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Effect of Enamel Etching Time on Roughness and Bond Strength

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Clinical Relevance

Traditionally, etch-and-rinse adhesive systems have used phosphoric acid to condition enamel surfaces and successfully create a strong, durable bond to resin-based materials. Newer adhesive systems now use acid monomers to bond resin materials to both enamel and dentin. These newer adhesives do not provide the same degree of bonding to enamel as etch-and-rinse systems, and extending the application time does not improve their performance.

SUMMARY

The current study examined the effect of different enamel conditioning times on surface roughness and bond strength using an etch-and-rinse system and four self-etch adhesives. Surface roughness (Ra) and composite to enamel shear bond strengths (SBS) were determined following the treatment of flat ground human enamel (4000 grit) with five adhesive systems: 1) Adper Single Bond Plus (SBP), 2) Adper Prompt L-Pop (PLP), 3) Clearfil SE Bond (CSE), 4) Clearfil S[3] Bond (CS3) and 5) Xeno IV (X4), using recommended treatment times and an extended treatment time of 60 seconds (n=10/group). Control groups were also included for Ra (4000 grit surface) and SBS (no enamel treatment and Adper Scotchbond Multi-Purpose Adhesive). For surface roughness measurements, the phosphoric acid conditioner of the SBP etch-and-rinse system was rinsed from the surface with an air-water spray, and the other four self-etch adhesive agents were removed with alternating rinses of water and acetone. A Proscan 2000 non-contact profilometer was used to determine Ra values. Composite (Z100) to enamel bond strengths (24 hours) were determined using Ultradent fixtures and they were debonded with a crosshead speed of 1 mm/minute. The data were analyzed with ANOVA and Fisher's LSD post-hoc test. The etch-and-

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rinse system (SBP) produced the highest Ra (µm) and SBS (MPa) using both the recommended treatment time (0.352 ± 0.028 µm and 40.5 ± 6.1 MPa) and the extended treatment time (0.733 ± 0.122 µm and 44.2 ± 8.2 MPa). The Ra and SBS of the etch-and-rinse system were significantly greater (*p*<0.05) than all the self-etch systems and controls. Increasing the treatment time with phosphoric acid (SBP) and PLP produced greater surface roughness (*p*<0.05) but did not result in significantly higher bond strengths (*p*>0.05).

Table 1: *Enamel Treatment Times for Surface Roughness Measurements*

Adhesive System	Time (seconds)		
	Recommended	Extended	Selected
SBP	15	60	30
PLP	15	60	30
CSE	20	60	40
CS3	20	60	40
X4	30	60	15

Table 2: *Enamel Treatment Times for Shear Bond Strength Testing*

Adhesive System	Time (seconds)	
	Recommended	Extended
SBP	15	60
PLP	15	60
CSE	20	60
CS3	20	60
X4	30	60

INTRODUCTION

Phosphoric acids ranging from 32% to 40% have been used for decades to bond resin-based materials to enamel. The micro-mechanical retention of resin materials into enamel porosity, resulting from acid etching, creates a strong and durable bond.

In recent years, self-etch adhesive systems have been developed and introduced to the profession. Self-etch adhesives are promoted primarily to increase efficiency in patient treatment and reduce the time for bonding procedures. These adhesives use acidic monomers to condition tooth structures rather than traditional phosphoric acids; however, they do not produce the same degree of porosity in enamel surfaces as that attained with phosphoric acid etching.[1-3]

The bond strengths of resin composite to enamel determined in the laboratory generally have been lower with self-etch adhesives when compared with etch-and-rinse systems.[4-6] Pre-etching enamel with phosphoric acid has been shown to increase the bond strength of self-etch systems.[7-14] These observations suggest that reduced etching of enamel may compromise optimum bond strength and durability. Fatigue testing of enamel bonds has also shown that self-etch adhesives do not perform to the same level as traditional phosphoric acid conditioning systems.[15-17]

Currently, there is limited information about the effects of extending the enamel treatment time of self-etch adhesives. The current study examined the effect of different enamel conditioning times on surface roughness and shear bond strength using an etch-and-rinse system and self-etch adhesives.

METHODS AND MATERIALS

Enamel surface roughness (Ra) and resin composite to enamel shear bond strengths (SBS) were determined with five adhesive systems: 1) Adper Single Bond Plus (3M ESPE, St Paul, MN, USA)—(SBP), 2) Adper Prompt L-Pop (3M ESPE)—(PLP), 3) Clearfil SE Bond (Kuraray Medical Inc, Okayama, Japan)—(CSE), 4) Clearfil S^3 Bond (Kuraray Medical Inc)—(CS3) and 5) Xeno IV (Dentsply Caulk, Milford, DE, USA)—(X4). Extracted human molars were sectioned mesio-distally and approximately two-thirds of the apical root structure was removed. The buccal and lingual coronal sections were then mounted with Triad DuaLine (Dentsply International, York, PA, USA) in custom brass fixtures designed for use with an abrasive polishing system to create a flat enamel surface. The enamel was ground flat using a water coolant and a sequence of carbide polishing papers to create a final surface of 4000 grit (Struers Inc, Cleveland, OH, USA).

Surface Roughness

Enamel roughness was measured to monitor the change in morphology due to etching the surface. Flat ground (4000 grit) enamel surfaces were used to determine enamel surface roughness resulting from use of the five adhesive systems and a control group that did not receive treatment. Ten specimens each were used for the control (4000 grit surface) and three treatment times were used with each of the adhesive systems: 1) recommended treatment time, 2) extended treatment time (60 seconds) and 3) selected treatment time (intermediate time between recommended and extended treatment time or a shorter time than the recommended time, depending on the system) to develop a relationship among the treatment times. The treatment times for the adhesive systems are listed in Table 1.

Phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for the SBP system was applied to the enamel surfaces for the treatment times specified in Table 1. The acid gel was were then rinsed from the surface with an air-water spray from a three-way dental syringe and air-dried. The self-etch adhesives were applied to the surfaces for the recommended treatment times (Table 1) according to manufacturers' directions. Additional applications were used for the extended treatment times (PLP [60 seconds]—initial application for 15 seconds, additional applications at 15 seconds, 30 seconds and 45 seconds). The self-etch agents were then removed from the surface using alternating washes of distilled water and acetone to remove all soluble organic and inorganic products, air-dried and immediately examined.

The surface roughness of the treated enamel surfaces and the control specimens was determined using a Proscan 2000 non-contact profilometer and Proscan software (Scantron Industrial Products, Ltd, Taunton, England). The profilometer scan was 3.0 mm in length and 0.75 mm in height. The scan set-up parameters were as follows: scan rate = 300 Hz, average = 4 and step size = 0.005 mm. A cut-off filter of 0.25 mm was used to determine the Ra value.

Shear Bond Strength

Ten specimens each were used to determine the enamel shear bond strengths using the recommended treatment time and an extended treatment time (60 seconds) for the five adhesive systems. A control group (4000 grit surface) without any enamel surface treatment was bonded using Adper Scotchbond Multi-Purpose Adhesive (3M ESPE). Treatment times for enamel conditioning for the adhesive systems are shown in Table 2.

The agents were applied according to the manufacturers' directions for the recommended treatment times and additional applications were used for the extended groups in the same manner as the surface roughness tests. The adhesives were polymerized according to the manufacturers' guidelines with a Spectrum 800 Curing Unit (Dentsply Caulk) set at 600 mW/cm^2.

Z100 (3M ESPE) resin composite was bonded to the treated enamel surfaces using an Ultradent bonding fixture (Ultradent Products, South Jordan, UT USA). The composite cylinders (2.35 mm in diameter and approximately 2 mm in length) were polymerized for 40 seconds. The bonded specimens were stored for 24 hours in distilled water at 37°C. The specimens were loaded to failure (1 mm per minute) using an Ultradent shearing fixture in an Instron test frame (Instron, Norwood, MA, USA) with an MTS ReNew Upgrade Package and TestWorks software (MTS Systems Corporation, Eden Prairie, MN, USA). The debonded specimens were examined with an optical microscope (20x) to assess the failure sites.

Data Analysis

A two-way Analysis of Variance (ANOVA) and Fisher's LSD post hoc test were conducted for both surface roughness and bond strength to determine if there was a difference among the treatment groups. The factors for the ANOVA tests were: 1) adhesive and 2) treatment time (recommended and extended).

RESULTS

The ANOVA for enamel surface roughness (Table 3), using the five adhesive agents at the recommended and extended treatment times, along with the control, revealed a significant effect for the individual factors of adhesive (p=0.000) and treatment time (p=0.000). There also was a significant effect (p=0.000) for the interaction of adhesive and treatment time.

The surface roughness created by enamel treatment with phosphoric acid (SBP) was significantly greater (p<0.05) than the control and the four self-etch adhesives (Table 4) at the recommended treatment time and the four self-etch adhesives at the extended treatment time. The surface roughness of enamel treated with PLP also was significantly greater (p<0.05) than CSE, CS3 and X4 at recommended and extended treatment times, and the latter three were not significantly different from the control surface. The only treatment agents to produce significantly higher Ra value for the extended treatment time, compared with the recommended treatment time, were phosphoric acid (SBP) and PLP.

Regression lines of surface roughness for the five adhesive systems in the study, using the three treatment times (Table 1), are shown in Figure 1. The slope

Table 3: *Analysis of Variance—Surface Roughness*

Source	Sum-of-Squares	df	Mean-Square	F-Ratio	P
Adhesive	3.108	5	0.622	391.772	0.000
Time	0.169	1	0.169	106.299	0.000
Adhesive*Time	0.570	5	0.114	71.834	0.000

Table 4: *Enamel Surface Roughness Measurements*

	Recommended Treatment Time (RT)		Extended Treatment Time (ET)	
System	Time (seconds)	Ra (um)	Time (seconds)	Ra (um)
SBP	15	0.352 ± 0.028 a	60	0.733 ± 0.122 a*
PLP	15	0.250 ± 0.031 b	60	0.298 ± 0.034 b*
CSE	20	0.123 ± 0.010 c	60	0.134 ± 0.012 c
CS3	20	0.102 ± 0.014 c	60	0.107 ± 0.018 c
X4	30	0.107 ± 0.010 c	60	0.110 ± 0.012 c
Control	—	0.099 ± 0.012 c		

Values in the same column with same letter are not different (p>0.05).
**=significant difference (p<0.05) between RT and ET.*

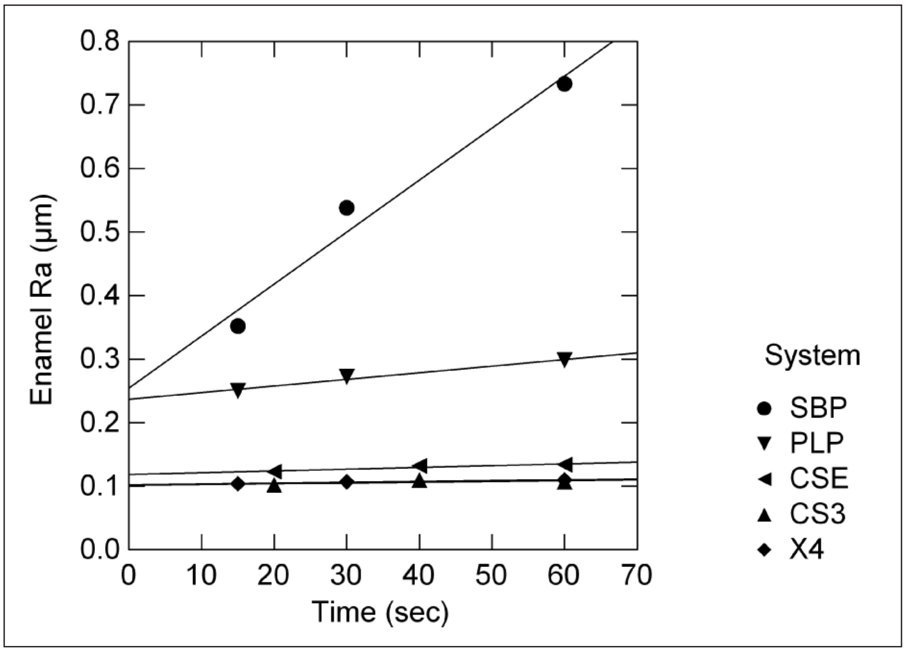

Figure 1: *Regression lines of Ra values generated from various enamel surface treatment times with five adhesive systems.*

of the lines was significant ($p<0.05$) for both SBP and PLP, but not for CSE, CS3 and X4.

The ANOVA for shear bond strength (Table 5) using the five adhesive agents at the recommended and extended treatment times and the control revealed a significant effect for the individual factors of adhesive (p=0.000) and treatment time (p=0.039) but not for the interaction of adhesive and treatment time (p=0.38).

The shear bond strengths for the recommended and extended treatment times are shown in Table 6. The enamel shear bond strength of the etch-and-rinse system (SBP) was significantly higher ($p<0.05$) than the four self-etch systems at both the recommended and extended treatment times. All five adhesive systems produced bond strengths that were significantly greater than the control. None of the adhesive systems showed a significant increase in bond strength ($p<0.05$) when the treatment time was extended to 60 seconds. The failure site locations are reported as a percentage in Table 7.

Table 5: *Analysis of Variance—Shear Bond Strength*

Source	Sum-of-Squares	df	Mean-Square	F-Ratio	P
Adhesive	19713.877	5	3942.775	217.109	0.000
Time	79.056	1	79.056	4.353	0.039
Adhesive*Time	61.951	5	12.390	0.682	0.638

Table 6: *Enamel Shear Bond Strengths*

Recommended Treatment Time (RT)			Extended Treatment Time (ET)	
System	Time (seconds)	SBS (MPa)	Time (seconds)	SBS (MPa)
SBP	15	40.5 ± 6.1 a	60	44.2 ± 8.2 a
PLP	15	30.7 ± 4.3 b	60	32.9 ± 3.6 bc
CSE	20	29.1 ± 2.7 bc	60	29.3 ± 5.9 cd
CS3	20	25.7 ± 2.4 c	60	26.4 ± 3.4 de
X4	30	20.9 ± 3.1 d	60	23.9 ± 3.5 e
Control	—	0.2 ± 0.1 e		

Values in same column with the same letter are not different (p>0.05).

Table 7: *Failure Site Percentage*

	Recommended Treatment Time (RT)			Extended Treatment Time (ET)		
System	Adhesive	Cohesive	Mixed*	Adhesive	Cohesive	Mixed*
SBP	90%	–	10% A/C	80%	20% (Enamel)	–
PLP	80%	–	20% A/C	100%	–	–
CSE	100%	–	–	100%	–	–
CS3	90%	10% (Enamel)	–	90%	–	10% A/C
X4	80%	–	20% A/C	100%	–	–
Control	100%	–	–			

**A/C=adhesive & cohesive in composite.*

DISCUSSION

Enamel bonding has traditionally been dependent upon the infiltration of resin into surface porosity created by acid conditioning agents. In the past, phosphoric acid has been the principal etchant used for enamel conditioning. More recently, newer systems have been introduced, which use a single treatment step to condition or etch both the enamel and dentin surfaces with agents other than phosphoric acid. These systems are generally referred to as self-etch adhesives. The primary benefit of using these new adhesive systems is a reduction in the number of steps in the bonding procedure.

Studies have shown that the resultant enamel surface is not etched to the same degree with self-etch adhesive systems when compared with traditional phosphoric acid conditioning.[1-3] Concern has been expressed that the resulting bond to enamel surfaces produced by self-etch adhesives may not be as durable as etch-and-rinse systems.

The current study indicates that enamel surfaces generated using self-etch adhesive systems are different from an enamel surface conditioned with phosphoric acid. The mean enamel surface roughness (Ra) created by applying a 37% phosphoric acid gel for 15 seconds (recommended time) and 60 seconds (extended time) was significantly greater ($p<0.05$) than that produced by the four self-etch systems when used at the recommended time and extended time (60 seconds). Phosphoric acid etching of enamel and PLP were the only treatments that showed a significant increase ($p<0.05$) in surface roughness when the recommended time group was compared to the extended time group. Legler, Retief and Bradley[18] also showed a progressive increase in enamel surface roughness using a 37% phosphoric acid solution for 15, 30 and 60 seconds on 600 grit flat ground surfaces.

Further evidence of the superior bonding surface created by the phosphoric acid treatment of enamel as compared with self-etch systems is found in the strength of the enamel bond of a resin composite. The enamel bond strength of the etch-and-rinse system was significantly higher when used for 15 or 60 seconds than the self-etch systems when used for the recommended time and the extended treatment time (60 seconds). Increased treatment times did not result in significantly greater bond strengths, even when the Ra showed significant increases.

When the acid etch technique was introduced to dentistry, the standard enamel conditioning time was 60 seconds. Studies in the mid-1980s found that a reduction in enamel etching time to 15 seconds did not significantly reduce the bond strength of a resin composite or orthodontic brackets bonded to enamel.[19-20] In the current study, extending the etch time of phosphoric acid or the treatment time of the four self-etch adhesives did not result in markedly higher shear bond strengths. However, extending the enamel treatment time did result in a small increase in the mean bond strength for the adhesives used in this study. Sixty seconds was used for the extended treatment time in the current study, because it approaches the practical time limit for this type of clinical procedure.

CONCLUSIONS

Non-contact profilometery demonstrated significant differences ($p<0.05$) in enamel roughness between surfaces conditioned with phosphoric acid when compared to self-etch adhesive agents. Enamel shear bond strengths were significantly higher on surfaces conditioned with phosphoric acid when compared to surfaces treated with self-etch adhesive systems. Increasing the treatment time with phosphoric acid and the acid monomer in Adper Prompt L-Pop produced greater surface roughness ($p<0.05$) but did not result in significantly higher bond strengths ($p>0.05$). Extending the treatment time with CSE, CS3 and X4 also did not significantly increase bond strengths. Phosphoric acid treatment of enamel appears to be much more effective than acidic monomers for bonding resin-based materials.

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