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3M™ Adper™ Single Bond Plus Adhesive

Technical Product Profile

Table of Contents

Introduction

Product Description

3M™ Adper™ Single Bond *Plus* Adhesive, based on the clinically proven 3M™ Adper™ Single Bond Adhesive is a total etch, visible-light activated dental bonding agent incorporating 10 percent by weight of 5nm diameter silica filler. As with the original Adper Single Bond Adhesive, Adper Single Bond *Plus* adhesive is indicated for direct light-cured restorative material as well as for the treatment of cervical sensitivity. Adper Single Bond *Plus* Adhesive may also be used for bonding laboratory fabricated composite or porcelain veneers when used with 3M™ RelyX™ Veneer Cement.

Adper Single Bond *Plus* Adhesive is available in a vial. The vial incorporates a “pinch & flip” cap design that minimizes the messiness associated with typical threaded caps. Unlike the standard black, opaque vial used by manufacturers to shield the photoinitiator from all ambient light, the Adper Single Bond *Plus* vial was designed to shield predominantly the visible light absorbed by the photoinitiator. This design allows for a unique translucent vial that permits visual inspection of the contents.

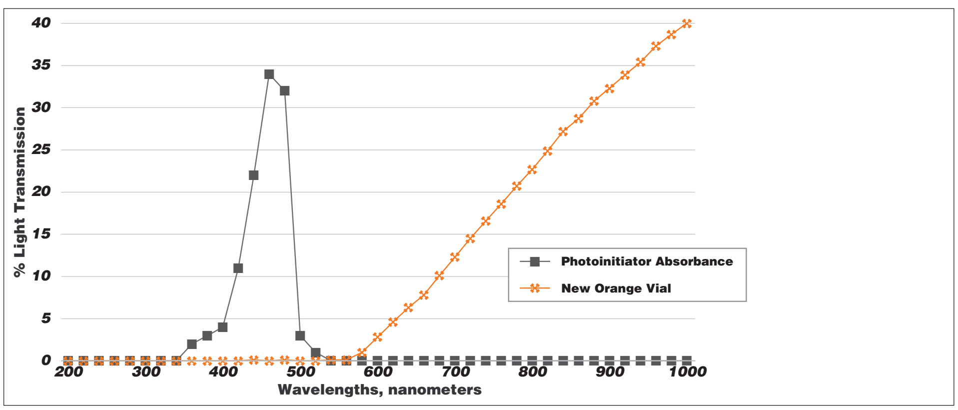


Figure 1. Selective light absorption of Adper Single Bond Plus vial.
Source: 3M internal data

Composition

3M™ Adper™ Single Bond *Plus* Adhesive

With the exception of the silica nanofiller, Adper Single Bond *Plus* adhesive contains the same components as the original Adper Single Bond adhesive: BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids (see structure in figure 2) first introduced in Vitrebond™ Glass Ionomer liner/base and also utilized in 3M™ Adper™ Scotchbond™ Multi-Purpose Adhesive, 3M™ RelyX™ Luting Plus Cement and Vitremer™ Core/Restorative. Incorporation of the polyalkenoic acid into the 3M™ Adper™ Scotchbond™ Multi-Purpose primer has been shown to aid in resisting the detrimental effect of moisture in an elevated relative humidity environment (Fundingsland et al. 1992). The proprietary photoinitiator system allows for a fast 10-second light cure.

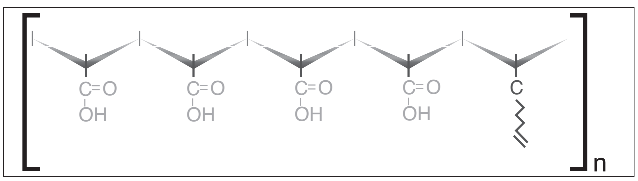


Figure 2. Vitrebond polyalkenoic acid copolymer

Nanotechnology in Adper Single Bond *Plus* Adhesive

Adper Single Bond *Plus* adhesive incorporates 10% by weight of 5 nanometer-diameter spherical silica particles. These silane treated particles are incorporated into the adhesive through a process that prevents agglomeration. As discrete particles, their extremely small size keeps them in colloidal suspension. This means you never have to shake Adper Single Bond *Plus* adhesive prior to using. In contrast, larger filler particles incorporated into some adhesives are at risk of settling out of solution. Such adhesives require routine shaking before use.

Figure 3 demonstrates this difference in suspension stability between the nanoparticles in Adper Single Bond *Plus* adhesive and the much larger particles averaging 0.4 microns in diameter in Optibond Solo Plus™ adhesive (filled 15% by weight).

Approximately four grams of each adhesive was dispensed into separate glass vials and tightly capped: Optibond Solo Plus adhesive was vigorously shaken for 10 seconds prior to dispensing. Within one week of observation, a phase separation in Optibond Solo Plus adhesive became evident. This separation progressed to that shown in Figure 3, which shows Optibond Solo Plus adhesive after storing undisturbed for approximately 4 months. Conversely, the vial containing Adper Single Bond *Plus* adhesive shows no separation after storing undisturbed for approximately 10 months.

Figure 3. (l) Nanofiller in 3M™ Adper™ Single Bond Plus Adhesive remains in solution; (r) filler in Optibond Solo Plus™ does not.

3M™ Scotchbond™ Universal Etchant

Prior to applying 3M™ Adper™ Single Bond *Plus*, enamel and dentin require a separate etch with phosphoric acid. 3M™ Scotchbond™ Universal Etchant is available for the selective enamel etch and total-etch placement modes. Scotchbond Universal adhesive etchant superficially demineralizes the enamel and dentin in preparation for bonding. The etchant is 32% phosphoric acid by weight and has a pH of approximately 0.1. The viscosity of the phosphoric acid gel etchant is modified with fumed silica and a water-soluble polymer. Scotchbond Universal Etchant has a distinct blue color to assist in complete rinsing away of the etchant in order to avoid remnants of fumed silica or phosphoric acid on the prepared tooth surface.

3M™ RelyX™ Ceramic Primer

RelyX Ceramic Primer is a prehydrolyzed, single-phase silane specifically designed to enhance the bond to ceramic surfaces. Test results have also suggested some benefit of application to metals. RelyX Ceramic Primer also has a three-year shelf life at room temperature. The ceramic primer is required for indirect bonding applications such as porcelain veneers, crown and bridge, inlay/onlays and for porcelain repair.

Indications for Use

Adper Single Bond *Plus* Adhesive is indicated for use in the following types of restorations.

- Direct light-cured composite/compomer restorations
- Root surface desensitization
- Porcelain/composite repair
- Porcelain veneers (when used with 3M™ RelyX™ Veneer Cement)

Background

Modern adhesives for bonding restorative materials to tooth structure encompass a range of strategies. The original 3M™ Single Bond Adhesive, introduced in 1997, offered the dental profession the performance of a multi-bottle system in an easy to use, time saving single bottle system.

Adper Single Bond *Plus* Adhesive offers all the features and benefits of the clinically proven 3M™ Adper™ Single Bond adhesive and introduces new nanofiller tecnology.

Bonding Mechanism

Current dental adhesives rely on a micromechanical bond for adhesion to enamel and dentin. With total etch adhesives the mineral phase of enamel and dentin (hydroxyapatite) is superficially demineralized by an acidic agent in a separate step prior to application of the adhesive. The acid used is typically a gelled phosphoric acid with a concentration of approximately 32%.

On enamel, acid etching greatly increases the surface area available for bonding. Low viscosity, polymerizable resins penetrate the porosities revealed by the etching procedure forming an interlocking mechanical bond after curing. These interlocking "resin tags" can be seen in Figure 4 which shows a cross-section of the enamel/adhesive bond interface produced with 3M™ Adper™ Single Bond *Plus* adhesive.

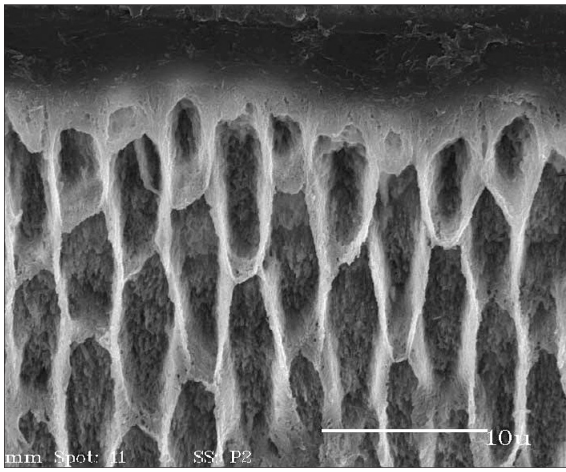


Figure 4. Scanning electron photomicrograph of 3M™ Adper™ Single Bond Plus Adhesive bond to enamel. Enamel has been partially dissolved during microscopy specimen preparation to reveal "resin tags" penetrating into etched enamel. (image from Patricia N.R. Pereira).

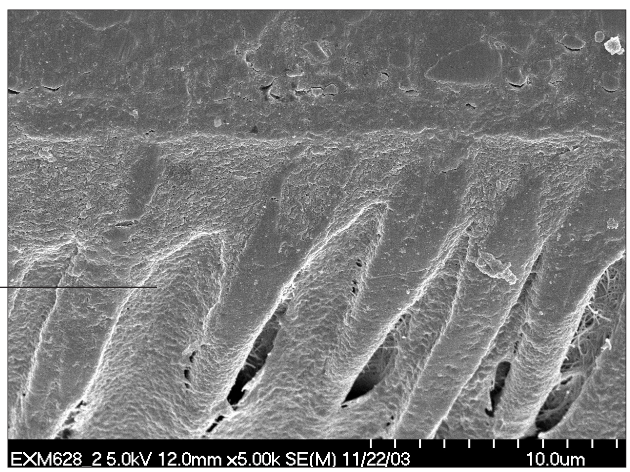


Figure 5. Scanning electron photomicrograph of 3M™ Adper™ Single Bond Plus Adhesive bond to dentin (image from J. Perdigo & M. Lopes, University of Minnesota).

A similar process involving dissolution of the mineral phase and an interlocking adhesive layer occurs when bonding to dentin. A 15 second etch of prepared dentin completely dissolves the smear layer and removes the mineral phase to a depth of around 5 microns. A collagen, or protein phase, resistant to acid dissolution is present at the surface after washing off the etchant. Penetration of the adhesive into this collagenous phase and its subsequent polymerization provides an interlocking layer commonly referred to as the "hybrid layer." The dentinal hybrid layer is revealed in a scanning electron micrograph in Figure 5 for Adper Single Bond *Plus* Adhesive.

Role of Inorganic Filler in Single Solution, Total-Etch Adhesives

To ensure optimum penetration of single solution adhesives into the collagen network, the adhesive resins are dissolved in a volatile solvent. After application, the solvent is evaporated with a stream of air leaving the adhesive layer ready for light curing. It has been suggested that the presence of filler in these relatively low viscosity adhesives helps develop a uniform adhesive film and "stabilizes" the hybrid layer (Inoue). This theory may be especially significant in instances where an overly aggressive drying technique is practiced.

The filler density in cured films of adhesives was explored by transmission electron microscopy (TEM) at 3M Laboratories. In these images, the electron dense areas (grey to black shaded areas) represent filler. It is readily observed that the cured film of 3M™ Adper™ Single Bond *Plus* Adhesive (Figure 6) exhibits the greatest filler density of the four, total etch, single solution adhesives. Of interest is the relatively low filler density in the cured film of Excite® adhesive (Figure 7) and the lack of homogeneity in both filler density and particle size in Optibond Solo Plus™ adhesive (Figure 9). The bar in each image represents 100nm.

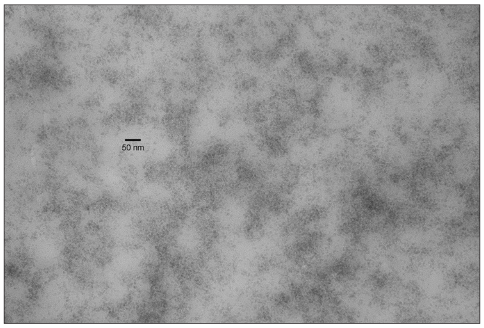


Figure 6. 3M™ Adper™ Single Bond Plus Adhesive.

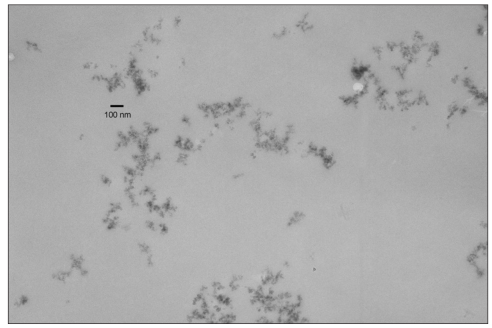


Figure 7. Excite® adhesive

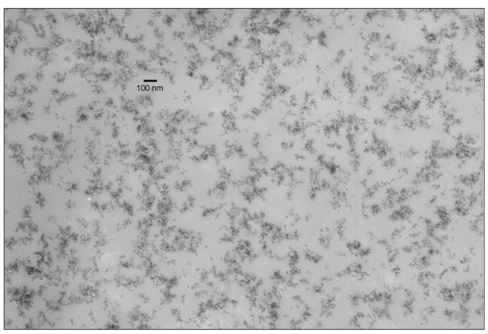


Figure 8. 3M™ Prime & Bond® NT™ Adhesive

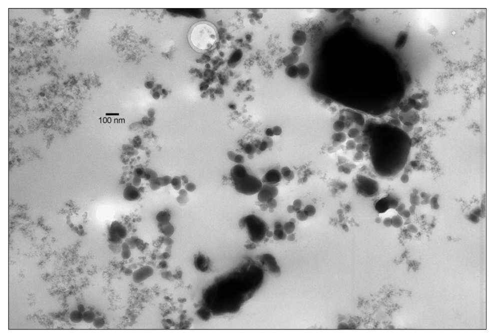
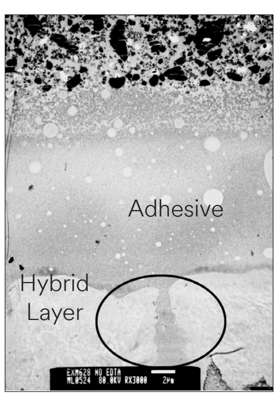


Figure 9. Optibond Solo Plus™ adhesive

Figure 10 represents a TEM photomicrograph of 3M™ Filtek™ Flow Flowable Restorative bonded to dentin with Adper Single Bond *Plus* adhesive. The zirconia silica particles of Filtek Flow restorative, are readily observed in the uppermost layer. The homogeneous nanofilled layer of Adper Single Bond *Plus* Adhesive is also observed. Below the adhesive layer is the "hybrid layer" consisting of interlocking collagen fibrils and cured resin. An adhesive resin tag can be seen bisecting the hybrid layer (circled). The resin tag has a similar electron density as the adhesive layer above indicating a continuous nanofiller concentration. At higher magnification, the particulate nanofiller within the resin tag is observed. It is also significant to note that the periphery of the tubules have been "hybridized" and that the hybrid layer extends the full depth of the demineralized zone. **In other words, complete adaptation between the tooth and restorative material has been achieved.** This result forms the foundation for a pain-free, long lasting restoration.

Photomicrograph of 3M™ Adper™ Single Bond *Plus* Adhesive bond to dentin. Images from J. Perdigo & M. Lopes, University of Minnesota

Fig. 10. Transmission electron



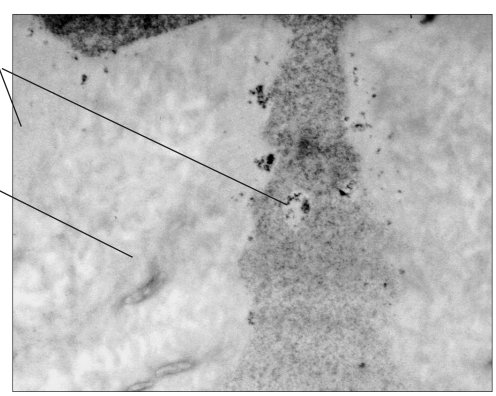


Fig 10b. Magnified area of Figure 10 showing penetration of nanofilled resin intodentinal tubule.

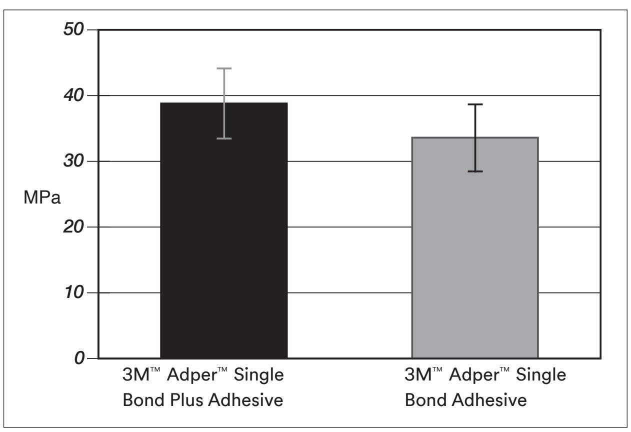
Physical Properties

Bond Strength Measurements

A number of test methodologies were used to explore the bond strength of 3M™ Adper™ Single Bond *Plus* Adhesive to various dental substrates. Internal laboratory data was generated using two different shear test methodologies. The methods differ primarily in the diameter of the bonded sample and the configuration of the fixture that applies the shear force. The independent laboratory study presented was performed using a microtensile method. Due to differences in sample geometry and the applied force of these methods, different bond strengths are expected for similarly bonded substrates.

Enamel and Dentin Bond Strengths. Notched edge shear test.

The graph below depicts pooled results from a multi-user dentin bond strength test performed using a notched edge shear test. In this test 3M™ Filtek™ Z250 Restorative buttons with a diameter of 2.38 microns were bonded to bovine dentin with 3M™ Adper™ Single Bond Adhesive and Adper Single Bond *Plus* Adhesive using their respective instructions (with Adper Single Bond *Plus* adhesive, two to three coats of adhesive are applied over 15 seconds; this is modified from Adper Single Bond adhesive which instructed two sequential coats). Each operator bonded 20 samples. Samples were stored in water at 37° C for 24 hours before testing. Three of the four operators obtained statistically higher bond strength with Adper Single Bond *Plus* adhesive, while for the other operator, the bonds strength of the two adhesives were equivalent. The pooled results for all operators revealed a p-value of 0.000.

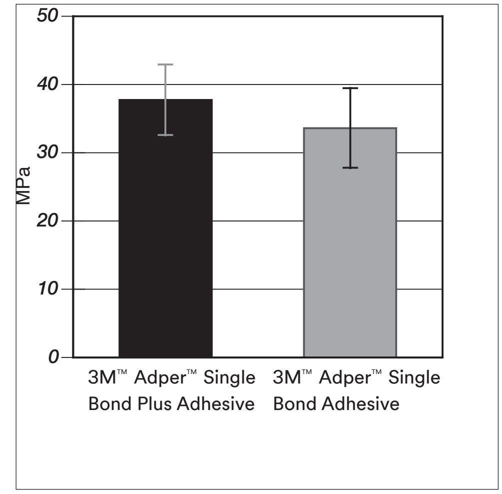


Source: 3M Internal data

Figure 11. Pooled multi-user dentin bond strength (MPa)

The above results represent enhanced performance of Adper Single Bond *Plus* adhesive due to incorporation of nanofiller and the modified application instructions noted above.

To determine the effect of the incorporation of nanofiller alone under more rigorous conditions, two operators performed a similar study where the two adhesives were applied using the instructions for Adper Single Bond *Plus* adhesive. This study design negates the effect of the application protocol on the results. For this study, each operator bonded ten samples. To challenge the adhesive, an aggressive ten-second air dry of the adhesive was used prior to light-curing. The pooled results, shown in Figure 12, reveal that incorporation of nanofiller has resulted in significantly higher dentin bond strength (p=0.04).



Source: 3M Internal data

Figure 12. Dentin bond strengths—effect of filler.

3M™ Adper™ Single Bond *Plus* Adhesive was also tested for adhesion to various substrates using a wire loop shear method. In this method, an approximate 5mm diameter button is bonded to the substrate and a shear force is applied with a wire loop until failure. For the direct light-cure indications, 3M™ Filtek™ Z250 Restorative is bonded to the substrate of interest—enamel, dentin, cured composite, porcelain or metal. The latter three substrates are relevant for composite or porcelain repair. To simulate indirect restorations, a porcelain or metal button is bonded to enamel or dentin using Adper Single Bond *Plus* Adhesive and 3M™ RelyX™ Veneer Cement. With RelyX Veneer Cement, the adhesive and cement are light-cured together. Results are shown in Table 1. Comparable bond strengths were found for Adper Single Bond *Plus* adhesive and 3M™ Adper™ Single Bond Adhesive using this method.

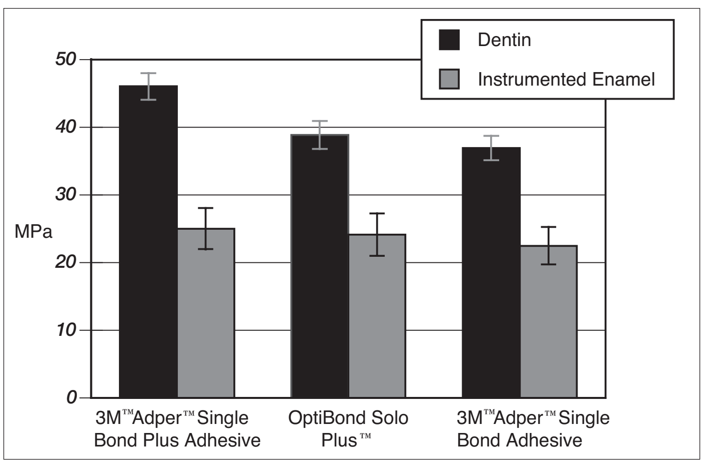
Table 1. Shear bond strengths in MPa for direct and indirect indications (wire loop shear method).

	3M ™Adper™ Single Bond *Plus* Adhesive	3M™Adper™ Single Bond Adhesive
Direct indications		
Composite to enamel	29.7 ± 6.0	26.6 ± 3.3
Composite to dentin	28.9 ± 1.8	23.8 ± 5.1
Composite to composite (Z100™ Restorative)	20.5 ± 3.5	23.8 ± 1.7
Composite to porcelain	13.5 ± 5.2	15.6 ± 2.2
Composite to semi-metal	17.0 ± 3.8	21.9 ± 3.1
Indirect indications		
Porcelain to enamel with RelyX Veneer Cement	27.4 ± 8.1	30.9 ± 8.5
Porcelain to dentin with RelyX Veneer Cement	22.4 ± 5.2	22.6 ± 6.1

Independent Investigations

Microtensile Bond Strength

A microtensile bond strength experiment was performed at the University of Minnesota comparing 3M™ Adper™ Single Bond *Plus* Adhesive to 3M™ Adper™ Single Bond Adhesive and Optibond Solo Plus™ adhesive. Twenty-four extracted human third molars were prepared for bonding, 12 each for enamel and dentin. Enamel was prepared by roughening with a diamond bur for five seconds while dentin samples were prepared by cutting the crown off with a diamond saw exposing middle dentin. 3M™ Filtek™ Z250 Restorative was bonded to the substrates using the above named adhesives followed by sectioning into rectangular sticks with cross-sectional areas of $0.7mm^2$ for dentin and $1.6mm^2$ for enamel. Each stick was then tested under a tensile load using an Instron testing machine. Figure 13 depicts the results.



Source: Dr. J. Perdigao, University of Minnesota

Figure 13. Enamel and Dentin Microtensile Bond Strength (MPa)

While the enamel bond strengths were found to be equivalent for the three adhesives tested, the dentin bond strength for Adper Single Bond *Plus* adhesive was significantly greater than the other two adhesives.

Management of Post-operative Sensitivity

Sensitivity following restorative procedures is, in many cases, associated with an incomplete dentinal seal. Often referred to as "dentinal sensitivity", its origin has been associated with movement of dentinal tubular fluid in a coronal direction at a rate that stimulates pulpal pain receptors (Brannstrom). For adhesive restorative procedures, the formation of a fully sealed dentinal hybrid layer will prevent this rapid outward flow of tubule fluid and greatly minimize the risk for post-operative sensitivity if not eliminate it altogether. The following considerations are helpful in achieving this goal with 3M™ Adper™ Single Bond *Plus* Adhesive.

Isolation with rubber dam is highly recommended.

Contamination of the preparation during adhesive placement of any adhesive may potentially compromise the dentinal seal leading not only to post-operative sensitivity, but also possible margin discoloration and lack of long-term retention.

Use of 3M™ Vitrebond™ Light Cure Glass Ionomer Liner/Base

The resin-modified glass ionomer, 3M™ Vitrebond™ Light Cure Glass Ionomer Liner/Base , is recommended for the management of post-operative sensitivity.

Vitrebond liner/base can be used routinely to seal the dentin of class I and II restorations since it is with these restorations where post-operative sensitivity often occurs. It is also recommended for dentin bonding in areas of deep cavity excavation where the bond with total etch adhesives may be compromised due to increased dentinal fluid.

Adhesive Application

For optimum penetration of the adhesive into dentin following the etching step, the dentin must remain moist. Dehydration of the etched dentinal surface will cause the collagen fibrils to collapse resulting in a reduction in the porosity of this surface. Consequently, the collagen layer in this state will inhibit the penetration of adhesive throughout this layer and compromise the integrity of the dentinal seal. Following the few simple steps described below will promote complete dentinal sealing and minimize potential for post-operative sensitivity.

- Use of compressed air is not recommended to remove pooled water remaining after the etch step.
- Blot excess moisture from the preparation using a cotton pellet or mini-sponge. The dentinal surface should appear glistening without pooling of water.
- Apply adhesive immediately after blotting. Evacuators may hasten dentinal surface dehydration. If application is delayed, rewet and blot as above.
- Use generous amounts of adhesive. Two to three coats of Adper Single Bond *Plus* adhesive are recommended. While two coats should be sufficient in most cases, an additional coat may be beneficial in cases where inadvertent pooling of water occur.

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