Dentin/enamel adhesives: review of the literature

Edward J. Swift, Jr, DMD, MS

Dr. Swift is professor and chair, Department of Operative Dentistry, University of North Carolina, Chapel Hill, NC. Correspond with Dr. Swift at ed_swift@dentistry.unc.edu

Abstract

This paper describes the development of dentin bonding systems, and describes the current strategies for bonding composite resin materials to dentin. Two main strategies are available — total-etch or self-etch — and each has unique advantages and disadvantages. For each category, simplified systems that reduce the number of application steps are available. Currently, the market is moving towards self-etching materials, largely because these are associated with less post-operative tooth sensitivity. However, the clinical performance of most of these materials is not yet proven.(*Pediatr Dent*. 2002;24:456-461)

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Enamel bonding has been widely and successfully used in dentistry for over 20 years, but reliable dentin bonding has been possible only during the last decade. A wide assortment of dentin/enamel adhesives is available on the market today. However, despite the larger number of commercial products, only 2 distinct strategies are currently recognized as successful approaches for bonding resin-based composite to dentin. Each of the 2 basic strategies can be used in either a more traditional or a simplified approach; thus, 4 types of modern dentin adhesives can be described. None of the four types is "perfect," and each has its own peculiar strengths and weaknesses. The practicing pediatric dentist must evaluate these strengths and weaknesses and the available scientific evidence about clinical performance carefully to choose the appropriate system(s) for use in his or her practice.

Enamel bonding

The foundation for modern adhesive dentistry was laid in 1955, when Buonocore reported that acids could be used to alter the surface of enamel to "render it more receptive to adhesion."[1] Basing his work on the common industrial use of phosphoric acid to improve adhesion of paints and acrylic coatings to metal surfaces, Buonocore discovered that acrylic resin could be bonded to human enamel that was conditioned with 85% phosphoric acid for 30 seconds. He predicted that this "bonding" technique could be used in various dental procedures, including Class III and Class V restorations and pit and fissure sealants.

Enamel bonding did not become widely used until more than 20 years after Buonocore's first publication on the topic. However, the technique is now commonplace and has revolutionized the practice of restorative dentistry as well as related disciplines such as esthetic, preventive and pediatric dentistry and orthodontics.

Problems in bonding to dentin

Interest in adhesion of restorative materials to dentin actually predated Buonocore's 1955 report on enamel etching and bonding.[2] However, bonding of resins to dentin is far more difficult and less predictable than bonding to enamel. Dentin not only has a more complex histologic structure than enamel, but also varies more with location. On average, enamel is 92% inorganic hydroxyapatite by volume, and dentin is only 45% inorganic. Dentinal hydroxyapatite crystals are randomly arranged in an organic matrix that consists primarily of collagen, and are not regularly arranged as they are in enamel.[3]

Dentin should be considered not as a separate entity, but as part of a complex with the pulp. It contains numerous fluid-filled channels or tubules that run from the pulp to the dentinoenamel junction (DEJ). The relative area of dentin occupied by tubules decreases towards the DEJ from approximately 45,000 per mm^2 at the pulp to about 20,000 per mm^2 at the DEJ in coronal dentin. Investigators have calculated that tubules occupy 22%-28% of the cross-sectional area near the pulp and only 1%-4% near the enamel.[4,5] An odontoblastic process extends from the pulp into the inner portion of each tubule.[6] The plasma-like fluid in the tubules is under a slight but constant outward pressure from the pulp. The intrapulpal pressure is estimated to be 25-30 mm Hg (or 34-40 cm H_2O).[7] Variations in dentin structure and composition occur not only with differences in depth, but also from region to region of the tooth. The permeability characteristics (or hydraulic conductance) of dentin clearly illustrate these regional variations. For example, the permeability of occlusal dentin is higher over the pulp horns than at the center of the occlusal surface, proximal dentin is more permeable than occlusal dentin and coronal dentin is more permeable than root dentin.[8,9]

When materials are bonded to dentin exposed during tooth preparation, the inherent complexity of the dentin morphology is further complicated by the formation of a "smear layer."[10] The smear layer consists of debris that is burnished against, and bound to, the dentin surface during instrumentation. Depending in part on the type of cutting instrument used, the smear layer is typically just 0.5-5.0 μm thick, but it occludes the orifices of the dentinal tubules. Although the smear layer acts as a "diffusion barrier" that decreases dentinal permeability, it also can be considered an obstruction that prevents resin from reaching the underlying dentin substrate.[11]

Alterations in the mineral content and structure of dentin—as in caries-affected or sclerotic areas—represent another source of difficulty in bonding resins to dentin.[12] Resin penetration into sclerotic dentin is less than in normal dentin, and this may compromise the outcome of bonding procedures.

Development of dentin adhesives

Begun in the 1950s, research on dentin bonding continued at a slow pace through the 1960s and 1970s and culminated in the introduction of the first commercial dentin adhesive in 1975. However, this product had very poor clinical results when used to restore cervical erosion lesions without mechanical retention.[13]

A "second generation" of dentin bonding agents was introduced for clinical use in the early 1980s. While a few of these products are still available (Dual-Cure Scotchbond, 3M ESPE; Bondlite, Kerr), they are no longer widely used. Most were halophosphorous esters of unfilled resins such as Bis-GMA (bisphenol A-glycidyl methacrylate) or HEMA (hydroxyethyl methacrylate). They bonded to dentin via surface wetting and interaction between their phosphate groups and calcium ions in the smear layer.[14] Shear dentin bond strengths were only about 1-10 MPa,[14,15] and were too weak to counteract the polymerization shrinkage of composite resin.[16] In clinical trials, fairly high percentages of cervical restorations were lost in 1 or 2 years.[17,18] A major reason for the poor performance of these adhesives is that they bonded to the smear layer rather than to the dentin itself. Thus, their bond strength was limited by the cohesive strength of the smear layer or by the weak and unstable adhesion of the smear layer to the underlying dentin.[19]

A third generation of adhesives was introduced in the mid- to late 1980s, and these either modified or removed the smear layer to permit resin penetration into the underlying dentin. Prominent examples of this generation including Scotchbond 2 (3M ESPE), Gluma (Bayer), Tenure (Den-Mat Corporation), Prisma Universal Bond 2 and 3 (Dentsply Caulk), Syntac Classic (Ivoclar Vivadent) and XR Bonding System (Kerr), which usually were greater than those of the second-generation agents. Clinically, 3-year studies of Scotchbond 2 and Gluma in cervical areas indicated that these systems also had considerably better clinical performance (retention, marginal integrity, etc) than earlier adhesives. However, they did not nearly approach the desired goal of 100% retention.[17,18]

Current strategies for resin-dentin bonding

Total-etch adhesives

In the United States, the current era of resin-dentin bonding began in the late 1980s, with the introduction of the "total-etch" concept. Based on the work of Fusayama and others in Japan,[20] Bertolotti and Kanca proposed a technique that involved phosphoric acid-etching of dentin as well as enamel, followed by the application of relatively hydrophilic resins that had recently become available.[21,22] The total-etch technique was considered quite controversial at first, as earlier research had suggested that dentin etching would damage the pulp.[23] Dentists in the United States and most other regions of the world had been taught that dentin etching was taboo. Eventually, the earlier studies were reinterpreted, new research was performed and the total-etch technique became widely accepted as both safe and effective.

Many commercial products based on the total-etch technique were developed and released in the early 1990s. Several of those products, including All-Bond 2 (Bisco, Inc.), OptiBond FL (Kerr), Perma Quick (Ultradent Products) and Scotchbond Multi-Purpose (3M ESPE), remain available today. Although chemical composition and application techniques vary from one material to the next, these adhesive systems all include 3 fundamental steps for achieving a bond of resin to dentin.[24-26]

The first step, phosphoric acid-etching, removes the smear layer, opens the dentinal tubules and decalcifies the intertubular and peritubular dentin. The depth of decalcification is affected by the pH, concentration, viscosity and application time of the etchant. Hydroxyapatite crystals are dissolved, leaving a collagen meshwork that can collapse and shrink due to the loss of inorganic support. (Preventing this collapse is an important consideration for total-etch adhesive systems, and will be discussed later in this section.)

After the etchant is rinsed off, a primer containing a solvent such as acetone, ethanol and/or water and one or more bifunctional resin monomers is applied. Primer resins such as hydroxyethylmethacrylate (HEMA) contain 2 functional groups—a hydrophilic group and a hydrophobic group. The hydrophilic group has an affinity for the dentin surface, and the hydrophobic (methacrylate) group has an affinity for resin. The primer wets and penetrates the collagen meshwork and also increases surface energy, and therefore wettability, of the dentin.

A bonding agent is applied and penetrates into the primed dentin. The bonding agent typically contains a hydrophobic resin such as Bis-GMA, but many also contain a more hydrophilic resin such as HEMA to improve wetting. Although most bonding agents are unfilled, specific products (eg, OptiBond FL) contain filler particles, as some evidence suggests that filled resins provide stress relief at the tooth-restoration interface. The bonding agent copolymerizes with the primer to form an intermingled layer of collagen fibers and resin called the "hybrid layer," "resin-reinforced zone" or "resin-infiltrated layer." This hybrid layer, which was first described by Nakabayashi et al, in 1982,[27] has been considered the most important factor for ensuring a good bond between resin and dentin. However, several studies have indicated that successful bonding can be achieved even if collagen is removed by sodium hypochlorite or collagenase after etching and before resin application. Resin penetration into partially demineralized dentin and irregularities within the dentin just beneath the collagen meshwork is obviously important, and in fact may be as important or more important than formation of the classically described hybrid layer.[28,29]

Scanning electron microscopy (SEM) evaluations of bonded interfaces reveal that many adhesives form long resin tags within the dentinal tubules of extracted teeth. The tags typically have an impressive appearance and may convey some information about the wetting ability of a given material. Resin tags also have been observed after bonding resin to dentin in vital teeth in vivo. If the sides of these resin tags are firmly bonded to the tubule walls, they can contribute to the overall bond of resin to dentin.[30]

High bond strengths have been reported for the 3-step, total-etch adhesives; in fact, dentin bond strengths sometimes have exceeded enamel bond strengths.[31-33] Performance in microleakage tests has also been generally good.[34] In addition to laboratory studies, a number of clinical trials have now been reported on this group of adhesives. Retention rates of Class V restorations without mechanical retention have been in the range of 69%-100% in studies of up to 3 years.[18,35-41]

Because the etch/prime/bond adhesives require multiple applications, there are numerous opportunities for errors to occur.[42] Therefore, manufacturers have attempted to simplify the systems, and many have developed so-called "one-bottle" systems. While these still require etching as the first step, the primer and bonding functions are combined into a single solution; hence, the term "one-bottle." Over the last several years, these products—including familiar ones such as Prime & Bond NT (Dentsply Caulk), OptiBond Solo (Kerr) and Single Bond (3M ESPE)—have been the most widely used adhesives.

One-bottle adhesives contain mixtures of hydrophilic and hydrophobic resins carried in a solvent such as acetone, ethanol or (rarely) water. Their bonding mechanism is the same as that of the 3-step, total-etch systems, and—like some of the 3-step systems—nearly all require a moist bonding technique.[43]

When dentin is etched, the surface is depleted of the hydroxyapatite crystals that support the collagen framework. Thus, etching leaves a porous, collagen-rich surface that can collapse if dried, limiting penetration of resins applied to that surface. In a moist-bonding technique, the surface is not dried after etching and rinsing, and, therefore, the collagen remains in position and behaves almost as a sponge. The acetone or ethanol solvent displaces water and carries the resins into the collagen.[44,45]

If the surface must be dried—eg, to check the enamel etch—it should be remoistened. Various materials have been tested as rewetting agents, including water, which does not rewet the surface rapidly. Better alternatives are aqueous solutions of HEMA such as Aqua-Prep (Bisco, Inc.) or Gluma Desensitizer (Heraeus Kulzer).[46,47] The latter also contains glutaraldehyde, which might stabilize the collagen layer, thus facilitating resin penetration.[47]

As with the 3-step, total-etch systems, the one-bottle systems generally have demonstrated good performance in laboratory testing of dentin bond strengths and marginal seal.[33,48-50] Most also bond extremely well to either dry or moist enamel. Unfortunately, only a few clinical trials have been reported on the one-bottle systems. However, the studies that have been published generally have reported good results. A recent study on two such adhesives reported a retention rate of about 90% for Class V restorations placed without mechanical retention.[51]

Despite the good laboratory and clinical performance of the total-etch adhesives, their use results in a frustratingly high incidence of postoperative sensitivity. Once the dentin is etched, it must be sealed well, which is not always possible under clinical conditions. The problem of postoperative sensitivity is most common in situations that magnify the effects of composite polymerization shrinkage. An example of this is a simple Class I posterior composite restoration. The Class I has a configuration factor (or C-factor) of 5, which indicates that the ratio of bonded to unbonded walls is 5:1.[52] When the composite shrinks during polymerization, some stress relief occurs at the occlusal (unbonded) surface, but inevitably some stress also occurs at the bonded interfaces. Furthermore, most of the dentin bonding occurs at a single location, the pulpal floor. The entire circumference of the restoration is bonded to enamel. If the bond of resin to the enamel periphery exceeds the bond to the dentin, the composite may partially debond from the pulpal floor, leaving a gap between resin and dentin. When the patient functions on the tooth, hydraulic forces within the fluid-filled gap and underlying tubules stimulate pulpal nerve endings, causing a sensation of sensitivity or pain.[53]

Self-etch systems

Largely because of this continuing problem with total-etch adhesives, much of the current product development and clinician interest is focused on self-etching systems. Although only recently gaining popularity in the United States, self-etch systems have been available in Japan for several years. The original self-etch systems included two steps—an acidic, self-etching primer followed by a separate bonding resin. Some of the newer systems are considered "all-in-one," and contain etch, prime and bond functions in a single solution. The former group of materials can be described as self-etch primer systems, and the latter can be called self-etch adhesives.

Examples of current 2-step, or self-etch primer, systems include Clearfil SE Bond and Clearfil Liner Bond IIV (both from Kuraray). The former is intended for use with direct restorations only, while the latter is designed for more universal use. Unfortunately, little research has been published concerning these relatively new systems. They are simple to use and, anecdotally, are associated with very little postoperative sensitivity.

Much research has been published on the performance of an earlier system from the same manufacturer (Liner Bond II).[54-57] However, it should be noted that the chemistry of the newer materials is somewhat different from that of this earlier system.[57] Based on research with the older systems, the chief concern about the self-etch systems is that they might not etch enamel effectively. Bonding to uninstrumented enamel is particularly challenging, so enamel should be instrumented in some way before etching with these systems.[55,59] Furthermore, one in vitro study suggests that the initial bond might deteriorate significantly with aging, which could lead to premature clinical failures.[59] Bonding of self-etch systems to sclerotic or caries-affected dentin also might be problematic.[60,61]

The most recent developments in dentin bonding have been in the area of the self-etch adhesives, or all-in-one systems such as Prompt L-Pop (3M ESPE), One Up Bond F (Tokuyama and J. Morita USA) and Touch & Bond (Parkell). These materials, particularly Prompt L-Pop, have rapidly gained popularity in response to their simplicity of use and low incidence of postoperative sensitivity.

Very little independent research has been published on these new materials, and most of it has been on Prompt L-Pop. Perhaps surprisingly, Prompt appears to bond more strongly to enamel than to dentin. However, this finding can be explained by its pH, which is unusually low for a self-etch agent. One clinical trial of Class I posterior composites reported essentially zero postoperative sensitivity, compared with 20% for restorations placed using Prime & Bond, a total-etch adhesive.[62] Six-month retention rates of 76%-100% have been reported for Class V restorations placed using Prompt as the adhesive.[63-65] Such data should be regarded as very preliminary at best. Eighteen- to 36-month studies will provide much more information about the long-term reliability of this or any other adhesive.

Bonding to primary teeth

The ultrastructure of primary dentin presents challenges that go beyond those encountered in permanent teeth. For example, primary dentin has even greater regional variation than permanent dentin, and has been described as containing more, and in some cases unusually large, dentinal tubules.[66] Much less is known about the performance of dentin adhesives in primary teeth than in permanent teeth—if for no other reason than the fact that primary teeth are difficult to obtain in sufficient quantity for research. In the competition for exfoliated teeth, the tooth fairy usually wins the competition over the dental researcher. Studies with earlier generation of adhesive systems routinely reported lower bond strengths to primary than to permanent dentin.[67,68] However, recent studies of total-etch adhesives have revealed similar bond strengths to both types of dentin.[69]

Summary

Two primary strategies are currently available for bonding resin to dentin: total-etch and self-etch. For each strategy, simplified approaches are available, so that 4 distinct categories of dentin adhesives can be identified: (1) 3-step, total-etch; (2) total-etch using combined primer/adhesive ("one-bottle"); (3) self-etch primers with a separate bonding agent; and (4) self-etch, or "all-in-one," adhesives. Each category has advantages and disadvantages, so none is clearly better than the others, although much more laboratory and clinical data are available concerning the total-etch systems. At present, the profession seems to be moving in the direction of the self-etch, long-term adhesives, but the clinical performance of these materials is not yet proven.

Regardless of the bonding approach the clinician selects, he or she must be aware that proper technique and attention to detail are critical to success.[70-72] In addition, dentin is a highly variable substrate, and this variability may present problems in specific cases.

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