Adhesive Bond Strengths to Enamel and Dentin Using Recommended and Extended Treatment Times

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Clinical Relevance

Self-etch adhesive systems are promoted as being more efficient for bonding procedures by using fewer treatment steps to condition tooth surfaces for bonding resin-based materials. Concern has been expressed regarding the ability of the newer self-etch adhesives to yield strong, durable bonds when compared to more traditional etch-and-rinse systems. Extending the treatment time of etch-and-rinse and self-etch adhesives does not appear to result in relevant increases in the bond strength of resin composites to enamel or dentin.

SUMMARY

This study examined the effect of different enamel and dentin conditioning times on the shear bond strength of a resin composite using etchand-rinse and self-etch adhesive systems. Shear bond strengths were determined following treatment of flat ground human enamel and dentin

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surfaces (4000 grit) with 11 adhesive systems: 1) AdheSE One Viva Pen-(ASE), 2) Adper Prompt L-Pop-(PLP), 3) Adper Single Bond Plus-(SBP), 4) Clearfil SE Bond-(CSE), 5) Clearfil S³ Bond-(CS3), 6) OptiBond All-In-One-(OBA), 7) OptiBond Solo Plus-(OBS), 8) Peak SE-(PSE), 9) Xeno IV-(X4), 10) Xeno V-(X5) and 11) XP Bond-(XPB) using recommended treatment times and an extended treatment time of 60 seconds (n=10/group). Composite (Z100) to enamel and dentin bond strengths (24 hours) were determined using Ultradent fixtures and debonded with a crosshead speed of 1 mm/minute. The data were analyzed with a three-way Analysis of Variance (ANOVA) and Fisher's LSD post hoc test. The highest shear bond strengths (MPa) to enamel were achieved by the three etch-and-rinse systems at both the recommended treatment time $(SBP-40.5 \pm 6.1; XPB-38.7 \pm 3.7; OBS-35.2 \pm 6.2)$ and the extended treatment time (SBP-44.5 \pm 8.1; $XPB-40.9 \pm 5.7$; OBS-35.0 ± 4.5). Extending the enamel treatment time did not produce a significant change (p>0.05) in bond strength for the 11 adhesive systems tested. OBS generated the highest (46.2 ± 7.9) bond strengths to dentin at the recommended treatment time. At the extended treatment time X4 (42.2 ± 11.7) , PSE (42.1 ± 9.7) and OBS (41.4 ± 8.0) produced the highest bond strengths to dentin. The bond strength change between recommend and extended treatment times was significant (p<0.05) for PSE, but the other 10 systems did not exhibit any significant change.

INTRODUCTION

Buonocore¹ introduced the acid-etch technique to the dental profession in 1955. The concept involved creating microporosity in enamel tooth surfaces by using acid conditioning, then bonding resin-based materials to this altered surface. The initial bonding systems were introduced into clinical practice in the early 1970s and were developed for enamel bonding only. These early systems were used primarily for sealant placement and repair of fractured teeth. The typical etching time for enamel was 60 seconds and phosphoric acid concentrations generally ranged from 32% to 40%. A strong, durable bond of resin-based materials was created using the phosphoric acid conditioning of enamel.

Studies have shown that the conditioning time for enamel bonding could be reduced from 60 seconds to 15 seconds and could still develop a strong, durable bond.²⁵ Steady advancement in adhesive systems continued and, by the late 1980s, dentin bonding became possible with the introduction of the total-etch technique. As materials and techniques for dentin bonding evolved, a 15-second phosphoric acid treatment time also yielded good bond values to this substrate.⁶⁸ The total-etch or etch-and-rinse technique generally uses a phosphoric acid that is applied, then rinsed with water before an adhesive resin is applied. Unlike enamel bonding, which is achieved with relative ease, bonding to dentin has continued to be a challenge.⁸

Part of the challenge in bonding to dentin when compared to enamel is the difference in the substrates. Enamel is homogeneous in nature and is primarily composed of hydroxyapatite. Etchants dissolve hydroxyapatite crystals in enamel, creating pits by which the adhesive resin is readily absorbed by capillary attraction, creating macrotags of resin that envelop the individually exposed hydroxyapatite crystals. Additionally, resin microtags extend within tiny etch pits in the enamel prism cores.8 Resin tags in the interprismatic spaces provide for the majority of micromechanical adhesion.8-9 In comparison, dentin is heterogeneous, consisting of hydroxyapatite and collagen. The degree of mineral content in dentin is quite variable, depending on whether it is near the dentino-enamel junction or deeper in close proximity to the pulp. 10 Overall, the water content of dentin is significantly higher than enamel, posing another challenge to adhesive bonding.8 The acid conditioning of dentin leaves a micro-porous scaffold of collagen fibrils after most or all of the hydroxyapatite is eliminated. Adhesive resin microtags infiltrate and mechanically interlock within this microretentive collagen network.⁸⁻⁹

Etch-and-rinse adhesive systems typically utilize a strong phosphoric acid etchant (32%-40%) with a pH≈1.0, followed by a primer and/or a bonding resin to promote adhesion. Three-step and two-step etch-andrinse systems are available, with the latter combining the primer and bonding resin into a single step. Increasing the application time of acid conditioning agents on enamel can lead to the formation of an insoluble reaction product and a resultant weak bond. An upper limit of 60 seconds for etch-and-rinse system application time is generally well agreed upon; however, some disagreement regarding the lower limit exists. Extending the application time of an acid on dentin can lead to a deeper demineralization that may adversely affect the penetration of adhesive resins. 11 With most etch-and-rinse adhesive systems, the dentin substrate must be left in a moist condition to facilitate maximum penetration of the resin monomers around the collagen exposed in the acid conditioning process. 12-13 The application time for adhesive resins used with etch-andrinse systems is typically 15-20 seconds to facilitate full penetration via capillary action in the conditioned substrate.13

Newer self-etch systems use acid monomers that are applied but typically not rinsed from the tooth surfaces prior to bonding. With these systems, substrate demineralization and resin penetration occur simultaneously. One advantage of self-etch materials is a reduction in treatment time required for the bonding procedure. In addition, there is no ambiguity about the remaining moisture in dentin, as these systems contain water and are not dependent on surface moisture in dentin. Self-etch adhesive systems are available as oneor two-step systems. One-step self-etch adhesive systems combine the etchant, primer and adhesive, while two-step self-etch adhesive systems have a separate component for the adhesive. Self-etch adhesive systems utilize weaker hydrophilic acidic monomers to infiltrate and modify the smear layer, after which bonding resins remain incorporated into the hybridized demineralized dentin.¹⁴ Contact times for etching typically vary between 15 and 30 seconds, and some studies have advocated increasing the application time to allow etching through thick smear layers. 15 However, concern has been expressed that these newer systems do not provide the same degree of enamel porosity and resin penetration to that created by traditional phosphoric acid etching.13-18 It has been demonstrated that some phosphate esters form a covalent bond with residual mineral in the dentin and enamel substrates, but the long114 Operative Dentistry

term stability of this bond formed during the bonding procedure may not be stable.^{9,19}

Etch-and-rinse adhesive systems provide bonding to both enamel and dentin that has proven clinical effectiveness with consistent long-term results.20 However, the procedure involves multiple steps and is perceived to be time consuming and as having technical issues related to over-etching dentin and achieving optimal wetness for bonding to the dentin surface. Self-etch adhesives, on the other hand, are advocated for their ease of use, reduced post-op sensitivity and decreased technique sensitivity. Elimination of the rinse phase decreases chairside time. Unfortunately, data relating to the long-term effects of incorporating the dissolved hydroxyapatite and residual smear layer into the bond and/or the effects of residual primer/adhesive solvent is limited. Also, there is a general trend towards lower enamel bond strengths for self-etch adhesives when compared to etch-and-rinse systems. 21-22 Currently, adhesive-related research is primarily focused on dentin bonding; however, the effectiveness of bonding resin composite to enamel should not be overlooked, especially with these newer adhesive systems.

Barkmeier and others²² examined the degree of surface porosity created by four self-etch systems and an etch-and rinse system (phosphoric acid conditioning) by determining the surface roughness (Ra value) of enamel using several treatment times. The 35% phosphoric acid conditioning produced significantly greater surface roughness than that achieved with any of the self-etch systems. The same study also examined the effect of increasing conditioning time on both surface roughness and shear bond strength of a resin composite to enamel. The increased conditioning time increased surface roughness for the phosphoric acid etch and one self-etch system, but this increase did not result in appreciably higher bond strengths.

In an effort to understand the relationship between conditioning time and the bond strength of a resin composite to enamel and dentin, the investigation in this area was continued. The purpose of the current study was to expand the number of self-etch and etch-andrinse adhesives system evaluated in a previous study and include both enamel and dentin substrates.

METHODS AND MATERIALS

Shear Bond Strength

The shear bond strengths of a resin composite to both enamel and dentin were determined with 11 adhesive systems using the recommended treatment time and an extended treatment time of 60 seconds (n=10/group): 1) AdheSE One Viva Pen (Ivoclar Vivadent AG, Schaan, Liechtenstein)–(ASE), 2) Adper Prompt L-Pop (3M ESPE, St Paul, MN, USA)–(PLP), 3) Adper Single Bond Plus (3M ESPE)–(SBP), 4)

Clearfil SE Bond (Kuraray Medical Inc, Okayama, Japan)–(CSE), 5) Clearfil S³ Bond (Kuraray Medical Inc)–(CS3), 6) OptiBond All-In-One (Kerr USA, Orange, CA, USA)–(OBA), 7) OptiBond Solo Plus (Kerr USA)–(OBS), 8) Peak SE (Ultradent Products Inc, South Jordan, UT, USA)–(PSE), 9) Xeno IV (Dentsply Caulk, Milford, DE, USA)–(X4), 10) Xeno V (Dentsply DeTrey GmbH, Konstanz, Germany)–(X5) and 11) XP Bond (Dentsply DeTrey GmbH)–(XPB). Of the 11 systems evaluated in the current study, three were etchand-rinse adhesives and eight were self-etch adhesives (Table 1).

Extracted human molars were sectioned mesio-distally, then 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 brass fixtures that were custom designed for use with an abrasive polishing system to create flat ground bonding sites on enamel or dentin. The enamel and dentin bonding sites were prepared to a final surface of 4000 grit using a grinding wheel (Ecomet 4 Variable Speed Grinder-Polisher, Buehler, Lake Bluff, IL, USA) with a water coolant and a sequence of carbide polishing papers (Struers Inc, Cleveland, OH, USA).

Forty specimens were prepared for each of the 11 adhesive systems (a total of 440 specimens) and divided into groups of 10. Enamel and dentin shear bond strengths using the recommended surface conditioning treatment time and extended treatment time (60 seconds) were determined using 10 teeth for each treatment group (Table 2).

The agents were applied according to the manufacturers' directions for the recommended treatment times to the enamel or dentin bonding sites. For the three etch-and-rinse systems, the recommended etching time with phosphoric acid was 15 seconds, and this time was extended to 60 seconds for the extended treatment time groups (Table 2). For the self-etch adhesives, additional applications were used for the extended treatment time (60-second) groups (PLP [60 seconds]–initial application for 15 seconds, additional applications at 15 seconds, 30 seconds and 45 seconds). The adhesive agents were polymerized according to the manufacturers' guidelines with a Spectrum 800 Curing Unit (Dentsply Caulk) set at 600 mW/cm².

An Ultradent bonding fixture (Ultradent Products Inc) was used to bond Z100 resin composite (3M ESPE) to the conditioned enamel and dentin surfaces. The composite cylinders (2.35 mm in diameter and approximately 2 mm in length) were polymerized for 40 seconds with the Spectrum 800 unit. The bonded specimens were stored for 24 hours in distilled water at 37°C. Following storage, the specimens were loaded to failure (1 mm per minute) using an Ultradent shearing

Table 1: Adhesive Materials and	d Type of Sys	tem
Adhesive	Code	System/Etchant
AdheSE One Viva Pen (Lot #K14311)	ASE	Self-etch/One component-one-step system
Adper Prompt L-Pop (Lot #255652)	PLP	Self-etch/Two components mixed-one-step system
Adper Single Bond Plus Etchant (Lot #7JL) Adhesive (Lot #7KY)	SBP	Etch-and-rinse/35 wt% phosphoric acid
Clearfil SE Primer (Lot #00625A) Bond (Lot #00885A)	CSE	Self-etch/Primer and adhesive-two-step system
Clearfil S³ (Lot #00069A)	CS3	Self-etch/One component-one-step system
OptiBond All-In-One (Lot #2731588)	ОВА	Self-etch/One component-one-step system
OptiBond Solo Plus Etchant (Lot #2731637) Adhesive (Lot #2730675)	OBS	Etch-and-rinse/37.5 wt% phosphoric acid
Peak SE Primer (Lot #D1C8) Adhesive (Lot #A078)	PSE	Self-etch/Primer and adhesive-two-step system
Xeno IV (Lot #061102)	X4	Self-etch/One component-one-step system
Xeno V (Lot #0703004018)	X5	Self-etch/One component-one-step system
XP Bond Etchant (Lot #0610002830) Adhesive (Lot #0612001498)	XPB	Etch-and-rinse/36 wt% phosphoric acid

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.

Table 2: Treatment Times for Shear Bond Strength Testing Time (seconds) **Adhesive System** Recommended Extended ASE 30 60 PLP 15 60 SBP 15 60 **CSE** 20 60 CS3 20 60 OBA 40 60 OBS 15 60 **PSE** 20 60 X4 30 60 X5 20 60 **XPB** 60 15

Data Analysis

The data were analyzed using a three-way Analysis of Variance (ANOVA) and Fisher's LSD post hoc test. The factors for the ANOVA tests were: 1) adhesive systems, 2) surface (enamel and dentin) and 3) time (recommended and extended).

RESULTS

The three-way ANOVA for resin composite to enamel and dentin shear bond strengths (Table 3) using the 11 adhesive systems at the recommended and extended treatment times revealed the following: 1) there was a significant effect for the factors of adhesive system (p=0.000) and surface (p=0.000) but not for treatment time (p=0.115); 2) the interaction of adhesive system and surface was significant (p=0.000), but the interactions of adhesive system and (p=0.271), and surface and time (p=0.494) were

not significant; 3) the interaction of adhesive system, surface and time did not exhibit a significant effect (p=0.313).

Enamel Bonds

The enamel shear bond strengths for the recommended and extended treatment times are shown in Tables 4 and 5. The failure site locations for the enamel groups are reported as a percentage in Table 6. The etch-and-rinse systems SBP, XPB and OBS produced higher bond strengths to enamel than the self-etch systems at both the recommended and extended treatment times. The mean shear bond strength (MPa) for these systems ranged from 35.2 ± 6.2 to 40.5 ± 6.1 for the recommended treatment time and from 35.0 ± 4.5 to 44.5 ± 8.1 for the extended time. The bond strengths of two of the three etch-and-rinse systems (SBP and XPB) were significantly greater (p<0.05) than all the self-etch systems at both the recommended and extended treatment times. The bond strengths of the self-etch systems ranged from 20.9 ± 3.1 to 33.9 ± 4.6 at the recommended time and from 23.8 ± 2.9 to $32.9 \pm$ 3.6 for the extended time.

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Table 3: Multiple Analysis of Variance					
Source	Sum-of-Squares	df	Mean-Square	F-Ratio	Р
Adhesive	3821.850	10	382.185	10.991	0.000
Surface	2431.718	1	2431.718	69.931	0.000
Time	86.596	1	86.596	2.490	0.115
Adhesive*Surface	7831.903	10	783.190	22.523	0.000
Adhesive*Time	427.233	10	42.723	1.229	0.271
Surface*Time	16.275	1	16.275	0.468	0.494
Adhesive*Surface*Time	404.880	10	40.488	1.164	0.313

Table 4: Enamel Shear Bond Strength—Recommended Treatment Times				
System	Recommended Time (RT) (seconds)	Enamel Bond Strength (MPa ± SD)		
SBP	15	40.5 ± 6.1 a		
XPB	15	38.7 ± 3.7 a		
OBS	15	35.2 ± 6.2 ab		
PSE	20	33.9 ± 4.6 b		
PLP	15	30.7 ± 4.3 bc		
CSE	20	29.1 ± 2.7 c		
X5	20	27.8 ± 2.4 c		
ASE	30	27.4 ± 3.9 c		
OBA	40	26.2 ± 2.8 c		
CS3	20	25.7 ± 2.4 cd		
X4	30	20.9 ± 3.1 d		
Groups with the	same letter are not statistically different (p>0.0	5).		

Table 5: Enamel Shear Bond Strength—Extended Treatment Times				
System	Extended Time (ET) (seconds)	Enamel Bond Strength (MPa ± SD)		
SBP	60	44.5 ± 8.1 a		
XPB	60	40.9 ± 5.7 a		
OBS	60	35.0 ± 4.5 b		
PLP	60	32.9 ± 3.6 bc		
PSE	60	32.6 ± 4.1 bc		
X5	60	29.8 ± 4.9 cd		
ASE	60	29.6 ± 3.2 cd		
CSE	60	29.3 ± 5.9 cd		
CS3	60	26.4 ± 3.4 de		
X4	60	23.9 ± 3.5 e		
OBA	60	23.8 ± 2.9 e		
Groups with the	same letter are not statistically different (p>0.0	05).		

Although the enamel bond strengths tended to increase slightly when the treatment time was extended, there was not a significant increase (p>0.05) in bond strength when the recommended treatment time groups were compared to the extended treatment groups for the same system.

Dentin Bonds

The dentin shear bond strengths for the recommended and extended treatment times are shown in Tables 7

and 8. The failure site locations for the dentin groups are reported as a percentage in Table 9.

For the recommended treatment time, the etch-and-rinse system OBS had the highest bond strength (46.2 ± 7.9); however, for the extended treatment time, the self-etch adhesive system X4 had the highest dentin shear bond strength (42.2 ± 11.7). The other two etch-and-rinse systems (SBP and XPB) were in the lower three positions for dentin bond strengths for the recommended time and the

lowest two positions for the extended treatment time. OBS also produced a bond strength to dentin above 40 MPa (41.4 ± 8.0) when the treatment time was extended.

Two self-etch systems (CSE and X5) generated bond strengths above 40 MPa using the recommended time and X4 and PSE produced bond strengths above 40 MPa using the extended treatment time. PSE was the only system in the study that demonstrated a significantly (p<0.05) greater bond strength when the recommended treatment time dentin group was compared to the extended treatment time.

DISCUSSION

Clinicians are faced with many challenges when selecting a dental adhesive system to bond resin-based materials to mineralized tooth structures. The current trend in adhesive dentistry is focused on using systems that are simple to use and have minimal chair time. Earlier generation systems were typically multi-step systems that utilized phosphoric acid (PA) conditioning of the mineralized substrates before a primer and/or adhesive resin was applied. These total-etch or etch-and-rinse systems produced durable long-term bonds to enamel and ushered-in various approaches for bonding to dentin.

The acid conditioning time recommended for both enamel and dentin with phosphoric acid gels commonly employed with etch-and-rinse systems has been 15 seconds. Studies have shown that extending this time, particularly for dentin, produces morphological changes that may decrease the ability of adhesive resin to bond to the demineralized surface. 23-24

In the current study, the three etch-and-rinse systems, SBP, XPB and OBS, generated the highest bond strength to ground enamel using a 15-second condi-

System	Re	Recommended Time (RT)			Extended Treatment Time (ET)		
	Adhesive	Cohesive	Mixed*	Adhesive	Cohesive	Mixed*	
ASE	90%	10% C/E	-	100%	-	-	
PLP	80%	-	20% A/C	100%	-	-	
SBP	90%	-	10% A/C	100%	-	-	
CSE	100%	-	-	90%	-	10% A/C	
CS3	90%	10% C/E	-	80%	20% C/E	-	
OBA	100%	-	-	100%	-	-	
OBS	80%	20% C/E		80%	20% C/E		
PSE	100%	-	-	100%	-	-	
X4	100%	-	-	100%	-	-	
X5	100%	-	-	90%	10% C/E	-	
XPB	80%	20% C/E	-	70%	30% C/E	-	

Table 7: Dentin Shear Bond Strength—Recommended Treatment Times System **Extended Time (RT) Dentin Bond Strength** (seconds) (MPa ± SD) OBS 15 $46.2 \pm 7.9 a$ 40.5 ± 10.3 b CSE 20 X5 20 40.4 ± 7.8 bc X4 30 $37.7 \pm 4.3 bc$ PSE 36.4 ± 4.5 bcd 20 OBA 40 35.2 ± 5.3 cde $32.7 \pm 5.4 def$ CS3 20 ASE 30 32.0 ± 9.0 def SBP 15 31.4 ± 3.1 def PLP 15 30.3 ± 7.9 ef XPB 15 $27.9 \pm 3.6 f$

tioning time with their respective phosphoric acid etchant. Extending the conditioning time to 60 seconds did not result in significant changes (p>0.05) in bond strengths with these systems. Six of the eight self-etch systems produced bond strengths to enamel that were significantly (p<0.05) lower than the etch-and-rinse adhesives. Extending the enamel treatment time of the

Groups with the same letter are not statistically different (p>0.05).

Table 8: Dentin Shear Bond Strength—Extended Treatment Times System **Extended Time (ET) Dentin Bond Strength** (seconds) (MPa ± SD) X4 60 42.2 ± 11.7 a PSE 42.1 ± 9.7 a* 60 OBS 60 41.4 ± 8.0 ab CSE 60 $37.9 \pm 9.5 \text{ ab}$ X5 60 $36.6 \pm 7.1 bc$ OBA 60 35.7 ± 5.7 cd 60 **ASE** 34.4 ± 6.2 cde PLP 60 33.7 ± 6.8 cde CS3 60 32.2 ± 4.1 cde XPB 60 $30.5 \pm 4.4 de$ 29.5 ± 4.5 e 60

Groups with the same letter are not statistically different (p>0.05). *Significant difference (p<0.05) between RT and ET bond strength groups self-etch adhesive did not result in significant changes in bond strength.

It is interesting to note that the OBS etchand-rinse system yielded the highest bond strength to dentin when the recommended conditioning time was used, but the other two etch-and-rinse systems, SBP and XPB, ranked ninth and eleventh of the 11 systems evaluated (Table 7). When the phosphoric acid conditioning time on dentin was extended to 60 seconds, OBS ranked third and XPB and SBP ranked tenth and eleventh of the 11 systems tested (Table 8).

The results clearly demonstrate that the etch-and-rinse systems produce high bond strengths to enamel, but one of the systems

(OBS) also ranked high for bonding to dentin when compared to the self-etch systems in the study (Tables 7 and 8).

Increasing the conditioning time of enamel or dentin surfaces did not markedly change the bond strength of a resin-based material to the treated surfaces. Of all the systems tested, only PSE showed a significant

> increase (p<0.05) when the treatment time on dentin was extended to 60 seconds. While the increase in bond strength with PSE was statistically significant compared to the recommended treatment time, it may not be clinically relevant. While studies have shown that extending the conditioning time on dentin may result in morphological changes detrimental to resin infiltration into the demineralized surface with subsequent formation of an optimal hybrid zone, the current study did not demonstrate lower bond strengths to dentin when extending the conditioning time to 60 seconds.

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	Recommended Time (RT)			Extended Treatment Time (ET)		
System	Adhesive	Cohesive	Mixed*	Adhesive	Cohesive	Mixed*
ASE	90%	10% C/D	-	100%	-	-
PLP	100%	-	-	50%	50% CD	-
SBP	70%	-	30% A/C	90%	10%C/D	-
CSE	100%	-	-	100%	-	-
CS3	70%		30% A/C	90%	10% C/D	-
OBA	40%	60% C/D	-	50%	50% C/D	-
OBS	100%		-	80%	20% C/D	
PSE	80%	20% C/D	-	100%	-	-
X4	90%		10% A/C	80%	20% C/D	-
X5	40%	50% C/D	10% A/C	30%	30% C/D	40% A/C
XPB	80%	20% C/D	-	90%	10% C/D	

CONCLUSIONS

Phosphoric acid conditioning of enamel yields a higher bond strength of a resin-based composite restorative material when compared to acidic monomers. Excellent bond strengths to dentin are achieved with both phosphoric acid and acid monomer conditioning. Extending the conditioning time of both etch-and-rinse and self-etch adhesive systems on enamel and dentin does not greatly influence bond strengths.

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