an increase in the etching time.

Conclusion: The present study showed that etching of tooth for 30 seconds produced good surface roughness and optimal surface loss which is favourable for bonding the brackets.

INTRODUCTION

Buonocore[1] in 1955 introduced the acid etching technique to the field of dentistry by using phosphoric acid, which facilitated a strong adhesion between composite and enamel. Newman[2] later on in 1964 proposed this pre treatment technique for the bonding of orthodontic brackets. Hence the bonding of brackets is based on alteration of the enamel surface applying the standard protocol that is acid etching. Ideally an optimal orthodontic bonding system should integrate maximum bond strength with minimal damage to the enamel.[3]

Etching induces a selective demineralization that increases the free surface energy, porosity, and increase in surface area of the enamel due to roughening.[4] Enamel surfaces treated by phosphoric acid is transformed from a smooth surface into an irregular one. Micromechanical retention is promoted as the resin penetrates between rods and crystals of this microporous layer creating adhesion with the enamel.[5] Hydroxyapatite crystals are

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individually encapsulated by infiltrated resin creating microtags[6] and establishes the hybrid layer which promotes a nanoretention mechanism between the dental structure and the resinous material.[7] Although etching with phosphoric acid has the advantage of a high level of bracket bond strength,[8] a potential disadvantage of it is the demineralization of the most superficial layer, which renders the surface of enamel more prone to long-term acid attack leading to caries, especially around the orthodontic attachments.

The most routinely used acid is 37% *o*-phosphoric acid, for a period of 15 to 30 seconds, wherein the enamel loss is typically in the region of 8.8 to 16.4 microns.[9] However, wide variations in loss of enamel surface, from as little as 10 to 30 microns to as much as 170 microns, have been proclaimed.[10] Traditionally enamel etching time upto 60 seconds was considered optimum for bonding but several studies done thereafter showed etching time less than 30 seconds yields peak quality of the etched enamel.[11]

Thus the purpose of this study is to evaluate an optimal etching time with maximum surface roughness in order to minimize the enamel loss without compromising the bond strength.

The null hypothesis of this study is that different etching time using 37% phosphoric acid does produce same surface roughness of enamel and enamel loss.

MATERIAL AND METHODS

The present in-vitro study was done on extracted premolar teeth which were collected and stored in saline. The teeth were cleaned and then polished with pumice and prophylactic brush using a contra- angled micromotor handpiece for 10 seconds to remove any residual plaque or stains. Etching was done to evaluate the surface roughness along with enamel loss by varying etching time. The etchant used was 37% phosphoric acid gel (d-tech). The following calibrating tools were used for this study:

1. Profilometer (Mitutoyo-Japan, Model SJ 210)
2. Profile Projector (Sipcon Measuring Systems Model No. AVI-IMG-3D)

METHODOLOGY

1. Samples were randomly divided into 4 different groups each containing 10 teeth.
2. Teeth were embedded vertically in acrylic blocks upto the cemento-enamel junction so that only the crown portion was exposed with a 0.8 mm stainless steel wire, serving as a reference point. (figure A)
3. 37% phosphoric acid etchant was applied over the buccal surface of the tooth sample for different time intervals as per the groups assigned.
4. Group 1, 2, 3 & 4 were etched with etching gel for 10, 20, 30 & 40 seconds respectively.
5. The application area was rinsed thoroughly with distilled water for 5 seconds.
6. It was then air dried for 5 seconds by gently blowing with air syringe.
7. All the samples were tested for surface roughness using a profilometer before and after the etching. (figure B & C)
8. All the samples were examined under a profile projector before and after the etching to assess the tooth surface for enamel loss. (figure E) The distances were measured from the upper limit of the marking made by a permanent marker pen on the reference wire.

RESULTS

Surface Roughness and Depth

There was **statistically highly significant** (p<0.01)increase in the surface roughness (μm) from pre etching values to 10 seconds, 20 seconds , 30 seconds and 40 seconds of after etching values as seen in **Wilcoxon Signed Rank test (Table no.1).**

Table 1: Comparison of pre and post etching Surface roughness (μm) at different time intervals

	10 sec: Pre to Post Etching	20 sec: Pre to Post Etching	30 sec: Pre to Post Etching	40 sec: Pre to Post Etching
Z	-2.805	-2.803	-2.803	-2.803
P value	.005	.005	.005	.005

Wilcoxon Signed Ranks Test

There was **statistically very highly significant** (p=0.001) difference of surface roughness between 10 seconds and 20 seconds etching group and also between 20 and 30 seconds etching group while there was statistically no significant (p>0.05) difference of surface roughness between 30 seconds and 40 seconds etching group **Mann-Whitney U test (Table no. 2).**

Table 2: Comparison Surface Roughness (μm) after etching between different time intervals

(I) Group	(J) Group	Z	P value
10 sec	20 sec	-3.252	0.001
20 sec	30 sec	-3.780	<0.001
30 sec	40 sec	-0.227	.820

Mann-Whitney U test

Surface roughness (μm) changes after 10 seconds, 20 seconds , 30 seconds and 40 seconds after etching in each sample shows an increasing order upto 30 seconds thereafter decreasing or remaining constant at 40 seconds.

Mean surface roughness after 20 seconds of etching was greater by 0.042 μm compared to 10 seconds etching. After 30 seconds of etching surface roughness increased by 0.146 μm compared to that of 20 seconds of etching. Mean surface roughness after 40 seconds increased just by 0.004 μm than that of 30 seconds of etching.

The **depth of etch** can be rightly derived from the formula Ra (in microns) = $\frac{y1+y2+y3+\cdots yN}{N}$ where y1, y2, y3 ...yN are the ordinates measured on both sides of the mean line and N are the number of ordinates indicating that the depth of etch can be adequately measured. (figure D) The average depth of etch will be twice the average surface roughness measured thus indicating that etching depth is highest at 30 seconds as compared to the other groups.

Thus, it is concluded that enamel surface roughness is a function of time upto 30 seconds i.e. **surface roughness is directly proportional to the etching time upto 30 seconds following which the roughness practically either remains constant or decreases.**

Surface Enamel Loss

There was **statistically highly   significant** (p<0.01) surface loss (mm) from pre etching to 10 seconds, 20 seconds , 30 seconds and 40 seconds each of after etching as seen in **Wilcoxon Signed Rank test (Table no.3).**

Table 3: Comparison of pre and post etching Surface loss (mm) at different time intervals

	10 sec: Pre to Post Etching	20 sec: Pre to Post Etching	30 sec: Pre to Post Etching	40 sec: Pre to Post Etching
Z	-2.810	-2.818	-2.821	-2.807
P value	.005	.005	.005	.005

Wilcoxon Signed Ranks Test

There was **statistically very highly significant** (p<0.001) difference of surface loss between 10 seconds and 20 seconds etching group, between 20 seconds and 30 seconds group and also between 30 seconds and 40 seconds group. Thus showing increasing surface loss with the increasing duration of etching as derived by **Mann-Whitney U test (Table no. 4).**

Table 4: Comparison Surface loss (mm) after etching between different time intervals

(I) Group	(J) Group	Z	P value
10 sec	20 sec	-3.807	<0.001
20 sec	30 sec	-3.707	<0.001
30 sec	40 sec	-3.804	<0.001

Mann-Whitney U test

Mean Surface loss after 20 seconds of etching was greater by 0.006 mm i.e. 6 microns compared to 10 seconds etching. After 30 seconds of etching surface loss was more by 0.004 mm i.e. 4 microns compared to that of 20 seconds of etching. Mean surface loss after 40 seconds was more by 0.007 mm i.e. 7 microns than that of 30 seconds of etching. There was increasing mean surface loss with increase of duration.

Concluding that **the surface enamel loss is directly proportional to the etching time i.e. when etching time is increased there is increase in enamel loss.**
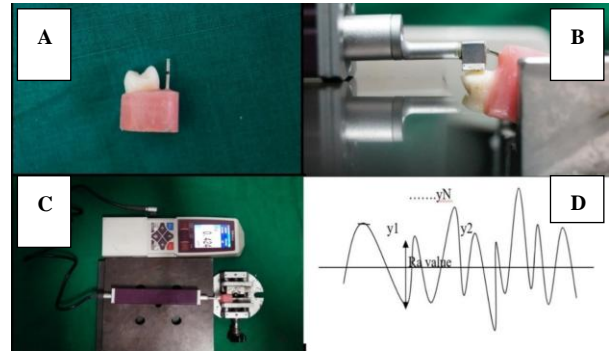
FigureA -Tooth Embedded in Self Cure Acrylic Resin with a Reference wire

Figure B - Profilometer

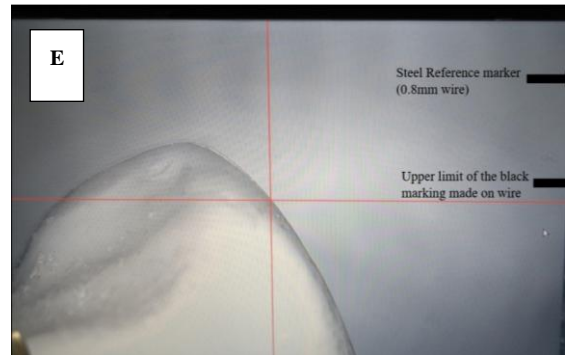Figure C – Readings of Profilometer shown on the digital recording machine

Figure E – Tooth Projection displayed on a Screen attached to a Profile Projector

DISCUSSION

The acid etch technique for bonding orthodontic brackets has revolutionized the field of orthodontics and has brought about a paramount change in approach to clinical practice . The enamel etching time is crucial in providing clinical success in adhesive dentistry which have been reduced from 60 seconds time using 30–40% of phosphoric acid to an etching time as brief as 15 seconds. Minimizing the etching time imparts several advantages as it saves chairside time without compromising the adhesive performance, also because acid etching causes superficial tissue loss, it is desirable that minimal tooth structure be dissolved; therefore, optimal acid-application time is advocated.[13] In vitro studies have demonstrated that a 15-seconds of acid etching time is also adequate for orthodontic adhesive procedures.[14,15] However, there are alternate reports that

suggest an optimum application time of 30 seconds for 37% phosphoric acid solutions.[16] The acid etching causes washout of inorganic minerals from the surface of the tooth and hence the amount of time the tooth structure is exposed to acid can affect the underlying physical and mechanical properties of the surface as well. Because of the uncertainties in these variables, it is important to test the application time of commercial phosphoric acid solutions in vitro which will provide adequate roughness of the enamel along with least enamel loss. Accordingly, based on the previous studies, different time intervals for etching in the range of 10–40 seconds has been considered in the study. And this is one of the main variables in our study as surface loss is directly proportional to it whereas surface roughness is also directly proportional but till 30 sec after which the roughness practically remains constant even after increasing the time.

An important variable in etching of enamel is the tissue to be etched itself, i.e. the subtle differences in enamel characteristics influence the ability of an acid conditioner to properly demineralise it. Because of higher inorganic content, the intact enamel surface presents some unique features. The young permanent teeth have an aprismatic layer of approximately 30 μm that covers the entire crown.[4] This layer is lost with time; however, the hard tissue of the teeth becomes more mineralized when exposed to the oral cavity in patients with equilibrium in the demineralization process. This causes the surface layer of enamel to present hypermineralization features when compared with the inner enamel.[17] These two differences can influence the features of the etching pattern and result in less homogeneous etching patterns,[12] compromising the quality of bonding. It was also found that prismless zone of enamel was frequently seen at the cervical third of the permanent teeth.[18]

The crystal orientation in prismless enamel is exclusive in a way that it is oriented perpendicular to the surface enamel as compared to the three dimensional spread in the prismatic enamel. The unidirectional orientation of crystals and their relatively dense arrangement in prismless enamel causes a relatively uniform dissolution and creation of limited random porosity. Subsequently, limited resin penetration, manifested by short resinous tags. Therefore, prismatic enamel allows a greater penetration and mechanical interlocking of resins than does the prismless type.[19] In our study, the surface roughness seems to be increasing with etching duration upto the extent of 30 seconds of etching thereafter the roughness remains constant.

When phosphoric acid is applied to the dental enamel surface, it dissolves loosely attached layers or organic films. The outer 5 to 50 micron ends of the enamel prisms are selectively dissolved as well.[5] As a result, microscopic pores and enamel crystallites are exposed, resulting in a retentive surface for the primer.[1] Etching causes several changes of the enamel surface namely (a) a tremendous increase in surface area due to the etching action; (b) the exposure of the organic framework of enamel which serves as a network, in and about which the resin can adhere; (c) a new surface formation due to precipitation of new substance, for instance, calcium oxalate, organic tungstate complex, and so on, to which the resin might adhere provided the etchant contains these ions; (d) the removal of old, fully reacted, and inert enamel surface, exposing a fresh, reactive surface which is more favorable for adhesion; and (e) the presence of an adsorbed layer of highly polar phosphate groups on the enamel surface, derived from the acid used.[1] It is assumed that the increase in the number of pores with an increase in the surface irregularities develops a larger

exposed area of enamel, which creates a greater area for bonding and higher subsequent bond strengths.[17]

Eventually, too much of exposure to acid will result in enamel crystallites to be gradually dissolved and broken down until the enamel structure is destroyed. It is considered that the once-opened enamel pores will slowly become obliterated by collapsing enamel structures, which serves as an explanation for decreased enamel roughness at 40 seconds in our study. The microporous enamel surface thus created by etching with phosphoric acid plays a vital role in the bond strength of restorative dental materials for primary and permanent teeth. Hence it is necessary for the etchant to be effective enough to create adequate surface roughness with minimal loss of enamel layer and without undue weakening of the enamel prisms.

1. Considering these facts, an attempt has been made in our study to determine the optimal etching time so that there is minimum damage of the enamel. In our study it is seen that enamel surface loss is a function of etching time i.e. with increase in the period of etching statistically significant enamel loss is seen. To date, most of the studies examining the surface morphology of etched enamel were limited to qualitative evaluations with Scanning electron microscopy (SEM). Microscopic analysis provides only visualization of surface morphology, entailing the use of further methods to determine the extent of surface modifications quantitatively. Microscopic techniques lack a quantitative scale and hence cannot be used for the comparative evaluation of surface roughness of the treated tooth surfaces.[20] The use of descriptive terms such as "surface roughness" creates the need for a proper quantitative tool for accurate estimation of such roughness.

2. A tool especially engineered for this kind of evaluation is a "Profilometer" or a "Surface Roughness Tester". Profilometric assessment of a surface allows an objective determination, and thus profilometry was the main testing instrument used in their study.[20] Therefore, surface profilometry is used in this study to obtain quantitative data of the etched enamel surfaces which can be easily compared at different time intervals using 37% phosphoric acid.

Profilometry is a widely accepted method to examine the surface characteristics. It is a common method to determine surface configuration which includes a non-invasive approach. Besides, in this system the overall roughness is specified by a metric average value, which allows for a statistical evaluation.

A profile projector is an optical measuring tool which magnifies a sample's surface aspect to allow measurement on a linear/circular scale. A profile projector is also referred to as an optical comparator, or also known as a shadowgraph. A profile projector projects a magnified profile image of an area or feature of a work piece onto a screen most commonly using diascopic illumination. Dimensions can be computed directly on the screen or compared to a standard reference at the correct magnification. For precision, it is important that the magnification does not change with perspective, i.e. its position or the view point of the operator. Telecentric lenses are, therefore, highly preferrable. The screen often has a grid which can often be rotated through 360 degrees to align with an edge as presented on the screen. Point positions, measurements, and calculations may also be brought about by using a simple digital read out device. A computer may be added to a profile projector system for edge detection, thereupon eliminating some human errors. Because of the directness and the high degree of reproducibility of

the measurement technique i.e. the Profile Projector used in this study, it was thought to be more accurate than the others mentioned. Hence, considering the several advantages of this direct measurement technique it has been used in our study for evaluating the enamel loss using a Profile Projector or a Visual Inspection System, Sipcon Measuring Systems, India.

As, surface roughness of enamel remains the same at 30 and 40 seconds but the surface loss of enamel keeps increasing with increase in time . So 30 seconds is considered to be the ideal time for etching in this study as it produces maximum surface roughness and optimal loss of surface enamel loss. Hence, the null hypothesis of this study that different etching time using 37% phosphoric acid produces same surface roughness of enamel and enamel loss stands rejected.

CONCLUSION

It is certainly desirable to dissolve only the minimum possible amount of enamel from the tooth surface, and hence only the minimum etching time consistent with obtaining optimum surface roughness should be used. As concluded in our study 30 seconds serves as the optimal etching time where highest surface roughness is observed besides minimal possible enamel loss.

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