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The Microtensile Bond Strength of Self-etching Adhesives to Ground Enamel

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Clinical Relevance

Bond strength to ground enamel of single component self-etching adhesive systems was lower than that of etch-and-rinse and self-etching primer systems.

SUMMARY

It is uncertain whether single-phase self-etching adhesives form bonds to enamel as reliable as those of etch-and-rinse adhesives. This study compared the microtensile bond strengths to ground enamel of three self-etching adhesive systems, a self-etching primer system and an etch-and-rinse adhesive system. Human enamel was ground flat with 320-grit silicone carbide paper. The self-etching adhesives iBond (Heraeus Kulzer), Prompt L-Pop (3M ESPE) and Xeno III (Caulk/Dentsply), the adhesive with a self-etching primer Clearfil SE Bond (Kuraray) and the etch-and-rinse adhesive Scotchbond Multipur-pose (3M/ESPE) were applied as directed, followed by a core of the same manufacturers' hybrid resin composite. A microtensile bond strength evaluation was performed after 48 hours of water storage, using untrimmed beams approximately 0.9 mm² in cross-sectional area at a crosshead speed of 0.6 mm/minute. There were no pretest failures in any group, and failures were predominately adhesive or mixed. Adhesion to enamel of Clearfil SE was not significantly different from Scotchbond Multi-Purpose, while the three self-etching adhesive systems demonstrated significantly lower bond strengths (One-way ANOVA, Tukey-Kramer Multiple-Comparison Test, *p*<0.00001).

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DOI: 10.2341/05-39

INTRODUCTION

For many years, the application of resin to enamel, which has been etched with phosphoric acid, has produced a well-sealed interface that is sufficiently strong to retain most resin composite restorations. In recent years, self-etching resins containing acidic methacrylates have been introduced in an attempt to eliminate the steps of etching and rinsing. These methacrylates are intended to produce simultaneous demineralization

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of the enamel surface and infiltration of the resin into the resulting surface topography and produce resin tags and micromechanical bonds comparable to those evident with conventional etching. Self-etching resins are of two types: self-etching primers, which are applied prior to an adhesive layer, and self-etching adhesives, which combine these two steps.

Whether these newer products produce the same superior margins along enamel walls as their etch-and-rinse predecessors is of critical importance. Clinical data is limited to the evaluation of postoperative sensitivity in Class I or II resin composite restorations, which could indicate a poor marginal seal, and to observation of the enamel margins of Class I and II and non-retentive Class V resin composite restorations in which poorly-sealed margins may discolor or become carious. Representative clinical studies have not demonstrated significant differences in the marginal discoloration of non-retentive Class V restorations between self-etching primers, self-etching adhesives and etch-and-rinse adhesives (Burrow & Tyas, 2003; Turkun, 2003; van Dijken, 2004) or among these three classes of adhesive in marginal discoloration or postoperative sensitivity in Class I and II restorations (Denehy & others, 2002; Perdigão, Geraldeli & Hodges, 2003; Yip & others, 2003).

Unfortunately, the strength of the enamel bond cannot be adequately evaluated in clinical studies of Class I, II and V restorations, because it is largely retained through mechanical retention or dentin adhesion, and the results would likely not generalize to clinical situations highly dependent on enamel adhesion, such as non-retentive Class IV restorations. Laboratory studies of bond strength to ground enamel demonstrate that self-etching resins produce lower bond strengths to ground enamel than etch-and-rinse adhesives, with self-etching adhesives lower than self-etching primers (Inoue & others, 2001; De Munck & others, 2003, 2005b). This effect is more pronounced on unground enamel (Pashley & Tay, 2001; Perdigão & Geraldeli, 2003). All of the studies previously cited include just one or two self-etching products, thus, many materials have not been evaluated.

This study compared the microtensile bond strength to ground enamel of three self-etching adhesive systems to a self-etching primer system and an etch-and-rinse adhesive system.

METHODS AND MATERIALS

The extracted teeth were collected in accordance with human subjects' regulations at the Medical College of Georgia. Twenty-five third molars stored in water saturated with thymol at 4°C were used within three months of extraction. Based on a previous study that showed no difference between facial and lingual enamel bond strengths (Shono & others, 1997), the occlusal halves of the facial and lingual surfaces of each tooth were ground flat using 320-grit silicon carbide paper under running water at 60 rpm on a polishing machine (Ecomet 3, Buehler, Ltd, Lake Bluff, IL, USA). These flat areas were prepared in a plane estimated to be parallel to the underlying dentino-enamel junction.

Four commercial self-etching resin systems were selected for the study: Adper Prompt L-Pop (3M ESPE, St Paul, MN, USA), iBond GI (Heraeus Kulzer, Inc, Armonk, NY, USA), Xeno III (Caulk/Dentsply, Milford, DE, USA) and Clearfil SE Bond (Kuraray Co, Ltd, Osaka, Japan). The first three are self-etching adhesives, while the fourth is a self-etching primer to which a layer of adhesive is applied. The etch-and-rinse system Adper Scotchbond Multi-Purpose (3M ESPE) was used as a control. Each adhesive system was applied according to the manufacturer's instructions and is listed in Table 1. For the self-etching resins, in order to completely evaporate solvents, air drying was continued for approximately 15 seconds past a "dried" appearance of the resin layer. The sample size was five teeth for each adhesive system.

Following application of the adhesives, approximately 6-mm thick cores of one of the same manufacturer's hybrid resin composites were built incrementally on the flattened etched enamel of each tooth. The resin composites used were Z 250 (3M ESPE), Venus (Heraeus Kulzer), TPH Spectrum (Caulk/Dentsply) and AP-X (Kuraray). Increment thickness was limited to 2 mm, and curing was accomplished from all directions using a fast halogen light source (VIP, BISCO, Inc, Schaumburg, IL, USA) for a total of 5 minutes of curing per specimen. Light output of 600 mW/cm^2 was verified throughout the study, using the unit's built-in radiometer.

After storage in distilled water at 37°C for 24 hours, the restored teeth were hemisected occluso-gingivally along the mesio-distal axis. Each half-tooth was then sectioned facio-lingually into serial slabs approximately 0.9-mm thick using a slow-speed water-cooled diamond saw (Isomet, Buehler, Ltd). Each slab was then sectioned perpendicular to the flat-ground enamel into resin composite, enamel and dentin beams approximately 0.9 x 0.9 mm in cross section according to the "non-trimming" version of the microtensile test (Shono & others, 1999). Each tooth yielded 5 to 10 beams for bond strength evaluation.

After 24 hours of additional storage in distilled water at 37°C, the dimensions of each beam were measured with a digital caliper (Absolute Digimatic Model CD 6" CS, Mitutoyo Corp, Kanagawa, Japan) accurate to ± 5 µm. The beams were then affixed to a Ciucchi device (Kuraray) using Zapit cyanoacrylate glue (Dental Ventures of America, Corona, CA, USA) and tested to failure under tension in a universal testing machine (Vitrodyne V1000, Chatillon, Largo, FL, USA) at a crosshead speed of 0.6 mm/minute. The types of failure

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Table 1: *Manufacturer, Instructions/Technique, Composition, and pH for Each Adhesive System*

	Technique	Composition	pH
Clearfil SE Bond Lot #61129			
Primer	Apply 20 seconds, air dry	10-methacryloyloxy decyl dihydrogenphosphate (MDP) 2-hydroxyethyl methacrylate (HEMA), water	1.9
Bond	Apply, light cure	MDP, Bis-GMA, HEMA	
Clearfil AP-X Lot #801AA	(Resin Composite)		
(Kuraray, Osaka, Japan)			
Adper ScotchBond MultiPurpose Plus Lot #3AH/3NG			
Etch	15 seconds, rinse	37% phosphoric acid	1
Primer	Apply, dry	HEMA, water Copolymer of acrylic and itaconic acids	
Adhesive	Apply, light cure	Bisphenol A diglycidyl ether dimethacrylate, HEMA	
Filtek Z 250 Lot #20030509	(Resin Composite)		
(3M ESPE, St Paul, MN, USA)			
Xeno III Lot #0303001499			
	Mix, apply thick coating, 30 seconds, air dry, light cure	HEMA, water, ethanol, 2,6-Di-tert-butyl-p hydroxy toluene Pyro-EMA-SK, PEM-F, Urethane dimethacrylate, EPD, p-diethyl amine ethyl benzoate	1
TPH Spektrum Lot #0301281	(Resin Composite)		
(Caulk/Dentsply Milford, DE, USA)			
Adper Prompt L-Pop Lot #149354			
	Mix, apply with agitation 15 seconds, air dry, reapply, light cure	methacrylated phosphoric acid esters, water Bisphenol A diglycidyl ether dimethacrylate, HEMA	0.8
Filtek Z 250 Lot #20030509 (3M ESPE, St Paul, MN, USA)	(Resin Composite)		
iBond GI Lot #010042			
	Apply three coats with agitation, 30 seconds, air dry, light cure	4-META, Urethane dimethacrylate, Glutaraldehyde, acetone, water	1.6
Venus Lot #180026	(Resin Composite)		
(Heraeus Kulzer, Armonk, NY, USA)			

were observed at 2.5x magnification and categorized as adhesive, cohesive or mixed.

In addition to microtensile testing, fractured interfaces from each group with high, average and low bond strengths were selected for microscopic evaluation. The enamel of five additional teeth was also ground as previously described, and the etching component of each adhesive system was applied as directed, except for the self-etching resins, which were removed with copious absolute ethanol instead of being light cured. These teeth and the selected fractured beams were then dehydrated in alcohol and sputter-coated. The fractured beams were examined at 80x magnification, and the etched enamel surfaces were examined at 5000x magnification in a SEM (Model XL30 FEG, Philips Electronics, Eindhoven, The Netherlands) operated at 10kV.

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Bond strength was calculated and data obtained for the five subgroups were statistically analyzed using a one-way ANOVA, followed by a Tukey-Kramer Multiple Comparison Test at a 5% confidence level.

RESULTS

Data for each subgroup were found to be normally distributed. Adhesion to ground enamel of the Clearfil SE self-etching primer system was not significantly different from Scotchbond Multi-Purpose, while the three self-etching adhesive systems demonstrated significantly lower bond strengths, with no significant differences among them (p<0.0001). There were no pretest failures in any group. Overall, failures were predominately adhesive or mixed. The incidence of cohesive failures in dentin, enamel or along the dentino-enamel junction was more common with increasing bond strength. Complete results are presented in Table 2.

SEM evaluation showed that conventional phosphoric acid etching produced the greatest topographical changes in the enamel surface (Figure 1). Among the self-etching resins, there appeared to be no correlation between pH and increased surface topography or between microscopic appearance of the etched enamel

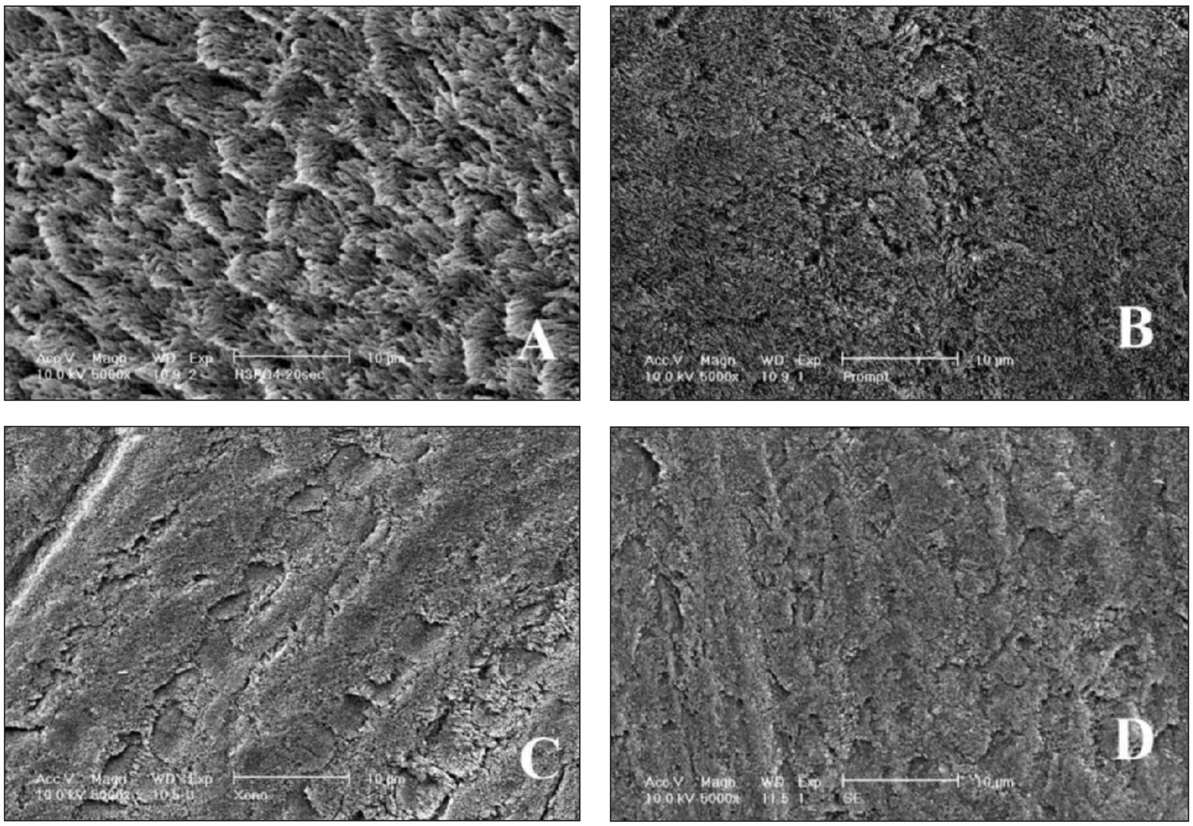

Figure 1: *SEM images of enamel topography produced by (A) conventional phosphoric acid etching, (B) and (C) two relatively acidic self-etching adhesives (Prompt L-Pop and Xeno III, respectively), which show differing amounts of surface etching, and (D) a relatively mild pH self-etching primer (SE Bond). There was no apparent correlation between pH, surface topography and enamel bond strength (original magnification = 5000x).*

Table 2: *Resin/Enamel Microtensile Bond Strengths*

	(teeth)	(beams)	Bond Strength-MPa (SD)	Failure Type		
	n	n		a	m-e	c-de
Clearfil SE	5	37	57.2 (13.1)[a]	16	9	12
ScotchBond MultiPurpose	5	34	52.2 (11.3)[a]	16	4	14
Xeno III	5	43	38.4 (11.0)[b]	24	8	11
Prompt L-Pop	5	47	37.2 (11.2)[b]	26	14	7
iBond	5	29	34.9 (14.0)[b]	20	4	5

Groups with different letters significantly different, one-way ANOVA, Tukey-Kramer Multiple-Comparison Test, p<0.05
(Failure types: a = adhesive; m-e = mixed-enamel; c-de = cohesive-dentin/enamel)

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and bond strength. For all the adhesive systems, there was no obvious correlation between bond strength and microscopic fracture pattern. Fractured interfaces in general appeared to have failed primarily through the unfilled resin layer, with remnants of resin composite attached to the tooth structure for most specimens, even those judged visually to have failed adhesively.

DISCUSSION

For resin-based adhesives, the primary mechanism of adhesion to enamel has long been micromechanical, with acid etching greatly increasing the surface area, and a stable, well-polymerized resin infiltrated into the surface topography. This can be augmented by secondary bonds, which have formed between hydrophilic resins and enamel.

The results of this study are in accordance with earlier reports, in that single-phase self-etching resins do not produce as good a bond with enamel as etch-and-rinse adhesives. In both this study and in an earlier report (De Munck & others, 2005a), there is little correlation between the pH of the adhesive and bond strength. Since some self-etching resins, such as Prompt L-Pop, appear to adequately etch enamel but yield relatively low bond strengths, it appears that the resin infiltrated into the enamel surface does not form a strong polymer. Studies of such products have demonstrated the presence of water-rich phases that are detrimental to strength and stability (Van Landuyt & others, 2005). Conversely, an adhesive such as Clearfil SE can produce relatively high bond strengths with minimal changes in surface topography compared to etching, which must result from additional secondary bonding. Although promising, it is not known whether this greater reliance on secondary bonds will equal the long-term stability of the micromechanical bonding of conventional etching. The authors believe this should be addressed through clinical studies that focus on enamel bond strength and that the use of self-etching resins alone is contraindicated in clinical situations where maximum enamel adhesion is needed.

For this study, the authors elected to pair only adhesives and resin composites from the same manufacturer for each group of specimens, because this would be the most likely way that resins would be purchased by clinicians. This also eliminated the risk of incompatibility between any of the adhesives and a single resin composite, but dictated that the study was an evaluation of adhesive systems rather than of the adhesives themselves. Like all adhesives and resin composites that evaluate microtensile bond strength to enamel, this study is limited by adhesives approaching and exceeding the tensile strength of enamel. This produces failures at other sites in the specimens instead of the interface being studied, particularly through the enamel itself and along the dentino-enamel junction, which occurred about half of the time for the two materials that demonstrated the highest bond strengths. The value of the SEM evaluation of fractured interfaces is also limited by this, as there is little to be gained from observing cohesive fractures through tooth structure.

CONCLUSIONS

Only the adhesive system that used a self-etching primer demonstrated an enamel bond strength comparable to the etch-and-rinse adhesive system.

Acknowledgements

This work was supported in part by grants R01 DE 014911 and R01 DE 015306 (PI: David H Pashley) from the NIH/National Institute of Dental and Craniofacial Research and by Heraeus-Kulzer, Inc.

(Received 3 March 2005)

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teeth restored before restoration and at the end of the study.

At 48 months, 66% of restorations placed with Elite Fil in carious and non-carious lesion were present. Retention rates for Elite Flo restorations were 65% and 47% for the carious and non-carious lesions, respectively. Post-operative sensitivity was absent in all patients. Vitality was maintained on all teeth treated at 48 months. A decrease in quality of the restorations was recorded with USPHS criteria, with no statistically significant difference between materials tested.

The 2 materials examined in this study performed poorly in the restorations of cervical lesions. Some have suggested that low elastic modulus materials are more compatible with cervical tooth flexure. Interestingly, Elite Flo, with a low modulus of elasticity, performed no better than a traditional resin-based hybrid composite. In addition, its rate of failure increased when used in non-carious cervical lesions where abfraction is often a factor.

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CORRECTION

In *Operative Dentistry* **31-3**, the article, "The Microtensile Bond Strength of Self-etching Adhesives to Ground Enamel," was incorrectly identified as DOI: 10.2341/0539. The correct DOI is: 10:2341-0538.

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