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Effect of Selective Etch on the Bond Strength of Composite to Enamel Using a Silorane Adhesive

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Clinical Relevance

The selective etch of enamel with phosphoric acid may improve the bond strength of silorane adhesives.

SUMMARY

An improvement in bond strength to enamel has been demonstrated with the use of phosphoric acid prior to bonding with self-etch methacrylate-based adhesive agents. No research has evaluated the effect of phosphoric-acid etching of enamel with a newer self-etch silorane adhesive. The purpose of this study was to evaluate the shear-bond strength of composite to enamel using the self-etch silorane adhesive compared to other self-etching methacrylate-based adhesives, with or without a separate application of phosphoric acid. Bovine incisors were sectioned using a diamond saw and mounted in plastic pipe. The bonding agents were applied to flattened enamel surfaces with or without the application of 35% phosphoric acid. The bonded tooth specimens were inserted beneath a mold, and composite was placed incrementally and light cured. The specimens were stored for 24 hours and six months in water and tested in shear. Data were analyzed with a three-way analysis of variance (ANOVA) to evaluate the effects of surface treatment, adhesive agent, or time on the bond strength of composite to bovine enamel (α=0.05). Significant differences were found between the groups based on surface treatment ($p<0.01$) or adhesive agent ($p<0.01$), but not on time ($p=0.19$), with no significant interactions ($p>0.14$). Phosphoric-acid etching of bovine enamel significantly increased the bond strength of the self-etch methacrylate and the silorane adhesives. The methacrylate-based adhesives had significantly greater bond strength to enamel than the silorane adhesive.

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INTRODUCTION

Enamel and dentin adhesive bonding agents have changed significantly over the past few decades with the trend towards simplification and ease of use and placement. Spanning seven generations, these materials require anywhere from one to three steps. Currently, there are two major categories of methacrylate-based adhesives: etch-and-rinse and self-etch.[1] Etch-and-rinse adhesives have been available since the early 1990s and are divided into three- and two-step systems. The three-step adhesives require a separate acidic conditioner, primer, and bonding resin. To reduce the placement time and complexity of three-step adhesives, manufacturers combined the primer and resin components to create a simplified two-step system. Self-etch adhesives were more recently introduced and are divided into two- and one-step systems. Two-step self-etch adhesives combine the acidic conditioner with the primer in the initial step and use a bonding resin in the second step. Even further reduction in the number of steps came with the introduction of one-step self-etch adhesives with the acidified primer and bonding resin placed in one simplified step.[1,2]

With the evolution of self-etch adhesive systems, there was a concern that the manufacturers were sacrificing the strength of the bond to enamel by using a weaker acidified primer in order to eliminate one step in the procedure. Laboratory studies have demonstrated that self-etch adhesives produce lower bond strength to enamel compared to etch-and-rinse adhesives.[3] More significantly, clinical studies have shown significantly less marginal defects and staining with selective etching of enamel with phosphoric acid when using a self-etch adhesive agent.[4] Van Meerbeek and Yoshihara[7] state that the use of phosphoric acid on enamel currently remains necessary to maintain the most durable bond to the interface and to protect the more vulnerable bond to dentin against degradation.

A completely different alternative to conventional methacrylate-based composites and adhesives has been introduced recently that is based on a ring-opening silorane (low-shrinking) monomer (Filtek LS, 3M ESPE, St Paul, MN, USA) formed by the chemical building blocks of siloxanes and oxiranes. According to the manufacturer, siloxane is characterized by hydrophobicity, and the oxirane polymer is known for low shrinkage.[8] In the polymerization process of silorane composites, the ring-shaped molecules open and approach the "neighbor" molecules. The expansion of the ring before polymerization has been shown to decrease the overall polymerization shrinkage to an average of 1.0% - 1.5%.[9] On the other hand, methacrylate-based resins connect by shifting closer together in a linear response, resulting in a greater loss of volume.[8] In a recent study, it was found that the silorane-based composite, Filtek LS, had the lowest polymerization shrinkage when compared to methacrylate-based composites, even though they shared similar physical properties.[9] The LS System Adhesive is a two-step self-etch bonding agent developed exclusively to work with the new silorane restorative composite. The LS System Adhesive Primer is hydrophilic and adheres to tooth structure. The LS System Adhesive Bond adheres to both the hydrophilic primed tooth structure and to the hydrophobic silorane-based Filtek LS composite.[9] The Filtek LS System Adhesive belongs to a similar two-step, self-etch adhesive strategy as other two-step self-etch methacrylate-based adhesives, such as Clearfil SE Bond (Kuraray, New York, NY, USA), and OptiBond XTR (Kerr, Orange, CA, USA). No research has been done evaluating the effect of selective etching using phosphoric acid on the bond strength of the new self-etch silorane adhesive and the unique low-shrinking Filtek LS silorane restorative material to enamel.

Recent systematic reviews of the literature reported the average annual failure rate of noncarious cervical lesions bonded with different dental adhesives and restored with composite resin. The mild two-step self-etch adhesives (eg, Clearfil SE Bond) were found to be the most effective clinically.[10,11] Clearfil SE Bond is composed of a mildly acidic self-etch primer containing 10-MDP (methacryloyloxydecyl dihydrogen phosphate), the active acidic functional monomer for demineralizing the enamel and the dentin. The acidic monomer is not as strong as phosphoric acid.[6] Although no significant difference in retention rates in restored noncarious lesions using Clearfil SE Bond with or without the selective etching of enamel were found in a recent 13-year clinical study by Peumans and others,[4] less marginal discoloration and better marginal integrity were found in the selective etch group. OptiBond XTR is a recently introduced two-step, self-etch adhesive that employs Kerr's ternary solvent system and a filled adhesive with an optimized formulation to reportedly produce outstanding adhesion for direct and indirect procedures.[12] Limited research is available evaluating this new two-step self-etching adhesive.

The purpose of this study was to evaluate the shear bond strength of composite to enamel using the two-step, self-etch silorane adhesive (LS System Adhesive) compared to other two-step self-etch methacrylate-based adhesives (Clearfil SE Bond, OptiBond XTR) with and without a separate application of phosphoric acid. The null hypothesis tested was that there would be no difference in shear bond strength of composite restorative material to bovine enamel based on 1) surface treatment, 2) adhesive agent, or 3) time.

METHODS AND MATERIALS

One hundred twenty freshly extracted bovine incisors (Animal Technologies, Tyler, TX, USA) were stored in 0.5% chloramine-T and used within three months of purchase. The crowns were sectioned in a buccolingual direction at the cementoenamel junction to remove the root using a water-cooled diamond saw (Isomet 5000, Buehler, Lake Bluff, IL, USA). Retention cuts were placed in the lingual surface of the crown to prevent tooth dislodgement during shear testing. The teeth were mounted in polyvinylchloride pipe using dental stone and bis-acryl resin. After the stone and bis-acryl resin was set, a small area of the exposed enamel was flattened using a diamond wheel bur mounted in a drill press (Proxxon, Hickory, NC, USA) and smoothed using 600-grit silicon-carbide paper (Norton Abrasives, Worchester, MA, USA).

The enamel specimens were divided into six groups with 20 specimens each in order to compare the shear bond strength of the different adhesives and surface treatments over time. The adhesives were applied as a two-step, self-etch bonding agent with and without the selective enamel-etch technique. For the groups using a selective enamel-etch technique, 35% phosphoric-acid gel etchant (Kerr) was applied to the enamel for 15 seconds, rinsed with water for 15 seconds, then lightly air dried for three seconds. The adhesives were then applied according to the manufacturer's instructions (Table 1) and light cured with a visible light-curing unit (Bluephase G2, Ivoclar Vivadent, Amherst, NY, USA). Irradiance was determined with a radiometer (Bluephase Meter, Ivoclar Vivadent) and was considered acceptable if greater than 1000 mW/cm^2.

After application of the adhesive, the bonded specimens were placed in a jig (Ultradent Products, South Jordan, UT, USA) and secured beneath a white plastic mold. The bonded area was limited to the 2.4 mm circle determined by the mold. The teeth treated with LS System Adhesive were restored with Filtek LS composite, and the teeth treated with Clearfil SE Bond and OptiBond XTR were restored with Filtek Supreme Ultra (3M ESPE) composite. All materials were used according to manufacturer's instructions. The composite restorative materials were incrementally placed in two increments to a height of 3-4 mm. Each layer was polymerized as recommended by the manufacturer with the visible light curing unit. The specimens were stored for 24 hours and 6 months in distilled water at 37°C in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL, USA).

The bond strength was tested in shear mode with a knife-edge blade in a universal testing machine (Model 5943, Instron, Norwood, MA, USA) at a crosshead speed of 1 mm/min until failure. Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure in newtons divided by the specimen cross-sectional surface area. The mean and standard deviation were determined per group. Following testing, the specimens were examined under a 10× microscope to determine the fracture mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the enamel or composite, or 3) mixed (combination of adhesive and cohesive) in enamel or composite. Data were analyzed with a three-way analysis of variance (ANOVA) with Tukey post-hoc test to evaluate the effects of adhesive agent, surface treatment, or time on the bond strength of composite to bovine enamel ($\alpha=0.05$).

RESULTS

Significant differences were found between the groups based on surface treatment ($p<0.01$) or adhesive agent ($p<0.01$), but not on time ($p=0.19$), with no significant interactions ($p>0.14$) as shown in Table 2. The Clearfil SE Bond and OptiBond XTR groups had significantly higher bond strengths to enamel than the LS System Adhesive group as shown in Figure 1. Etching the enamel with phosphoric acid prior to placement of the adhesives resulted in significantly higher bond strength. There was no significant difference in bond strength with any of the adhesives between 24 hours and six months of storage in water. Etching of the enamel was associated with more mixed or cohesive fractures (Figure 2).

DISCUSSION

An improvement in bond strength to enamel has been demonstrated with the use of phosphoric acid prior to bonding with self-etch methacrylate-based adhesive agents. No research has evaluated the effect of phosphoric-acid etching of enamel with the newer self-etch silorane adhesive, Filtek LS System Adhesive. The unique curing mechanism and chemistry of Filtek LS required the development of a dedicated adhesive. Filtek LS must be used with the LS System Adhesive, a two-step, self-etch bonding

Table 1: *Composition and Technique Guide for Filtek LS System Adhesive, Clearfil SE Bond, and OptiBond XTR as reported by the Manufacturers*

Adhesive System	Component	Composition	Application
LS System Adhesive	Primer	Hydroxyethyl methacrylate Bisphenol A glycidyl methacrylate Water Ethanol Phosphoric acid-methacryloxy-hexylesters Silane treated silica Hexanediol dimethacrylate Copolymer of acrylic and itaconic acid (Dimethylamino) ethyl methacrylate Camphorquinone Phosphine oxide	Apply with brush for 15 s Expose to air stream Cure 10 s
	Adhesive	Substituted dimethacrylate Silane treated silica Triethylene glycol dimethacrylate Phosphoric acid methacryloxy-hexylesters Camphorquinone Hexanediol dimethacrylate	Agitate bottle Apply to tooth surface Expose to air stream Cure 10 s
Clearfil SE Bond	Primer	Hydroxyethyl methacrylate Methacryloyloxydecl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Camphorquinone Water Accelerators Dyes Others	Apply for 20 s on tooth surface Evaporate volatile ingredients with a mild air stream
	Adhesive	Bisphenol A glycidyl methacrylate Hydroxyethyl methacrylate Methacryloyloxydecyl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Colloidal silica Camphorquinone Initiators Accelerators Others	Apply to tooth surface Expose to air stream Cure 10 s
OptiBond XTR	Primer	Glycerol phosphate dimethacrylate Hydrophilic co-monomers Camphorquinone Water Ethanol Acetone	Apply for 20 s; dry thin for 5 s
	Adhesive	Resin monomers Hydroxyethyl methacrylate Camphorquinone Inorganic fillers Ethanol	Apply with light brushing motion for 15 s; light cure for 10 s

Table 2: *Mean Shear Bond Strength and Statistical Analysis**

Bonding Agent	Shear Bond Strength (SD), MPa				
	Self-Etch		Etch-and-Rinse		Bonding Agent Total
	24 Hours	6 Months	24 Hours	6 Months	
Clearfil SE Bond	20.9 (2.9)	21.9 (4.8)	29.8 (4.2)	28.8 (6.5)	25.3 (4.6)^{B}
LS System Adhesive	16.3 (3.8)	14.2 (3.3)	18.9 (3.5)	20.4 (4.8)	17.5 (3.9)^{A}
OptiBond XTR	19.5 (2.6)	25.4 (7.3)	29.5 (3.2)	30.9 (7.9)	26.3 (5.3)^{B}
Surface treatment total	19.7 (5.6)^{a}		26.4 (7.0)^{b}		

**Groups with the same upper case letter per column or lower case letter per row are not significantly different ($p>0.05$).*

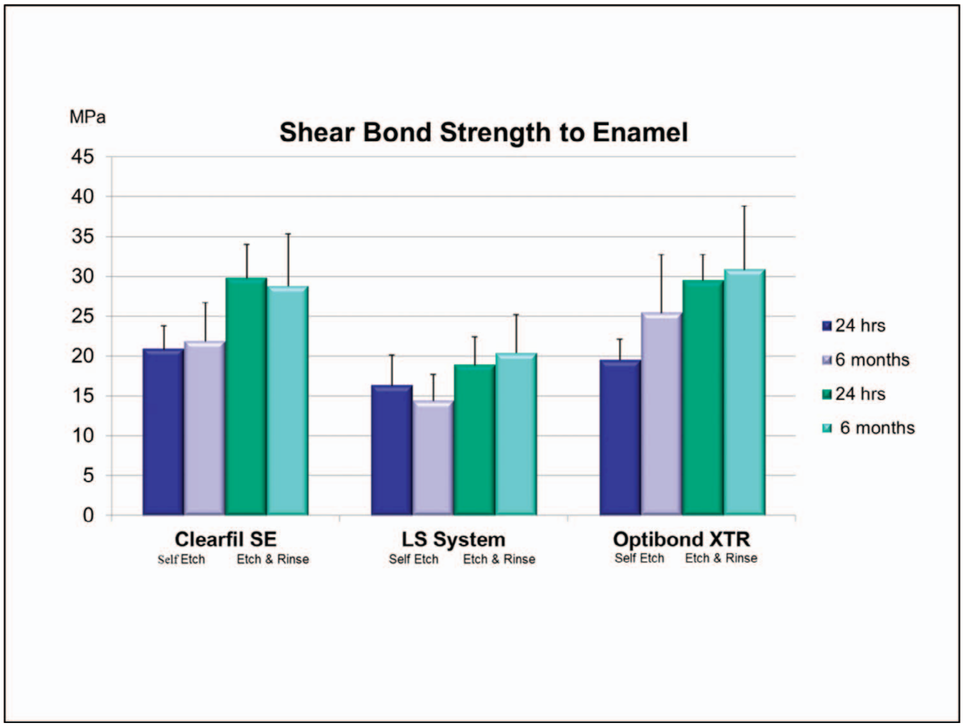

Figure 1. *Mean shear bond strength of adhesives applied in self-etch and etch-and-rinse modes over time. Error bars represent 1 standard deviation.*

agent. Self-etch adhesives have gained significant popularity recently due to their ease of use compared to the older generations of etch-and-rinse adhesives. However, the etch-and-rinse adhesives have been considered the "gold standard" when bonding to enamel due to the predictable etch pattern created from the relatively lower pH of the phosphoric acid.[13]

In this study, a significant difference was found based on surface treatment. Etching with phosphoric acid resulted in significantly higher bond strength to

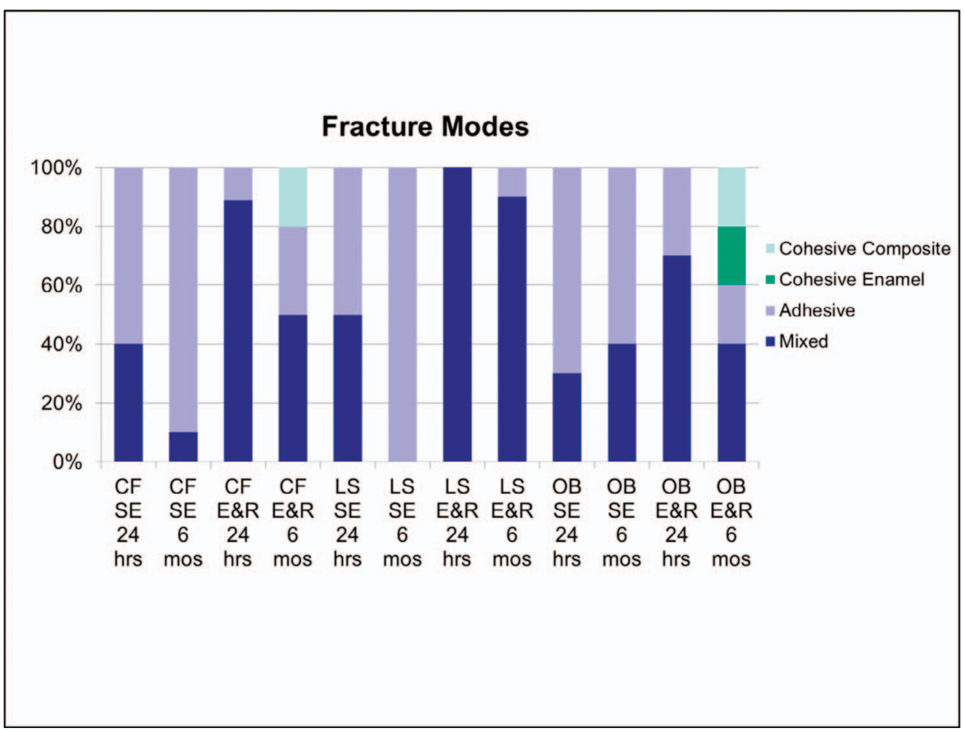

Figure 2. *Fracture mode of adhesive bonding agents over time.*

enamel, and therefore the first hypothesis was rejected. Also, phosphoric-acid etching of enamel resulted in more mixed and cohesive fractures, which is associated with a more stable interface.[14] Etch-and-rinse systems are characterized by an initial etching step with a stronger acid, usually 32%-37% phosphoric acid at a pH of approximately 0.9, to completely etch enamel and dentin. The alternative self-etch approach produces a shallower and less retentive enamel-etching pattern.[6] The degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer. Although not evaluated in this study, some strong self-etch adhesives (pH<1) create demineralized enamel that is similar to that created by phosphoric acid; whereas, some mild self-etching adhesives have demonstrated reduced bond strength to enamel in laboratory studies.[15] Although stronger self-etch adhesives may improve the bond to enamel, their high acidity may lead to overetching of dentin and subsequent loss of mineral content. Mild self-etch adhesives, as used in this study, may form ionic bonds between their functional monomers and the calcium of hydroxyapatite in dentin.[7] To maximize the bond to enamel without compromising the bond to dentin, selective etching of enamel with phosphoric acid prior to the application of a mild self-etch adhesive may be the most efficacious approach to adhesion to tooth structure.[7,16] According to Peumans and others,[4] selective etching of enamel may reduce marginal defects that can negatively influence the esthetic performance of direct composite restorations, especially in large visible enamel surfaces such as Class IV restorations.

A significant difference based on adhesive agent was also found in this study. The use of Clearfil SE Bond or OptiBond XTR resulted in significantly greater bond to enamel than the LS System Adhesive, and therefore the second null hypothesis was also rejected (Figure 1). Clearfil SE Bond and OptiBond XTR were chosen specifically for this study based on the fact that they are two-step self-etch adhesives similar to the LS System Adhesive. Clearfil SE Bond has been shown in laboratory studies to have consistently stronger bond strengths to enamel than other two-step, self-etch adhesive agents.[10,11] No clinical studies are available evaluating the clinical performance of the relatively newer OptiBond XTR adhesive. However, a recent laboratory study by Meharry and others[17] found no significant difference in bond strength to enamel between Clearfil SE Bond and OptiBond XTR similar to the results of this study. The LS System Adhesive has a higher pH (2.7) than Clearfil SE Bond (2.0) or OptiBond XTR (1.6).[8] The relatively lower pH of Clearfil SE Bond and OptiBond XTR may have contributed to their stronger bond to enamel compared to the LS System Adhesive.[17] OptiBond XTR is considered a mild self-etch adhesive with an initial pH of 2.4; but the pH drops to 1.6 during the primer application. The primer contains a three-part solvent of water, ethanol, and acetone that reportedly enhances the self-etch capability by facilitating penetration of the hydrophilic monomers into tooth structure.[17] Clearfil SE Bond contains the functional monomer, 10-MDP, vs the LS System Adhesive's Vitrebond copolymer, although both are stated to have the same purpose of bonding to tooth structure.[13] However, the monomer 10-MDP contains phosphate groups capable of producing ionic bonds with calcium in hydroxyapatite.[18]

No significant difference was found in bond strength based on storage time, and therefore the third null hypothesis was not rejected. Water sorption, in addition to masticatory stresses, salivary enzymes, and changes in temperature and pH, are thought to be major factors in destabilizing the adhesive-tooth interface over time.[17] Simplified bonding agents, with the hydrophilic primer and adhesive combined together, are potentially more susceptible to hydrolytic degradation over time. However, the adhesive bonding agents evaluated in this study are less susceptible to hydrolysis over time due to the separate primer and adhesive application.[2] Six months is a relatively short time to evaluate the effects of hydrolysis on bond strength. Longer storage times may be necessary to elucidate any changes in the more stable adhesive agents evaluated in this study.

Very limited *in vivo* research has been published evaluating the clinical performance of Filtek LS. The majority of short-term clinical studies have found similar results between Filtek LS and methacrylate-based materials in Class I and Class II restorations, suggesting no real benefit in the use of the low-shrinkage silorane-based composite restorative material.[13,19-23] Although the polymerization shrinkage of Filtek LS has been shown in laboratory studies to be the lowest among composite-based restorative materials, there is evidence to suggest that the higher elastic modulus or stiffness of Filtek LS may increase the polymerization shrinkage stress generated during placement in cavity preparations. Also, recent studies by Baracco and others[13,22] found that restorations restored using an etch-and-rinse adhesive and methacrylate-based composite had better marginal adaptation than those restored with Filtek LS and the self-etching LS System Adhesive. No research has been completed evaluating the effect of selective etch on the clinical performance of Filtek LS with LS System Adhesive. The results of this laboratory study suggest that it may be advantageous to etch the enamel with phosphoric acid prior to the placement of the unique self-etch LS System Adhesive and Filtek LS composite restorative material. *In vivo* studies are necessary to determine if the greater bond strengths achieved with phosphoric-acid etching of the enamel prior to the placement of LS System Adhesive is clinically significant.

CONCLUSION

Phosphoric-acid etching of bovine enamel significantly increased the bond strength of the two-step, self-etch methacrylate and silorane adhesives. The methacrylate-based adhesives had significantly greater bond strength to enamel than the silorane adhesive.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of JBSA-Lackland, TX Institutional Review Board. The approval code issued for this study is FWH20130020A.

Disclosure

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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