Enamel and Dentin Bond Strengths of a New Self-Etch Adhesive System

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ABSTRACT

Statement of Problem: Self-etch adhesives typically are mildly acidic and therefore less effective than etch-and-rinse adhesives for bonding to enamel.

Purpose: The purpose of this study was to evaluate the enamel and dentin shear bond strengths of a new two-step self-etch adhesive system, OptiBond XTR (Kerr Corporation, Orange, CA, USA).

Materials and Methods: The labial surfaces of 80 bovine teeth were ground to create flat, 600-grit enamel or dentin surfaces. Composite was bonded to enamel or dentin using the new two-step self-etch system or a three-step etch-and-rinse (OptiBond FL, Kerr), two-step self-etch (Clearfil SE Bond, Kuraray America, Houston, TX, USA), or one-step self-etch adhesive (Xeno IV, Dentsply Caulk, Milford, DE, USA). Following storage in water for 24 hours, shear bond strengths were determined using a universal testing machine. The enamel and dentin data sets were subjected to separate analysis of variance and Tukey's tests. Scanning electron microscopy was used to evaluate the effects of each system on enamel.

Results: Mean shear bond strengths to enamel ranged from 18.1 MPa for Xeno IV to 41.0 MPa for OptiBond FL. On dentin, the means ranged from 33.3 MPa for OptiBond FL to 47.1 MPa for Clearfil SE Bond. OptiBond XTR performed as well as Clearfil SE Bond on dentin and as well as OptiBond FL on enamel. Field emission scanning electron microscope revealed that OptiBond XTR produced an enamel etch pattern that was less defined than that of OptiBond FL (37.5% phosphoric acid) but more defined than that of Clearfil SE Bond or Xeno IV.

Conclusion: The new two-step self-etch adhesive system formed excellent bonds to enamel and dentin in vitro.

CLINICAL SIGNIFICANCE

OptiBond XTR, a new two-step self-etch adhesive system, is a promising material for bonding to enamel as well as to dentin.

(J Esthet Restor Dent 23:390–398, 2011)

INTRODUCTION

Adhesion of resin-based materials to dentin requires three steps: etching (conditioning), priming, and bonding.[1] The first group of contemporary dentin adhesive systems was introduced about two decades ago and was based on the "total-etch" concept using phosphoric acid to etch dentin and enamel simultaneously. These systems, now commonly described as three-step etch-and-rinse

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The study was reported at the 89th General Session of the International Association for Dental Research, San Diego, CA, USA, March 18, 2011.

 DOI 10.1111/j.1708-8240.2011.00465.x

adhesives, have proved to be very versatile and clinically successful.[1,2]

Although many experts consider a three-step etch-and-rinse system such as OptiBond FL (Kerr Corporation, Orange, CA, USA) to be the "gold standard" for bonding,[3] clinicians frequently prefer simplified products. The simplified options include the two-step etch-and-rinse systems that combine priming and bonding functions in a single solution, the two-step self-etch systems that combine the conditioning and priming functions, and the all-in-one adhesives that deliver the three essential components in a single solution.[1,2]

One of the clinically proven approaches for simplification is epitomized by Clearfil SE Bond (Kuraray America, Houston, TX, USA), a two-step system that includes a self-etch primer and a separate adhesive.[4] Clearfil provides a strong, stable bond to dentin,[5,6] but is only mildly acidic and therefore provides only a moderate bond to enamel or even a weak bond if the enamel has not been instrumented in some way.[7,8] In fact, some experts recommend selective phosphoric acid-etching of enamel margins before application of the Clearfil self-etch primer.[9] In contrast, the manufacturer of a new two-step self-etch primer system, OptiBond XTR (Kerr), claims that it effectively etches and bonds not only to dentin but also to enamel, thus eliminating the need for a separate acid-etching step.

The purpose of this study was to determine the shear bond strengths (SBS) of OptiBond XTR to enamel and dentin. For comparison, OptiBond FL, Clearfil SE Bond, and Xeno IV (Dentsply Caulk, Milford, DE, USA) were tested on the same substrates. These three materials used for comparison purposes were chosen as representative of the three-step etch-and-rinse, two-step self-etch, and one-step self-etch adhesive systems, respectively. The specific hypothesis tested was that OptiBond XTR would have enamel and dentin bond strengths similar to those of established etch-and-rinse and self-etch adhesives.

TABLE 1. Adhesives tested in the study

Material	Major Components
OptiBond XTR Kerr Corporation two-step self-etch	Primer: GPDM, hydrophilic co-monomers, water/ethanol, acetone Adhesive: resin monomers, inorganic fillers, ethanol
OptiBond FL Kerr Corporation three-step etch-and-rinse	Etchant: 37.5% phosphoric acid Primer: HEMA, GPDM, PAMM, ethanol, water Adhesive: TEGDMA, UDMA, GPDM, HEMA, Bis-GMA, hydrophobic dimethacrylate, fillers (15%)
Clearfil SE Bond Kuraray Dental two-step self-etch	Primer: 10-MDP, HEMA, water Adhesive: Bis-GMA, 10-MDP, HEMA, hydrophobic dimethacrylate, colloidal silica
Xeno IV Dentsply Caulk one-step self-etch	Adhesive: methacrylate resins, UDMA, PENTA, acetone

Bis-GMA = bisphenol A diglycidyl methacrylate; GPDM = glycerol phosphate dimethacrylate; HEMA = 2-hydroxyethyl methacrylate; MDP = methacryloyloxydecyl dihydrogen phosphate; PAMM; phthalic acid monoethyl methacrylate; PENTA = dipentaerythritol pentaacrylate phosphate; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate.

MATERIALS AND METHODS

Eighty bovine incisors were used in this study. The teeth were debrided and examined to ensure that they were free of defects. Crowns were separated from the roots using an Isomet diamond saw (Buehler Ltd., Lake Bluff, IL, USA) under running water. The facial surfaces of the crowns were ground mechanically (Ecomet 3, Buehler Ltd.) under running water with 600-grit silicon carbide (SiC) paper to obtain a flat enamel or superficial dentin surface. Specimens were randomly assigned to eight groups of 10 teeth each to be treated with four adhesive systems—OptiBond XTR, OptiBond FL, Clearfil SE Bond, or Xeno IV (see Table 1 for detailed information about the materials).

All bonding procedures were performed according to manufacturers' instructions. For OptiBond XTR, the self-etch primer was applied using a microbrush with a scrubbing motion for 20 seconds. It was thinned using medium air pressure. The adhesive was applied using a

 DOI 10.1111/j.1708-8240.2011.00465.x

light brushing motion for 15 seconds, thinned using medium to strong air pressure, and light-activated for 10 seconds.

For OptiBond FL, the dentin or enamel surface was etched with 37.5% phosphoric acid gel for 15 seconds. The etchant was rinsed. The surface was left moist by blotting the dentin with a KimWipe (Kimberly-Clark Corporation, Irving, TX, USA) laboratory tissue rather than drying with compressed air. The primer was applied using a microbrush with a light brushing motion for 15 seconds. After air-drying, the adhesive was applied using a light brushing motion. It was lightly air-thinned and light-activated for 20 seconds.

For Clearfil SE Bond, the self-etch primer was applied with a brush and left on the surface for 20 seconds. It was lightly air-dried and followed by application of the adhesive using a microbrush. The adhesive was thinned lightly with air and was light-activated for 10 seconds.

For Xeno IV, two coats of the adhesive were applied by scrubbing with a microbrush for 15 to 20 seconds each time. The solvent was removed by air-drying, and the adhesive was reapplied if the surface was not shiny after drying. It was light-activated for 10 seconds.

After completion of the adhesive application, composite resin was applied using the Ultradent specimen former (Ultradent Products, South Jordan, UT, USA), which includes a split Teflon mold with an internal diameter of 2.38 mm. Filtek Z250 (3M ESPE, St. Paul, MN, USA) was placed in a single approximately 2-mm increment, which was light-activated for 20 seconds. Visible light-activation procedures were accomplished using an Ultra-Lume LED 5 (Ultradent Products) device at a minimum intensity of 500 mW/cm^2.

Following polymerization, specimens were stored in distilled water for 24 hours at 37°C. Shear bond strengths were determined using a model 4411 universal testing machine (Instron Corporation, Norwood, MA, USA) with a hollow notch shearing device at a crosshead speed of 1 mm/min. Bond-strength values were calculated by dividing the peak break force (*N*) by the cross-sectional area of the bonded interface and were expressed in MPa units.

After the bond-strength testing was completed, two extracted human molars were used for field emission scanning electron microscopy (FESEM) evaluation of the effects of the materials on enamel. Crowns were separated from roots and sectioned mesiodistally. Facial and lingual halves were further sectioned into two 2 × 1-mm rectangles, which were embedded in epoxy resin. After setting of the epoxy resin, specimens were ground flat with 600-grit SiC paper. Enamel specimens were further polished with 1,200-grit SiC and Sof-Lex disks (3M ESPE) to create smooth surfaces. The primers of OptiBond XTR and Clearfil SE Bond, and Xeno IV were applied as previously described. After application, the materials were rinsed off with acetone. To ensure complete removal of the resins, specimens were further placed in an ultrasonic bath with acetone for 15 minutes. Phosphoric acid, part of the OptiBond FL system, was used as control. Any moisture was removed from the specimens by placing them in a desiccator under vacuum.

Specimens were mounted on 13-mm aluminum stubs using carbon-adhesive tabs and coated with gold–palladium alloy (60:40) to a thickness of 10 nm using a Hummer X Sputter Coater (Anatech USA, Union City, CA, USA). Images were collected using a Zeiss Supra 25 FESEM (Carl Zeiss SMT, Inc., Peabody, MA, USA), operating at 5 kV, 10 μm aperture, and at a working distance of 10 mm.

Bond-strength data were analyzed using the JMP8 statistical software package (SAS Institute Inc., Cary, NC, USA). Enamel and dentin data sets were analyzed separately using analysis of variance and Tukey's post-hoc test. All statistical tests were performed at the 95% confidence level. FESEM images were qualitatively analyzed.

RESULTS

Enamel and dentin shear bond strength data are summarized in Tables 2 and 3. On enamel, mean shear

 DOI 10.1111/j.1708-8240.2011.00465.x

TABLE 2. Shear bond strengths to bovine enamel

Adhesive	Mean (MPa ± SD)			
OptiBond FL	41.0 (5.5)	A	B	
OptiBond XTR	40.4 (3.3)	A	B	
Clearfil SE Bond	33.2 (3.8)		B	
Xeno IV	18.1 (3.9)			C

Same capital letters within a column denote means that are not significantly different ($p > 0.05$).

TABLE 3. Shear bond strengths to bovine dentin

Adhesive	Mean (MPa ± SD)			
Clearfil SE Bond	47.1 (7.6)	A		
OptiBond XTR	45.1 (5.6)	A		
Xeno IV	39.5 (10.9)	A	B	
OptiBond FL	33.3 (9.0)		B	C

Same capital letters within a column denote means that are not significantly different ($p > 0.05$).

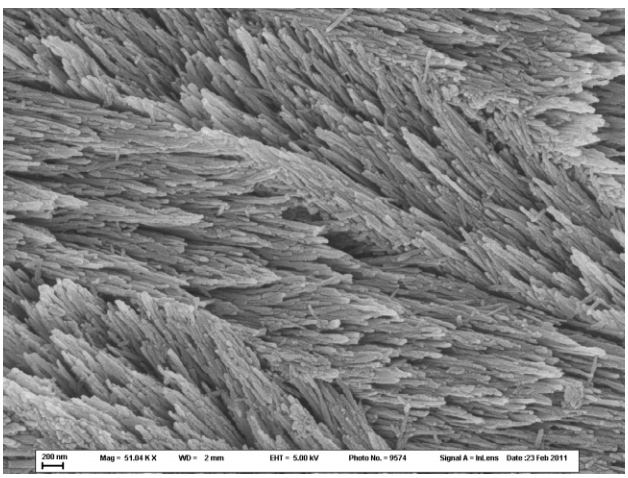

FIGURE 1. FESEM image of enamel surface after 15-second etch with 37.5% phosphoric acid. 50,000× magnification.

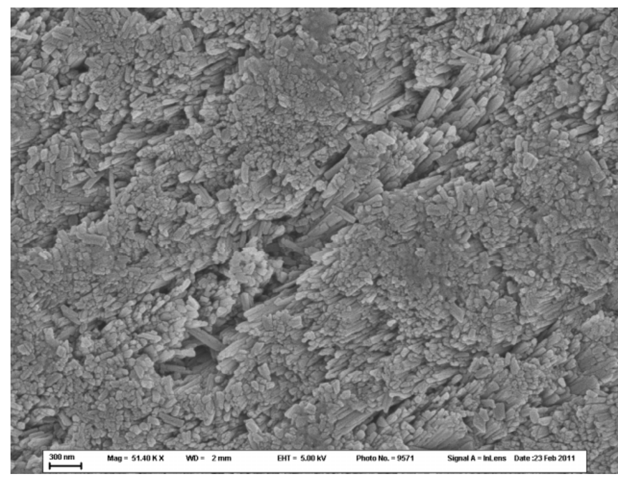

FIGURE 2. FESEM image of enamel surface after 20-second application (undisturbed) of Clearfil SE Primer. 50,000× magnification.

bond strengths ranged from 18.1 MPa for Xeno IV to 41.0 MPa for OptiBond FL. On dentin, the means ranged from 33.3 MPa for OptiBond FL to 47.1 MPa for Clearfil SE Bond. Although there was considerable statistical overlap in both data sets, the test material (OptiBond XTR) was in the highest statistical grouping for both enamel and dentin. All bond failures were adhesive.

Representative FESEM images of enamel surfaces can be seen in Figures 1–5. Figure 1 shows the effects of 37.5% phosphoric acid (component of OptiBond FL), with extensive demineralization of the enamel rods. The effects of the self-etch adhesives can be seen in Figures 2–4. Clearfil SE Bond (Figure 2) showed a mild demineralization of the enamel, whereas OptiBond XTR had a pattern that closer resembled phosphoric acid-etching. Xeno IV, shown in Figure 4, produced minimal alteration of the enamel surface. Figure 5 shows the intact surface of the control polished specimens.

DISCUSSION

The new self-etch adhesive system tested in this study, OptiBond XTR, performed well on both dentin and enamel. On dentin, its mean bond strength was not significantly different from that of Clearfil SE Bond, a proven standard for self-etch primer systems. On ground enamel, its mean bond strength was not statistically different from that of OptiBond FL, which uses 37.5% phosphoric acid to etch enamel. These

 DOI 10.1111/j.1708-8240.2011.00465.x

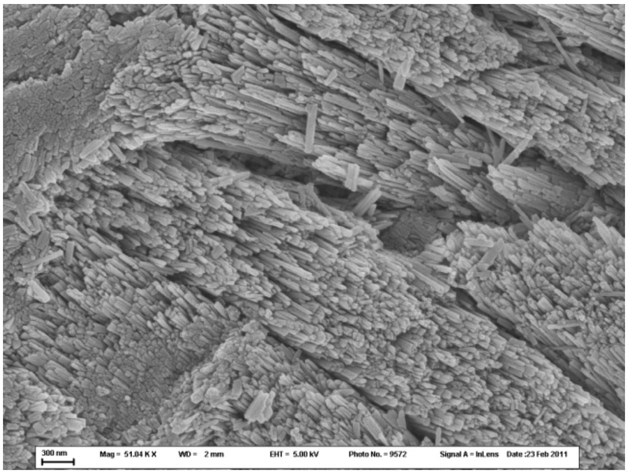

FIGURE 3. FESEM image of enamel surface after 20-second application (scrubbing motion) of OptiBond XTR. 50,000× magnification.

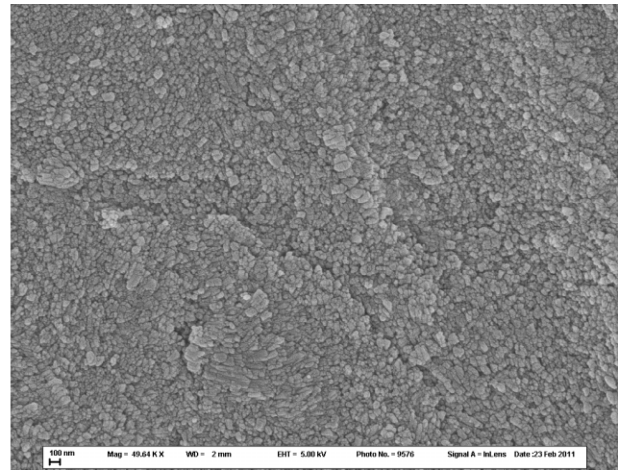

FIGURE 4. FESEM image of enamel surface after 15-second application (scrubbing motion) of Xeno IV. 50,000× magnification.

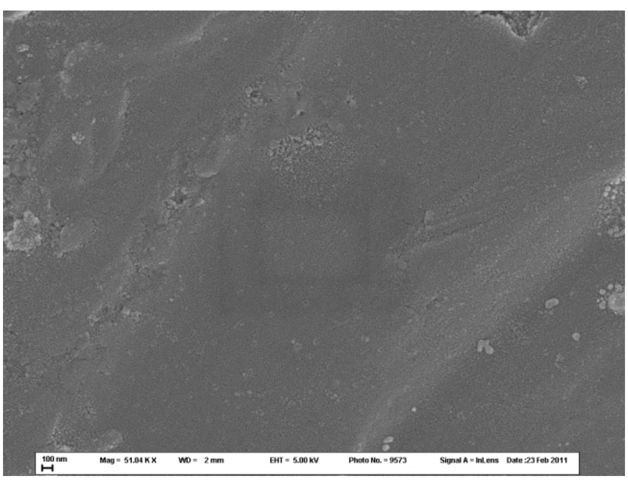

FIGURE 5. FESEM image of untreated specimen (control), polished with 1,200-grit SiC paper and Sof-Lex disks. 50,000× magnification.

results require acceptance of the test hypothesis that OptiBond XTR would produce enamel and dentin bond strengths similar to those of established etch-and-rinse and self-etch adhesives. No published studies of OptiBond XTR are available for comparison.

According to the manufacturer (E. Qian, personal communication), the pH of OptiBond XTR primer is 2.4 until it is dispensed. Acetone rapidly evaporates from the material, increasing the concentration of glycerol phosphate dimethacrylate and thereby reducing the pH to 1.6. By comparison, the pH of Clearfil SE Bond primer measured in the same laboratory is a relatively mild 2.5 (although lower pH values have been reported elsewhere[7]). The roughly 10-fold difference in the acidity of these two primers could account for the differences in their enamel etch patterns and shear bond strengths. (As shown in Table 2, the mean enamel bond strength of OptiBond XTR was about 20% greater than that of Clearfil SE Bond, but these were in the same "B" statistical grouping.)

Two of the adhesive systems used for comparison in this study (OptiBond FL and Clearfil SE Bond) were selected because of their proven long-term clinical performance. The third (Xeno IV) was chosen as representative of the all-in-one type of adhesive.

OptiBond FL is a three-step etch-and-rinse system that bonds well to both enamel and dentin in the laboratory, even over extended storage periods.[10,11] In restorations of noncarious cervical lesions, its clinical performance has been excellent. Clinical trials have reported retention rates of approximately 90% at 12–13 years, with mostly acceptable marginal quality.[3,12]

Similarly, Clearfil SE Bond has demonstrated excellent performance, with nearly 100% retention

 DOI 10.1111/j.1708-8240.2011.00465.x

in noncarious cervical lesions at 8 years.[4] This material contains a functional monomer, 10-methacryloyloxydecyl dihydrogen phosphate, which forms a water-stable ionic bond to residual hydroxyapatite in the dentin surface,[5] contributing to its durable bond strength and described clinical performance.[6] However, because of its mild pH, it does not etch enamel aggressively and therefore its bond strength to enamel, particularly uninstrumented enamel, is only moderately strong.[13] Therefore, some experts recommend selectively etching enamel margins of a preparation first,[9] which does improve marginal quality.[3]

As the newest comparison material evaluated in this study, less is known about the clinical performance of the Xeno IV adhesive. One study of orthodontic brackets bonded to enamel reported that bond strengths achieved by Xeno IV were similar to those achieved by etching with phosphoric acid.[14] However, another study reported that its enamel bond strength was much lower than that of an etch-and-rinse control.[15]

On dentin, studies have reported that the bond strength of Xeno IV was significantly less than that of two-step etch-and-rinse system Prime & Bond NT (Dentsply Caulk) or the three-step etch-and-rinse system Scotchbond Multi-Purpose (3M ESPE).[15,16] Another study reported that Xeno IV bonded as well as several other all-in-one adhesives, but none was as effective as Clearfil SE Bond.[17]

It is worth noting that the bond strengths determined in this study represent "immediate" bond strengths. The performance of OptiBond XTR should be evaluated further with long-term storage in the laboratory and with clinical trials.

CONCLUSION

Within the limits of this in vitro study, the two-step self-etch adhesive system OptiBond XTR provided excellent bond strengths to both dentin and enamel. The long-term durability of those bonds was not evaluated in this study.

DISCLOSURE AND ACKNOWLEDGEMENT

The authors have no financial interest in the companies whose materials are included in this article.

The study was supported by the Kerr Corporation.

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This article is accompanied by commentary, Enamel and Dentin Bond Strengths of a New Self-Etch Adhesive System, Jorge Perdigão, DMD, MS, PhD

DOI 10.1111/j.1708-8240.2011.00466.x

