ORIGINAL RESEARCH

Comparative Assessment of pH and Degree of Surface Roughness of Enamel When Etched with Five Commercially Available Etchants: An *In Vitro* Study

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Abstract

Aim: To evaluate the pH and degree of surface roughness caused by five commercially and readily available etchants on tooth enamel.

Materials and methods: Five different etchants were chosen. An electric pH meter was utilized to test the pH of the etchants employed. Fifteen maxillary bicuspids that had been extracted were cleansed and stored in thymol solution. The samples were sorted into five groups of three each. A noncontact profilometer was employed to assess the microsurface changes of the pre-etched enamel. The teeth were then etched for 30 seconds with respect to the group to which they belonged before being cleaned and dried. The surface roughness after etching was analyzed, measured and values were tabulated. Descriptive statistics and paired *t*-test were done.

Results: The pH of the etchants and surface roughness of the enamel are varied across the five groups, though they have the same composition of 37% orthophosphoric acid. Etchant from Group C was found to be most acidic while the one manufactured by Group E was least acidic. Ivoclar, DPI, and DTECH showed a statistically significant value in surface roughness parameter post-etching ($p < 0.05$). A statistical difference that was significant was observed with the Kruskal–Wallis test for surface roughness parameter ($p < 0.05$).

Conclusion: All five etchants had varied pH and the amount of surface roughness was also varied though the composition was the same. Further elemental analysis of these etchants has to be done to validate the results obtained.

Clinical significance: Etchants of the same composition should ideally produce the same effect on the tooth enamel surface, but etchants from different manufacturers produce different levels of surface roughness which could be due to differences in the composition of the prepared etchant. The study was conducted to assist in making an educated selection about the most cost-effective but efficient etchant for clinical application.

Keywords: Acid etching, Orthophosphoric acid, Surface roughness.

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Introduction

Resins and resin types of cement have been used in the field of dentistry since their advent in the 1950s,[1] with acid etching of tooth enamel surface being an important step in bonding. Enamel conditioning or acid etching creates microporosities on the tooth surface that increase the enamel surface energy.[2] Acid etching induces a selective demineralization, which raises the density of the material, enamel porosity, free surface energy, and provides an increased surface for bonding of resins.[3] The ability of the resin to infiltrate between the rods and crystals is responsible for its adhesion to enamel,[4] resulting in micromechanical retention. These resins encapsulate hydroxyapatite crystals individually forming micro tags and hence a hybrid layer,[5] that promotes a nano retention mechanism between the enamel and resin.[6,7] The formed Micro tags effectively contribute to the adhesion of the resin to dental enamel.[8]

The chemical composition of the enamel, the form, pH, concentration of acids, and the etching time all influence the retention characteristics of the etched enamel surface.[7–10] Many studies have been published comparing etching patterns by different etching materials at varying etching times.[11] Literature also comparing etching patterns with self-etch primers and conventional two-step etching process has also been well documented.[11–15] When the enamel was surface treated, the bond strength of orthodontic attachments was examined with 35–37% orthophosphoric acid for varied time intervals of 15–90 seconds thoroughly in the past.[16–18] Most studies to date have focused on evaluating the different etching patterns, the influence of etchant, and etching time on bond strength.[19–21] However, no research has been conducted to date to analyze and compare the pH and surface roughness of commercially available and widely used etching methods. Prime manufactured by Prime Dental Products (Thane, Maharashtra), ANABOND manufactured by Anabond Stedman Pharma Research (Chennai, Tamil Nadu), D-tech manufactured by D-tech Orthodontics (Pune, Maharashtra), and DPI manufactured by DPI (Mumbai, Maharashtra) are some of the common and economical options of etchants available in India. Ivoclar manufactured by Ivoclar Vivadent

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(United States of America) is a comparatively less economical and less commonly used etchant.

Surface roughness is defined as imperfections, or tiny projections and indentations, on a surface that affect wetting, adhesion quality, and other properties. Despite the fact that micromechanical roughness is required for effective enamel adherence,[22,23] the precise etched enamel properties involved, as well as the conditions under which metrical scale adhesion occurs, are unclear. Surface roughness has an unidentified effect on adhesion.[23] The polymer bonding will be stronger if the enamel is roughened and intimate contact is made between the adhesive and the adherent.[24,25] Following the introduction of enamel surface treatment using acids to promote resin adherence to enamel, there has been a substantial amount of research into dental materials to improve bond strength,[24,25] but none into the etched enamel surface topography.[26] The surface roughness of human dental enamel is an essential characteristic that has yet to be extensively studied.[26] In this study the aim was to determine the pH of five commercially available etchants of the same composition and their effect on the enamel by measuring the degree of roughness caused by each and correlating if the pH of the material has an impact on the surface roughness of enamel produced post-etching.

Materials and Methods

The current *in vitro* study was conducted at Saveetha Dental College and Hospitals, Saveetha University, Chennai, during the months of September 2021 to December 2021. With reference to the Bhandari et al.[2] study, the required sample was calculated using the G power software at a power of $P = 95$. A total of 15 samples with 3 in each group of etchants was used. The study was conducted by two investigators and only one of the investigators was responsible for sample collection and preparation of the samples and conduction of the study.

Determination of pH of the Etchants

Five etchants were included in this study. Each was designated as a group for further reference in the study.

Group A: 37% orthophosphoric acid by DPI (DPI, Mumbai, Maharashtra),
Group B: 37% orthophosphoric acid by DTECH (D-tech Orthodontics, Pune, Maharashtra),
Group C: 37% orthophosphoric acid by Prime (Prime Dental Products, Thane, Maharashtra),
Group D: 37% orthophosphoric acid by ANABOND (Anabond Stedman Pharma Research, Chennai, Tamil Nadu),
Group E: 37% orthophosphoric acid by Ivoclar (Ivoclar Vivadent, United States of America).

The five etchants chosen in this study were first subjected to pH analysis. An electronic pH meter (ELICO pH Meter Table Top, Model No: LI120) was chosen for this purpose.

Each of the five etchants chosen in this study was squeezed out into smaller holders, into which the pH meter tip was inserted to determine the pH of the etchant. The values were noted.

Sample Preparation

This study employed 15 human maxillary bicuspids that had been extracted for orthodontic or other dental treatment procedures. Visual examination was done to assess the quality of the tooth before including the sample. Teeth without decay, fracture, tooth wear, fluorosis were included in this study. To avoid bacterial development and dehydration during the storage of samples, the extracted teeth were rinsed and cleansed under running water and kept in a 0.1% Thymol solution. Just before the use of the sample for the study, the teeth were polished with pumice slurry, cleaned, washed, and air-dried. This was done to remove any residual thymol solution (Fig. 1).

After that, the teeth were sorted into five groups (Fig. 2), each with three samples ($n = 15$).

In each group, for 20 seconds, the tooth surface was treated with the respective etchant, washed well, rinsed, and air-dried until the enamel surface had a white frosty look.

Determination of Surface Roughness

The pretreatment surface changes in terms of microsurface changes of the enamel, Ra (μm), were recorded with a noncontact stylus profilometer after the teeth had been prepped and polished (Fig. 3). These values were noted as pretreatment readings. Depending on which group the teeth belonged to, the enamel tooth surface was etched. All teeth were treated and etched for 20–30 seconds before being washed with running water and air-dried. The surface roughness was noted posttreatment using the same methodology

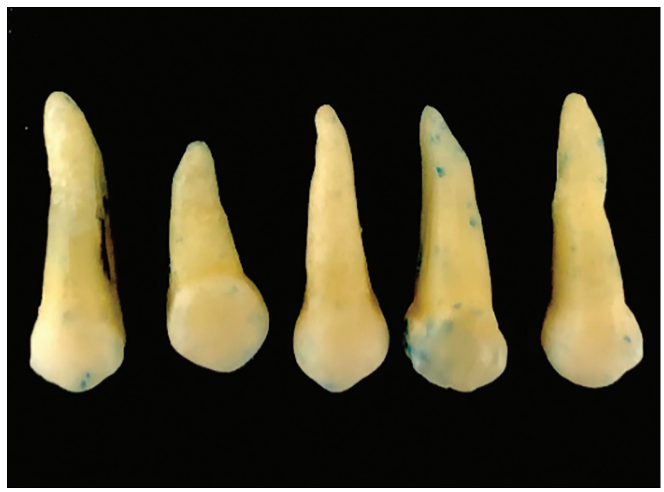

Fig. 1: Prepared premolar teeth surfaces for etching using 37% orthophosphoric acid

Fig. 2: Five commercially available etchants used. From left to right, ANABOND, DPI, PRIME, DTECH, and Ivoclar

as used for pretreatment measurements. The values were tabulated and an average Ra (μm) was obtained for each group.

Statistics Analysis

For evaluating the data using statistics, the data collected for each group was entered in Microsoft Excel and transferred into SPSS software (23.0). Each group's before and post data were compared using descriptive statistics and paired *t*-tests (Table 1). The Kruskal-Wallis test was employed to assess differences between and within groups.

Results

pH Determination

The pH of the five included studies has been graphically demonstrated in Figure 4. From the analysis done using an electronic pH meter, it could be inferred that the pH of all etchants included in this study was acidic in nature. Group C etchant was more acidic (0.2) when compared to the other etchants (1.5–2.5) included in this study.

Surface Roughness Evaluation

Table 1 summarizes the descriptive data (mean SD) of enamel surface roughness before and after etching. The average surface roughness observed before and after etching with group A was 0.19 ± 0.005 μm and 0.17 ± 0.005 μm, respectively, with group B was 0.20 ± 0.005 μm and 0.18 ± 0.005 μm, respectively, with group C was 0.20 ± 0.10 μm and 0.17 ± 0.005 μm, respectively, with group D was 0.18 ± 0.015 μm and 0.18 ± 0.00 μm respectively, and with the group E was 0.18 ± 0.005 μm and 0.19 ± 0.005 μm, respectively. It can be observed that all etchants produce a similar amount of surface change of the enamel post-etching.

On statistical analysis using a paired sample *t*-test, a statistically significant difference with $p < 0.05$ was observed for groups A, B, and E etchants with reference to surface changes/roughness observed post-etching of enamel surface. Table 1 depicts the values of paired sample *t*-test.

Kruskal-Wallis test was done to observe differences between the five groups of etchants involved in this study. On statistical analysis, it was observed that a statistically significant difference ($p < 0.041$) was observed across the five groups post-etching of enamel surface. Table 2 displays the results of the Kruskal-Wallis test.

With reference to results obtained with statistical analysis, it could be inferred that Group C etchant was the most acidic, whereas etchant from Group E was shown to be the least acidic. Surface roughness after etching was statistically significant between Groups A, B, and E ($p < 0.05$). A statistically significant difference was observed with the Kruskal-Wallis test for surface roughness parameter ($p < 0.05$).

Fig. 3: Noncontact stylus profilometer used for assessing surface roughness

Discussion

In the present study, the pH and the effect of five commercially and most commonly available etchants on the surface roughness of enamel post surface conditioning were assessed using a noncontact profilometer. Our results showed that the pH of the etchant by group C (Prime) was more acidic when compared to the remaining four groups. The pH of group C was close to 0.2. Groups A and B had a similar pH of around 1.55 and 1.63, respectively, whereas groups D and E had a pH of about 2.16 and 2.5, respectively. It has been studied previously that acids used for etching of enamel surfaces produce microporosities by the dissolution of inorganic structures that facilitate the penetration of monomer/resin to create resin tags.[1] Highly acidic nature of the etchant can result in increased dissolution and demineralization of organic compounds of enamel leading to the collapse of the prism matrix of the enamel structure that could interfere with the bonding of resins.[1] The differences in the pH of any material could be attributed to its composition. Any product or material is made up of additional agents other than the

Table 1: Descriptive statistics of surface roughness pre- and post-etching

Etchant	*Pre and Post*	*Mean ± standard deviation [surface roughness, Ra (μm)]*	*Sig (two-tailed) p value Paired sample t test*
DPI (Group A)	Pre	0.19 ± 0.005	0.038
	Post	0.17 ± 0.005	
DTECH (Group B)	Pre	0.20 ± 0.005	0.020
	Post	0.18 ± 0.005	
Prime (Group C)	Pre	0.20 ± 0.10	0.118
	Post	0.17 ± 0.005	
ANABOND (Group D)	Pre	0.18 ± 0.015	0.529
	Post	0.18 ± 0.00	
Ivoclar (Group E)	Pre	0.18 ± 0.005	0.042
	Post	0.19 ± 0.005	

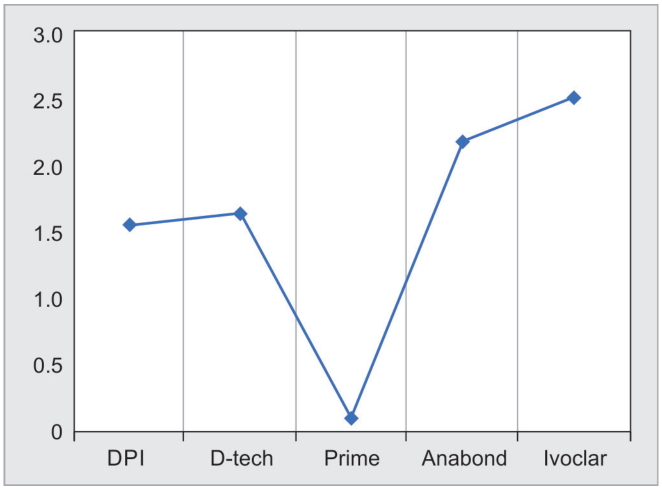

Fig. 4: Graphical representation of pH of all five etchants included

Table 2: Kruskal-Wallis test

	Post-treatment
Chi-square	9.953
df	4
Asymp. sig.	0.041

key component. Etchants constitute preservatives, coloring agents, and viscosity enhancers that could alter the overall effect of the 37% orthophosphoric acid used. This could be one such reason for the difference in the pH observed across the involved groups. In order to substantiate this result, an elemental analysis studying and evaluating the constituents of each etchant should be carried out.

With reference to surface roughness, groups A, B, and E had a significant change in enamel surface microstructure after etching with 37% orthophosphoric acid ($p < 0.05$). This indicated that changes in surface structure post-etching exist that enable efficient resin bonding.

Kruskal-Wallis test was done to correlate differences across the five groups. Due to a decreased sample size, this statistical test was employed. A statistically significant difference was observed in roughness ($p < 0.05$) across the five groups.

To our knowledge, no previous study had correlated the relation between the pH and the degree of surface roughness of enamel caused by the etchants. From this study, it can be observed that a pH too low or too high might nullify the effect of the etchant as it collapses the prism matrix. A pH around 1.5 allows enough acidic environment for demineralization of the tooth enamel hydroxyapatite crystals allowing just enough surface changes to form resin tags and allow better retention of the resin.

Another important point to be noted is that though all the chosen etchants have the same amount of phosphoric acid i.e., 37%, their pH is varied. This might be attributable to changes in the etchant's composition. Electronic pH meters provided various advantages over the conventional methods of pH determination. The advantages include that it is a fast and simple process, the pH meter is portable and in comparison with color strip or pH indicator the values are more accurate.[27] But this also poses disadvantages such as calibration of the pH meter before use which can be influenced by temperature and carbon dioxide absorption, deposits on electrode membrane which could interfere with the readings obtained. In order to confirm the obtained results, further investigations should be done to evaluate the etchant and its impact on the pH and consequently the surface roughness of enamel.[27]

Post-etching, the surface roughness in this study was evaluated using a noncontact profilometer. It should be noted that this method is a conventional method of assessment of surface roughness but it also poses some limitations. Firstly, the profilometer has limited lateral resolution due to the radius of the stylus tip and it usually cannot measure the surface changes rapidly.[28] Hence alternate standardized methods to evaluate the surface changes needs to be employed. Scanning electron microscopy could be best used to evaluate the characteristic of the enamel surface post-etching. This helps evaluate structures at nano and micro levels and also helps determine the etching pattern of enamel. Hence these updated methods need to be employed to substantiate the results.

The main objective of this study was to evaluate the difference in enamel surface microstructure post-etching using five etchants from different manufacturers having the same composition of 37% orthophosphoric acid. The limitation of this study is decreased sample size across all five groups and hence a study with more samples should be performed. An elemental analysis should be done to evaluate for differences in the composition of etchant that could influence the pH of the sample. Also, better methods of assessing the pH of material should be employed. An scanning electron microscope (SEM) analysis should also be done post-etching to assess if the type of etching pattern obtained also influences the bond strength of the resin. Also, a shear bond strength assessment should be done to justify the efficiency of the etchant used for the preparation of microporosities.

Conclusion and Clinical Significance

The pH of the etchants and surface roughness of the enamel are varied across the five groups, though they have the same composition of 37% orthophosphoric acid. Etchant from Prime (Group C) was found to be most acidic while the one manufactured by Ivoclar (Group E) was least acidic. Groups A, B, and E showed a significant difference in surface roughness post-etching $p < 0.05$. The Kruskal-Wallis test showed a difference in surface roughness ($p < 0.05$). Further studies should be done to assist in making an educated selection about the most cost-effective but efficient etchant for clinical application.

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