Dentinal Adhesion

by Karl F. Leinfelder, DDS, MS

The Quest for the Perfect Adhesive

Bonding to tooth structure has been a long-term objective for both researchers and manufacturers alike. The first success in adhesive bonding came about through the efforts of a Swiss chemist by the name of Hagar. Working with an acrylic resin restorative material identified as Sevriton, he was actually the first individual to generate adhesion of resin to dentinal surfaces. The next successful effort was achieved by Michael Buonocore who demonstrated the ability for bonding methyl methacrylate resin to the surface of enamel. Subsequent efforts by many investigators have identified optimum types of acids, concentrations and times of application. Currently when etching enamel surfaces, most clinicians use a 37 percent concentration of phosphoric acid for 15 to 20 seconds.

The quest for an agent that would successfully bond to dentin has been considerably more time-consuming. Over the years, the adhesive bond strength has gradually risen. Currently, the successful dentin adhesives exhibit shear bond strengths of 20 to 25MPa. Such a level is necessary to overcome the contraction forces exhibited by the overlying composite resins during the

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polymerization process. Values that are appreciably lower will result in micro-leakage, debonding of the restoration, marginal discoloration, post-operative sensitivity and secondary caries.

Bonding Mechanism

The bonding mechanisms of composite resin to enamel and to dentin are considerably different. In the case of enamel, the phosphoric acid superficially removes a portion of the enamel rod prisms. The depth of the etched enamel defect ranges from 15 to 25 microns. The eroded surface is then filled and sealed with a low viscosity composite resin. Bonding to enamel essentially is micro-mechanical.

Adhesion to dentin is considerably different than it is to enamel. While the enamel consists mainly of hydroxyapatite, dentin consists of both hydroxyapatite as well as collagen. Histologically, the collagen fibers within the dentin are surrounded by the hydroxyapatite. Using phosphoric acid (37%), a portion of the hydroxyapatite is removed, leaving spaces or voids between the collagen bundles. A low viscosity resin is then inserted onto the surface. Due to its excellent wetting characteristics, the bonding agent penetrates into the surface and fills up the spaces left by the dissolution of the hydroxyapatite.

When the phosphoric acid is placed by the clinician, the acid penetrates into the surface of the dentin by about 10 microns. The depth of penetration into the dentinal tubules, however, is closer to 100 microns.

The bonding to dentin actually is a substitution of a highly wettable resin monomer for hydroxyapatite crystals, which surround the collagen bundles. Identified as hybridization, the process was first described by Nobuo Nakabayashi in Japan. Nearly all dentin bonding procedures carried out at the present time are based upon this principle.

Types of Dentin Bonding Agents

Presently there are four types or generations of dentin bonding agents:

- Generation Four: separate bottles of a) acid, b) primer and c) adhesive
- Generation Five: a) acid and b) combined primer and adhesive
- Generation Six: a) combined acid and primer and b) adhesive
- Generation Seven: all components in one bottle

While there are four generations of dentin bonding agents, they can all be placed into two groups. These can be identified as total-etch and self-etch. The total-etch technique (generations four and five) consists of first eliminating the hydroxyapatite by means of an acid etching agent, followed by the infiltration into the evacuated zones by the dentin bonding agent. The self-etch systems (generations six and seven) consist of simultaneously eliminating the hydroxyapatite and at the same time filling up the evacuated spaces. The basic advantage to the self-etch system is that it provides greater protection against post-operative sensitivity. According to the *Dental Advisor,* it is the system of choice when using posterior composite resins. The logic behind this finding is that post-operative sensitivity is associated more with Class I and II cavity preparations than it is with anterior restorations of composite resins.

When using the total-etch dentin bonding agents, the acid, as already mentioned, creates microscopic voids within the dentin. This condition is generated by extracting the hydroxyapatite crystals. Since the subsequent application of the dentin adhesive might not totally fill up these evacuated sites, the odontoblastic processes might remain exposed. This, in turn, can result in post-operative sensitivity. In the case of the self-etching agents, the evacuated crystalline sites are filled simultaneously with the bonding agent. As a result, the potential for exposed odontoblastic process is not a possibility.

The self-etching dentin bonding agents have become quite popular in North America. There are a couple of reasons for this. The first is the near absence of post-operative sensitivity. The second is relative simplification of technique, particularly in comparison to the fourth generation (total-etch) bonding system. In spite of the advantage, there are numerous disadvantages associated with their use. The first and perhaps most serious of these is a less-than-ideal etching of the enamel. When first introduced, particularly in the case of the seventh generation dentin bonding agent, some of the restorations exhibited a thick brown line at the restoration-tooth interface. It turns out that the discoloration resulted from an inadequate etching of the enamel, which consequently led to leakage along the margins.

A couple of suggestions have been offered for eliminating the problem. The first consists of etching the enamel portion of the preparation using phosphoric acid (37%) for 15 seconds. This then is followed by application of the dentin bonding agent to the rest of the preparation (dentin). Care should be taken to restrict the phosphoric acid etching to the enamel only. The second recommendation is to use the self-etch dentin bonding agent on the enamel for 30 seconds and the dentinal portion of the preparation for 15 seconds. The added 15 seconds (30 seconds total) of application to the enamel appears to have solved the problem.

Another clinical problem associated with the self-etching bonding agents is that the initial shear bond strength is somewhat lower than that associated with the total-etch systems. The reason is not clear. Also the clinical significance has not been identified. Additionally, the extended bond strength of the self-etch bonding systems tends to decrease over an extended period of time as compared to those classified as total-etch.

Incomplete Hybridization

While the new generations (four through seven) of dentin bonding agents are very effective in their ability to bond to

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dentin surfaces, they all are technique-sensitive. Failure to follow the manufacturer's recommendation for use might result in clinical failure. It has been demonstrated that not following the prescribed techniques of application can result in incomplete hybridization. Specifically while the superficial surface of the dentin might be hybridized, there might be a zone beyond this region that is only partially hybridized. The most probable cause is failure of the dentin bonding agent to penetrate the vacancies created by the evacuated hydroxyapatite. Under such conditions the collagenous fibers remain unprotected and eventually undergo a biologic degradation. Such a condition is characterized by eventual micro-leakage (nano-leakage), delayed sensitivity and debonding.

One or more techniques have been recommended to minimize the potential for incomplete hybridization. The techniques depend upon the type of bonding system being employed. In the case of the "total-etch" bonding systems the following technique is suggested:

1. Applying the phosphoric acid gel to the entire preparation (dentin and enamel) for a period of 15 to 20 seconds.
2. Wash thoroughly (water and compressed air) for several seconds.
3. Disperse air on the wetted surface so that a slight glistening (reflection of light) can be seen. Or blot the wetted surface with dry cotton to remove any excess water. (Dentin bonding agents are hygroscopic and need moisture to effectively penetrate the surface.)
4. Apply the primer (fourth generation) by swabbing the surface of the preparation continuously for 15 seconds. After air dispersing, repeat the process.
5. Apply the adhesive (15 seconds) and then air disperse.
6. In the case of the fifth-generation bonding agent (primer and adhesive are in the same bottle) apply the bonding agent; after 15 seconds air disperse; repeat the process.
7. Light-cure for 15 seconds.

When dealing with the "self-etch" bonding agents (generations six and seven), a specific technique has been recommended for proper bonding to enamel as well as dentin.

1. The first (as already mentioned) consists of etching the enamel surfaces of the preparation only with a 37% gel concentration for 15 seconds.
2. Wash and air disperse.
3. Next, place the dentin bonding agent onto all the surfaces (enamel and dentin) for 15 seconds and air disperse.
4. Place the adhesive and air disperse after 15 seconds.
5. Cure with the light for 15 seconds.

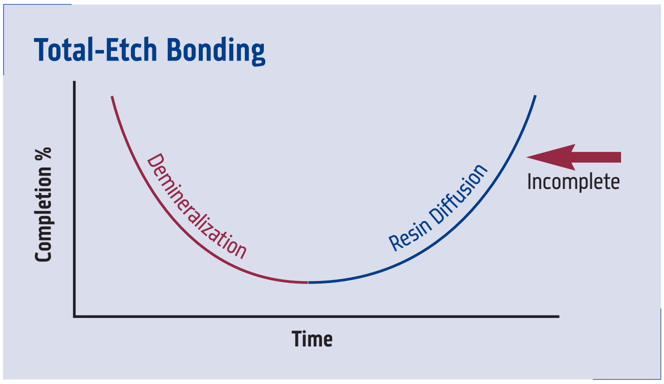
An alternative method (used less frequently) for successful bonding of self-etching bonding agents consists of the following:

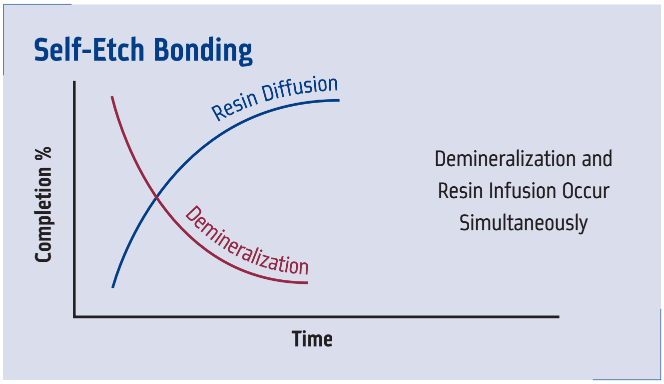
1. Place the bonding agent on the enamel only for 15 seconds.
2. Next, place the bonding agent on the dentinal portion of the preparation for 15 seconds, then air disperse. This technique automatically leaves the bonding agent on the enamel twice as long as the dentin thereby producing good etching and bonding to the enamel.
3. Place the adhesive and air disperse after 15 seconds.
4. Light cure for 15 seconds.

Note: When using the seventh-generation dentin bonding agent, everything is in one bottle.

While the dentin bonding agents have been quite successful for numerous reasons, they remain one of the more technique-sensitive materials used by the clinician. It is highly recommended that the practitioner follow the manufacturer's recommendations for any one of the described systems.

As already mentioned, the potential for post-operative sensitivity is less likely with the self-etching bonding agents than it is with the total-etch systems. One of the basic reasons can be related to the manner in which the crystalline structure is removed from the dentin. In the case of the self-etching systems, the extraction of the hydroxyapatite and the infiltration of the resinous bonding agent occur nearly simultaneously. In the case of the total-etch technique, the extraction of the hydroxyapatite is completed before the resin infiltration is initiated. Such a condition yields the potential for uncompleted sealing and thereby access to the odontoblastic processes. A graphic illustration of the etching and resin infiltration associated with the two different types of etching and infiltration are depicted in the two illustrations (Figs. 1 & 2).





Post-operative Sensitivity

Post-restorative pain is nothing new to the clinician. In the case of amalgam for example, it is quite common for the patient to complain of sensitivity when drinking cold water. This sensitivity commonly starts immediately after insertion of the restoration but gradually tapers off within two weeks or so. The cause probably relates to a microscopic space between the amalgam and the walls of the preparation. It is proposed that this space approximates 10 to 12 microns when the restoration is inserted but then fills in with corrosion products over a period of time.

Pain can also be experienced after the insertion of composite resins involving the occlusal surfaces. While the mechanism for this occurrence might be due to a number of factors, it is probable that it can be related to less-than-ideal marginal adaptation. Failure to use a flowable composite resin in conjunction with a highly viscous (packable) composite resin might lead to less-than-ideal marginal adaptation. Masticatory forces on restorations in which the margins are not entirely sealed might introduce hydraulic forces on the odontoblastic processes.

Other factors that might contribute to sensitivity include polymerization shrinkage of the composite resin, microbial invasion, temperature reduction, pulpal hyperemia and transmission of ions of salt or sugars onto the surfaces of the odontoblastic processes. Post-operative sensitivity associated with an ideal cavity preparation and routinely placed posterior composite resin is commonly related to the odontoblastic processes (OP). Located between the pulpal surface and the pre-dentin, the odontoblast extends its process into the dentinal tubules. The depth of penetration is about one-third the thickness of the dentin itself. The process according to Brännström is quite sensitive to negative pressures. Interestingly however, it is not sensitive to pressures that are positive.

Several examples can be given. Directing air to the surface of a cavity preparation that was generated without anesthesia will almost invariably induce a painful sensation. The air, which is forced across the surface of the tubules causes a slight evaporation of the fluids surrounding the odontoblastic process. This, in turn, will create a negative pressure on the processes, which results in a painful response. The pain will continue until the odontoblastic fluids distal to the effected site replace the evaporated fluids and thereby return the pressure to normal.

Introducing cold temperatures to the odontoblastic process is a major contributor to pain. Drinking cold liquids causes a contraction of the fluids surrounding the processes. The resultant negative pressure on the odontoblastic process causes pain. Application of cold liquids or ice on the cervical region of teeth in which the tissues have receded is a common example. The pain disappears when the processes return to normal body temperature

There are several things that can be done to prevent post-operative sensitivity. The first of these is to use either a flowable composite resin or a glass ionomer (Fuji II) under the composite resin restoration. The flowable composite resin controls the amount of contraction forces generated by the composite resin whereas the glass ionomer prevents micro-leakage. Exhibiting a coefficient of thermal expansion that is the same or similar to tooth structure produces the latter characteristic. Incidentally, another positive feature of the glass ionomer is that it releases fluoride ions, which are not only incorporated into the tooth structure but kill micro-organisms. It is interesting to note that glass ionomers uniquely do not lose substantial substance in the process.

If post-operative sensitivity continues to be a problem in spite of addressing all the factors just discussed, one should consider adopting a sixth-generation (self-etch) bonding agent. Since decalcification of the hydroxyapatite and infiltration of the dentin bonding agent occur nearly simultaneously, the potential for exposed odontoblastic processes is, for all practical purposes, avoided. Clinicians in the United States have overwhelmingly done so when dealing with posterior composite resins.

Currently Available Dentin Bonding Agents

Due to the clinical success and increasing modifications, the number of dentinal adhesives now on the market is quite impressive. Furthermore, for each type or generation of material

Commonly Used Dentinal Adhesives

Some of the more commonly used agents, their respective manufacturers and the generation to which they belong are listed below:

Generation Four
- All-Bond 2 (Bisco)
- Bond-It (Pentron)
- Optibond (Sybron/Kerr)
- ScotchBond MP (3M ESPE)
- Tenure (Den-Mat)

Generation Five
- Bond-1 (Pentron)
- ExciTE (Ivoclar)
- Gluma Comfort Bond (Heraeus/Kulzer)
- OptiBond FL (Sybron/Kerr)
- Prime & Bond (Dentsply/Caulk)
- Single Bond (3M ESPE)

Generation Six
- AdheSE (Ivoclar)
- Nano-Bond (Pentron)
- Prompt-L-Pop (3M ESPE)
- SE Bond (Kuraray)
- Simplicity (Apex)
- Xeno III (Dentsply/Caulk)

Generation Seven
- All-In-One (Sybron/Kerr)
- AQ Bond (Sun Medical)
- Clearfil S3 (Kuraray)
- G-Bond (GC America)
- iBond (Heraeus Kulzer)
- Xeno IV (Dentsply/Caulk)

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there is a modification of the instructions for use. As a result it can be quite confusing for the clinician.

The directions for each generation of material can be appreciably different while the instructions for each material within a generation might be only slightly different.

Degradation of Bond Strength

It's been more than 10 years since it was first determined that bond strengths associated with the hybridization process might not stable.[1] In a clinical study, the researchers restored a fairly large number of primary teeth. They used a conventional composite resin and a currently available dentin bonding agent. At a time when the teeth were ready to exfoliate, the teeth were extracted. Using a modified sectioning technique the investigators determined the shear bond strength after various times of clinical service. They also evaluated the hybrid zone for possible changes in the hybrid zone and surrounding areas using scanning electron microscopy. Surprisingly all the teeth underwent a definite reduction in shear bond strength. At the end of a two- to three-year period as a clinical restoration, the average reduction in shear bond strength was nearly 75 percent. Furthermore, the micro-structural studies revealed a deterioration of the collagenous structures. The general findings of this study have been replicated a significant number of times.

Clinically, this means that there is a lack of stability in the region of the hybrid zone. A biological degradation probably means that the clinician should not necessarily depend upon hybridization as the sole means of retention. Mechanical retention with the appropriate instrumentation is still recommended. For example, restoration of abfracted lesions should still involve a generation of undercuts on both the gingival and incisal/occlusal aspects of the cavity preparation. It is for this reason that surfaces of a veneer preparation (resin or ceramic) should consist of enamel (or mostly enamel) rather than dentin. Based upon most recent investigations, the long-term bond strengths of enamel are superior to those of dentin.

There is a possible solution to the problem of instability associated with the use of dentin bonding agents. At least two different studies[2] have demonstrated that there is now a product on the market designed to prevent the degradation process. Identified as Hemaseal & Cide Desensitizer, the product is marketed by Advantage Dental Products. The product is rather easy to use. The procedure consists of etching, washing and then drying. This is followed by application of the Hemaseal with a brush, suctioning off the excess and finally applying the dentin bonding agent.

Final Comments: Incompatibility

The introduction of the fifth-generation dentin bonding agent as a substitute for the fourth-generation system was met with a great deal of excitement. The complexities in technique associated with the earlier product were appreciable. The combination of the primer with the adhesive component certainly made the procedure of application much simpler. Unfortunately, as it turns out, the newer system exhibited a number of problems not associated with its predecessor. The first of these was a higher incidence of post-operative sensitivity. The second was a potential for incompatibility between the bonding agent and dual-cured core materials as well as dual-cure luting agents. Specifically, when some of these agents were used with self-setting cements or core materials, setting would not occur. In the process of combining the primer with the adhesive, it was necessary to reduce the acidity. This, in effect, neutralized the tertiary amine in the bonding agent. When used in conjunction with dual-cure composite-based materials setting might not occur. The problem can be rectified by adding a drop of tertiary amine, which comes in a small cylindrically shaped bottle and is included in some of the fifth-generation dentin bonding packages. ■

1. *Hashimoto, 2000*
2. *Donmez, Hebling*

Author's Bio

Dr. Karl F. Leinfelder earned both his Doctor of Dental Surgery and Master of Science (dental materials) degrees from Marquette University. He joined the faculty of dentistry at the University of North Carolina in 1970. In 1983, he joined the School of Dentistry at the University of Alabama and is the recipient of the Joseph Volker Chair. He also served as Chairman of the Department of Biomaterials until 1994. Presently he holds positions at both universities; adjunct professor at University of North Carolina and professor emeritus at the University of Alabama. Dr. Leinfelder has published more than 275 papers on restorative materials, authored more than 150 scientific presentations, two textbooks on restorative systems and has lectured nationally and internationally on clinical biomaterials.

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1. When phosphoric acid is placed on the surface of dentin, the depth of penetration is about how many microns?
   a. 10
   b. 25
   c. 50
   d. 100

2. Who was the first clinician to generate adhesion to enamel tooth structure?
   a. JF Flagg
   b. Buonocore
   c. Skinner
   d. Black

3. The shear bond strength of dentin adhesives to dentinal surfaces is approximately how many megapascals?
   a. 5
   b. 20
   c. 50
   d. 100

4. What is the nomenclature of the bonding system that uses phosphoric acid as the first step in the bonding procedure?
   a. Self-etch
   b. Total-etch
   c. Hybridization
   d. Single bottle

5. The basic advantage to the self-etch adhesive dentin bonding agent is which of the following?
   a. Faster application
   b. Less expensive
   c. Little or no post-operative sensitivity
   d. Better bond strengths

6. Which generation dentin bonding agent contains all the ingredients in one bottle?
   a. Fourth
   b. Fifth
   c. Sixth
   d. Seventh

7. What is one of the disadvantages of the self-etching dentin bonding agents?
   a. Potential for less-than-optimal etching of the enamel
   b. Shorter shelf life
   c. Inferior clinical performance
   d. Possibly caustic to soft tissues

8. Failure to make certain that the dentin bonding agent sufficiently penetrates the etched dentinal surface might result in which of the following problems?
   a. Post-operative sensitivity
   b. Decreased bond strength
   c. Debonding of the restoration
   d. All of the above

9. The potential for eliminating or significantly reducing post-operative sensitivity occurs under which of the following conditions?
   a. Etching the preparation for at least 60 seconds
   b. Infiltrating the dentin bonding agent simultaneously with the decalcification process
   c. Drying the etched preparation for at least 30 seconds
   d. Failure to refrigerate the dentin bonding agent continuously

10. The odontoblastic process is quite sensitive to negative pressures on its surface. Which of the following conditions can contribute to such a condition?
   a. Cold temperatures
   b. Microbial invasion
   c. Desiccation
   d. All of the above

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