



# Critical Appraisal

## LONGEVITY OF RESIN BONDS TO DENTIN

Author and Associate Editor

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*Much contemporary esthetic and restorative dentistry relies on bonding of resin-based materials to tooth structure. Adhesion of resin materials to enamel has proved to be extremely strong and reliable since the concept was first introduced by Buonocore in 1955 and widely accepted by clinicians 20–25 years later. Enamel bonding is a simple and predictable process, because enamel presents a relatively homogeneous and largely inorganic substrate. Dentin, in contrast, is a heterogeneous tissue containing a large proportion of organic material and fluid, and is a difficult and variable substrate for bonding. Although clinically effective dentin adhesives have been available now for over 15 years, questions remain about the long-term durability of resin bonds to dentin. This Critical Appraisal reviews some of the relevant literature on this topic.*

## THE EFFECT OF SIX YEARS OF WATER STORAGE ON RESIN COMPOSITE BONDING TO HUMAN DENTIN

R. Frankenberger, W.O. Strobel, U. Lohbauer, N. Krämer, A. Petschelt

*Journal of Biomedical Research B: Applied Biomaterials* 2004 (69B:25–32)

### ABSTRACT

**Objective:** The purpose of this study was to evaluate the effects of water storage for up to 6 years on the push-out bond strength and marginal adaptation of dentin adhesives.

**Materials and Methods:** Disks of 2-mm thickness were cut from the mid-coronal level of 360 extracted human third molars. A 2.3-diameter cylindrical cavity was prepared in the center of each disk. The dentin disks were embedded in an extrusion device using a temporary restorative material.

They were assigned to eight groups of 45 for treatment with various adhesives.

The adhesives used in the study were Syntac Classic (Ivoclar Vivadent, Schaan, Liechtenstein) with and without phosphoric acid etching, A.R.T. Bond (Coltène, Alstätten, Switzerland) with and without etching, Scotchbond Multi-Purpose (3M ESPE, St. Paul, MN, USA), EBS (3M ESPE, Seefeld, Germany), Prime & Bond 2.0 (Dentsply DeTrey, Konstanz, Germany), and Syntac Single-Component (Ivoclar Vivadent).

Prime & Bond and Syntac Single-Component are both two-step etch-and-rinse systems that deliver primer and bonding agent simultaneously. The others include separate primer and bonding agent steps.

After adhesive application, the cavities were restored with composite. The specimens were polished to 600 grit and stored in distilled water at 37°C for 1 day, 90 days, or 6 years. After storage, the specimens were thermocycled and then subjected to push-out testing. Using a universal testing

machine, this was accomplished by applying a rod to the composite until failure. Failure was defined as the loss of 30% of the maximum push-out force. Push-out bond strength was determined by calculating the quotient of maximum load and adhesion area.

Impressions of the 6-year specimens were made after 1 and 90 days, and 1, 2, 3, 4, 5, and 6 years. Epoxy replicas were made and sputter-coated with gold. The restoration margins were analyzed using scanning electron microscopy at 200× magnification). A quantitative analysis of gap-free margins versus margins with gaps or irregularities was made using an image analysis system. Margin quality was expressed as the percentage of gap-free margins relative to the entire marginal length.

**Results:** At 1 day, mean bond strengths ranged from 26.3 MPa for Prime & Bond 2.0 to 33.8 MPa

for A.R.T. with etching. After 90 days of water storage, the mean bond strength of all adhesives remained constant. However, at 6 years, the bond strength of every adhesive had declined significantly. The loss of bond strength was more pronounced for the one-bottle systems than for the three-step systems.

In general, the marginal quality of the three-step systems was better than that of the two-step systems. Margins began to deteriorate more rapidly with the simpler systems and stabilized at approximately 50% continuous, gap-free margins. For the more complex systems, the gap-free margin percentage was approximately 80%.

**Conclusions:** Etch-and-rinse multi-step adhesives provide higher bond strengths and better marginal adaptation than two-step systems over 6 years of storage in water.

#### 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 purpose of this study was to evaluate the bond durability and interfacial morphology of two self-etch adhesive systems aged in the

laboratory and in the oral environment for 1 year.

**Materials and Methods:** The adhesives tested in this study were Clearfil SE Bond and Clearfil

#### COMMENTARY

This study reports two important findings. First, bonds of resin-based materials to dentin are not stable and will degrade as a result of prolonged water exposure. Second, the more complex systems—those that etch, prime, and bond as separate steps in sequence—are more resistant to the effects of water. Based on these findings, and rightfully so, the authors recommend the use of multistep adhesive when restoring preparations that lack enamel margins.

#### SUGGESTED READING

Armstrong SR, Vargas MA, Fang Q, Laffoon JE. 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. *Dent Mater* 2003;5:47–56.

Burrow MF, Harada N, Kitasako Y, et al. Seven-year dentin bond strengths of a total- and self-etch system. *Eur J Oral Sci* 2005;113:265–70.

Protect Bond (both from Kuraray, Tokyo, Japan). Both systems include a water- and ethanol-based self-etching primer and a filled bonding agent. The chemistry of the two systems is similar

except that Protect Bond contains fluoride and a modified antibacterial monomer.

Volunteer subjects with one or more erupted, unrestored third molars scheduled for extraction were included in the study. Standard Class I preparations (3 mm in depth) were made in the teeth and restored under rubber dam isolation. The preparations were restored using one of the adhesives, followed by a thin layer of a flowable resin liner and two increments of a hybrid restorative composite. The restored teeth were extracted following either 24 hours or 1 year.

After extraction, each tooth was sectioned into  $0.9 \times 0.9$  mm beams for microtensile testing. Bond testing was done using a universal testing machine. Failure modes were examined at  $30\times$  magnification and were classified as adhesive, cohesive, or mixed.

In vitro specimens were prepared and restored using extracted third molars. These were stored in artificial saliva at  $37^{\circ}\text{C}$  for either 24 hours or 1 year. Microtensile specimens were obtained and tested in the same manner as the in vivo specimens.

In addition to the microtensile specimens, other specimens were evaluated using transmission

electron microscopy (TEM). The TEM was used specifically to examine nanoleakage at the resin–dentin interfaces.

**Results:** For Clearfil SE Bond, the mean in vitro bond strengths were 33.9 MPa at 24 hours, and 21.4 MPa at 1 year. The corresponding means for in vivo specimens were 21.3 and 13.8 MPa. For Clearfil Protect Bond, the in vivo mean at 24 hours was 17.9 MPa, and the mean at 1 year was 18.6. In vitro, the respective means were 28.1 and 28.3 MPa. The proportion of adhesive failures for both materials was greater at 1 year than at 24 hours.

Interfacial “water trees” were observed at 1 year but not at 24 hours. These channels were more extensive in Clearfil SE Bond and might have been formed by slow water sorption through the adhesives that expedited leaching of hydrolytic resin compounds.

**Conclusions:** Degradation of self-etch adhesives has a similar mechanism in vitro and in vivo.

#### COMMENTARY

This study demonstrates that resin–dentin bonds degrade over time, whether in the mouth or in the laboratory. While the authors conclude that the mechanism of degradation is the same in both locations, it seems that the oral

environment presents much harsher conditions than the laboratory environment. For both adhesives, both the early and later bond strengths were higher for the in vitro specimens than for the in vivo specimens.

An interesting finding of this study was the difference in stability of the adhesives. Clearfil Protect Bond was similar to Clearfil SE Bond in that its in vitro adhesion was better than its in vivo adhesion. However, while adhesion of Clearfil SE Bond deteriorated in both environments, Clearfil Protect Bond did not. The authors attribute this phenomenon to the presence of fluoride in its formulation. They speculate that fluoride might reduce the solubility of calcium phosphates within the hybridized smear layer and hybrid layer, resulting in a more stable bond.

#### SUGGESTED READING

Hashimoto M, Ohno H, Kaga M, et al. In vivo degradation of resin–dentin bonds in humans over 1 to 3 years. *J Dent Res* 2000;79:1385–91.

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.

# EFFECT OF WATER STORAGE ON THE BONDING EFFECTIVENESS OF 6 ADHESIVES TO CLASS I CAVITY DENTIN

J. De Munck, K. Shirai, Y. Yoshida, S. Inoue, K.L. Van Landuyt, P. Lambrechts, K. Suzuki, H. Shintani, B. Van Meerbeek

*Operative Dentistry* 2006 (31:456–65)

## ABSTRACT

**Objective:** The aim of this study was to determine the bonding effectiveness of six adhesives, including two glass ionomers, representing three different approaches using prepared cavities and water storage.

**Materials and Methods:** Standard box-type Class I restorations (4.5-mm deep) were prepared in extracted human third molars. The prepared specimens were randomly assigned to six groups for treatment with the various adhesives, which included OptiBond FL (three-step etch-and-rinse, Kerr, Orange, CA, USA), Scotchbond 1 (known in the United States as Single Bond, a two-step etch-and-rinse material, 3M ESPE), Clearfil SE Bond (two-step self-etching primer system, Kuraray), Adper Prompt (“all-in-one” self-etch adhesive, 3M ESPE), FujiBond LC (two-step resin-modified glass ionomer adhesive, GC, Tokyo, Japan), and Reactmer (one-step RMGI adhesive, Shofu, Kyoto, Japan). All preparations were restored using Z100 composite (3M ESPE) in an incremental technique.

The specimens in each group were divided into three subgroups for microtensile bond strength (MTBS) testing under three different conditions. First, the bond strengths were determined after 24 hours of storage in water. Second, bond strengths were determined after 1 year of water storage. In both cases, the microtensile specimens were formed *after* storage. For the third subgroup of each material, the microtensile specimens were formed *before* 1 year of water storage. Thus, the resin–dentin interface was protected by circumferential bonded enamel in the first two conditions but was exposed directly to water in the third.

**Results:** At 24 hours, mean bond strengths for the resin-based adhesives ranged from 7.2 MPa for Adper Prompt to 51.5 for OptiBond FL. The glass ionomer bond strengths were 4.0 MPa for Reactmer, and 19.9 MPa for FujiBond LC. At 1 year, for specimens in which the dentin had not been exposed directly to water, the means ranged from 0 MPa for Scotchbond 1 to 40.7 MPa for OptiBond FL. The bond strengths of all resin-based adhesives had decreased significantly. The bond

strength of FujiBond LC was similar to its 24-hour mean, and the bond strength of Reactmer had actually increased. For the specimens with direct exposure to water for 1 year, the means were zero for both Scotchbond 1 and Prompt, and 43.6 MPa for OptiBond FL. FujiBond LC was close to zero, and Reactmer’s mean was very close to that of the 24-hour mean.

**Conclusions:** The three-step etch-and-rinse adhesive was the least sensitive to water degradation. The only one approaching this “gold standard” was the two-step self-etching primer system. In general, the use of simplified systems did not improve bonding performance, especially over the long term.

## COMMENTARY

Some of the results are a bit difficult to explain. For example, why did the bond strength of Reactmer improve from 24 hours to 1 year? Nevertheless, this study comes from a highly regarded research group and confirms the results of other studies that have shown that the three-step etch-and-rinse adhesives remain the “gold standard” for resin bonding to dentin, at least in terms of maintaining bond

strength when exposed to water. A recent clinical trial found that the clinical performance of OptiBond Dual-Cure (Kerr), a material similar to the OptiBond FL used in this study, was excellent at 12 years. In the present study, the self-etch primer system was second best for its resistance to water. The

simplified etch-and-rinse and self-etch adhesives did not perform well at all.

#### SUGGESTED READING

De Munck J, Van Meerbeek B, Yoshida Y, et al. Four-year water degradation of total-etch adhesives bonded to dentin. *J Dent Res* 2003;82:136–40.

Shirai K, De Munck J, Yoshida Y, et al. Effect of cavity configuration and aging on the

bonding effectiveness of six adhesives to dentin. *Dent Mater* 2005;21:110–24.

Breschi L, Mazzoni A, Ruggeri A, et al. Dental adhesion review: aging and stability of the bonded interface. *Dent Mater* 2008;24:90–101.

Wilder AD, Swift EJ, Heymann HO, et al. 12-year clinical evaluation of a three-step dentin adhesive in non-carious cervical lesions. *J Am Dent Assoc* 2009;140:526–35.

### RESISTANCE OF TEN CONTEMPORARY ADHESIVES TO RESIN-DENTINE BOND DEGRADATION

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*Journal Dentistry* 2008 (36:163–9)

#### ABSTRACT

**Objective:** The objective of this study was to determine the resistance of resin-dentin bonds created by 10 adhesive systems to degradation after 24 hours, and 6 and 12 months of storage in water.

**Materials and Methods:** The adhesives tested in this study represented a range of different types, including two-step etch-and-rinse, self-etching primers, and all-in-one adhesives. Some of the products would not be familiar to dentists in the United States, but several are popular products—including Single Bond (3M ESPE), Prime & Bond NT (Dentsply Caulk, Milford, DE, USA), Clearfil SE Bond (Kuraray), and Prompt L-Pop (3M ESPE).

The occlusal portions of 60 extracted human molars were ground flat to expose flat dentin

surfaces. The teeth were randomly assigned to 10 groups of six for treatment with the various adhesives using their respective manufacturers' directions. Composite crowns were built up on the dentin, and the specimens were stored in water for 24 hours, 6 months, or 1 year. Because the specimens were stored intact, the resin-dentin interface was exposed to water only indirectly—that is, through the enamel. At the designated times, specimens were sectioned into small beams for MTBS testing, which was accomplished using a universal testing machine. Fractured specimens were examined at 40× magnification to evaluate the failure mode.

**Results:** The 24-hour bond strengths of Single Bond, Prime & Bond NT, Clearfil SE Bond, and Prompt L-Pop were 41.0, 38.4, 40.2, and 18.4 MPa, respectively.

Bond strengths generally declined with storage time. At 1 year, Single Bond had a mean bond strength of 39.0 MPa, and Clearfil had a mean of 32.0 MPa. These were the only adhesives for which bond strengths were not significantly lower at 1 year than at 24 hours. At 1 year, some of the self-etch materials had premature failures so that bond strength could not even be measured.

**Conclusions:** Etch-and-rinse adhesive and a mild two-step self-etch adhesive were the only adhesives able to maintain consistent bond strengths after 1 year of indirect water exposure.

#### COMMENTARY

Numerous studies on resin bond durability have begun to appear in the literature. The present study reports results that are not atypical. Bond strengths tend to decline with time, particularly when the

dentin interface is not protected by a circumference of bonded enamel. In this study, the good results achieved with the etch-and-rinse material Single Bond can be partly attributed to its effectiveness in enamel bonding. The excellent enamel seal provided by this material, and others like it, provides some protection for the underlying dentin bonds. Clearfil SE Bond uses a mildly acidic self-etching

primer and does not provide the same sort of enamel bond. Its mildly acidic nature helps provide a stable dentin bond that probably involves at least some chemical bonding to residual mineral in the hybrid layer.

#### SUGGESTED READING

Carrilho MR, Carvalho RM, Tay FR, et al. Durability of resin–dentin bonds related to water and oil storage. *Am J Dent* 2005;18:315–9.

Toledano M, Osorio R, Osorio E, et al. Durability of resin–dentin bonds: effects of direct/indirect exposure and storage media. *Dent Mater* 2007;23:885–92.

Erhardt MC, Toledano M, Osorio R, Pimenta LA. Histomorphologic characterization and bond strength evaluation of caries-affected dentin/resin interfaces: effects of long-term water exposure. *Dent Mater* 2008;24:786–98.

Reis AF, Giannini M, Pereira PN. Effects of a peripheral enamel bond on the long-term effectiveness of dentin bonding agents exposed to water in vitro. *Biomed Mater Res B Appl Biomater* 2008;84B:10–7.

#### THE BOTTOM LINE

Effective adhesion of resin-based materials to dentin requires three distinct functions: etching or conditioning, priming the surface with hydrophilic resins, and application of a hydrophobic resin. The systems that perform these functions in sequence are the three-step etch-and-rinse adhesives. Although these have proven to be the “gold standard” in dentin bonding, the desire for improved clinical efficiency and less complexity has resulted in the development of simplified methods for bonding. Simplified products combine two or even all three of the required functions into a single step.

As the research studies reviewed in this *Critical Appraisal* demonstrate, all resin bonds to dentin tend to degrade with time. The precise mechanisms of this degradation are only beginning to be understood, and are beyond the scope of this piece.

Regardless of the reasons for bond deterioration, the restorative dentist must be aware of it and understand its implications. The first implication is the most obvious—bonded restorations cannot be expected to last forever!

Clinically, the importance of enamel cannot be overstated. With all of the emphasis on “dentin bonding” in the scientific literature and elsewhere, we tend to forget that enamel is the better and preferred substrate for bonding. The presence of enamel (e.g., at the gingival margin of a Class II restoration) contributes greatly to the longevity of a bonded restoration. When the enamel is effectively bonded, it protects the underlying and more fragile bonds of resin to dentin from the deleterious effects of water in the oral environment.

Also, the more complex bonding systems provide more reliable and durable bonds to dentin than the simplified systems do. The three-step etch-and-rinse systems remain the proven gold standard for bonding, especially when the dentin is not protected by a circumference of enamel. The second choice for bonding in the absence of peripheral enamel is the two-step, self-etching primer systems. For purposes of longevity, the least desirable approach is the use of the simplified all-in-one systems. It is entirely likely that these materials will continue to improve, but the current all-in-one systems do not provide the most reliable bond of resin to either dentin or enamel.

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