

Compare the Effects of Different Etching Times on the Etching Pattern, Etching Depth, and Bond Strength in Permanent Teeth Using 37 % Phosphoric Acid and Enlight Light Cure Bonding Material

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Abstract

Background: Present study was done to compare the effects of different etching times on the etching pattern, etching depth, and bond strength in permanent teeth using 37 % phosphoric acid and Enlight light cure bonding material.

Methodology: The study was done on total sample of 115 extracted premolar teeth. Samples were divided into five groups according to different etching time of 15, 30, 60, 90, 120 seconds. These were further subdivided into three subgroups for etch pattern, etch depth, and bond strength measurement.

Result: Showed that highest mean etch depth was recorded in Group E (120 seconds etching) of $15.88 \pm 2.58 \mu\text{m}$ and least etch depth in Group A (15 seconds etching)

of $8.87 \pm 2.25 \mu\text{m}$. Highest mean shear bond strength was recorded for Group C (60 seconds etching) of $17.06 \pm 3.80 \text{Mpa}$ and least was observed in Group E (120 seconds etching) of $9.78 \pm 3.7 \text{Mpa}$.

Conclusion: Above findings strongly suggest that 15 seconds etching and 30 seconds etching time should be the most preferred procedures to obtain clinically acceptable mean shear bond strength with minimum loss of enamel mineral. We strongly recommend these approaches.

Keywords: Etching time, Etching pattern, Etching depth, Bond strength, 37 % phosphoric acid, Enlight light cure.

Introduction

The 1955 invention of the acid etching process by Buonocore¹ has had a significant impact on dentistry and

orthodontics, revolutionizing the field by streamlining restorative, aesthetic, and orthodontic bonding treatments and increasing their effectiveness by directly attaching to tooth enamel.²

It is a recognized clinical practice to bind orthodontic attachments to acid-etched enamel. The acid that is most frequently used to etch before bonding is phosphoric acid. The application of phosphoric acid to enamel surfaces, as demonstrated by Buonocore¹ in 1955, results in a porous surface layer that aids the interlocking of composite resin and enamel when it is penetrated by a low-viscosity resin-bonding agent.

Phosphoric acid was normally best used in enamel surfaces that were retentive at concentrations of 30–40%.

⁽³⁾ A monocalcium phosphate monohydrate was formed at concentrations higher than 50%, which prevented further dissolving. At concentrations as low as 10%, the phosphoric acid solution had no negative impacts on bond strength^{4,5} but it did lessen surface damage to enamel by lowering the amount of mineral component breakdown and depth of penetration.

Numerous investigations have been conducted subsequently to standardise the acid concentrations^{6,7,8} as well as the period of administration.^{9,10} The suggested duration for 85% phosphoric acid enamel etching was 30 seconds when it was first introduced in 1955. One When it was originally used in a clinical setting in the 1960s, it was then expanded to 60 seconds^(11, 12) The application was condensed to a 30-second form in the 1980s, and it is still accepted today.^{13,14,15} Since complete bonding saves time without sacrificing the strength of the adhesive connection, the majority of adhesive system makers have advised 15 seconds.

According to Conniff and Hamby¹⁶ and Silverstone^{17,18} proper etching patterns on permanent enamel required an etching period of 120 s. Legler et al.¹⁹ used varying

phosphoric acid concentrations (37%, 15%, and 5%) and etching times (60, 30, and 15 seconds) to assess the depth of etch. Enamel bond strength properties, types of enamel conditioners, and acid concentrations between enamel and the orthodontic bracket depend on a number of variables, such as the actual etching time, adhesive composition, bracket base design, bracket material, oral environment, and clinician skill. A common cause of bond failure during the bonding process is moisture contamination. Regardless, even though the oral cavity's unfavourable damp environment is where the polymer adhesive system functions, sufficient bond strength is still required.

Tests measuring shear bond strength have been performed to compare the bond strengths of various materials, including composite, enamel, and dentin.^{20,21, 22} Insufficient bonding and larger spaces between the restoration and the tooth are linked to low shear bond strength.²³ It is crucial to keep in mind that elevated bond strength levels carry some risk, as they could result in cracks in the enamel during the debonding process.^{24,25,26}

Retief et al.²⁷ outlined the several elements that contribute to the ideal binding strength. He demonstrated that binding strengths as low as 138 kg/cm² (13.53 MPa) can result in enamel fractures. Smukta et al.²⁸ discovered no appreciable variations in the forces needed to remove the sealant from permanent enamel after 60, 120, and 240 seconds of acid application. It was thought that it would be beneficial to ascertain the shortest duration required to etch permanent enamel while yet preserving good sealant retention in light of these contradictory findings. The current study used 37% phosphoric acid and Enlight light cure bonding material to investigate the effects of various etching times on the etching pattern, etching depth, and bond strength in permanent teeth.

Material and methodology

The current study was conducted in Bhopal, Madhya Pradesh, India, at MANIT College. In all, 115 removed premolar teeth were utilised in this study. Prior to test methods, they were stored in a thymol (0.1%) solution after being carefully cleaned with ordinary water, as described by Galil and Wright²⁹, Redford et al.³⁰ and Cal-Neto and Miguel.³¹ Weekly changes were made to the solution to stop the growth of bacteria. Teeth free of discoloration, abrasions, cavities, and microscopic fissures were chosen for the investigation. After that, the chosen teeth were placed in identical 20 by 23 mm acrylic blocks, as instructed by Attilio MD et al.³²

Extracted premolar teeth were equally divided in five groups of 23 teeth each, according to different etching time Group A 15 seconds etching, Group B 30 seconds etching, Group C 60 seconds etching, Group D 90 seconds etching, Group D 120 seconds etching. These main groups were further subdivided into three subgroups having (a.) 5 sample size for etch pattern, (b.) 3 sample size for etch depth, and (c.) 15 sample size for bond strength respectively. The teeth were submerged in the color coded rectangular bases of cold cure acrylic with full crown exposed to facilitate conduction of study. The etchant used was 37% orthophosphoric acid (H_3PO_4) which was applied using a 00 camel's hair brush. Each window were covered completely with 1 ml of acid and agitated continuously for the specified time. The surfaces then were rinsed with distilled H_2O for 15 sec and air dried for 30 sec. The sealant that was used was Enlight light cure material with standard light cure procedure. All procedures of etching, bonding were done by a single operator (GC) to prevent inter operator error.

Testing of etch pattern: Twenty-Five teeth were etched and were viewed with SEM at 1,800 X magnification to examine etch patterns after different etching times. The

buccal surfaces were then etched using 37% phosphoric acid for different etching period according to five groups washed with distilled water and dried with chip blower. The samples were then evaluated by a single operator to avoid inter operator error at 1,800 X magnification under Scanning Electron Microscope (Quanta 200 EDS, FEI, Netherlands).

Testing of etch depth: Scanning Electron Microscope has been used by many investigators^{29,30,31} to evaluate etch depth penetrating the enamel after etching with 37% phosphoric acid. The prepared specimen are demineralized in 10 - 40% hydrochloric acid and then mounted on aluminium stubs, sputter coated with gold and evaluated under SEM for depth of craters penetrating enamel after etching.

Testing of shear bond strength: The blocks were secured in the jaws of Instron machine model 3382 (manufactured by Instron India, Chennai, Tamil Naidu, India). The jig was placed parallel to the buccal surface of tooth. The jig was moved for debonding at a cross head speed of 1 mm/ minute. The bond strength was measured as a force reading at which the brackets were debonded.

Statistical Analysis: The data was entered into the excel sheet. The data was analyzed using SPSS (Statistical Package for Social Sciences) 25.0 version, IBM, Chicago. Mean values and SD was calculated. The ANOVA test was used for comparison. P value ≤ 0.05 will be considered statistically significant. Confidence interval will be set at 95%.

Result

Etching pattern: The etching pattern was assessed by seeing etched enamel surface at 1,800 X magnification under Scanning Electron Microscope. Subgroup A1 specimen Presented slightly roughened enamel surface with interspersed shallow depression, and pitting. We

term this as Type 6 etching pattern. The subgroup B1 specimen Presented more roughened surface with loss of superficial enamel having deeper depressions, and small circular voids uniformly distributed throughout the etched area. We term this as Type 7 etching pattern, subgroup C1 specimen Presented uniformly more roughened surface with somewhat linear raised ridges with interspersed enlarged pitting, uniformly distributed throughout the etched area. We term this as Type 8 etching pattern. The subgroup D1 specimen Presented prominent curvi-linear ridges with crater like depressions of the prisms and interspersed circular or voids uniformly distributed throughout the etched area. We term this as Type 9 etching pattern. The subgroup E1 Presented ill-defined ridges, crater like depressions of the prisms, more enlarged round voids uniformly distributed somehow same as Group D but with loss of sharpness probably due to more loss of mineral content. We term this as Type 10 etching pattern. (Figure 1)

Etch depth: Table I Presents mean etch depth of five groups according to different etching time along with range of values observed and standard deviation for each group required for statistical differences Group E (120 seconds etching) had highest etch depth of $15.88 \pm 2.58 \mu\text{m}$ which was followed by Group D (90 seconds etching) with $14.16 \pm 2.17 \mu\text{m}$. Group A (15 seconds etching) had least etch depth of $8.87 \pm 2.25 \mu\text{m}$. Means were significantly different in all five groups according to F value, P value and E value ($P=2.211$, E-11).

Table II showed that Mean difference between group A and B was $1.18 \mu\text{m}$, which was not statistically significant ($P=0.150$), Mean difference between Group A and C was $3.56 \mu\text{m}$, which was not statistically significant ($P=3.465$), Mean difference between group A and D was $5.29 \mu\text{m}$, which was not statistically significant ($P=2.2719$). Mean difference between group A and E was

$6.29 \mu\text{m}$, which was not statistically significant ($P=1.321$), Mean difference between group Band C was $3.64 \mu\text{m}$, which was statistically significant ($P=0.004$), Mean difference between group B and E was $4.11 \mu\text{m}$, which was not statistically significant ($P=1.79$), Mean difference between group C and D was $1.73 \mu\text{m}$, which was statistically significant ($P=0.05$). Mean difference between group C and E was $3.45 \mu\text{m}$, which was statistically significant ($P=0.001$), Mean difference between group D and E was $1.72 \mu\text{m}$, which was not statistically significantly ($P=0.07887$).

Shear bond strength: Table III Presents mean of shear bond strength of five groups according to different etching time along with range of values observed and standard deviation for each group required for statistical inferences. Group C (60 seconds etching) had highest shear bond strength i.e $17.06 \pm 3.80 \text{ Mpa}$ which was followed by Group D (90 seconds etching) having $15.91 \pm 5.16 \text{ Mpa}$. Group E (120 seconds etching) had least shear bond strength which was $9.78 \pm 3.7 \text{ Mpa}$. Means were significantly different in all five groups ($P=9.48 \times 10^{-6}$).

Discussion

This work compared how various etching times affected the permanent teeth's etching pattern, etching depth, depth of resin tag, and bond strength using 37% phosphoric acid and Enlight light cure bonding material. Three different etching pattern types were identified and categorised by Silverstone et al.¹⁸ Prism cores of enamel were removed preferentially from Type 1. The second design, known as Type 2, involved removing the prisms' periphery while leaving the prism cores largely intact. Areas in Type 3 matched those in Types 1 and 2. In addition to the types 1, 2, and 3 that Silverstone et al.¹⁸ identified, Galil KA and Wright GZ⁽²⁹⁾ described types 4 and 5.

The following five etch patterns were examined under a SEM at a magnification of 1,800 X in the current investigation. These five etch patterns are indicated by the numbers 6, 7, 8, 9, and 10 above and beyond the three that Silverstone et al²¹ and Galil and Wright have discussed. (36) The reason behind this is that our etch designs were not previously disclosed. Each of them generated a surface that was clinically acceptable in terms of bond strength and adequately retentive. As a result, they ought to be categorized with etch patterns.

The results of this investigation strongly imply that the best practices for achieving a mean shear bond strength that is clinically appropriate while minimizing enamel mineral loss are 15 seconds of etching and 30 seconds of etching time. Johnston CD et al.³³ discovered that there was a considerable increase in the likelihood of bond survival at the adhesive contact between the resin and the buccal enamel of the molars when the etching period was extended from 15 to 30 seconds. The probability of bond survival improved with an additional 60 seconds of etching time, although not to a statistically significant degree. In our investigation, etching for 60 seconds resulted in a noticeably stronger connection, which was followed by etching for 30 and then 15 seconds, respectively.

The results of Legler LR et al¹⁹ calculations showed that the range of etch depths was 27.1 microns when using 37% H3PO4 for 60 seconds and 3.5 microns when using 5% H3PO4 for 15 seconds. Etch depth measurements showed a similar trend. Between the calculated and measured depths of etch, a highly significant correlation was found.

The findings of Nguyen TT et al³⁴ demonstrated that following 15 and 30 seconds of etching, the surface area and volume of the pores increased. This increment diminished at sixty seconds. In contrast, it seems that

etching reduces pore size. There was no discernible relationship between shear strength and varying etching times. Clinically viable shear strength values were demonstrated by samples that were etched for 15, 30, and 60 seconds.

In the present study the etch depth (Total Range – 8.87 to 15.88µm) was comparable to that reported by Redford et al³⁰ upto 60 seconds etching was found lesser than that found in the study by Legler LR et al¹⁹, Devanna R and Keluskar KM.³⁵ The literature has reported creation of roughened porous surface with 5 to 50µm deep craters created by dissolution of interprismatic material in the enamel by etching with phosphoric acid. Though etch depth was less deep in the present study, the shear bond strength obtained was adequate for all samples, indicating that depth of 50µm is not required to obtain good bond strength. Hence, results of present study endorse the observation of Redford et al³⁰ that removal of 50µm of enamel seems to be unwarranted.

In the present study the shear bond strength (Total Range – 9.78 to 17.06Mpa) was found greater than that found in the study by Johnston CD et al.³³ Also; the shear bond strength found in the present study was lesser than that found in the study by Attilio MD.³² This difference could be attributed to difference in methodologies. However, in all our samples the shear bond strength was adequate to obtain desired bond strength.

Al-Suleiman M et al³⁶ results showed that the shear bond strength with 25% phosphoric acid concentration was significantly increased ($p < 0.05$). However, they didn't found significant difference between two etching times (30 & 60 sec) ($p > 0.05$). The shear bond strength with 37% phosphoric acid concentration was significantly decreased ($p < 0.05$) and decreased more when we delayed the application time ($p < 0.05$). The results of their study suggested that a 25% phosphoric acid

concentration with 60 sec etching duration obtained the higher bond strength.

Alavi S et al³⁷ reported that the mean shear bond strength of Er: YAG laser group (16.61 ± 7.7 MPa) was not significantly different from that of the acid-etched group (18.86 ± 6.09 MPa) ($P = 0.41$). There was no significant difference in the ARI scores between two groups ($P = 0.08$). However, in the laser group, more adhesive remained on the brackets, which is not suitable for orthodontic purposes.

Conclusion

Above findings strongly suggest that 15 seconds etching and 30 seconds etching time should be the most preferred procedures to obtain clinically acceptable mean shear bond strength with minimum loss of enamel mineral. We strongly recommend these approaches.

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Legends Figures and Tables

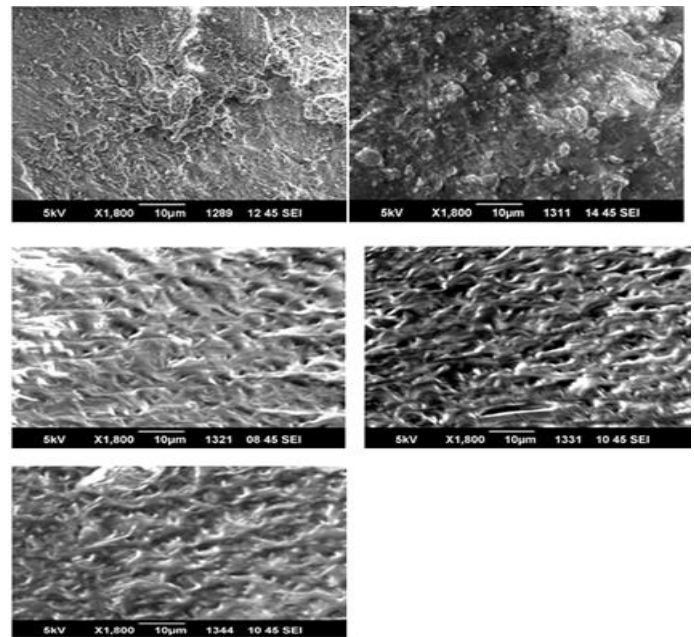


Figure 1: SEM photograph of Group A 15 seconds etching (Subgroup A1), Group B 30 seconds etching (Subgroup B1), Group C 60 seconds etching (Subgroup C1), Group D 90 seconds etching (Subgroup D3), Group E 120 seconds etching (Subgroup E1) at 1800 X magnification

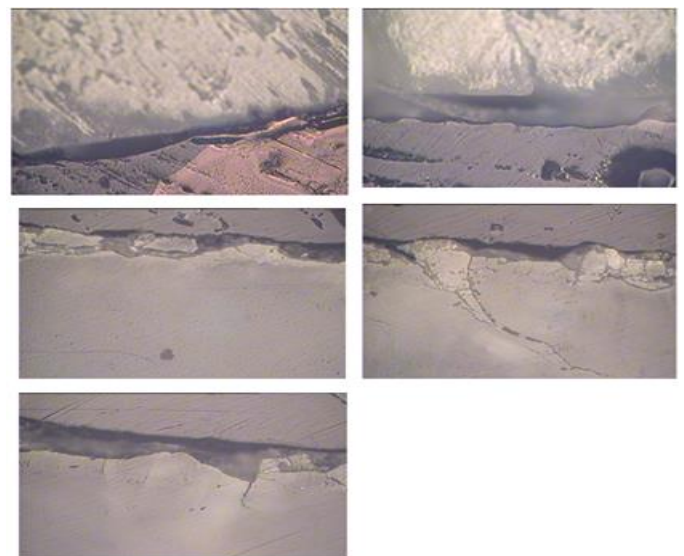


Figure 2: Microscopic view of etch depth at 100 X Magnification of group A (Subgroup A2) , group B (Subgroup B2) , group C (Subgroup C2), group D (Subgroup D2), group E (Subgroup E2) seen under optical microscope.

Table 1: Mean Etch Depth of Group A (15 seconds etching), Group B (30 seconds etching), Group C (60 seconds etching), Group D (90 seconds etching) and Group E (120 seconds etching)

Groups	Etch Depth (µm)		Range µm	Anova 'F' Value	'P' Value
	Mean	S.D			
Group A	8.87	2.25	5.26-12.65	22.49	2.211 E-11
Group B	10.05	1.74	7.73-13.21		
Group C	12.43	2.10	8.30-16.45		
Group D	14.16	2.17	10.82-18.19		
Group E	15.88	2.58	12.14-21.60		

Table 2: Comparison of mean difference between Group A (15 seconds etching), Group B (30 seconds etching), Group C (60 seconds etching), Group D (90 seconds etching) and Group E (120 seconds etching) according to Etch Depth

Groups	Mean Difference	Unpaired student 't' test value	'P' Value
Group A Vs B	1.18	1.48358	0.150
Group A Vs C	3.56	4.1656	3.465
Group A Vs D	5.29	6.09211	2.717
Group A Vs E	6.29	7.36501	1.321
Group B Vs C	3.64	3.15583	0.004*
Group B Vs D	4.11	5.33278	1.793
Group B Vs E	5.83	6.7494	5.564
Group C Vs D	1.73	2.05833	0.050*
Group C Vs E	3.45	3.72578	0.001*
Group D Vs E	1.72	1.8354	0.078

*statistically significant

Table 3: Mean Shear Bond strength of Group A (15 seconds etching), Group B (30 seconds etching), Group C (60 seconds etching), Group D (90 seconds etching) and Group E (120 seconds etching).

Groups	Shear Bond Strength (Mpa)		Range	Anova 'F' Value	'P' Value
	Mean	S.D			
Group A	12.988	3.28	6.42-19.26	8.68	9.48 x 10 ⁻⁶
Group B	13.416	1.76	10.70-17.12		
Group C	17.06	3.80	8.56-23.55		
Group D	15.91	5.16	4.28-14.98		
Group E	9.78	3.77	8.56-29.97		