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ORAL DIAGNOSIS RESTORATIVE DENTISTRY
IMPLANTS RESEARCH METHODOLOGY SPECIAL PATIENT CARE
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Bond strength of composite to dentin and enamel using self-etching adhesive systems

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To determine the shear bond strength of 10 self-etching adhesive systems to dentin and enamel, flat bonding sites were prepared on 216 extracted human molar teeth, using 600-grit silicon carbide paper to expose dentin or enamel. Following the application of each adhesive system, bonded assemblies of Spectrum TPH were prepared using a gelatin capsule matrix (n = 24). After 24 hours of storage in water at 37°C, 12 specimens from each enamel and dentin group were debonded. This was followed by thermocycling for 6,000 cycles between water baths at 5°C and 55°C, with a

dwell time of 20 seconds in each bath. Specimens were loaded to failure in a testing machine with a crosshead speed of 5 mm/ minute. Statistical analysis included a one-way ANOVA and least square difference *post hoc* test. There were significant differences in shear bond strength for both enamel and dentin among the materials tested (p < 0.05).

Received: March 7, 2008 Accepted: May 9, 2008

dhesive dentistry is a rapidly changing and evolving field. In 1955, Buonocore introduced the concept of treating the enamel chemically to alter its surface characteristics and allow acrylic resins to adhere to the enamel surface of the tooth.1 Etching the enamel with phosphoric acid produced microroughness, allowing resin bonding via micromechanical retention. Acid conditioning of the enamel surface gave way to etch-and-rinse techniques, in which both the enamel and dentin surfaces are acid-conditioned and the acid is rinsed to allow resin to adhere to both enamel and dentin surfaces. Effective dentin bonding (that is, resulting in shear bond strengths of 17 MPa or more) has been a considerably greater technical challenge than enamel bonding.² Early concerns regarding adverse effects on pulpal health from acid conditioning of dentin have proven to be unfounded, provided the pulp can be sealed or an adequate thickness of overlying dentin remains.^{3,4}

The strategy for modern dentin bonding systems centers on the

creation of a hybrid resin-reinforced layer for dentin adhesion. This layer requires partial demineralization of the dentin layer, exposing collagen fibrils.⁵ At that point, the adhesive monomers are infiltrated in and around the residual collagen and mineral. With traditional etch-and-rinse adhesive systems, this infiltration technique requires moisture on the dentin surface to support collagen fibers, thus allowing for adequate resin penetration to generate a mineral/collagen/resin interface.

Determining appropriate dentin moisture content can be a problem when placing a bonded restoration. An overly wet dentin surface may result in emulsification and cause voids to form in the primer; conversely, a desiccated dentin surface causes collagen fiber collapse, reduced resin penetration, and voids and gaps under the restorative material. Moist bonding with the latest etch-and-rinse hydrophilic bonding systems can produce laboratory bond strengths ranging from 17–24 MPa, with correspondingly favorable clinical results.^{6,7}

Self-etching adhesive systems characterized by acidic monomers that are not rinsed from the tooth surface have become popular due to their purported simplified technique, which requires fewer steps and eliminates clinical judgment regarding residual dentin moisture. These systems act by conditioning, demineralizing, and infiltrating the enamel and the dentin simultaneously.8 The smear layer is altered but not removed and rinsing is not indicated. Eliminating the separate etch-and-rinse step may decrease the risk of overconditioning dentin, minimize the problem of inadequate penetration of adhesive monomers, and reduce the risk of postoperative sensitivity.

Because the dentinal smear layer is dissolved and incorporated into the hybrid adhesive layer, the hydrolytic stability of these systems has been called into question.⁹ The lower immediate enamel bond strength values and the long-term consistency of the enamel bond with these systems also are areas of concern. Generally, enamel shear bond values are lower than those generated with Table 1. Mean shear bond strengths of self-etching adhesives to enamel (MPa). Groups designated by a common symbol were statistically similar at a confidence level of 95%.

Product	MPa
Clearfil S3	24.0 ± 4.3^{a}
Xeno V	$23.6 \pm 3.9^{a,b}$
AdheSE One	$23.2 \pm 4.1^{a,b}$
Clearfil SE Bond	$22.3 \pm 5.8^{a,b}$
iBond SE	$21.4 \pm 4.2^{a,b,c}$
Xeno IV	$21.3\pm1.9^{a,b,c}$
Optibond All-in-One	$20.0 \pm 3.1^{b,c}$
Adper Prompt L-Pop	$18.4 \pm 4.8^{\circ}$
G-Bond	$17.8 \pm 2.9^{c,d}$
iBond	11.8 ± 2.9^{d}

Table 2. Mean shear bond strengths of self-etching adhesives to dentin (MPa). Groups designated by a common symbol were statistically similar at a confidence level of 95%.

Product	MPa
Xeno IV	25.8 ± 2.7^{a}
Optibond All-in-One	23.9 ± 3.9^{a}
Xeno V	$23.7 \pm 3.9^{\circ}$
Clearfil SE Bond	$23.7 \pm 4.3^{\circ}$
iBond	16.4 ± 3.8^{b}
AdheSE One	$14.5 \pm 4.0^{b,c}$
G-Bond	$14.4 \pm 3.3^{b,c}$
Clearfil S3	$13.8 \pm 4.1^{b,c,d}$
Adper Prompt L-Pop	$12.7 \pm 3.5^{c,d}$
iBond SE	10.4 ± 4.0^{d}

etch-and-rinse systems and some clinical results have indicated enamel marginal breakdown of restorations placed with these adhesives.¹⁰

Bond strength testing is used as a screening tool to help dentists understand and predict the clinical behavior of adhesives. This laboratory study sought to determine the shear bond strength to dentin and enamel of 10 newer generation selfetching adhesive systems.

Materials and methods

Flat bonding sites were prepared on 240 extracted human molar teeth, using 600 grit silicon carbide paper to expose dentin and enamel. Ten "self-etching" adhesives were tested: Xeno IV (Dentsply Caulk, Milford, DE; 800.532.2855), Xeno V (Dentsply DeTrey, Konstanz, Germany; 49.0.7531.583.158), Adper Prompt L-Pop (3M ESPE, St. Paul, MN; 888.364.3577), Optibond All-in-One (Kerr Dental, Orange, CA; 800.537.7123), AdheSE One (Ivoclar Vivadent, Amherst, NY; 800.533.6825), iBond SelfEtch (Heraeus Kulzer, South Bend, IN; 800.431.1785), Clearfil S3 (Kuraray Dental, New York, NY; 800.879.1676), G-Bond (GC America, Alsip, IL; 800.323.7063), Clearfil SE Bond (Kuraray Dental), and iBond with GLUMA (Heraeus Kulzer).

Each adhesive system was applied according to manufacturers' instructions; at that point, bonded assemblies of Spectrum TPH composite resin (Dentsply Caulk) were prepared using a gelatin capsule matrix. A total of 24 samples were prepared for each adhesive system, with 12 bonded to dentin and 12 to enamel. All specimens in each group were stored in water for 24 hours at 37°C and immersed in a water bath at 5°C for 20 seconds. All specimens were subjected to thermocycling for 6,000 cycles and subsequently placed into another water bath at 55°C for 20 seconds. Following storage, specimens were mounted in acrylic and loaded to failure in an Instron Model 1123 testing machine (Instron Corp.,

Canton, MA; 800.564.8378) with a crosshead speed of 5 mm/minute.

Results

Table 1 reports mean shear bond strength values to enamel for each adhesive (in MPa), while Table 2 reports mean shear bond strength values to dentin. Based on one-way ANOVA, there were significant differences among the adhesives for both enamel and dentin groups. A pair-wise comparison was done using a least square difference (LSD) *post hoc* test. There were statistical differences among the materials for both enamel and dentin. Only four materials (Xeno IV, Xeno V, Optibond All-in-One, and Clearfil SE Bond) generated more than 17 MPa to dentin; conversely, all of the materials except for iBond exceeded 17 MPa for enamel.

Discussion

Self-etching adhesive systems rely on acidic monomers to demineralize and infiltrate enamel and dentin simultaneously. This acidity must be neutralized by the mineral in the tooth structure to allow the adhesive film to polymerize completely. Etch-and-rinse adhesives remove the smear layer and dissolved mineral during the rinsing step. The longterm hydrolytic stability of the selfetching adhesive systems remains an issue due to concerns about residual acidity and the fact that these systems do not remove the smear layer.

Previous articles evaluating the bond strength between the first commercially available self-etching adhesive systems and enamel showed a decrease in bond strength after storage and thermocycling.¹⁰ The results of the present study suggest that the more recently developed self-etching systems may offer hydrolytic stability similar to that reported for etch-andrinse systems. Neither water storage nor the application of 6,000 thermocycles significantly degraded the shear bond strength of the materials used in this study.

Incomplete infiltration of demineralized dentin has been proposed as one reason for postoperative sensitivity.9 Since self-etching primers demineralize and infiltrate the tooth structure simultaneously, some authors have advocated using self-etching systems to reduce the chance of postoperative sensitivity following the placement of resin restorations.9 However, other studies have shown poor retention results with resin adhesives.¹¹ There were significant differences in terms of in vitro dentin shear bond strength among the self-etching adhesive materials tested; however, no common factor accounts for the differential performance of the systems tested. While in vitro testing is not a definitive predictor of clinical behavior, four materials (Clearfil SE

Bond, Optibond All-in-One, Xeno IV, and Xeno V) generated similar shear bond strength values to etchand-rinse systems that have had a long history of clinical success.

Enamel bond strength values generated by etch-and-rinse systems typically are higher than dentin bond strengths. By comparison, self-etching systems produce enamel bond strength values that are similar to (and sometimes even lower than) dentin values. This change in the relative enamel/dentin bonding ratio may have a clinical impact on the quality of enamel margins. The ability of these systems to bond and seal to enamel warrants further investigation.

Conclusion

There were differences in shear bond strength to dentin and enamel among 10 self-etching adhesive systems. The highest values generated to dentin were similar to values generated for conventional etch-andrinse adhesives. The results of the present study suggest that Xeno IV, Xeno V, Optibond All in One, and Clearfil SE Bond may achieve clinical results similar to those produced by the well-established etch-andrinse adhesive systems when cavities are composed primarily of dentin. The significance of the relatively low enamel bond strengths (compared to etch-and-rinse adhesives) is not known. It may be appropriate for clinicians to be cautious with selfetching adhesives when enamel is the primary substrate in the bonding procedure.

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