

Effects of Surface Texture and Etching Time on Roughness and Bond Strength to Ground Enamel

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Abstract

Aim: The aim of this study was to assess the influence of surface texture and etching technique on surface roughness (Ra) and bond strength (BS) to enamel and to determine if a correlation exists between them.

Methods and Materials: Fifty enamel blocks were either roughened with 600-grit SiC paper or polished with diamond pastes. After establishing ten test groups (n=5), the initial Ra measurements, rough (R) and smooth (S) enamel surfaces were etched according to the following protocols: Group 1(R)/Group 2(S)- 35% phosphoric acid gel (H_3PO_4) for 15 seconds; Group 3(R)/Group 4(S)- 35% H_3PO_4 for 60 seconds; Group 5(R)/Group 6(S)- Clearfil SE Bond primer for 20 seconds; Group 7(R)/Group 8(S)- self-etching primer (SEP) for 60 seconds; Group 9(R)/Group 10(S)- 35% H_3PO_4 for 15 seconds + SEP for 20 seconds. After treatments, a new Ra measurement was performed and enamel surfaces were bonded with either Single Bond (Group 1 to Group 4) or Clearfil SE Bond (Group 5 to Group 10). Afterwards, specimens were prepared for the microtensile test. Ra values were analyzed using repeated-measures analysis of variance (ANOVA) and the BS values were analyzed using a two-way ANOVA and Tukey test (5%). Correlation between BS and Ra values was assessed using the Pearson's test.

Results: The application of SEP produced the lowest Ra values. No significant difference was detected between the BS values of polished and rough surfaces. No correlation was observed between Ra and BS values. Even though etching enamel with the SEP resulted in a surface with less roughness, similar BS values were observed for both self-etching and etch-and-rinse techniques.

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Conclusion: Within the limits of this study increasing the etching time or combining both etching techniques failed to improve the BS using SEP or etch-and-rinse systems.

Clinical Significance: Based on the findings of this study, there is no clinical justification for increasing the etching time or for combining the use of a SEP following the use of a 35% H_3PO_4 etchant to achieve a greater BS to ground enamel.

Keywords: Enamel, adhesive system, etching, roughness, bond strength, composite

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Introduction

Surface preparation for bonding of composite resins to dental hard tissues can be accomplished by either the etch-and-rinse method or by using a self-etching technique. The first approach utilizes a separate etching step to completely remove the smear layer and demineralizes the enamel and dentin to a depth of 3 to 7 μm .^{1,2} This strategy has proven to provide a strong and durable bond to enamel, and the technique is considered to be clinically sensitive for bonding to dentin.³⁻⁵ As a result, a second approach was developed in which self-etching primers (SEPs) or self-etching adhesives composed of acidic monomers are applied on the enamel and dentin without rinsing or moisture control.^{6,7}

SEPs have been widely accepted as a good alternative for bonding composite resin to dentin, however, controversy still remains regarding their use for bonding composite to enamel.⁸⁻¹⁰



This concern centers around the shallower demineralization pattern produced by SEPs compared to etch-and-rinse systems.^{1,10} However, in spite of the shallow demineralization a thin hybrid layer is still formed.¹¹ The retentive ability of etched enamel has been reported to be due to the increase in surface area and surface energy of etched enamel.¹²⁻¹⁶ The infiltration of adhesive resin into the micro-porosities of the acid etched enamel and encapsulation of enamel crystallites result in the formation of a resin-enamel interdiffusion zone or enamel hybrid layer.^{7,17}

The effect of different enamel surface textures on bonding to enamel using an etch-and-rinse system has been assessed with no difference being observed for surfaces with different degrees of roughness.¹⁸ While it is known bonding to unground enamel using SEPs is not as reliable as bonding to ground enamel,^{9,19} little is known about the influence of the surface texture of enamel on the bond strength (BS) using SEP systems.

The aims of this study were to (1) examine enamel surface roughness (Ra), BS, and micromorphological characteristics after application of a SEP and an etch-and-rinse system and to (2) verify if there is a correlation between Ra and BS values. Adhesives were applied according to manufacturers' instructions, with extended application times, and associating the etch-and-rinse and self-etching techniques. The null hypotheses to be tested were as follows:

- There is no difference in Ra produced by different etching techniques
- Specimens with rough and smooth surfaces present no difference in Ra after etching

- There is no difference in the BS produced by different etching techniques
- Specimens with roughened and smooth surfaces present no difference in BS
- There is no correlation between Ra and BS

Methods and Materials

Twenty-five freshly extracted sound third molars were used in this study. Teeth were obtained using protocols evaluated and approved by the appropriate institutional review board of the Piracicaba Dental School at the University of Campinas, SP, Brazil along with the informed consent of the donors (protocol # 012/2003). The crown of each tooth was sectioned in a mesio-distal direction (Figure 1A), then each part of the crown was set in an acrylic block which was then secured in a ISOMET 1000 precision slow speed water cooled diamond saw (Buehler Ltd., Lake Bluff, IL, USA) with two parallel disks located 5 mm from each other. Each part of the crown was then cut in the incisal-lingual and in the mesio-

distal direction (Figure 1B), resulting in fifty blocks with a 5 X 5 mm² enamel area exposed (Figure 1C).

The enamel blocks were then embedded in epoxy resin to facilitate handling during tooth surface preparation. To create specimens with roughened surfaces, half of the enamel blocks were roughened with 600 SiC paper (3M, Inc., Sumaré, SP, Brazil). The other half was roughened with 600 and 1200 grit SiC paper (3M) and polished with 6, 3, 1, 0.25 µm grits of diamond pastes using a polish cloth (smooth surfaces group) (Arotec S/A, Cotia, SP, Brazil). Each sample was ultrasonically cleaned in distilled water for 15 minutes, between different grits of diamond paste, for removal of any remaining debris.

Test Groups

Specimens were randomly assigned to ten test groups with five teeth per group, which were etched with 35% phosphoric acid gel (Scotchbond, 3M ESPE, St. Paul, MN, USA), Clearfil SE Bond

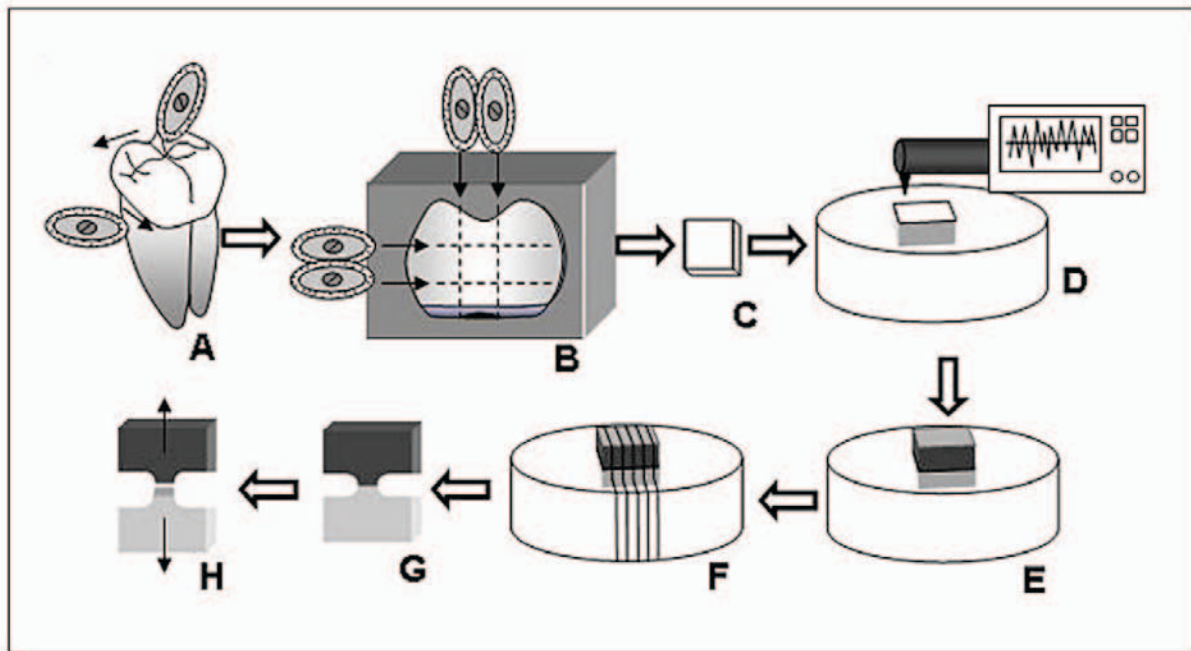


Figure 1. Schematic representation of the study methodology. **A.** Root removal and initial sectioning in a mesio-distal direction. **B.** Embedding the crown segments in acrylic resin blocks. **C.** Crown sectioning in the incisal-lingual and in the mesio-distal direction resulting in blocks with a 5 X 5 mm² enamel area. **D.** Embedding the acrylic blocks in epoxy resin and preparing the enamel surfaces to create either a smooth or rough surface as determined with a profilometer. After application of etchants and/or acidic primer, Ra was measured again. **E.** After the second profilometric reading, adhesive systems were applied and enamel surfaces were restored with composite resin. **F.** Restored specimens were sectioned into four 1 mm thick slabs. **G.** The slabs were trimmed to an hourglass shape with a cross-sectional area of approximately 0.8 mm². **H.** Specimens were debonded in tension at a crosshead speed of 0.5 mm/s.

primer (Kuraray Medical Inc., Tokyo, Japan), or with a combination of both. The following is the protocol for each group (n=5):

- **Groups 1 and 2:** A 35% solution of H_3PO_4 was applied for 15 seconds, rinsed for 15 seconds then air-dried for ten seconds.
- **Groups 3 and 4:** A 35% solution of H_3PO_4 was applied for 60 seconds, rinsed for 15 seconds then air-dried for ten seconds.
- **Groups 5 and 6:** Clearfil SE Bond primer was applied and rubbed for 20 seconds and gently air-dried for 20 seconds.
- **Groups 7 and 8:** Clearfil SE Bond primer was applied and rubbed for 60 seconds and gently air-dried for 20 seconds.
- **Groups 9 and 10:** A 35% solution of H_3PO_4 was applied for 15 seconds, rinsed for 15 seconds then air-dried for ten seconds followed by application of the Clearfil SE Bond primer, rubbed for 20 seconds, and gently air-dried for 20 seconds.

Surface Roughness (Ra) Measurements

Prior to etching, roughened and smooth enamel surfaces were washed in distilled water for 15 seconds and dried with an oil-free air stream. Baseline Ra measurements were performed on the specimens using a Surfcomer SE 1700 profilometer (Kosaka Corp, Tokyo, Japan) at a 0.05 mm/s speed, 2.5 mm length, and 0.25 mm cut-off. Three measurements were performed in different directions for each specimen surface. The average of these three measurements was used as an initial Ra value for the samples.

Immediately after etching, the three measurements were repeated along the same traces used for the initial Ra measurement. The average of the three measurements was used as a final Ra value for the etched samples (Figure 1D). Next, Single Bond (Groups 1 to 4) or Clearfil SE Bond bonding agents (Groups 5 to 10) were applied to the etched samples and light cured.

Microtensile Testing

Following the cure of the bonding agents, three increments of 2.0 mm thick TPH Spectrum composite resin (Dentsply Caulk, Milford, DE, USA) were applied to the samples to create a composite block approximately 6 mm high. A 3M XL2500 light curing unit (3M Espe, St. Paul, MN, USA) with an output of 650 mW/cm² was used to

polymerize resin-based materials for 20 seconds (Figure 1E).

The teeth were stored in distilled water for 24 hours then serially sectioned into four 1 mm thick slabs using the ISOMET 1000 saw (Figure 1F). The slabs were then trimmed to an hourglass shape with a cross-sectional area of approximately 0.8 mm² at the bonded interface (Figure 1G). The resulting specimens were tested in tension in a Instron 4411 universal testing machine (Instron Corp, Canton, MA, USA) at a cross-head speed of 0.5 mm/min until failure (Figure 1H). Maximal tensile load (KgF) was divided by the specimen cross-sectional area to express the results in Megapascals (MPa).

Scanning Electron Microscopy (SEM)

To determine mode of fracture, the enamel sides of the fractured specimens were mounted on aluminum stubs, gold sputter-coated (MED 010, BAL-TEC, Furstentum, Liechtenstein) then examined using a LEO 435 VP SEM (LEO Electron Microscopy Ltd., Cambridge, United Kingdom) at 200X and 400X magnifications.

Failure mode was classified into one of four types as follows:

- **Type 1.** Adhesive failure between the adhesive resin and enamel
- **Type 2.** Partial adhesive failure between enamel and the adhesive resin and partial cohesive failure in the adhesive resin
- **Type 3.** Partial cohesive failure in enamel
- **Type 4.** Cohesive failure in adhesive resin

Two additional specimens were prepared for the test groups for observation of the resin enamel interfaces. The interface was finished and polished with 6, 3, 1, and 0.25 μm -grit diamond paste using polishing cloths then demineralized with 35% H_3PO_4 for ten seconds. In order to examine the micromorphology after use of the different etching techniques, two specimens were etched in the same manner as the test groups without an application of adhesive resin. Etched enamel surfaces were then rinsed with acetone (three baths of ten minutes each) to dissolve away the SEP for the Clearfil SE Bond primer groups. Specimens were then sputter-coated and observed at 7,500X magnification at a 90° angle to the specimen surface.

Statistical Analysis

Ra data was statistically analyzed using the repeated measures analysis of variance (ANOVA) and Tukey tests. Microtensile BS (μ TBS) values were analyzed using the two-way ANOVA and Tukey tests. Pearson's correlation test was used to analyze the results to determine the existence of a possible correlation between Ra and BS means. All statistical analyses were performed at a pre-set significance level of 0.05 using SAS, version 8.0 software (SAS Institute, Cary, NC, USA).

Results

Mean Ra and μ TBS values are presented in Tables 1 and 2, respectively.

Surface Roughness (Ra) Measurements

The repeated measures ANOVA revealed statistically significant differences for the following factors:

- Surface Texture ($p=0.00001$)
- Etching ($p=0.00004$)
- Time before or after etching ($p=0.0004$)

Table 1. Mean (SD) Ra (μ m) of smooth and roughened enamel surfaces prior to and after etching.

	Smooth		Roughened (600 SiC paper)	
Etching	Before	After	Before	After
H ₃ PO ₄ 15s	0.14 (0.02) Ac	0.33 (0.02) Ba	0.23 (0.05) Ab	0.32 (0.04) Ba
H ₃ PO ₄ 60s	0.15 (0.03) Ac	0.37 (0.09) Aa	0.27 (0.02) Ab	0.39 (0.01) Aa
SEP 20s	0.13 (0.01) Ac	0.25 (0.02) Ca	0.24 (0.03) Ab	0.28 (0.04) Ca
SEP 60s	0.13 (0.02) Ac	0.24 (0.04) Ca	0.24 (0.02) Ab	0.31 (0.04) Ca
H ₃ PO ₄ + SEP	0.14 (0.03) Ac	0.39 (0.06) Aa	0.23 (0.05) Ab	0.38 (0.08) Aa

Means followed by different letters (upper case column, lower case row) differ among them using the Tukey test at the 0.05 confidence level. (H₃PO₄ – phosphoric acid; SEP – Clearfil SE Bond primer).

Table 2. Mean (SD) BS (MPa) to smooth and roughened surfaces after the different etching procedures.

Etching + Adhesive	Smooth		Roughened	Tukey
H ₃ PO ₄ 15s + Single Bond	21.16 (9.07)	NS	21.47 (7.56)	AB
H ₃ PO ₄ 60s + Single Bond	26.82 (3.85)	NS	24.39 (8.02)	A
SEP 20s + SEB	20.05 (7.90)	NS	21.91 (9.23)	AB
SEP 60s + SEB	19.63 (7.27)	NS	17.73 (7.44)	B
H ₃ PO ₄ + SEP(20s) + SEB	19.82 (5.32)	NS	23.04 (6.00)	AB

Means followed by different letters differ among them using the Tukey test at the 0.05 confidence level. NS. No significant differences between smooth and roughened surfaces (H₃PO₄ – phosphoric acid; SEP – Clearfil SE Bond primer; SEB – Clearfil SE Bond bonding agent).

- Double interactions between the factors “etching” X “time” and “surface texture” X “time” ($p < 0.0001$).

The triple interaction was not significant. The Tukey test revealed significant differences among groups ($p < 0.05$).

No significant difference ($p > 0.05$) was observed among groups (etching techniques) of roughened and smooth surface groups prior to etching treatments. Thus, the groups presented the same initial Ra before etching. Ra of groups abraded with 600 grit SiC paper were significantly higher than groups polished with diamond pastes ($p < 0.05$). However, no significant differences ($p < 0.05$) were observed between roughened and smooth surfaces groups after acid etching. All etching techniques produced an increased enamel Ra compared to baseline ($p < 0.05$). Etch-and-rinse system surfaces presented rougher surfaces ($p < 0.05$) than groups etched with the SEP (Table 1).

Microtensile Testing

For μ TBS, the two-way ANOVA revealed statistically significant differences for the factor “etching” ($p = 0.0142$), but failed to identify significant differences for the factor “surface

texture” and for the interaction between factors ($p > 0.05$). There were no statistically significant differences in μ TBS values between roughened and smooth surfaces groups.

The Tukey post-hoc test showed significant differences among the different etching techniques used on enamel ($p < 0.05$). Groups etched using etch-and-rinse systems for 60 seconds presented the highest μ TBS values but were only significantly different from the groups where the SEP was applied for 60 seconds. Except for this difference, all techniques presented similar BS values (Figure 2).

No correlation was observed between average Ra and BS values ($R^2 = 0.04$).

Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) observation of the mode of failure is shown in Figure 3. A large number of Type 1 failures were observed when the SEP was applied to smooth surfaces. Groups etched with H_3PO_4 for 60 seconds presented a considerable number of Type 4 failures.

Figures 4 to 8 show the morphologic surface and interfacial characteristics that resulted from the etching techniques used. The etch-and-rinse

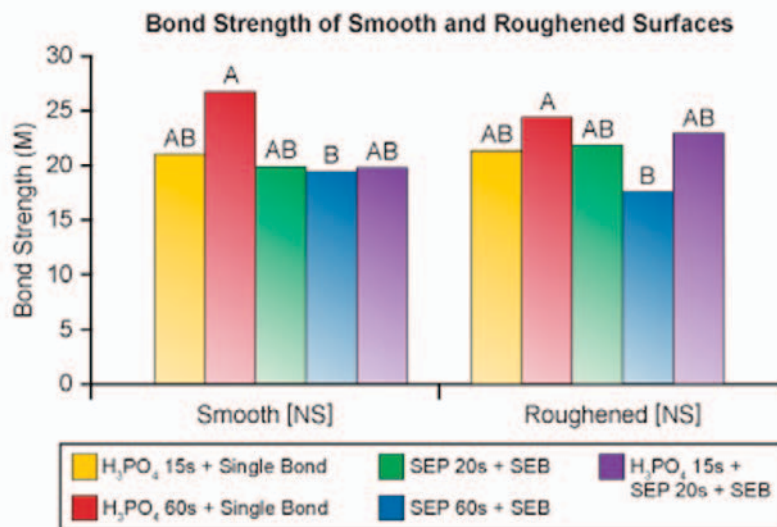


Figure 2. Bar graph of mean BS (MPa) to smooth and roughened surfaces after different etching procedures.

Note: NS indicates no significant difference between rough and smooth surfaces. ANOVA failed to identify significant differences for the factor “surface texture” and for the interaction between factors ($p > 0.05$).

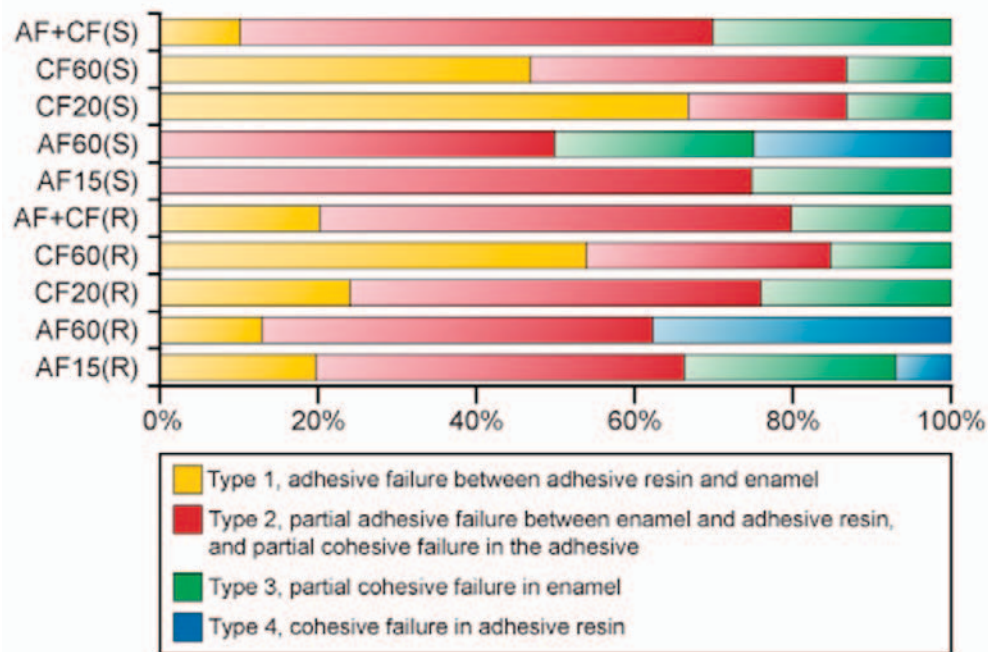


Figure 3. Failure modes of fractured specimens (%). (S) Smooth surfaces, (R) Roughened surfaces.

group specimens presented a characteristic Type II pattern with a well-defined interprismatic dissolution (Figures 4A, 4C, 5A, and 5C). No signs of the scratches produced by the SiC paper could be observed after etching (Figures 4C, 5C, and 8C). Interfacial analysis demonstrated long resin tags in the etch-and-rinse group's specimens (Figures 4B and 5B). A longer etching time resulted in a deeper etching pattern and longer resin tags (Figure 5B). When the SEP was applied, very mild surface alterations were noticed with a small dissolution of the interprismatic region (Figures 6A and 7A). An increased etching time did not necessarily produce a more defined etching pattern (Figure 7A). Shorter resin tags were observed for both etching times (Figures 6B and 7B). The scratches produced by the SiC paper could still be observed after application of the SEP (Figures 6C and 7C). The association of the 35% H_3PO_4 and the SEP techniques resulted in a surface similar to etch-and-rinse enamel (Figures 7A and 8C) with shorter resin tags than when a 35% solution of H_3PO_4 was applied alone (Figure 8B).

Discussion

The different techniques used in this study for etching enamel resulted in different Ra values.

Thus, the first null hypothesis was rejected. Surface profilometry and SEM evaluation demonstrated both 35% H_3PO_4 and SEP produced alterations on the enamel surface. However, different etching patterns were observed for the etching techniques studied. As expected, 35% H_3PO_4 produced the greatest alterations on enamel surface with a dissolution of enamel rods and porosities on exposed prism cores. An increased application time of 35% H_3PO_4 resulted in increased demineralization and, consequently, an increase in Ra.

Clearfil SE Bond Primer is mainly composed of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) which is an acidic monomer responsible for simultaneous demineralization and infiltration into tooth substrates. The pH of this SEP solution has been reported¹⁴ to be 2.0 while the pH of 35% H_3PO_4 is about 0.7.²⁰ Thus, the ability of SEP to demineralize enamel or dentin substrate is considerably reduced when compared to more aggressive acid solutions.^{9,10,17}

The present study demonstrated an extended application time (60 seconds) and did not increase the Ra when compared with the recommended application time (20 seconds).

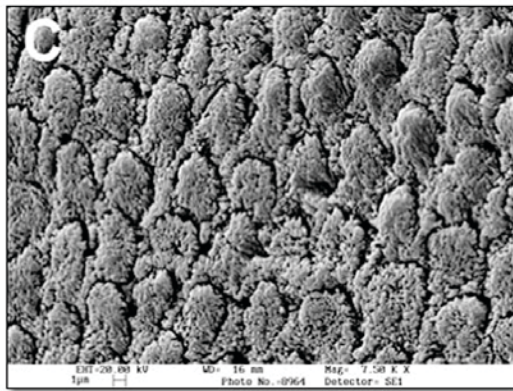
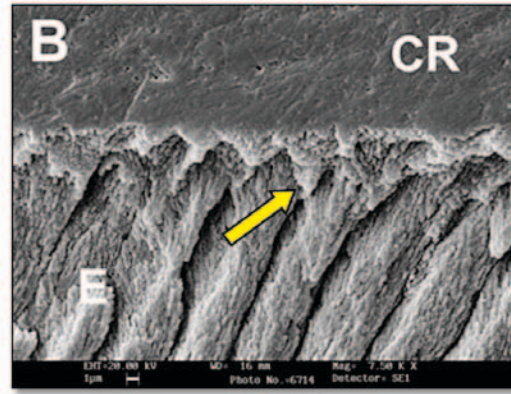
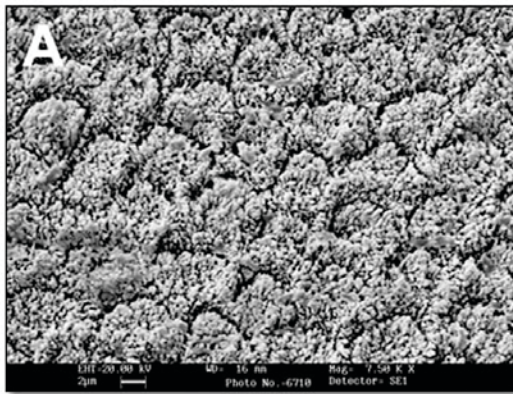


Figure 4A. SEM of a smooth surface etched with 35% H₃PO₄ for 15 seconds. Note the interprismatic dissolution of enamel. **B.** Resin-enamel interface produced by a 15 second application of 35% H₃PO₄ on a smooth surface. **C.** Roughened surface etched with 35% H₃PO₄ for 15 seconds. (CR- composite resin, E- enamel, the arrow indicates a resin tag). Original magnification X 7,500.

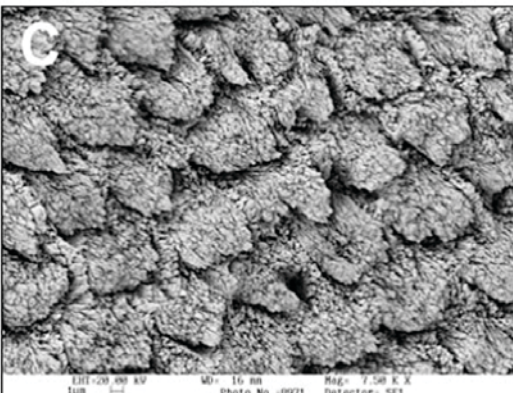
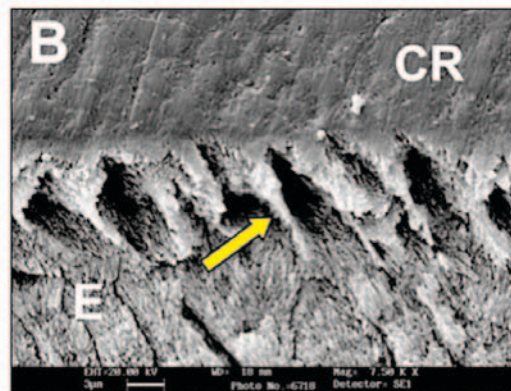
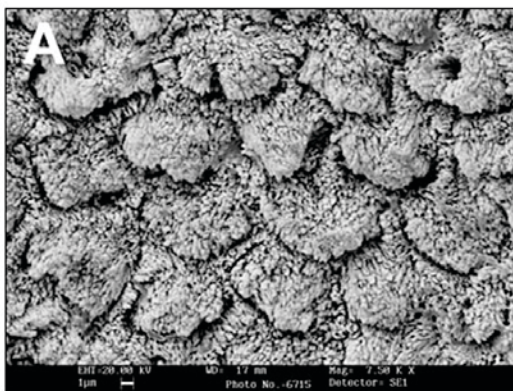


Figure 5A. SEM of a smooth surface etched with 35% H₃PO₄ for 60 seconds. A greater dissolution of interprismatic enamel can be observed. **B.** Resin-enamel interface produced by 60 second application of H₃PO₄ on a roughened surface. Longer resin tags were produced by the extended application time (arrow). **C.** Roughened surface etched with 35% H₃PO₄ for 60 seconds. (CR- composite resin, E- enamel). Original magnification X 7,500.

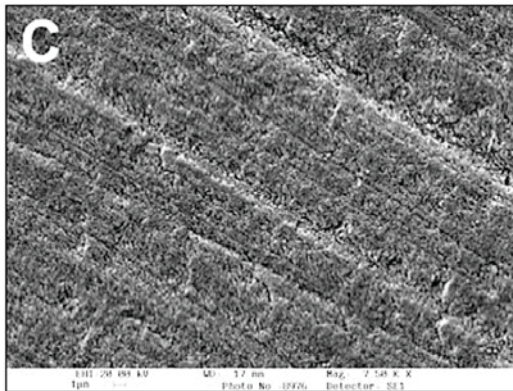
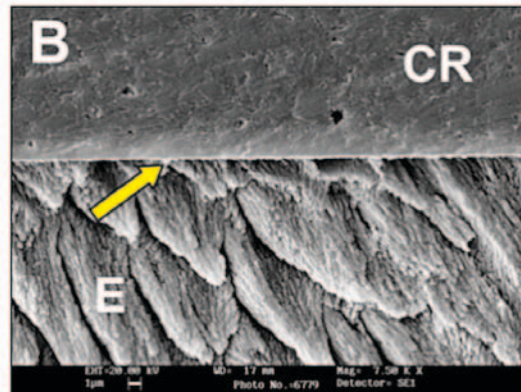
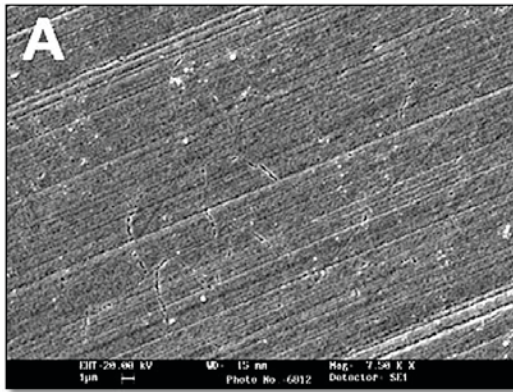


Figure 6A. SEM of a smooth surface etched with Clearfil SE Bond primer (SEP) for 20 seconds. A very mild etching pattern could be observed. **B.** Resin-enamel interface produced by Clearfil SE Bond systems applied as per manufacturer's instructions. Very tiny resin tags were observed (arrow). **C.** Roughened surface etched with SEP for 20 seconds. The scratches produced by the 600-grit SiC paper could be still observed. (CR- composite resin, E- enamel). Original magnification X 7,500.

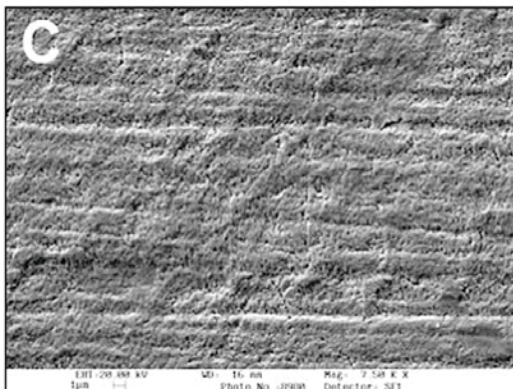
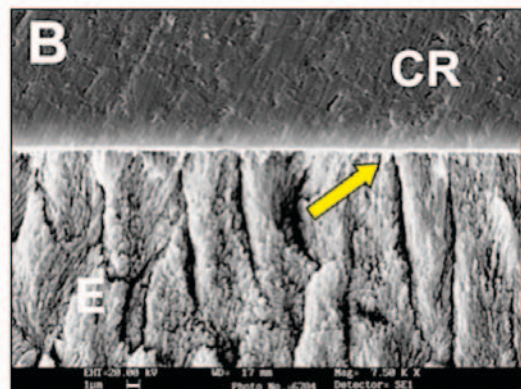
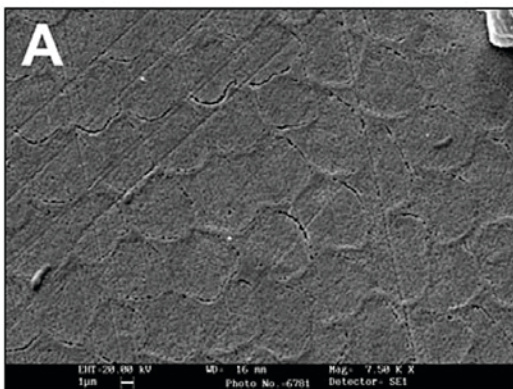


Figure 7A. SEM of a smooth surface etched with Clearfil SE Bond primer for 60 seconds. A very mild etching was also observed for this system. **B.** Resin-enamel interface produced by Clearfil SE Bond systems applied for 60 seconds. Very tiny resin tags were observed (arrow). **C.** Roughened surface etched with SEP for 60 seconds. The scratches produced by the 600-grit SiC paper could be still observed. (CR- composite resin, E- enamel). Original magnification X 7,500.

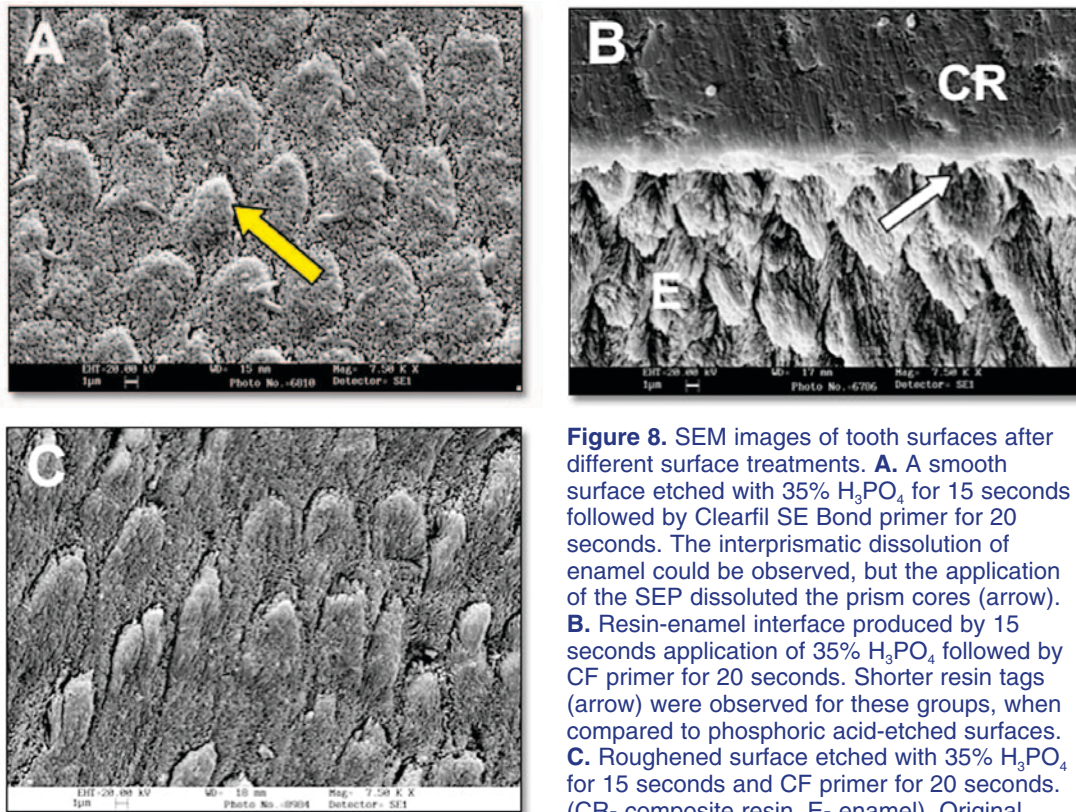


Figure 8. SEM images of tooth surfaces after different surface treatments. **A.** A smooth surface etched with 35% H_3PO_4 for 15 seconds followed by Clearfil SE Bond primer for 20 seconds. The interprismatic dissolution of enamel could be observed, but the application of the SEP dissolved the prism cores (arrow). **B.** Resin-enamel interface produced by 15 seconds application of 35% H_3PO_4 followed by CF primer for 20 seconds. Shorter resin tags (arrow) were observed for these groups, when compared to phosphoric acid-etched surfaces. **C.** Roughened surface etched with 35% H_3PO_4 for 15 seconds and CF primer for 20 seconds. (CR- composite resin, E- enamel). Original magnification X 7,500.

Even though surface profilometry did not detect a significant difference between groups treated with the SEP for 20 or 60 seconds, a slightly more defined etching pattern was observed for specimens treated with the SEP for 60 seconds (Figures 6A and 7A).

The SEM evaluation revealed the scratches produced by the 600-grit SiC paper were still apparent after application of the SEP (Figures 6C and 7C). However, a combination of both techniques (etch-and-rinse and self-etching) did increase the enamel Ra. Even though the initial Ra differed initially between roughened and smooth surfaces, no difference was observed between Ra values after conditioning the surfaces. These observations lead us to accept the second null hypothesis.

Interestingly, the results of the present study showed an increased Ra did not necessarily result in a higher BS as no correlation was observed between average Ra and BS. BS results are in accordance with other studies, which

demonstrated similar values when Clearfil SE Bond and etch-and-rinse systems were compared in enamel.^{11,21-23} Despite their different etching strategies, BSs for SEP systems have been reported as reliable as etch-and-rinse systems when applied over ground enamel surfaces.⁹

Recent ultramorphological studies have elucidated the bonding mechanisms of SEPs to enamel and compared them to pretreatment with phosphoric acid.^{1,11,17} While macro and micro resin tags are responsible for bonding to phosphoric acid-etched enamel, only microtags are found when SEPs are used. The mechanism of bonding to enamel with SEPs has been reported to be based on inter- and intracrystallite hybridization of the enamel surface, rather than resin tag formation.¹ Hashimoto et al.¹¹ demonstrated an infiltration of 0.6 to 0.7 μm into enamel using transmission electron microscopy. Despite this shallow resin penetration pattern, adequate resin-enamel BSs were recorded for SEPs. Thicker hybrid layers formed in dentin substrates have been shown to not necessarily produce a higher BS.²⁴ No attempt



was made to compare hybrid layer thickness and BS to enamel in the present study. However, it can be speculated this same concept also applies to enamel bonding.

An important finding in the present study is extended application times did not produce an increased BS. Surfaces etched with 35% H_3PO_4 promoted rougher surfaces and longer tags with extended application time (60 seconds). However, when comparing different H_3PO_4 concentrations, Shinchi et al.²⁵ demonstrated increased tag length contributed very little to the BS of resin enamel specimens. On the other hand, an increased application time did not produce rougher surfaces for Clearfil SE Bond groups. No improvement in BS values was achieved with extended application times. Most of the demineralizing capacity of the SEP appears to be buffered in the first 20 seconds of application by the high mineral content of the enamel. Thus, extended application times are not recommended for either of the etching systems.

The combination of both etching techniques has been shown to increase enamel BS²⁶ but not change dentin BS.⁶ However, the results of the present study do not support this hypothesis, since the BSs of the groups restored using only SEP were similar to those restored with 35% H_3PO_4 only, as well as those restored with the

combination of 35% H_3PO_4 pretreatment and SEP. Adding the acid-etching step increases the complexity of the procedure and defeats the intent of the simplified approach of SEP systems.²⁷ In a two year clinical trial, Van Meerbeek et al.²⁸ demonstrated no significant differences between cervical Class V restorations using Clearfil SE Bond with or without prior etching the enamel cavity margins with 40% H_3PO_4 . At the five year recall,²⁹ a higher number of marginal defects were observed for the non-etched group, but this was not critical for the overall clinical performance of the restorations.

Polishing dental substrates with diamond pastes produced a smooth surface without a smear layer.³⁰ The present study demonstrated no significant difference in BSs obtained with SEP applied on surfaces with no smear layer and surfaces produced with 600 grit SiC paper. However, it cannot be predicted how the SEP would perform if thicker smear layers had been present.

Conclusion

Based on the results of the present study rougher surfaces do not necessarily produce higher BSs even though a preceding application of H_3PO_4 does increase enamel Ra. Increased application times and/or a combination of the etch-and-rinse and self-etching techniques did not improve the BS for ground enamel. Further research is necessary in order to improve the ability of SEPs to produce reliable bonds to unground enamel.

Clinical Significance

Even though etching enamel with SEP resulted in a surface with less roughness, similar μ TBS values were observed for both self-etching and etch-and-rinse techniques when manufacturers' instructions were followed. Since increasing the etching time or using a combination of both techniques did not improve the BS using either a SEP or for the 35% H_3PO_4 acid-etch system, clinicians are probably wise not to waste valuable treatment time for no technical advantage.

References

1. Hannig M, Bock H, Bott B, Hoth-Hannig W. Inter-crystallite nanoretention of self-etching adhesives at enamel imaged by transmission electron microscopy. *Eur J Oral Sci.* 2002; 110:464-70.
2. Perdigão J, Lambrechts P, Van Meerbeek B, Tome AR, Vanherle G, Lopes AB. Morphological field emission-SEM study of the effect of six phosphoric acid etching agents on human dentin. *Dent Mater.* 1996; 12:262-271.
3. Ferrari M, Tay FR. Technique sensitivity in bonding to vital, acid-etched dentin. *Oper Dent.* 2003; 28:3-8.
4. Pereira PN, Okuda M, Sano H, Yoshikawa T, Burrow MF, Tagami J. Effect of intrinsic wetness and regional difference on dentin bond strength. *Dent Mater.* 1999; 15:46-53.
5. Tay FR, Gwinnett AJ, Wei SH. The overwet phenomenon: an optical, micromorphological study of surface moisture in the acid-conditioned, resin-dentin interface. *Am J Dent.* 1996; 9:43-48.
6. Chaves P, Giannini M, Ambrosano GMB. Influence of smear pretreatments on bond strength. *J Adhes Dent.* 2002; 4:191-196.
7. Nakabayashi N, Pashley DH. Hybridization of dental hard tissue. Quintessence Publishing Tokyo. 1998.
8. Ibarra G, Vargas MA, Armstrong SR, Cobb DS. Microtensile bond strength of self-etching adhesives to ground and unground enamel. *J Adhes Dent.* 2002; 4:115-24.
9. Perdigao J, Geraldini S. Bonding characteristics of self-etching adhesives to intact versus prepared enamel. *J Esthet Restor Dent.* 2003; 15:32-41.
10. Perdigão J, Lopes L, Lambrechts P, Leitão J, Van Meerbeek B. Effects of a self etching primer on enamel shear bond strengths and SEM morphology. *Am J Dent.* 1997; 10:141-146.
11. Hashimoto M, Ohno H, Yoshida E, Hori M, Sano H, Kaga M, Oguchi H. Resin-enamel bonds made with self-etching primers on ground enamel. *Eur J Oral Sci.* 2003; 111:447-53.
12. Silverstone LM, Saxton CA, Dogon IL, Fejerskov O. Variation in the pattern of acid etching of human dental enamel examined by scanning electron microscopy. *Caries Res.* 1975; 9:373-387.
13. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res.* 1955; 34:849-853.
14. Gwinnett AJ. Histologic changes in human enamel following treatment with acidic adhesive conditioning agents. *Arch Oral Biol.* 1971; 16:731-738.
15. Miyazaki M, Sato M, Onose H. Durability of enamel bond strength of simplified bonding systems. *Oper Dent.* 2000; 25:75-80.
16. Retief DH. Effect of conditioning the enamel surface with phosphoric acid. *J Dent Res.* 1973; 52:333-341.
17. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives Part II: etching effects on unground enamel. *Dent Mater.* 2001; 17:430-444.
18. Jung M, Wehlen LO, Klimek J. Surface roughness and bond strength of enamel to composite. *Dent Mater.* 1999; 15:250-256.
19. Kanemura N, Sano H, Tagami J. Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces. *J Dent.* 1999; 27:523-530.
20. Breschi L, Gobbi P, Mazzotti G, Ellis TH, Sacher E, Stangel I. Field emission in-lens SEM study of enamel and dentin. *J Biomed Mater Res.* 1999; 46:315-323.
21. Shimada Y, Iwamoto N, Kawashima M, Burrow MF, Tagami J. Shear bond strength of current adhesive systems to enamel, dentin and dentin-enamel junction region. *Oper Dent.* 2003; 28: 585-590.
22. Shimada Y, Kikushima D, Tagami J. Micro-shear bond strength of resin-bonding systems to cervical enamel. *Am J Dent.* 2002; 15:373-377.
23. Toledano M, Osorio R, de Leonardi G, Rosales-Leal JI, Ceballos L, Cabrerizo-Vilchez MA. Influence of self-etching primer on the resin adhesion to enamel and dentin. *Am J Dent.* 2001; 14:205-210.
24. Harada N, Yamada T, Inokoshi S, Tagami J. Tensile bond strengths and adhesive interfaces of ten dentin bonding systems. *J Medic Dent Sci.* 1998; 45:85-96.

25. Shinchi MJ, Soma K, Nakabayashi N. The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel. *Dent Mater.* 2000; 16: 324-329.
26. Torii Y, Itou K, Nishitani Y, Ishikawa K, Suzuki K. Effect of phosphoric acid etching prior to self-etching primer application on adhesion of resin composite to enamel and dentin. *Am J Dent.* 2002; 15:305-358.
27. Pereira PNR. Enamel-dentin bonding. *J Esthet Restor Dent.* 2003; 15:203.
28. Van Meerbeek B, Kanumilli P, De Munch J, Van Landuyt K, Lambrechts P, Peumans M. A randomized controlled study evaluating the effectiveness of a two-step self-etch adhesive with and without selective phosphoric-acid etching of enamel. *Dent Mater.* 2005; 21:375-383.
29. Peumans M, De Munch J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Five-year clinical effectiveness of a two-step self-etching adhesive. *J Adhes Dent.* 2007; 9:7-10.
30. Arrais CAG, Micheloni CD, Giannini M, Chan DCN. Occluding effect of dentifrices on dentinal tubules. *J Dent.* 2003; 31:59-66.

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