

Critical Appraisal

OPTIONS FOR DENTIN/ENAMEL BONDING: PART III

Author and Associate Editor

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Four categories of resin-based dentin/enamel adhesives are currently available. These include the three-step etch-&-rinse, "one-bottle" etch-&-rinse, two-step self-etch primer systems, and "all-in-one" self-etch adhesives. In consecutive issues of the Journal, the Critical Appraisal series is presenting salient publications on research in each of the categories. The first two installments focused on the etch-&-rinse systems. The series continues with this review of papers on the two-step self-etch primer systems.

ULTRASTRUCTURAL CORRELATES OF IN VIVO/IN VITRO BOND DEGRADATION IN SELF-ETCH ADHESIVES

N. Donmez, S. Belli, D.H. Pashley, F.R. Tay Journal of Dental Research (2005;84:355-9)

ABSTRACT

Objective: The objectives of this study were (1) to evaluate the in vitro and in vivo bond durability of two self-etch adhesive systems, and (2) to characterize morphological changes in resin-dentin interfaces aged in the oral environment and under laboratory conditions for 1 year.

Materials and Methods: For the in vivo portion of the study, patients with one or more erupted, unrestored third molars scheduled for extraction were selected. Class I preparations were made in 24 teeth under rubber dam isolation. The teeth were randomly divided into two groups for bonding with either Clearfil SE Bond or Clearfil Protect Bond (both Kuraray, Tokyo, Japan). Both systems include a mildly acidic self-etch primer. These are similar materials, but Protect Bond contains an antibacterial monomer and includes a fluoride-releasing bonding agent. The preparations were restored using a thin flowable resin liner and two increments of a light-activated hybrid composite.

The adhesive groups were divided into two sub-groups based on

extraction time—24 hours or 1 year following restoration. After extraction at each time, four teeth from each group were sectioned for microtensile bond strength (μ TBS) testing, which was accomplished using a universal testing machine.

To compare bond degradation in vivo and in vitro, the same restorative procedures were done in 25 extracted third molars. The restored teeth were stored in artificial saliva at 37°C, and were retrieved and sectioned for μ TBS testing at 24 hours or 1 year.

*Professor and Chair, Department of Operative Dentistry, University of North Carolina School of Dentistry, Chapel Hill, NC 27599-7450, USA Two specimens from each in vivo and in vitro group were processed for examination with transmission electron microscopy (TEM).

Results: For Clearfil SE Bond, both aging conditions and time had a significant effect on bond strengths. Under in vivo conditions, the mean μ TBS was 21.3 MPa at 24 hours but only 13.8 MPa at 1 year, a significant decrease. In vitro, the respective means were 33.9 MPa and 21.4 MPa, and the decrease over time was significant.

For Clearfil Protect Bond, only aging conditions—not time—significantly affected bond strengths. Bond strengths were greater under in vitro than under in vivo conditions but did not decrease with time in either case. In vivo, the 24-hour mean μ TBS was 17.9 MPa and the 1-year mean was 18.6 MPa. In vitro, the respective means were 28.1 MPa and 28.3 MPa. TEM showed that both adhesives formed thin, partially demineralized hybrid layers under both conditions. Nanoleakage could be observed even at 24 hours.

Conclusions: Bond strengths of both self-etch adhesive systems decreased over time under in vitro and in vivo conditions. Bond strengths were lower in vivo than in vitro, but the pattern of degradation was similar under both conditions.

COMMENTARY

This study is particularly interesting because it measured bond strengths achieved not only in the laboratory but also under clinical conditions. Not surprisingly, the results were better in the laboratory. Numerous clinical factors, including but not limited to dentin fluid movement and functional stresses, very likely have adverse effects on bonding efficacy. Some studies have reported that Clearfil SE Bond mediates a chemical bond to residual hydroxyapatite in the hybrid layer (see papers by Yoshida and colleagues as well as Fukegawa and colleagues under "Suggested Reading"), which potentially could improve the durability of resin-dentin bonds. That was not the case for SE Bond in this particular study, although a similar material (Clearfil Protect Bond) did provide a bond that was stable at 1 year.

SUGGESTED READING

- Armstrong SR, Vargas M, Fang Q, Laffoon J. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage. J Adhes Dent 2003;5:47–56.
- Fukegawa D, Hayakawa S, Yoshida Y, et al. Chemical interaction of phosphoric acid ester with hydroxyapatite. J Dent Res 2006;85:941–4.
- Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive performance of functional monomers. J Dent Res 2004;83:854–8.

HYDROLYTIC STABILITY OF SELF-ETCH ADHESIVES BONDED TO DENTIN

S. Inoue, K. Koshiro, Y. Yoshida, J. De Munck, K. Nagakane, K. Suzuki, H. Sano, B. Van Meerbeek *Journal of Dental Research* (2005;83:1160–4)

ABSTRACT

Objective: The purpose of this study was to evaluate the hydrolytic stability of three self-etch adhesive systems containing different functional monomers. Materials and Methods: Three two-step self-etch adhesive systems were used in this study—Clearfil MegaBond (Kuraray; marketed as Clearfil SE Bond outside Japan, and referred to by the latter name in this review), Unifil Bond (GC, Tokyo, Japan), and Clearfil Liner Bond II (Kuraray). The adhesive monomers in these materials are, respectively, 10-MDP, 4-MET, and phenyl-P. Each is a two-step system using a self-etch primer.

Occlusal enamel was sectioned from extracted human third molars to expose mid-coronal dentin, which was polished to 600-grit using silicon carbide abrasive paper to produce standardized smear layers. The three adhesives were applied according to manufacturers' instructions, and Z100 (3M ESPE, St. Paul, MN, USA) composite was applied and lightactivated in increments. Using a water-cooled diamond saw, each bonded specimen was sectioned into small slabs of approximately 1 mm cross-sectional area. Specimens were untouched (control) or were thermocycled 10,000, 20,000, 30,000, 50,000, or 100,000 times between 5°C and 55°C, with a dwell time of 60 seconds in each water bath. Three teeth were used for each condition (combination of thermocycling and adhesive type).

Microtensile bond strength (μ TBS) testing was performed using a desktop materials tester (EZ-Test, Shimadzu, Kyoto, Japan). Additional specimens of each adhesive (either controls or thermocycled 100,000 times) were fixed, dehydrated, and embedded in epoxy resin for transmission electron microscopy (TEM) analysis of resin-dentin interfaces.

Results: For the control groups, mean uTBS values were 37.9 MPa for Unifil Bond. 40.8 MPa for Clearfil SE Bond, and 44.7 MPa for Liner Bond II. Bond strengths of all three adhesives were reduced by thermocycling, but the difference was not statistically significant for SE Bond. Mean bond strengths declined to 23.2 MPa for Liner Bond II after 100,000 thermocycles and to 22.5 MPa for Unifil Bond. However, the mean bond strength of SE Bond declined only to 35.3 MPa. All of the adhesives produced shallow hybrid layers containing residual hydroxyapatite crystals, but this was particularly true for Clearfil SE Bond. The interfacial morphology of hybrid layers formed by SE Bond was similar regardless of whether the specimens had not been thermocycled or had been thermocycled 100,000 times.

Conclusions: Long-term durability of resin-dentin bonds might depend on chemical bonding of the functional monomer.

COMMENTARY

Penetration of resin-based adhesives into etched or conditioned dentin surfaces results in the formation of a hybrid layer, which is called "hybrid" because it is a mixture of resin and dentin. Some mild self-etch materials do not completely remove hydroxyapatite crystals from the dentin surface, and evidence suggests that some adhesive monomers might bond chemically with the residual hydroxyapatite. As the present study suggests, such chemical bonding might improve the durability of resin-dentin bonds.

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- Casagrande L, de Hipólito V, de Góes MF, et al. Bond strength and failure patterns of adhesive restorations in primary teeth aged in the oral environment. Am J Dent 2006;19:279–82.
- Osorio R, Pisani-Proenca J, Erhardt MC, et al. Resistance of ten contemporary adhesives to resin-dentine bond degradation. J Dent 2008;36:163–9.
- Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive performance of functional monomers. J Dent Res 2004;83:454–8.

FIVE-YEAR CLINICAL EFFECTIVENESS OF A TWO-STEP SELF-ETCHING ADHESIVE

M. Peumans, J. De Munck, K. Van Landuyt, P. Lambrechts, B. Van Meerbeek *Journal of Adhesive Dentistry* (2007;9:7-10)

ABSTRACT

Objective: This study was designed to evaluate the clinical performance of Clearfil SE Bond in restoration of Class V lesions.

Materials and Methods: Twentynine patients received two or four restorations of non-carious cervical lesions: the total number of restorations was 100. Clearfil SE Bond, which is a mildly acidic self-etch primer system, was used according to manufacturer's directions for half of the restorations. For the others, enamel was selectively etched using 40% phosphoric acid before the adhesive system was applied. The same composite resin material was used for all restorations. Tooth preparation involved roughening of the dentin surfaces with a coarse diamond and placement of a 1-2 mm bevel on enamel margins.

The restorations were evaluated after 6 months, and 1, 2, 3, and 5 years of clinical service. The recall rate was 84% at 5 years. Two evaluators rated each restoration using a standard set of criteria for retention, marginal integrity, marginal discoloration, recurrent caries, post-operative sensitivity, and preservation of tooth vitality.

Results: At 5 years, only one restoration had been lost from the group of restorations placed using manufacturer's directions, and none were lost from the selective etching group. In both groups, marginal integrity deteriorated over time. However, the deterioration was worse for the no-etch group, in which significantly more enamel margin defects were detected. Marginal discoloration was more frequent in the no-etch group. No recurrent caries, post-operative sensitivity, or loss of vitality was observed.

Conclusions: Marginal defects in the no-etch group were clinically acceptable and did not require any repair, indicating that the bonding effectiveness of Clearfil SE Bond remained relatively good despite the material's superficial interaction with enamel. Selective enamel etching improved marginal adaptation but did not affect the overall clinical performance of Class V restorations.

COMMENTARY

Although laboratory testing can be very useful for studying various aspects of adhesive performance and screening new materials during product development, the best evidence for efficacy of resin-based adhesives is found in controlled clinical trials. This particular study was a relatively long-term (5 years) clinical trial of the most popular self-etch primer system, Clearfil SE Bond. It was a typical clinical trial involving restoration of noncarious cervical lesions.

Clearfil SE Bond is known to be only mildly acidic, so one of the primary objectives of the study was to determine the durability of its enamel bond and whether selective acid-etching might improve durability. The authors concluded that the overall performance of the Class V restorations was similar in both groups. However, marginal integrity was better, and marginal discoloration was less frequent when enamel margins were etched before the adhesive system was applied.

Selective etching of enamel, therefore, is an acceptable technique for self-etch primer systems when the operator is concerned about enamel marginal integrity. Contact of the acid etchant with dentin should be avoided as much as possible because it can reduce adhesion of self-etch primers to dentin.

SUGGESTED READING

Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. Dent Mater 2001;17:430–44.

Peumans M, Kanumilli P, De Munck J, et al. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater 2005;21: 864–81.

Soares CJ, Castro CG, Santos Filho PC, da Mota AS. Effect of previous treatments on bond strength of two self-etching adhesive systems to dental substrate. J Adhes Dent 2007;9:291–6. Van Landuyt KL, Kanumilli P, De Munck J, et al. Bond strength of a mild self-etch adhesive with and without prior acidetching. J Dent 2006;34:77–85.

FOUR-YEAR WATER DEGRADATION OF A TOTAL-ETCH AND TWO SELF-ETCHING ADHESIVES BONDED TO DENTIN

A.I. Abdalla, A.J. Feilzer Journal of Dentistry (2008;36:611-7)

ABSTRACT

Objective: The purpose of this study was to evaluate the effect of direct and indirect water storage on the microtensile dentin bond strength (μ TBS) of one etch-&-rinse and two self-etch adhesives.

Materials and Methods: The materials used in this study were the one-bottle etch-&-rinse system Admira Bond (Voco, Cuxhaven, Germany), the self-etch primer system Clearfil SE Bond, and the all-in-one self-etch adhesive Hybrid Bond (Sun Medical, Shiga, Japan). Standardized Class I cavity preparations were made in the occlusal surfaces of 54 extracted human mandibular molars. The preparations extended approximately 1 mm into dentin and had all margins in enamel. The enamel margins were beveled.

The prepared teeth were divided into groups of 18, and the preparations were restored with composite resin following application of the adhesives according to manufacturers' directions. The restored teeth in each group were divided into three sub-groups based on time of storage in water. The storage times were 24 hours and 4 years. For the 24-hour group and one of the 4-year groups, the specimens were sectioned for µTBS after storage in water. In the other 4-year group, specimens were sectioned before storage in water. Thus, in this last group, resin-dentin interfaces were exposed directly to water. The bond strength test was done using a universal testing machine.

Results: At 24 hours, the mean μ TBS were 39 MPa for Admira Bond, 41 MPa for Clearfil SE Bond, and 37 MPa for Hybrid Bond. After 4 years without direct exposure of the resin–dentin interfaces to water, mean bond strengths had declined very little—the worst being a reduction of 5 MPa for Hybrid Bond. However, after four years of direct exposure to water, the bond strengths of all three adhesives were significantly lower—22 MPa for Admira Bond, 21 MPa for Clearfil SE Bond, and 12 MPa for Hybrid Bond.

Conclusions: Exposure to water causes deterioration of resin bonds to dentin. In restorations with margins in enamel, etching the enamel could protect the bond from this effect. However, when restoration margins extend into dentin or cementum, bonds are likely to degrade over time.

COMMENTARY

Laboratory studies on resin-dentin bond longevity tend to reach the same conclusion as this one—that is, when resin-dentin bonds are directly exposed to water, they deteriorate over time. In contrast, when the resin-dentin interface is protected by a bonded resinenamel interface, degradation is much slower. This is an important consideration for Class II posterior composites, where the gingival enamel is frequently lacking in quantity or quality.

The authors state in their conclusions that etching enamel margins can protect resin-dentin bonds, presumably implying that phosphoric acid-etching is required. However, their own results indicate that the enamel bond is important regardless of how it is achieved. Two of the three adhesives tested in this study did not use a separate acid-etching step and yet had stable dentin bonds when the enamel was present during storage.

Clearfil SE Bond did not perform as well in this study as it did in the study by Inoue and colleagues reviewed earlier. Another noteworthy, and perhaps unsurprising, finding is that the simplest adhesive was the one most affected by exposure to water.

SUGGESTED READING

- DeMunck J, Van Landuyt K, Peumans M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 2005;84:118–32.
- De Munck J, Shirai K, Yoshida Y, et al. Effect of water storage on the bonding effectiveness of 6 adhesives to Class I cavity dentin. Oper Dent 2006;30:456–65.

THE BOTTOM LINE

- Much of the research on self-etch primer systems has focused on one commercial product, Kuraray's Clearfil SE Bond.
- Mildly acidic self-etch primers such as Clearfil SE Bond form partially demineralized hybrid layers containing residual hydroxyapatite crystals. Because of this, there is potential for chemical interaction between adhesive monomers and hydroxyapatite that could improve the durability of resin–dentin bonds.
- Clinical trials have reported good results for self-etch primer systems.
- At least anecdotally, post-operative sensitivity occurs less frequently with self-etch primer systems than with etch-&-rinse systems.
- The bond of mildly-acidic self-etch primers to enamel can be improved by selective etching of the enamel before primer application. Contact of the etchant with dentin should be avoided as much as possible.

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