

A collection of scientific results

**AdperTM
PromptTM**

Self-Etch Adhesive

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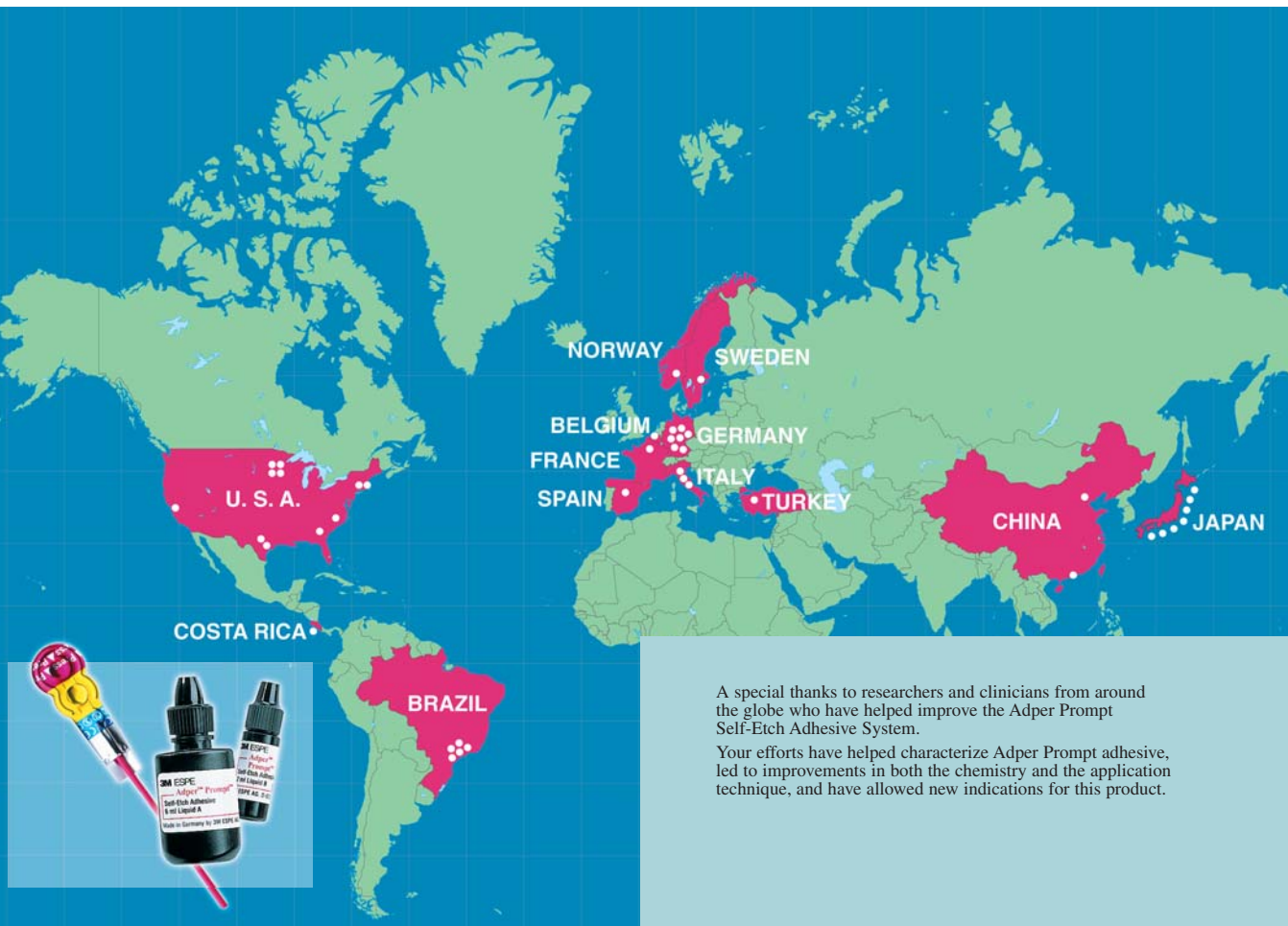
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In-Vivo Clinical Studies,
In-Vitro Research, Reviews
January 2002 — March 2004

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A special thanks to researchers and clinicians from around the globe who have helped improve the Adper Prompt Self-Etch Adhesive System.

Your efforts have helped characterize Adper Prompt adhesive, led to improvements in both the chemistry and the application technique, and have allowed new indications for this product.

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Introduction

Dear Dental Professional,

Adper™ Prompt Self-Etch Adhesive represents the culmination of years of experience in the area of dental adhesives. Originally introduced in 1999 as the Prompt™ L-Pop system, the unique unit-dose dispensing system and ease of application quickly established the product as one of the leading self-etch adhesive systems.

Product enhancements have subsequently been made in the photocuring chemistry as well as in the resin chemistry. These enhancements assured excellent performance with any type of curing light as well as improved the film forming capability, resulting in higher bond performance. Of course, low post-operative sensitivity continues to be an attribute of this self-etch system.

As in the past, Adper Prompt adhesive is indicated for use with light-cure composites and compomers. The unique ability of Adper Prompt adhesive to etch unprepared enamel has allowed the added indication of bonding light-cure pit and fissure sealants. In addition, the excellent film forming capability has allowed Adper Prompt adhesive to be recommended as a treatment for hypersensitive root dentition.

This booklet presents many of the independent test results on Adper Prompt adhesive from around the globe. As always, we have relied on our colleagues at universities and practices to aid in our research and development efforts. Adper Prompt adhesive displays excellent performance in a wide array of test protocols, from the laboratory setting to the clinical arena.



Dr. Oswald Gasser

Best Regards,
Dr. Oswald Gasser
Global Technical Director 3M ESPE

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Adper™ Prompt™ L-Pop™

1 Clinical Results (In-Vivo)

Clinical performance is the true test of a dental adhesive. In the laboratory setting, isolation is complete, visibility is perfect, and the surface is flat. Contrast this with the clinical setting, where isolation is variable, visibility is limited, and the surface is three-dimensional.

After placement of a restoration, the clinical setting stresses the restoration via thermal loading, occlusal forces stress the fatigue resistance of the bond, and various staining solutions from wine to espresso serve as continual indicators of the marginal integrity.

In the next few pages are summaries of clinical investigations into the performance of both the original Prompt™ adhesive product and the new Adper Prompt™ adhesive product. The first study, pertaining to the 3-year performance of the original formulation of Prompt adhesive, was included to provide a longer-term perspective on the clinical performance of this product. Studies on Adper Prompt adhesive are in progress, and early results are provided.

1. Clinical Results (In-Vivo)

Three Year Clinical Performance of Prompt L-Pop Self-Etch Adhesive

Authors: C. Munoz¹, J. Dunn¹, J. Fundingsland², and R. Richter³, ¹Loma Linda University, CA, USA, ²3M ESPE, St. Paul, MN, USA, ³3M ESPE, Seefeld, Germany
Reference: IADR 2004, Honolulu USA, #0541

Purpose: This investigation evaluated the clinical performance of a new self-etching bonding agent Prompt L-Pop (3M ESPE) over a three-year period.
Methods: Twenty-five Class III and Class V restorations were placed in 17 subjects. Fourteen of the restorations were maxillary and 11 were mandibular restorations. Twenty restorations were evaluated at 36 months. Five restorations were lost to follow-up. Following cavity preparation, the teeth were etched, restored with a hybrid composite, and polished following manufacturer's instructions. Marginal adaptation (MA), adhesive retention (AR), secondary caries (SC), marginal discoloration (MD), and sensitivity (SE) were evaluated.
Results: At 3 years, using a modified USPHS grading system the following results were found in percentage (%) (BL=Baseline):

	MA		AR		SC		MD		SE	
	BL	3Y	BL	3Y	BL	3Y	BL	3Y	BL	3Y
Alpha	72	75	100	95	100	95	100	90	100	100
Bravo	28	20	0	0	0	0	0	10	0	0
Charlie	0	5	0	0	0	5	0	0	0	0
Delta	0	0	0	5	0	0	0	0	0	0

Conclusions: 1) Categories MA, AR, and SC, were unchanged from baseline, with the exception of one restoration that was lost at the two-year recall. 2) MD showed a slight decrease in marginal discoloration. 3) No sensitivity was reported at either baseline or 3 years. 4) Overall clinical use of a self etching adhesive on Class III and V restorations were deemed acceptable for routine clinical use.
Aim of the Study: Clinical performance is the ultimate test for a dental material. This paper presents the 3-year recall data for 3M ESPE Prompt adhesive.
Result of the Study: The Prompt adhesive system displayed very good performance in all criteria at the 3-year recall. It should be noted that this study was conducted with a previous iteration of the Prompt adhesive, before improvements in both chemistry and technique which yielded the current 3M ESPE Adper™ Prompt™ Adhesive System.

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1. Clinical Results (In-Vivo)

Adper Prompt in Class I and Class II Restorations

Authors: Dr. William D. Browning, Augusta, USA

Reference: Unpublished Data

Dr. Bill Browning and colleagues at the Medical College of Georgia are conducting a two-year clinical study of Filtek™ Z250 Universal Restorative bonded with both 5th generation (3M ESPE Single Bond) and 6th generation (Adper Prompt) adhesives. Seventy-six patients are participating, and 206 restorations have been placed. Restorations are being placed in a general dental practice setting.

Adper Prompt has been placed in 18 Class I and 90 Class II restorations. Single Bond adhesive has been placed in 16 Class I and 85 Class II restorations.

Neither group experienced postoperative sensitivity.

Sensitivity is being measured by application of cold-water stimulus. Each patient receives a custom stent that directs the cold water to the tooth in question. At 1 week a downward trend in sensitivity to cold stimulus was noted. Preliminary, 3-month data indicated a continuation of this downward trend in sensitivity from the pre-operative condition. The authors speculated that this was indicative of good adaptation and sealing of the restoration.

This study will be followed over the years and summarized as data becomes available.

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1. Clinical Results (In-Vivo)

Treating Hypersensitive Root Dentin with Adper Prompt

Authors: Dr. Ronald Perry, Dr. Gerard Kugel, Boston, USA

Reference: Unpublished Data

The measured ability of Adper™ Prompt™ adhesive to reduce the permeability of patent dentinal tubules leads one to believe that such a treatment could be used clinically to treat exposed, hypersensitive root dentin.

A clinical study was conducted, with Dr. Gerard Kugel and Dr. Ronald Perry as investigators, to evaluate Adper Prompt as a treatment for exposed root surfaces. In this study the treatment is tested via application of cold water, air/water spray, and tactile stimuli.

A detailed description of both the study and the findings is provided later in this booklet. **To summarize, Adper Prompt proved effective at reducing sensitivity on exposed root dentin.**

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1. Clinical Results (In-Vivo)

1

**Bonding Class V Restorations
with Adper Prompt Self Etch Adhesive****Authors:** Professor Dr. Kurt Merte, Leipzig, Germany**Reference:** Unpublished Data

Retention of Class V restorations is challenging. First, the adhesive has to display excellent bond to dentin, as dentin typically comprises the majority of the bonding surface for this class of restoration. Perhaps even more challenging is the fact that this bond must be maintained in the presence of repetitive stress, as the flexation of the tooth during mastication creates stress at the interface between composite and tooth structure.

A study to evaluate the performance of Adper Prompt in Class V restorations has recently been initiated with Dr. Merte. To date, 60 restorations have been placed.

Baseline data is quite positive. **None of the 60 restorations have displayed postoperative sensitivity, indicating an excellent seal. All restorations scored Alpha, for marginal integrity, indicating an ability to resist polymerization stress.**

This study will be followed over the years and summarized as data becomes available.

Clinically Relevant Variables

2

Other variables besides adhesion can enter in the success or failure of a restoration. Multiple components in a system can increase the risk of misuse. Longer application times leave more opportunity for contamination.

In the following pages are several studies into clinically relevant parameters that could affect the clinical success of a direct restorative placement.

The simplicity of the Adper™ Prompt Adhesive System is highlighted and is related to operator variability. The times required to apply several adhesives are compared. Studies pertaining to effects on bacteria and on gingival tissue are also presented.

2. Clinically Relevant Variables

Operator Variability and Complexity of Dentin Bonding Agents

Authors: T. Jacobsen, Sahlgrenska Academy of Göteborg University, Sweden, K.J. Soderholm, University of Florida, Gainesville, USA, M. YANG, University of Florida, Gainesville, USA, and T.F. Watson, Guy's King's St Thomas' Dental Institute, King's College, London University, United Kingdom

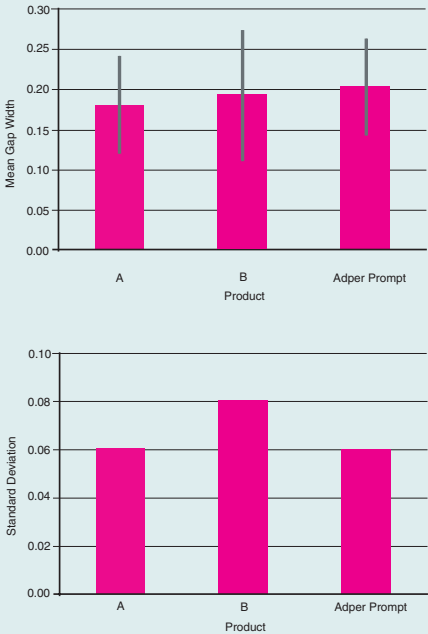
Reference: IADR 2003, Göteborg Sweden, #1278

Objective: To determine how different operators and bonding agents influence gap formation around dentin-bonded composites. **Methods:** Standardized dentin cavities (diameter=3mm, depth=1mm) were prepared in extracted human teeth (n=210), and 7 dentists evaluated 3 different bonding systems [A] 30 wt% phosphoric acid, 38 wt% HEMA in acetone, Scotchbond Multi-Purpose Adhesive (3M ESPE); B) 30 wt% phosphoric acid, 38 wt% HEMA in water, Scotchbond Multi-Purpose Adhesive; C) Adper Prompt L-Pop (3M ESPE)] by treating 10 teeth with each material. Bonding systems A&B were used according to the recommendations for Scotchbond Multi-Purpose, but where primer rubbing into the surface for 30s was emphasized and a 30s light-curing time of the adhesive was used. Bonding system C was used according to the manufacture's directions. One of the investigators restored all cavities with composite (Z100, 3M ESPE) and removed excess material to expose the cavity margins. One week after storage in room-temperature water, the maximum gap-width, expressed in percentages of the cavity diameter, was measured using a confocal microscope and statistically analyzed with PROC MIXED (SAS Inc.).

Results: The relative gap-width (%) for the three bonding systems were not statistically different (p=0.208). Mean (SD) were: A) 0.18 (0.06), B) 0.19 (0.08), and C) 0.20 (0.06). The variation due to the operator-material interaction was not negligible (p=0.057). Thus part of the operator variation (a random effect) was due to differences in materials. When the operator's variability (SD) within the different material were examined, group C had significantly smaller variability than the other two (A : B : C = 0.060 : 0.061 : 0.051, all with standard error 0.0032).

Conclusion: The operator variable is a more important consideration than the material variable. The one-bottle system performed better than the more complex A and B systems when variability rather than gap size was considered.

Dentin Bonding Agents



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2. Clinically Relevant Variables

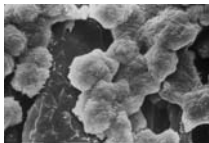
Effect of Adper Prompt Adhesive on Oral Bacteria

Authors: Dr. Susanne Kneist, Jena, Germany

Reference: Unpublished Data

Objectives: This study was designed to evaluate the effect of the 3M ESPE Adper Prompt system on 8 strains of bacteria.

Methods: The 8 strains of bacteria; *A. naeslundii*, *A. odontolyticus*, *S. sanguis*, *S. mutans*, *S. sobrinus*, *S. salivarius*, *L. casei*, *L. delbrueckii* ss lactis; were cultured under anaerobic conditions in Balmelli bouillon at 37°C. Strain suspension was suspended in liquefied balmelli agar and placed into a petri dish. After the agar had



S. Mutans

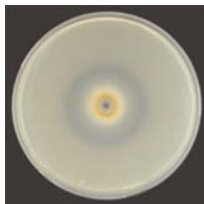
set, sample wells were prepared and filled with the test adhesive. Each component of the Adper Prompt system was tested individually, as well as mixed and in the form of a cured film.

Results: The individual components, mixed adhesive, the cured adhesive each displayed inhibition of both the plaque and the saliva microorganisms. Plaque bacteria were more inhibited in their growth than the saliva bacteria. *Actinomyces* was inhibited more strongly than streptococci, which in turn was inhibited more strongly than lactobacilli.

Conclusion: Both components of the Adper Prompt system as well as the mixed adhesive and cured film of adhesive displayed an antibacterial effect in vitro with respect to plaque and saliva bacteria.



Effect of cured adhesive on S. Salivarius.



Effect of mixed adhesive on S. Mutans.

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2. Clinically Relevant Variables

Irritation Testing of Dental Adhesives

Authors: J.E. Dahl, I.S. Dragland, and A. Wesmann, NIOM - Scandinavian Institute of Dental Materials, Haslum, Norway

Reference: IADR 2003, Göteborg Sweden, #0130

Dental adhesives that improve the bonding between the tooth and the restoration are extensively used. The techniques employed for placement of dental adhesive agents may result in accidental exposure to adjacent oral soft tissue of the patient.

Objective: To determine the potential of dental adhesive agents to evoke irritation of oral mucous membranes.

Method: The hens' egg test - chorioallantoic membrane (HET-CAM) was used to establish the agents' ability to cause immediate damage to the blood vessels of the chorioallantoic membrane of fertilized eggs. The type of injury observed during the five minutes exposure were rupture of the vessels, coagulation within the vessels and haemolysis of the vessels. An irritation score was calculated as an average of two experiments in triplicate based on the time of appearance of the different types of damage. Positive (0.1 M NaOH) and negative controls (saline) were included. Six commercially available agents marketed as "single-component", "one-step" or "self-etch" adhesives were randomly selected from the Scandinavian market: Adper Prompt L-Pop (3M ESPE) (A), ANA Single Bond (Nordiska Dental) (B), Gluma One Bond (Heraeus Kulzer) (C), ONE-STEP (Bisco Ltd) (D), Syntac Single-Component (Vivadent) (E), Xeno III (DENTSPLY DeTrey) (F). Products A and F also contained etching constituents whereas the etching component was separate for the other products and not included in the testing.

Results: Irritant reactions were observed for all of the tested dental adhesives. The irritation score for the different products were as follows: A: 8.1, B: 11.9, C: 18.1, D: 9.5, E: 13.7, F: 8.1. Two of the products (A and F), the so-called "self-etch" adhesives, were rated as moderate irritants (irritation score between 5 and 8.9) and the other four as severe irritants (irritation score between 9 and 21).

Conclusion: Inadvertent spill of adhesive agents may result in local damage to oral soft tissue.

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2. Clinically Relevant Variables

Comparison of Total Working Times of Self-etching Adhesives

Authors: M. Peuker, K. Janz, and J. Dubbe, 3M ESPE AG, Seefeld, Germany
Reference: IADR 2003, Göteborg Sweden, #0861

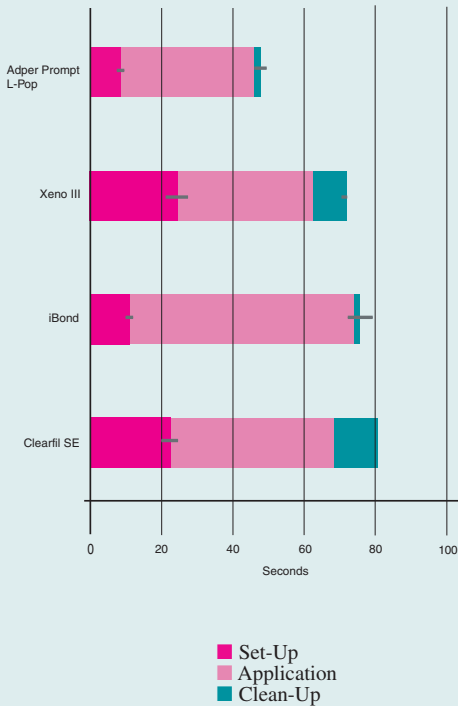
Objective: To compare the total working times, which consist of set-up, application and clean-up times, of a number of Self Etching Adhesives based on user instructions and handling tests by professionals.

Method: All the components of the manufacturers product – closed adhesive– and brush–containers, mixing wells, etc. – were initially placed on the dental assistant’s tray. The set-up time, measured with a stopwatch, started, e.g. with opening a vial and was completed when the assistant wet the application brush. The specific application time of each product was taken from each product’s instructions for use. The clean-up time consisted of the time it took to clean or dispose of the used components. Non disposable components then had to be disinfected. Disinfection methods and times vary and are indicated by “+disinfection”. All products set-up and clean-up times were determined three times by four different dental assistants.

Results: The total working time (sec.) is the sum of the mean values of (Set-up time ± standard deviation / Application time / Clean-up time ± standard deviation) A: Adper Prompt L-Pop (3M ESPE) ($8.8 \pm 0.87/38/1.7 \pm 0.65$) = 48.5; B: Clearfil SE Bond (Kuraray) ($22.7 \pm 2.23/46/12.2 \pm 3.41$) = 80.9+disinfection; C: iBond (Heraeus Kulzer) ($11.3 \pm 1.07/63/1.9 \pm 0.67$) = 76.2; D: XENO III (Dentsply) ($24.8 \pm 3.16/38/8.8 \pm 1.64$) = 71.6+disinfection. The set-up and clean-up times of the adhesives differ significantly ($p(\text{set-up}) < 0.05$ / $p(\text{cleanup}) < 0.05$) except for the homogeneous group (a) ($p > 0.05$) (Two-way ANOVA).

Conclusion: Set-up and clean-up times are significantly shorter with unit dose products like A and C. Adhesive A resulted in the shortest total working time of 48.5 seconds while B resulted in the longest working time of 80.9 seconds.

Total Working Time



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3 Desensitization of Hypersensitive Root Surfaces

Fluid movement in dentinal tubules is the generally accepted cause of sensitivity. This movement can be stimulated by drying, temperature shifts, as well as by compounds such as sugar (Brannstrom 1986). Studies indicate that hypersensitive root surfaces can have 8 times as many open tubules as non-sensitive dentin.

If open tubules are the cause of sensitivity, it makes sense that treatments that occlude sensitivity should be effective. Thus one of the first tests of a treatment should be the in vitro measurement of the effect of the treatment on sealing open dentinal tubules.

The final measurement is, of course, the clinical application. To determine the effectiveness of Adper™ Prompt Adhesive in treating hypersensitive root dentition a clinical study was conducted.

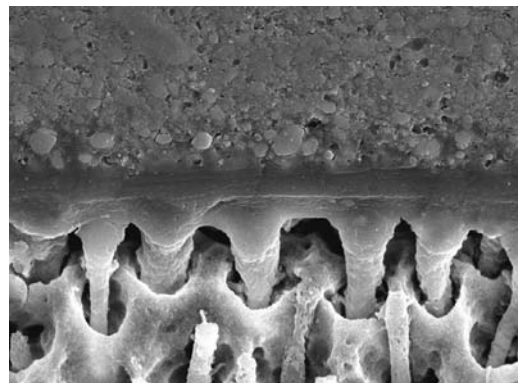
The next few pages summarize the in vitro and in vivo proof that allowed 3M ESPE to recommend Adper Prompt adhesive as an effective treatment for the common condition of exposed, hypersensitive root dentition.

3. Desensitization of Hypersensitive Root Surfaces

SEM Analysis of the Interface Between Adper Prompt and Dentin

Authors: Dr. Patricia Pereira, Chapel Hill, USA

Reference: Unpublished Data



This SEM, taken by Dr. Patricia Pereira, illustrates the ability of Adper Prompt to form a hybrid layer and to flow into open dentinal tubules. The interface between the composite and the dentin, via a uniform hybrid layer, is clearly visible.

While a uniformly bonded interface such as illustrated here tends to imply a good seal, further measurements are needed to both prove the ability to reduce permeability and, ultimately, provide an effective treatment.

[Image by Pereira; 2004]

Adper™ Prompt™ L-Pop™

Adper™ Prompt™ L-Pop™

3. Desensitization of Hypersensitive Root Surfaces

Effects of Dentin Desensitizing Agents on Dentin Permeability Under Different Application Conditions

Authors: K.-A. HILLER, A. SCHICKER, and G. SCHMALZ, University of Regensburg, Germany

Reference: AADR 2003, San Antonio USA, #0632
ANMERKUNG kontrolliert

Objectives: The purpose of this study was to compare the reduction of dentin permeability using dentin desensitizing agents under different application conditions.

Methods: 120 dentin slices were cut from bovine incisor teeth. Pulp-facing surfaces were acid etched (30s, 50% citric acid), surfaces far from the pulp were ground (600 grit) to 500 µm thickness. Dentin permeability (hydraulic conductance, Lp,m3/Ns) using aqua bidest at 0.7 m H2O, was measured for each specimen at baseline and after treatment using a split-chamber apparatus connected to a commercially available measurement unit (FloDec, DeMarco Engineering SA, Geneva). Materials were applied according to manufactures' instructions in three different ways: (1-pressure) Under pulp pressure simulation of 0.3 m H2O from the pulpal side and drying (30s air), and no pulp pressure simulation during application and (2-dry) dried (30s air) surface, or (3-moist) moist (removing water using a suction device, no air) surface. Slices served as their own controls. Materials tested comprised Gluma Desensitizer (Heraeus Kulzer, GD), Seal & Protect (Dentsply/DeTrey, SP), SuperSeal (Dexcel Pharma, SS), and Adper Prompt L-Pop (3M ESPE, AL). Test parameter was the percentage of reduction (P-RED) of Lp after treatment (100%=Lp before treatment). Mann-Whitney Test (p<=0.05) was used to evaluate differences between groups (10 samples).

Results: The median Lp before treatment varied between 1.01–2.02E-10m3/Ns for the 12 groups. Median PRED[%] of Lp (25-75% Quantiles) were:

Material	1-pressure	2-dry	3-moist
GD	34(26-42)	28(19-58)	28(17-33)
SP	48(30-61)	42(28-51)	38(33-48)
SS	38(25-47)	46(32-66)	25(17-40)
AL	55(39-70)	77(68-83)	41(27-65)

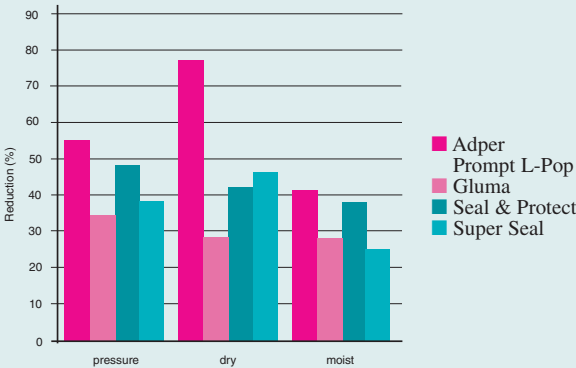
AL-dry showed the significantly highest reduction compared to all other treatment/material combinations. For other materials no influence of the application procedure could be detected (besides SS dry vs. moist). With moist application AL and SP showed similar reductions, both being significantly higher compared to other materials.

Conclusions: Application conditions may have an influence on the reduction of dentin permeability.

Aim of the Study: Tooth hypersensitivity e.g. in a cervical lesion can be eliminated by reducing the dentin permeability. Therefore it was the objective of this study to determine the reduction of dentin permeability after application of Adper Prompt L-Pop and desensitizing agents under different application conditions.

Results: When applied on a dried dentin surface, Adper Prompt L-Pop showed the significantly highest reduction compared to all other treatment / material combinations. On moist dentin surfaces Adper Prompt L-Pop and Super Seal showed similar reductions, both being significantly higher compared to other materials.

Effects of Dentin Desensitizing Agents on Dentin Permeability Under Different Application Conditions



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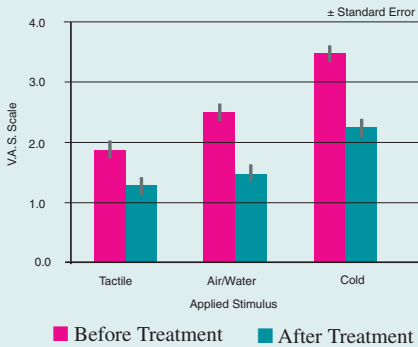
3. Desensitization of Hypersensitive Root Surfaces

Authors: Dr. Ronald Perry, Dr. Gerard Kugel, Boston, USA
Reference: Unpublished Data

The real test is, of course, the clinical result. To measure effectiveness in treating hypersensitive root surfaces, a randomized, controlled clinical study was conducted with Dr. G Kugel and Dr. R Perry, Boston, MA. In this study, hypersensitive dentin was treated with Adper Prompt adhesive. Three modes of stimuli, tactile, air/water, and cold, were applied to evaluate the effectiveness of the treatment. Patients used a Visual-Analog-Scale, or VAS, to indicate the level of discomfort caused by the stimulus.

Application of Adper Prompt adhesive resulted in a statistically significant decrease in hypersensitivity induced by tactile, air/water, or cold stimulus at all intervals up to 3 months.

Effect of Adper Prompt on Root Surface Hypersensitivity



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3. Desensitization of Hypersensitive Root Surfaces

Clinical practice based study

The effect of Adper Prompt L-Pop's sealing ability on the reduction of cervical hypersensitivities was evaluated in a clinical study run in general dental offices in 4 European countries.

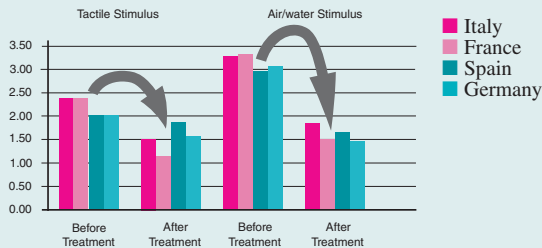
Design of the study

98 patients treated by 20 general dental practitioners in 4 European countries (Germany, France, Italy, Spain) participated in this study. The dentists were asked to score the level of tooth sensitivity for each patient after exposing the cervical area to tactile (dental probe) and air/water stimuli. The results were recorded on a Visual Analogue Scale (VAS), ranging from level 1 = no pain to 4 = very painful. Immediately after recording the baseline sensitivity Adper Prompt L-Pop was applied to the cervical tooth surface. Following light curing and removal of the oxygen inhibition zone the sensitivity levels were scored again.

Results of the study

The application of Adper Prompt L-Pop resulted in a significant reduction in sensitivity. This effect was more evident for the air/water stimuli, which generated higher pain responses at baseline. Adper Prompt L-Pop provided an effective, reliable and fast treatment for hypersensitive teeth through a perfect seal of the dentinal tubules. Patients appreciated that the positive effect was immediately noticeable and that there was no need for local anaesthetic. The application of Adper Prompt L-Pop was pain free, as the procedure did not require a phosphoric acid etching step.

Desensitization of Hypersensitive Root Surfaces

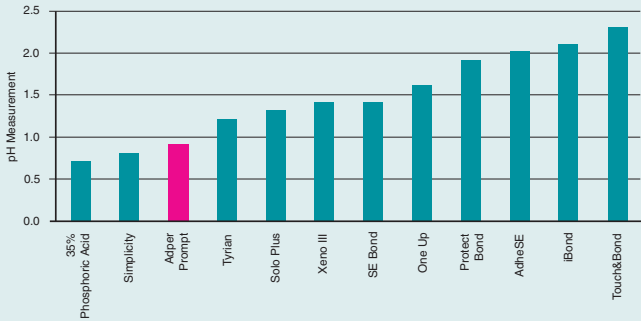


4 Bonding Sealants

One of the challenges inherent in the design of a self-etch adhesive is the fact that etching ability, or low pH, is contrary to chemical stability of aqueous methacrylate solutions. Adper™ Prompt Adhesive, both in L-Pop and vials, avoids this design constraint by separating the aqueous component from the acidic methacrylate components. **The important result is that Adper Prompt adhesive can be more acidic than other products, allowing the adhesive to be indicated for cut or uncut enamel.**

Thus it would be logical to assume that Adper Prompt adhesive would be a simple and effective solution for bonding light-cure sealants. To test this hypothesis, independent investigations into both adhesive and microleakage were conducted. Test results, presented in the next few pages, indicated bond strength equal to that of phosphoric acid-treated enamel, while microleakage results indicated even lower microleakage than with the conventional acid treatment.

pH Measurement of Self-Etch Adhesive Systems



4. Bonding Sealants

Ultrastructure of Resin-Enamel Bonds in Unground Enamel—Occlusal Fissures

Authors: F.R. TAY¹, S.H.Y. WEI², D.H. PASHLEY³, and R.M. CARVALHO⁴,
¹The University of Hong Kong, China, ²Medical College of Georgia, Augusta, USA, ³University of São Paulo, Bauru, Brazil

Reference: IADR 2003, Göteborg, Sweden, #0718

Objectives: Bonding to occlusal enamel fissures represents a special situation in bonding to unground enamel in which deep, narrow fissures with peripheral aprismatic enamel are not easily penetrable by phosphoric acid etchants. This study examined the ultrastructure of bonding to occlusal enamel fissures using phosphoric acid etching in combination with a fissure sealant, or a total-etch adhesive (One-Step, Bisco) followed by a fissure sealant, and two single-step self-etch adhesives (Adper Prompt, 3M ESPE and Xeno III, Dentsply DeTrey) followed by a fissure sealant.

Methods: Sections of bonded enamel fissures were polished, rinsed with phosphoric acid to bring surfaces into relief, and examined under dehydrated conditions with conventional SEM (for enamel structure) and under wet conditions with field emission-environmental SEM (for bond integrity). Extent of resin penetration into etched enamel was further supported by TEM examination of sections taken from stained, demineralized and unstained, undemineralized bonded specimens.

Results: All occlusal fissure walls examined were lined with remnant aprismatic enamel. Full penetration of resins into the bottom of the fissures were rarely observed, even with the adjunctive use of total-etch or self-etch adhesives. Phosphoric acid did not penetrate well into the fissures and although hybridization of the etched aprismatic enamel was observed with the use of a total-etch adhesive, etching was inconsistent and gaps were frequently observed. Entrapment of bacteria within fissural walls was also present. The more aggressive self-etch adhesive Adper Prompt created etching in aprismatic enamel that approached that of phosphoric acid etching. The less aggressive self-etch adhesive produced 1 mm thick hybrid layers in the aprismatic enamel fissural walls.

Conclusions: Self-etch adhesives penetrate occlusal fissures better than phosphoric acid and produce more uniform etching and hybridization of fissural walls. In all circumstances complete resin penetration into occlusal fissures cannot be a realistic expectation.

4. Bonding Sealants

Microleakage of Sealants Bonded with Adper Prompt Self-Etch Adhesive

Authors: Dr. Robert Feigal, Ann Arbor, USA

Reference: Unpublished Data

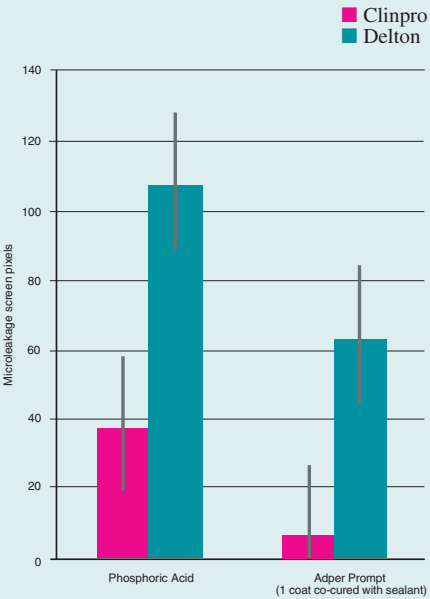
A microleakage study to compare Adper Prompt to phosphoric acid as a pretreatment for placement of light-cure sealants was conducted by Dr. Robert Feigal. Occlusal surfaces were cleaned with a dry bristle brush in order to remove organic debris. After adhesive and sealant application, teeth were stored in saline for a minimum of 24 hours thermocycling.

Following thermocycling, the specimens were placed in a 50% aqueous solution of silver nitrate for 2 hours in darkness followed by 8 hours in radiographic developer under fluorescent light to precipitate the silver nitrate leakage stain.

For microleakage quantification, the teeth were sectioned longitudinally in a buccal-lingual direction and three 1mm thick sections will be obtained from each tooth using a low speed diamond wheel saw. Both sides of each section were evaluated, so that for each tooth, six measures from separate points in the interface were recorded. Leakage was measured along the enamel-sealant interface by using a stereomicroscope at 20x power connected to a computer used to capture the image. The measurements were made on an Image Pro Plus program as microns of leakage from the external margin on each side of the sealant to the half-way point through the sealant interface. 6 measurements of buccal margin leakage and 6 measurements of lingual surface leakage were averaged for the mean leakage per tooth. A one-way ANOVA was used to determine main effects and a Fisher's Exact Test was used to determine differences between individual groups. All statistical tests will be run at a significance level of $p < 0.05$.

Results: The use of a single layer on Adper Prompt either with or without an adhesive cure offered statistically significant improvements (P.03) as compared to phosphoric acid.

Adper Prompt: Sealant Microleakage



*Note:
— Leakage measured in pixels. Divide by 96 to obtain mm.
— Treatment significant P.03; Lower leakage with Adper Prompt

Adper™ Prompt™ L-Pop™

Adper™ Prompt™

4. Bonding Sealants

Microtensile Bond Strengths of Sealants to Unprepared Enamel

Authors: J. PERDIGÃO¹, J. FUNDINGSLAND², S. DUARTE, Jr.³, and M.M. LOPES¹, ¹University of Minnesota School of Dentistry, Minneapolis, USA, ²3M/ESPE Dental Products, St Paul, MN, USA, ³University of Minnesota, Minneapolis, USA

Reference: IADR 2003, Göteborg, Sweden, #0863

Objectives: To characterize the adhesion of two pit-and-fissure sealants to unprepared enamel using 35% phosphoric acid vs. a self-etching bonding system (Adper Prompt L-Pop, PLP, 3M ESPE AG).

Methods: Proximal enamel surfaces of extracted molars were treated with one of the four conditioners shown in Table. One of two sealants (Clinpro, 3M ESPE; Delton, Dentsply) was applied in a thin layer (0.50 to 0.75 mm), followed by a composite buildup (Z250, 3M ESPE) to provide a gripping surface. Specimens were cut in X and Y directions in sticks with section of 0.7±0.1mm² and tested in an Instron at 1mm/min at 24h. µTBS data in MPa were analyzed with one- and two-way ANOVA/Tukey's (superscript letters, p<0.05).

Results: There was a significant difference between means for "sealant" at p<0.028 (Clinpro>Delton). For "surface etchant/conditioner", Groups A, C, and D resulted in similar bond strengths at p<0.05, while the means for Group B were statistically lower. The combinations Group C/Clinpro and Group D/Delton ranked in the highest statistical subset.

Conclusions: PLP applied in 2 coats and cured prior to sealant application is as effective for sealant bonding as either PLP applied in 1 coat and co-cured with the sealant or phosphoric acid etching. The application of PLP in 1 coat cured prior to sealant application is not recommended.

This project was supported by 3M ESPE.

Enamel Conditioning	Pooled Mean	Sealant	Mean±SE	N
Group A = 35% H ₃ PO ₄ for 15 sec, rinse, dry	15.57 ^{ab}	Clinpro	15.69 ^B ±1.240	35
		Delton	15.45 ^B ±1.60	40
Group B = PLP, 1 coat cured prior to application of sealant	9.49 ^c	Clinpro	9.77 ^C ±1.190	38
		Delton	9.22 ^C ±1.240	35
Group C = PLP, 2 coats cured prior to application of sealant	19.19 ^a	Clinpro	22.78 ^A ±0.998	54
		Delton	15.16 ^B ±1.059	48
Group D = PLP, 1 coat co-cured with the sealant	17.32 ^{ab}	Clinpro	16.60 ^B ±1.037	50
		Delton	18.03 ^{AB} ±1.027	51

Sealant Adhesion with Adper Prompt



5 Adhesion to Enamel and Dentin

This chapter presents results into the bonding capabilities of Adper™ Prompt™ Self-Etch Adhesive. Testing the ability of an adhesive to bond to enamel and dentin is perhaps the most popular in vitro test performed on a dental adhesive. Adhesion testing is used to design new adhesives, compare existing products, investigate variables such as the effects of moisture and contamination, and, ultimately to try to predict clinical performance. As the first study of this section (Re et, al.), illustrates, high bond values can be achieved by adhesives in 4th, 5th, or 6th generation products.

Unfortunately there is not a standard methodology for testing bond performance. There are many different test procedures, differing in sample preparation, storage and thermal stress, and test geometry. Thus it is important to look at several results in order to draw conclusions pertaining to performance. This chapter presents test results from many sources. The first section illustrates some of the independent tests that were conducted as the formulation of Prompt was modified to the current product Adper Prompt adhesive. Following this section are independent studies presenting both product comparisons as well as application variables.

5. Adhesion to Enamel and Dentin

Shear Bond Strength of Three Different Adhesive Systems

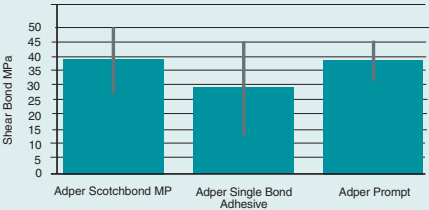
Authors: D. Re, D. Augusti, S. Semeraro, and M. Gagliani, University of Milan, Milano, Italy
Reference: IADR 2004, Honolulu USA, #1737
Objectives: The aim of this study was to evaluate the in vitro bond strength to dentin of three adhesive systems.
Methods: Thirty freshly extracted human teeth were mounted in acrylic molds and the facial surfaces were ground to expose middle dentin, which was polished to 600-grit. Teeth were randomly assigned to three groups (n=10), according to the bonding agent used: Scotchbond Multi-Purpose (3M ESPE); Scotchbond 1 (3M ESPE); Adper Prompt L-Pop (3M ESPE). Then the adhesive systems were applied by a single operator according to the manufacturer's instructions; the teeth were restored with composite resin Z100 (3M ESPE) and stored in distilled water at 37°C for 24 hours. SBS tests were performed using an Instron Machine at a cross head speed of 1mm/minute. The values were calculated in MPa and statistical analysis was performed using ANOVA.

Results:

SBS (MPa) n=10	Scotchbond Multi-Purpose	Scotchbond 1	Adper Prompt
Mean	39.2	29.16	38.48
SD	11.89	15.81	6.83
ANOVA		p<.05*	p=.0871

Conclusions: The multi-bottle SBM showed significantly (p<.05) higher bond strengths compared to SB1. No differences were found between the group SBM and LP. The self-etch adhesive LP showed the lowest standard deviation value.

Shear Bond to Dentin



Aim of the study: In this study the dentin bond values of three generations of adhesive by 3M ESPE were compared.
Result of the Study: All three generations displayed shear bond values to dentin in excess of 25 MPa.

Adper™ Prompt™

5. Adhesion to Enamel and Dentin

Changes to Original Prompt and Studies to Validate Improvement

Adper Prompt is one of the best examples of the synergy achieved by the combination of 3M Dental and ESPE to form 3M ESPE. Immediately after the merger, researchers at both centers collaborated to improve the performance of the Prompt adhesive system.

Modifications of the Adper Prompt adhesive system are illustrated by the figure below. The new formulation shares some of the design features of the Scotchbond Multi-Purpose dental adhesive system, with HEMA representing a widely used hydrophilic monomer, BisGMA to provide a durable cured film, and the unique polyalkenoic acid derivative developed for the Vitrebond™ light-cured glass ionomer liner-base. The next few pages are devoted to studies illustrating the effects of these improvements. Please note that several of these studies were conducted during the development phase of the product, and have not been published before.

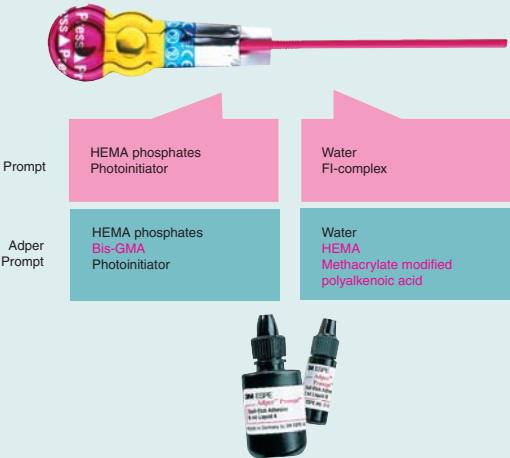
Adper™ Prompt™

5. Adhesion to Enamel and Dentin

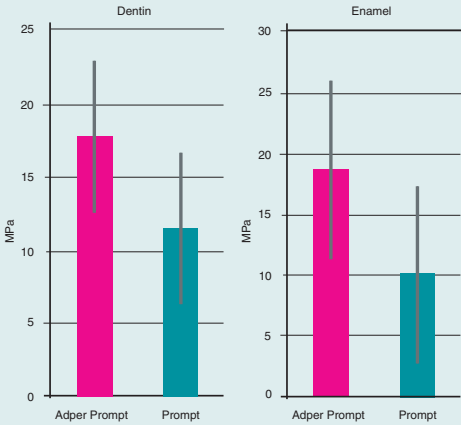
Microtensile Adhesion Comparison of Prompt and Adper Prompt

Authors: Dr. Bart vanMeerbeek, Leuven, Netherlands
Reference: Unpublished Data

Early in the development phase of the Adper Prompt system, Dr. Van Meerbeek used the microtensile approach to compare the adhesion of the original Prompt and the revised Adper Prompt. His test results indicated improvements in both the bond to enamel and the bond to dentin. These improvements were significant at the P .05 level.



Microtensile Bond to Dentin and Enamel



• Improvements significant at 0.05 level

Adper™ Prompt™

5. Adhesion to Enamel and Dentin

Shear Strength of Prompt and Adper Prompt

Authors: Dr. Heike Jung, Regensburg, Germany

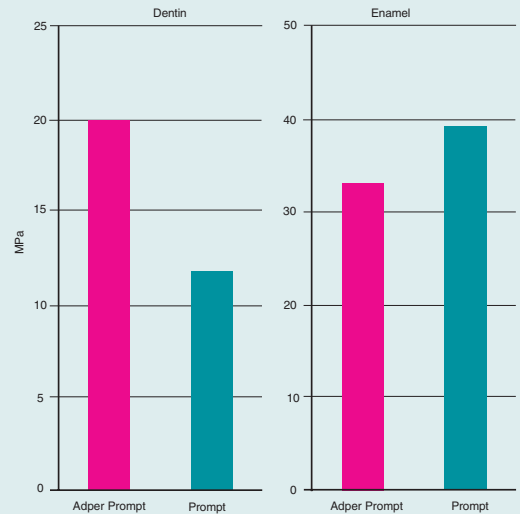
Reference: Unpublished Data

Dr. Jung used the shear bond technique to compare Prompt with the revised Adper Prompt system. Adper Prompt proved to be an improvement in bonding to dentin. Dr. Jung also felt that the new system created a better film on the tooth surface.

Quoting Dr. Jung “It was easier to create the uniform, shiny surface with Adper Prompt in comparison to Prompt L-Pop.”

Shear Bond to Dentin and Enamel

5



• Quote from author: “It was easier to create the uniform, shiny surface with Adper Prompt in comparison to Prompt L-Pop.”

Adper™ Prompt™ L-Pop™

Adper™ Prompt™ L-Pop™

5. Adhesion to Enamel and Dentin

Micro-Shear Bond Strengths of All-in-one Adhesives to Enamel and Dentin

Authors: S. Horiuchi¹, F. Nagano¹, W. Sasakawa¹, Y. Nakaoki¹, T. Ikeda¹, S. Uno¹, H. Sano¹, Y. Shimada², T. Nikaido², and J. Tagami², ¹Hokkaido U Dent, Sapporo, Japan, ²Tokyo Medical & Dental University, Graduate School, Faculty of Dentistry, Japan

Reference: IADR 2004, Honolulu USA, #0238

Objectives: Nowadays all-in-one adhesives, which are combined with etching, priming and bonding, have been clinically utilized for restorations of cavities with enamel-dentin margin. The purpose of this study is to compare the bond strengths of all-in-one adhesives to enamel and dentin substrate using micro-shear bond test (Shimada et al., JDR abstract, 2000).

Methods: The enamel or dentin disks were prepared by flat-grinding the occlusal surface of extracted human third molars. Three commercially available bonding systems and one experimental bonding system were used in this study: AQ Bond Plus (Sun Medical), Adper Prompt L-Pop (3M ESPE), XENO III (Dentsply-Sankin), and OBF-2 (Tokuyama). These adhesives were applied on the enamel or dentin surfaces according to manufacturers' instructions. Resin composite (Clearfil AP-X, Kuraray) was then mounted and light-cured for 40 seconds. After 24 hours immersion in water, a micro-shear bond test with a wire loop was carried out at a crosshead speed of 1.0 mm/minute to assess the micro-shear bond strength. Results were analyzed by one-way ANOVA and Tukey's HSD test (p<0.05).

Results: Micro-shear bond strengths (mean±standard deviation, in MPa) of tested adhesives were:

Adhesive	AQ Bond Plus	Adper Prompt L-Pop	Xeno III	OBF-2
Enamel	33.5±8.7	36.8±13.5	41.2±20.1	31.1±10.9
Dentin	37.4±10.7	28.4±6.9	29.9±7.5	32.5±14.7

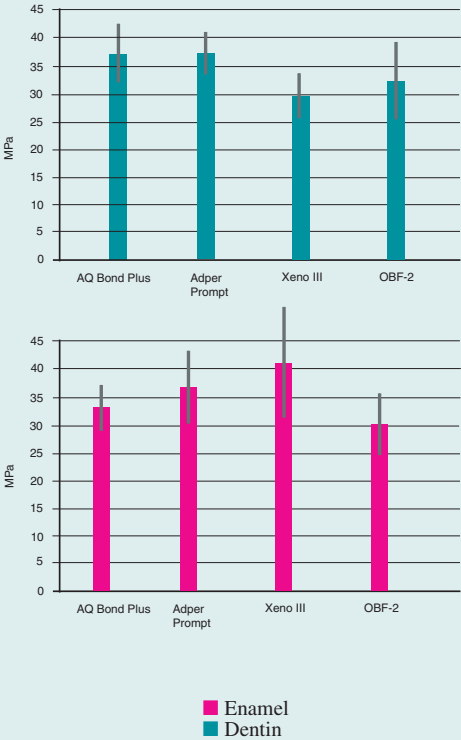
There was no statistically significant difference in the bond strength among all groups tested in this study.

Conclusions: The all-in-one adhesives used in this study showed the equivalent bond strength both for enamel and dentin.

Aim of the study: Many new self-etch systems are introduced to the market each year. This study compared the shear values of several of the most current systems.

Result of the Study: All of the systems displayed high shear bond values to enamel and dentin. Furthermore, bond values to enamel and dentin were very similar.

Micro-shear Bond Strengths



5. Adhesion to Enamel and Dentin

Bond Strength of Resin Adhesive Systems to Enamel and Dentin

Authors: T. Kimishima¹, Y. Nara¹, T. Eguro¹, T. Maseki¹, and I.L. Dogon², ¹Nippon Dental University, Tokyo, Japan, ²Harvard University, Forsyth Institute, Boston, MA, USA

Reference: IADR 2004, Honolulu USA, #3118

Objectives: The purpose of this study was to examine the tensile bond strength of one-step Adper Prompt L-Pop (3M ESPE), two-step self-etching adhesive systems [Clearfil SE Bond (Kuraray)], and one-bottle priming adhesive system [Single Bond (3M ESPE)]; to enamel and dentin of extracted human premolars using an original Portable Adhesion Tester (Nara Y et al., J. Dent Res. 75, SI #2943, 1996 etc.).

Methods: A standardized wedge shaped cavity was prepared in the cervical buccal side of the tooth. Dentin bond strength (DBS) test; was performed at the gingival dentine wall (n=8). Enamel bond strength (EBS) test; was performed at the beveled enamel (n=8). The test was performed immediately, after the system was applied to dentin or enamel, following manufacturer's direction, and combined with the original made composite resin (Kuraray) for PAT. The data were statistically analyzed using ANOVA, Tukey's q-Test and Weibull analysis.

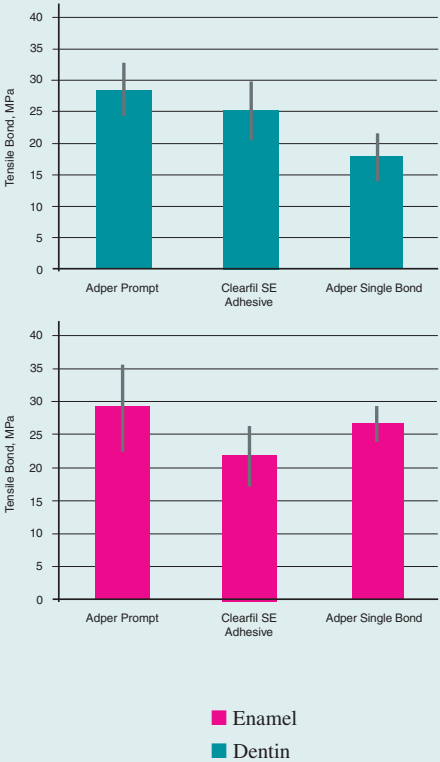
Results: Mean values (standard deviation) of EBS/DBS in MPa were; AL; 28.92 (6.57) / 28.39 (4.23), SE; 21.64 (4.56) / 25.00 (4.59), SB; 26.47 (2.72) / 17.68 (3.64). [EBS]; AL was statistically higher than SE (p<0.05). [DBS]; SB was lower than SE(p<0.05) and AL(p<0.01). EBS was higher than DBS in SB (p<0.01). Weibull modulus against EBS/DBS were; AL; 4.75 / 7.10, SE; 4.18 / 5.29, SB; 10.27 / 4.94. There was statistical difference between SB and the other two materials at enamel (p<0.01).

Conclusion: Although the EBS of each system showed equivalent in value, SB seemed to have better bonding quality, compare to the other systems. On the other hand, AL and SE DBS were higher than SB; however the quality of bonding were equivalent among the three systems.

Aim of the Study: The immediate bond value of adhesive systems is critical in counter-acting the contraction forces induced by polymerization shrinkage of dental composites. In this study the immediate tensile bond to enamel and dentin was tested using a novel methodology.

Result of the Study: Today's adhesives display excellent immediate bond values to both enamel and dentin.

Immediate Tensile Bond to Enamel and Dentin



5. Adhesion to Enamel and Dentin

Bond Strength of One-step and Two-step Resin Adhesive Systems to Enamel and Dentin

Authors: T. KIMISHIMA¹, Y. NARA¹, S. OSHITA¹, T. MASEKI¹, T. SUZUKI¹, I. KIZUKI¹, H. TANAKA¹, and L. DOGON², ¹The Nippon Dental University, Tokyo, Japan, ²Harvard School of Dental Medicine, Boston, USA ANMERKUNG kontrolliert

Reference: AADR 2003, San Antonio USA, #1630

Objectives: Purpose of this study was to examine the tensile bond strength of one-step (Adper Prompt L-Pop Self-Etch Adhesive (3M ESPE);AL, Prompt L-Pop(3M ESPE);PL, AQ Bond(Sun Medical);AQ, One-Up Bond F (Tokuyama);OB) and two-step resin adhesive systems(Clearfil SE Bond (Kuraray);SE, Single Bond (3M ESPE);SB) to enamel and dentine of extracted human premolars using an original Portable Adhesion Tester;(Nara et al., J. Dent Res. 75, SI #2943, 1996).

Methods: A wedge shaped cavity was prepared in the cervical of tooth. Enamel bond strength (E) test; was performed at beveled enamel prepared 2.0mm in width at occlusal margin (n=8). Dentine bond strength (D) test; at the gingival dentine wall (n=8). The system was applied to E or D following manufacturer's direction. Each system was combined with the same original composite resin (Kuraray) for the tester. The data were statistically analyzed using ANOVA and Tukey's q-Test.

Results: Mean values (SD) of the bond strength in MPa were; AL; E 28.92 (6.57) / D 28.39 (4.23), PL; E 27.52 (5.31) / D 26.67 (6.13), AQ; E 13.50 (3.06) / D 18.71 (4.01), OB; E 10.56 (3.24) / D 11.21 (3.82), SE; E 21.64 (4.56) / D 25.00 (4.59), SB; E 26.47 (2.72) / D 17.68 (3.64). <E> ; AQ and OB were statistically lower than the other systems (p<0.01). AL was higher than SE (p<0.05). <D> ; AL and PL were statistically higher than SB,OB and AQ (p<0.01, except AL/AQ; p<0.05). SE was higher than SB (p<0.01) and OB (p<0.05), and AQ was higher than OB (p<0.05). E was higher than D in SB (p<0.01), and D was higher than E in AQ (p<0.05).

Conclusions: AL, PL and SE showed high performance in both enamel and dentine bond strength test. Acid etching (i.e. SB) application seemed more adequate to enamel than dentine.

Aim of the Study: The objective of the study was to examine the tensile bond strength of one-step and two-step adhesive systems to enamel and dentin of extracted human premolars.

Results: Tensile bond strength of Adper Prompt L-Pop was higher than that of Clearfil SE. Adper Prompt L-Pop and Prompt L-Pop were on a higher statistical level than Single Bond, One-Up Bond F and AQ Bond. Adper Prompt L-Pop, Prompt L-Pop and Clearfil SE showed high performance in both enamel and dentin bond strength.

Bond Strength



5. Adhesion to Enamel and Dentin

Enamel Shear Bond Strength of Self-etching Adhesives

Authors: G.C. Lopes, F.C. Marson, L.C.C. Vieira, L.N. Baratieri, and M.A.C. Andrada, Universidade Federal de Santa Catarina, Florianópolis S.C, Brazil

Reference: IADR 2003, Göteborg, Sweden, #0346

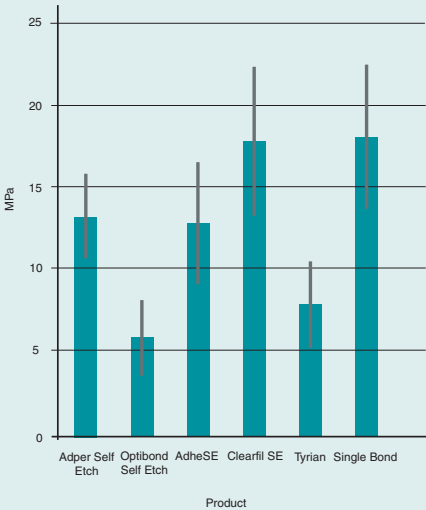
Objective: To compare the shear bond strength (SBS) to enamel with five self-etching primer systems and one total-etch one-bottle adhesive system.

Methods: Sixty freshly extracted bovine incisors were mounted, polished to 600-grit and randomly assigned to 6 groups (n=10): Adper Self Etch, 3M ESPE (AD), OptiBond Self-etching Primer, Kerr (OpS), AdheSE, Vivadent (ASE), Clearfil SE Bond, Kuraray (SE), Tyrian, Bisco (Ty) and SingleBond, 3M ESPE (SB) as control. The respective hybrid composite was applied in a #5 gelatin capsule and light-cured. After thermocycled (500x, 5-55° C, 60s dwell time), specimens were loaded in shear using an Instron at 5 mm/min. Mean bond strengths were analyzed with one-way ANOVA, followed by a Duncan's post-hoc test.

Results: SBS (mean ±SD): AD=13.0(±2.5)b; OpS=5.6(±2.3)c; SE=17.6(±4.5)a; Ty=7.6(±2.6)c; ASE=12.6(±3.7)b; SB=17.9(±4.4)a. Superscript letters indicate Duncan's homogeneous subsets. Only SE Bond showed similar enamel SBS than total-etch system (SB).

Conclusion: Shear Bond Strength to enamel with self-etching primers may depend on its specific composition.

Enamel Shear Bond



Adper™ Prompt™

5. Adhesion to Enamel and Dentin

Microtensile Bond Strength of new Self-etching Primer/Adhesives Systems

Authors: G.C. Lopes, M.C. Ribeiro, L.C.C. Vieira, and L.N. Baratieri, Universidade Federal de Santa Catarina, Florianópolis S.C, Brazil

Reference: IADR 2003, Göteborg, Sweden, #1447

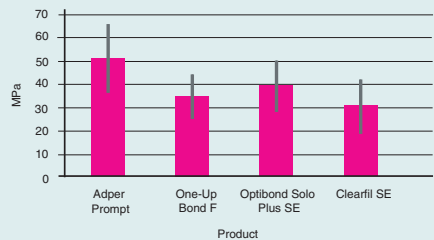
Objective: The aim of this in vitro study was to evaluate the microtensile bond strength (MTBS) to dentin with self-etching primers/adhesive systems.

Methods: Twenty human molars were transversally cut, polished to 600-grid. Teeth were randomly assigned to one of the groups: Adper Self Etch, 3M ESPE (AD) and One-Up Bond F, Tokuyama (OU) as self-etching adhesives; Clearfil SE Bond, Kuraray (SE) and Optibond Solo Plus - Self Etch, Kerr (OP) as self-etching primers. All adhesives were applied according to the manufacturer's instructions with its respective hybrid composites. After 24 h in water, the specimens were cut with a low-speed diamond saw in two perpendicular directions to obtain sticks with a cross section of approx. 0.35mm2(n=15). Mean bond strengths were analyzed with one-way ANOVA, followed by a Tukey's post hoc test.

Results: MTBS (mean ±SD): AD = 50.7 (±14.6)a; OU = 34.5 (±9.6)b; OP = 39.2 (±10.8)ab; SE = 30.3 (±11.5)b. Superscript letters indicate Tukey's homogeneous subsets. AD resulted in the highest mean dentin MTBS, not been different to OP. OP, AD and SE presented similar dentin MTBS.

Conclusion: The self-etching primers/adhesives tested in this project presented high bond strength to dentin.

Adhesion to Enamel and Dentin



Adper™ Prompt™

5. Adhesion to Enamel and Dentin

Microtensile Strength of Five Different Adhesive Systems

Authors: R.F. Mazuri, E.M. Souza¹, S. Vieira¹, S. Ignacio¹, and J.R. Saad², ¹Pontificia Universidade Catolica do Parana, Curitiba, Brazil, ²Unesp Araraquara Dental School, Brazil

Reference: IADR 2003, Göteborg, Sweden, #0336

Objectives: The aim of this study was to analyze the bond strength of five adhesive systems: Clearfil SE Bond - Kuraray Co. (SE); One Up bond F - Tokuyama (OU); Prime Bond NT - Caulk-Dentsply (NT); Single Bond - 3M ESPE (SB) Adper™ Prompt™ L-Pop™ - 3M-ESPE (AP).

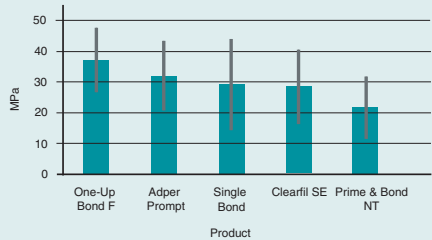
Methods: Ten freshly extracted human teeth were transversely wet-cut using a diamond disk in order to expose the occlusal dentin surface. Then the adhesive systems were applied according to the manufacturer's instructions and the teeth were restored with composite resin Z-100 (3M) and stored in distilled water at 37°C for 24 hours. A slow-speed diamond disk was used to prepare microtensile test specimens, which presented bonded area of 1 ± 0.02 mm². Each group resulted in thirty sticks, that were stored in distilled water at 37°C for 24 hours. The sticks were bonded on an universal testing machine at a cross head speed of 1mm/min.

Results: The following table shows microtensile bond strengths in MPa. Data were statistically analyzed using Kruskal-Wallis and Tukey Test (p 0.05). One up bond F (OU) and Adper™ Prompt™ L-Pop™ (AP) resulted in higher bond strength when compared to the other systems tested. The adhesive systems Clearfil SE Bond (SE), Prime Bond NT (NT) and Single Bond (SB) showed statistically similar bond strength values.

GROUPS n MEAN ± S.D. OU 30 36.73 ± 10.52 a AP 30 31.76 ± 11.30 a b SB 30 28.82 ± 14.74 b c SE 30 28.08 ± 12.09 b c NT 30 21.35 ± 10.22 c

Conclusion: Among the self-etching adhesive systems used in this study, One up bond F (OU) and Adper™ Prompt™ L-Pop™ (AP) showed the highest microtensile bond strength.

Microtensile Bond to Dentin



5. Adhesion to Enamel and Dentin

The Shear Bond Strength and Tensile Bond Strength of Adper Prompt on Primary Molars

Authors: Dr. Chang Yu; Beijing University

Reference: Unpublished Data

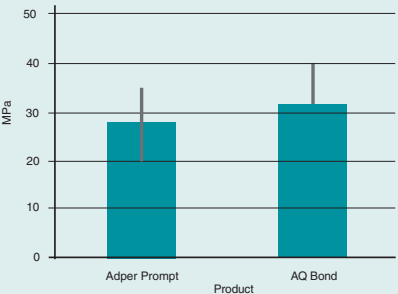
Objective: The aim of this study is to examine the shear bond strengths and tensile bond strengths of bonding systems on dentin of primary molars.

Methods and Materials: Dentin discs were prepared from extracted primary molars. The specimens were prepared with wet 600 SiC paper and divided randomly.

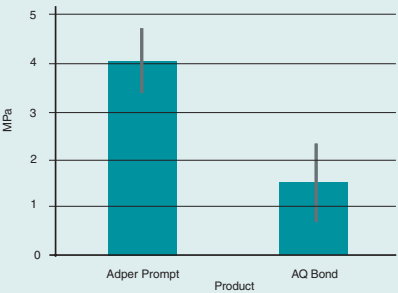
Results: The shear bond strengths were: 27.23±7.44MPa (Prompt L-Pop), 30.94±8.33Mpa (One-Coat Bond, Coltene). The tensile bond strengths were: 4.03±0.67MPa (Prompt L-Pop, 3M ESPE) 1.49 ± 0.82 MPa (One-Coat Bond, Coltene).

Conclusion: The shear bond strengths of Prompt L-Pop (3M ESPE) was similar to One-Coat Bond (Coltene). The tensile bond strength of Prompt L-Pop (3M ESPE) was much higher than those of the Coltene system (P=0.00, one-way ANOVA).

Shear Strength to Primary Dentin



Tensile Strength to Primary Dentin



5. Adhesion to Enamel and Dentin

Bond Strengths of Self and Separate-Etching Adhesive Systems

Authors: O. TULUNOGLU¹, I. TULUNOGLU², and N. HERSEK³, ¹Gazi Üniversitesi, Ankara, Turkey, ²Hacettepe University, Ankara, Turkey

Reference: 2004 Continental European Division of the IADR, Abstract 0263

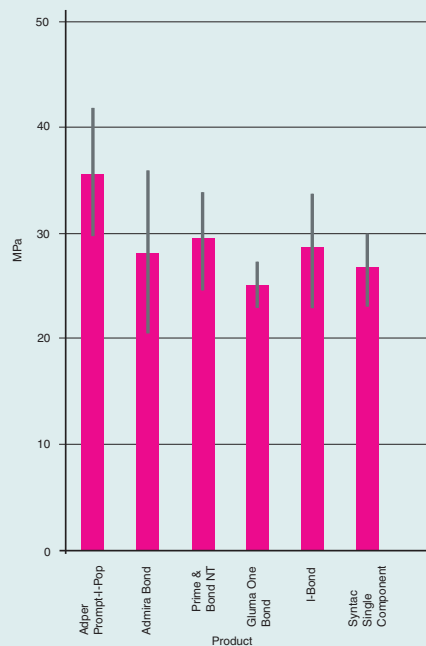
Objective: The purpose of this study was to evaluate the resin-dentin interfacial morphology and shear bond strength of several new dentin bonding systems classified as etch & rinse/total etch (Prime & Bond NT (Dentsply/De Trey, USA), Admira Bond (VOCO, W Germany), Gluma One Bond (Heraeus Kulzer), Syntac Single Component (Ivoclar/Vivadent USA)), and self etching (Adper Prompt-L-Pop (3M ESPE, USA), I-Bond (Heraeus Kulzer, Germany)) on the deep dentin of primary teeth at the end of one year storage period.

Methods: The occlusal surfaces of seventy-two recently extracted non-carious human primary molar teeth were abraded horizontally until a 1 mm residual dentine thickness was achieved. Composite resins were polymerised in clear PVC cylinders (1, 5mm Ø x 2 mm) on dentin specimens using one of six adhesive systems each representing a test group. All specimens were thermo cycled then stored in distilled water at 37°C for 365 days. The shear bond strength was calculated by dividing the peak failure loads by the bonding area. The data were statistically analysed using two-way ANOVA and Fisher's PLSD test at the 99% level of confidence. The cross-sectioned resin dentin interfaces were evaluated with SEM.

Results: The values of shear bond strengths for test groups were from higher to lower respectively as: Adper Prompt-L-Pop > Prime & Bond NT > I-Bond > Admira Bond > Gluma One Bond > Syntac Single Component. However, only the differences between Prompt-L-Pop vs. Syntac groups ($U=0$, $p=0.000$) and Prompt-L-Pop vs. Gluma groups ($U=1$, $p=0.000$) were statistically significant. SEM observation on sectioned surfaces of bonded specimens revealed seemingly equal length of resin tags in all groups.

Conclusion: There were not great differences amongst shear bond strengths of self etch and etch and rinse dentin bonding systems. In the long term self etch agents were more capable to penetrate into primary teeth dentine tubules.

Shear Bond to Primary Dentin



6. Technique Variables

Effect of Surface Moisture on Adper Prompt Self-Etch Adhesive

Authors: Dr. Lilliam Marie Pinzon, Dr. John M. Powers, Ph.D., Houston, USA

Reference: Unpublished Data

Study: A shear bond study was commissioned with Dr. John Powers to evaluate the effect of three levels of surface moisture on the bond to dentin. Adper Prompt and Prime & Bond NT were applied to surfaced dentin that was either dry, moist (blotted to remove excess moisture) or wet (water added to the surface).

Results: Adper Prompt bonded well on either dry or moist dentin. If the dentin was visibly wet bond performance was reduced. A wet surface may dilute the adhesive system.

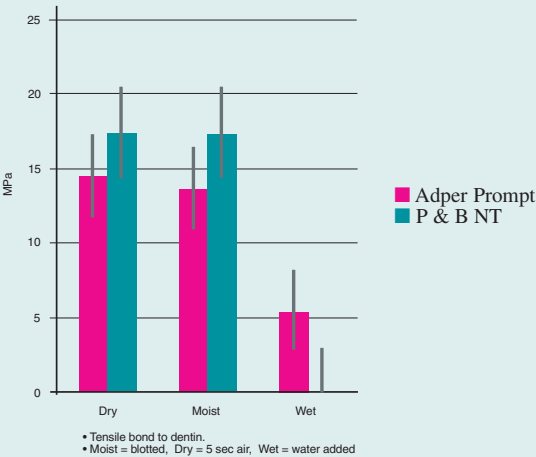
6 Technique Variables

In both clinical and laboratory settings, application technique variables can affect the bonding performance of an adhesive system. Much research has been published on the effect of surface moisture on adhesion; particularly in bonding to dentin. With the “total etch” systems, the hydration state of dentin after the etchant has been rinsed can drastically affect the ability of the adhesive to saturate the exposed collagen layer, resulting in different bonding performance. Self-etch systems are typically aqueous based. They also have the advantage of infiltrating dentin at the same time the dentin is being etched. These two attributes appear to allow a wider range of water content before bonding and may also account for reports of lower post-operative sensitivity.

Placement variables can affect any adhesive. Application technique, amount of adhesive, drying technique, and other procedural steps are of special interest.

In the following pages several studies into the effect of surface moisture, application of a second layer of adhesive, and drying technique are summarized.

Shear Bond to Dentin vs. Hydration State



6. Technique Variables

Effect of a Second Coat of Adhesive on Bond Strength

Authors: D. Carmona, and D. Lafuente, Universidad de Costa Rica, School of Dentistry, San Pedro, Costa Rica

Reference: IADR 2004, Honolulu USA, #0457

Purpose: To evaluate and compare the shear bond strength to superficial dentin of two different dentin adhesives, using two different application techniques.

Methods: A total of 20 healthy, recently extracted human molars were selected, embedded in acrylic, and polished using 600 grit SiC paper until superficial dentin was exposed. The following groups were established (n=5): (1.1) 3M ESPE Adper Prompt Self Etching System following manufacturer's instructions (1.2) 3M ESPE Adper Prompt Self Etching System two coats applied as before. (2.1) 37% Phosphoric acid and 3M ESPE Singlebond system, 2 coats brushed over the dentin surface each light cured for 20 seconds. (2.2) 37% Phosphoric acid and 3M ESPE Singlebond system, two coats rubbed over the dentin surface each light cured for 20 seconds. Then composite Z250 was light cured over the surface to form a 1mm diameter cylinder. The specimens were stored in a heating chamber in water at 37°C for a week before being tested in shear in the Universal Testing Machine (Instron 1000) at a crosshead speed of 0.1 cm/minute. Data was recorded in MPa and analyzed using a two way analysis of variance calculated at a 0.05 significance level. Tukey-Kramer intervals were 2.9 for comparisons between bonding agents and 22.4 between application techniques, also calculated at a 0.05 significance level.

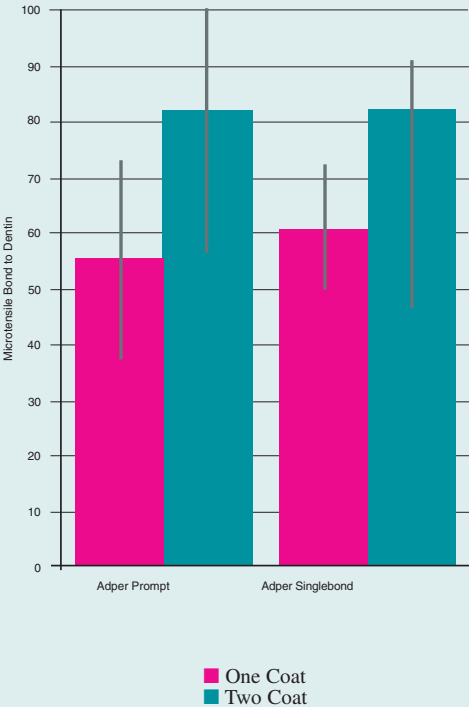
Results: Means and standard deviation in MPa using the suggested application technique were Adper Prompt 54.8 (17.6) and Singlebond 60.9 (11.3), and using a second coat the results were Adper Prompt 81.9 (25.8) and Singlebond 81.9 (35.8). If the application technique is changed, an increase in the bond strength was significant for both bonding agents. When compared by bonding agent, Singlebond showed a statistically higher bond strength than Adper Prompt.

Conclusions: A second application of the dentin bonding agent increases significantly the shear bond strength.

Aim of the study: Adhesion protocols often specify that the dentinal surface should have a shiny appearance after completion. Often, this calls for additional coats of adhesive. In this study, the effect of a second application of several adhesive systems was measured.

Result of the Study: Results varied between the tested products. For Adper Prompt, application of a second layer proved beneficial. Application of a second coat has been incorporated into the instructions for Adper Prompt. This second layer is applied and dried immediately after the first layer has been dried. A single light-cure is performed after the second layer has been dried.

Effect of Application of Second Adhesive Layer



Adper™ Prompt™ L-Pop™

6. Technique Variables

Effect of Double-Application of All-in-One Adhesives to Dentin Bonding

Authors: Y. Nakaoki¹, F. Nagano¹, S. Horiuchi¹, W. Sasakawa¹, T. Ikeda¹, S. Inoue¹, S. Uno¹, H. Sano¹, T. Ide¹, Y. Shimada², T. Nikaido², and J. Tagami², ¹Hokkaido U Dent, Sapporo, Japan, ²Tokyo Medical & Dental University, Japan

Reference: IADR 2004, Honolulu USA, #0029

Objectives: The clinical step of dentin bonding has been simplified with the development of all-in-one adhesives. Some of these adhesives are instructed as double application in bonding procedure and reported to show high bond strength to dentin. This study aims to evaluate the effect of double application of all-in-one adhesives to human dentin using micro-shear bond test (Shimada et al., JDR abstract, 2000).

Methods: The occlusal surfaces of extracted human third molars were ground perpendicular to the long axis of the tooth to expose a flat dentin surface. Three commercially available systems and one experimental bonding system were used in this study; newly developed OBF-2 (Tokuyama), Adper Prompt L-Pop (3M ESPE), REACTMER BOND (Shofu), and XENO III (Dentsply-Sankin). These adhesives were applied on the dentin surfaces according to the following method; manufacturers' instruction (single application) or experimental method (double application). Resin composite (Clearfil AP-X, Kuraray) was then mounted and light-cured for 40 seconds. After 24 hours immersion in water, a micro-shear bond test with a wire loop was carried out at a crosshead speed of 1.0 mm/minute to assess the micro-shear bond strength. Results were analyzed by one-way ANOVA and Tukey's HSD test ($p<0.05$).

Results: Micro-shear bond strengths (mean $\pm$ standard deviation in MPa) of tested adhesives were as follows.

Adhesive	OBF-2	Adper Prompt L-Pop	REACTMER BOND	Xeno III
Single-application	34.6 $\pm$ 4.9	22.7 $\pm$ 8.7	28.3 $\pm$ 6.7	30.3 $\pm$ 7.1
Double-application	32.5 $\pm$ 6.4	29.5 $\pm$ 9.2	27.2 $\pm$ 6.1	29.6 $\pm$ 6.4

The bonding strengths of OBF-2 (single-application and double-application) were significantly higher than single-application of PL.

Conclusions: Micro-shear bond strengths of all-in-one adhesive used in this study showed no significant difference between the single-application method and double-application method.

Aim of the study: Adhesion protocols often specify that the dentinal surface should have a shiny appearance after completion. Often, this calls for additional coats of adhesive. In this study, the effect of a second application of several adhesive systems was measured.

Result of the Study: Results varied between the tested products. For Adper Prompt, application of a second layer proved beneficial. Application of a second coat has been incorporated into the instructions for Adper Prompt. This second layer is applied and dried immediately after the first layer has been dried. A single light-cure is performed after the second layer has been dried.

Adper™ Prompt™

6. Technique Variables

Effect of Drying Technique

Authors: Dr. Ricardo M. Carvalho, São Paulo, Brazil

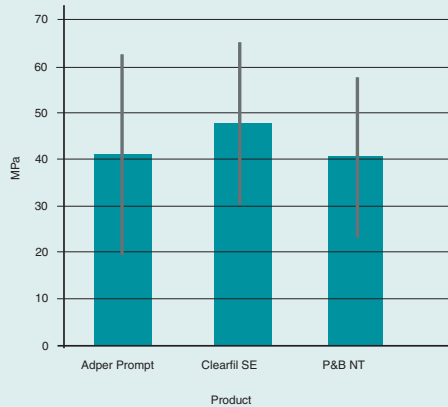
Reference: Unpublished Data

Dr. Ricardo M. Carvalho at the University of Sao Paulo conducted a study to evaluate the dentin bonding performance of three adhesive systems; Adper Prompt, Clearfil SE, and Prime & Bond NT. Testing was performed on extracted human molars.

During the evaluation of Adper Prompt the author noted the importance of a careful, thorough placement technique. With optimization of the drying technique, bond strength went from approximately 11 MPa to over 40 MPa.

The author obtained optimum results by placing a thin, even layer of adhesive rather than flooding the surface. Use of a gentle (rather than aggressive) air stream from a distance of approximately 10 cm allowed removal of the water inherent in the adhesive without displacing the adhesive system as a strong air blast might have done.

Microtensile Bond to Dentin



An important aspect of dentin bonding is the ability of an adhesive to resist the polymerization forces of the dental composite and maintain a sealed, continuous interface between tooth structure and composite. In the oral environment the ability to maintain marginal integrity will resist staining and ultimately, resist secondary decay.

As with adhesion tests, there are a myriad of ways to measure marginal integrity. One common method is to conduct a microleakage test. Variables in this type of study include the staining regimen, sample geometry, and thermal history. Often the seal of enamel and dentinal margins can be measured on the same sample. An alternative to a microleakage study is to use a microscopic technique such as SEM to measure continuous bonded interfaces.

In the next few pages are studies that challenged the ability of Adper™ Prompt Self-Etch Adhesive to maintain marginal integrity.

7. Marginal Integrity

Microleakage Evaluation of Enamel and Dentin Surfaces in Composite Restorations

Authors: D.A. Cavina, J.C. Gomes, O.M.M. Gomes, and A.L. Calixto, Universidade Estadual de Ponta Grossa, Ponta Grossa, Parana, Brazil

Reference: IADR 2004, Honolulu USA, #0506

Objective: Microleakage evaluation on enamel and dentin surfaces of self-etching adhesives in Class V restorations, with and without phosphoric acid etching.

Methods: Standardized Class V preparations were made on buccal and lingual surfaces of 28 human third molars with occlusal margins located in enamel and gingival margins located in dentin. The teeth were restored using Xeno III (Dentsply) and Adper Prompt (3M ESPE) adhesive systems and Filtek A110 (3M ESPE) composite. The teeth were thermocycled for 500 cycles and immersed in 50% silver nitrate solution for two hours. Then teeth were sectioned buccolingually through each restoration. The extend of dye penetration at margins were assessed under microscope at 20X power. The samples were divided into eight groups: GI – enamel + Xeno III + Filtek A110; GII – enamel + Phosphoric acid 37% + Xeno III + Filtek A110; GIII – dentin + Xeno III + Filtek A110; GIV – dentin + Phosphoric acid 37% + Xeno III + Filtek A110; GV – enamel + Adper Prompt + Filtek A110; GVI – enamel + Phosphoric acid 37% + Adper Prompt + Filtek A110; GVII dentin + Adper Prompt + Filtek A110 and GVIII – dentin + Phosphoric acid 37% + Adper Prompt + Filtek A110. Data were analyzed using Kruskal-Wallis and Dunn Tests ($p > 0.05$).

Results: There were no statistical differences among groups I, II, V, VI or among groups III, IV, VII, and VIII. When compared, groups with dentin and enamel margins displayed significant differences.

Conclusions: Gingival leakage was observed in all restorations while no leakage was observed in enamel margins. There were no significant differences at enamel and dentin margins when phosphoric acid etching was used or not before the self-etchings adhesive systems Xeno III (Dentsply) and Adper Prompt (3M ESPE).

7. Marginal Integrity

Microleakage at Gingival and Occlusal Margins

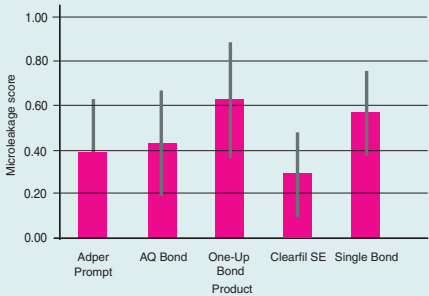
Authors: Dr. Yoichiro Nara, Tokyo, Japan
Reference: Unpublished Data

A microleakage evaluation comparing Adper Prompt to several self-etch products as well as a fifth generation product, Adper Single Bond, was conducted by Dr. Yoichiro Nara at Nippon Dental University, Tokyo.

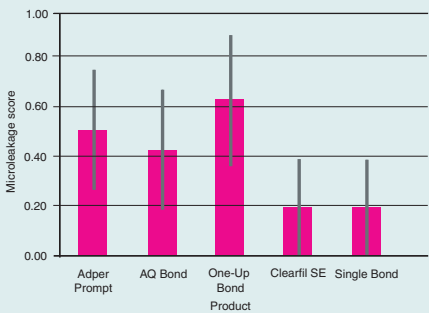
Dr. Nara uses a sophisticated technique to subject the extracted and restored teeth to thermal the mechanical stresses to better duplicate the oral environment. Standardized v-shaped Class V cavities were restored with the system in question. A cyclical load with maximum of 12 kgf and minimum of 0 kgf was applied at a 90 strokes per minute for a total of 1000 strokes. At the same time the samples were subjected to 125 cycles of thermal stress using water at 60 C and 4 C.

Results are summarized in the adjacent figures. Adper Prompt compared favorably to both 5th and 6th generation products in the ability to resist microleakage

Gingival Microleakage



Occlusal Microleakage



7. Marginal Integrity

Investigations into the Marginal Integrity of Adper Prompt Self-Etch Adhesive

Authors: Uwe Blunck, Berlin, Germany

Reference: Unpublished

Dr. Blunck used a micrographic methodology to detect and measure marginal gap formation. Samples were graded using the following criteria:

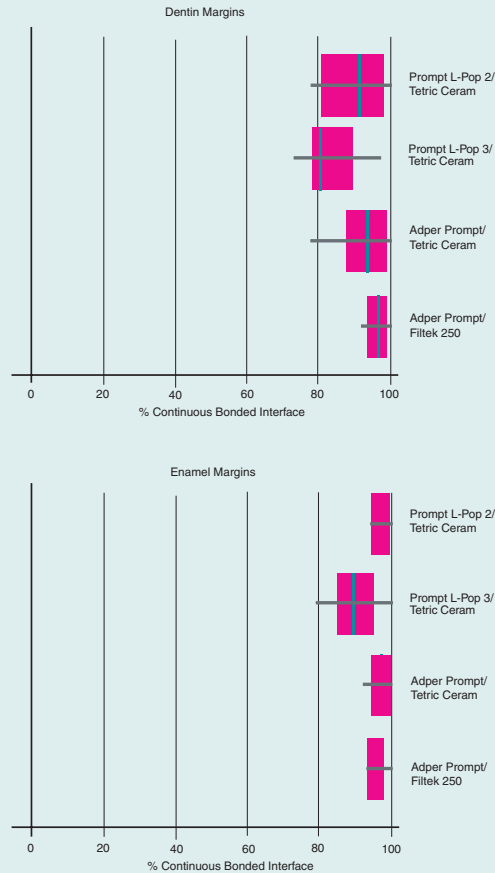
Rating	Definition
1	Margin not or hardly visible. No or minor marginal irregularities. No Gap.
2	No gap. Significant marginal irregularities.
3	Gap visible (up to 2µm). No marginal irregularities.
4	Significant gap (more than 2µm) with minor and/or significant marginal irregularities.
5	Gap between adhesive and restorative material.

7

In this investigation Dr. Blunck used 3 generations of the Prompt system, including Adper Prompt. Also included were two composites, Tetric Ceram and Filtek Z250. Filtek Z250 was designed to have lower polymerization shrinkage as well as lower residual stress than traditional hybrid composites, thus a comparison between composites is of interest.

Results of this study are presented on the facing page. Statistical analysis performed by Dr. Blunck did not reveal differences between study groups. All displayed a high rating for intact margins.

Dentinal and Enamel Marginal Integrity



7

7. Marginal Integrity

Microleakage of a New Restorative System in Posterior Teeth

Authors: S. Geraldeli, and J. Perdigao, University of Minnesota School of Dentistry, Minneapolis, USA

Reference: AADR 2003, San Antonio USA, #1276

Objectives: New developments in adhesive dentistry include self-etching adhesives and nanofilled composites. The null hypothesis tested in this in vitro study was that the combination of a self-etching adhesive, Adper Prompt (ADP, 3M ESPE), with a nanofilled composite (Filtek Supreme, SUP, 3M ESPE) would not result in greater microleakage than that obtained with phosphoric acid etching followed either by a nanofilled composite or an universal hybrid composite.

Methods: Class V's were prepared in the lingual and buccal aspects of thirty caries-free extracted third molars, with one margin in dentin/cementum and the other in enamel. Specimens were randomly assigned to three groups: (1) ADP+ SUP; (2) Single Bond (3M ESPE) + SUP; (3) Excite + Tetric Ceram (Ivoclar Vivadent). Specimens were isolated with nail polish except for a 1 mm-wide rim around the restoration, immersed in 0.5% basic fuchsin for 24 h at 37 C, sectioned, and evaluated for leakage (0-3 scale). Results: Medians (M) were analyzed with nonparametric tests (Mann-Whitney and Median tests, p£0.05, superscript letters):

Restorative	Enamel		Dentin	
	>M	£M	>M	£M
Adper Prompt + Supreme	7	3	6	4
Single Bond + Supreme	3	7	3	7
Excite + Tetric Ceram	3	7	6	4

The null hypothesis was accepted. Adper Prompt combined with Filtek Supreme resulted in statistically similar dentin (p>0.301) and enamel (p>0.114) microleakage scores than Single Bond + Filtek Supreme or Excite+Tetric Ceram.

Conclusions: The new restorative system resulted in enamel and dentin marginal sealing comparable to total-etch adhesives.

Interfacial Analysis

Scanning electron microscopy is one of the most powerful tools for characterization of dental adhesives. This tool allows the researcher to study the effects of adhesive systems (or individual components of the system) on the organic and inorganic components of dentin and enamel, the effect of hydration state, and provides insight into how the entire system reacts with tooth structure. In the following pages SEM studies of Adper™ Prompt Self-Etch Adhesive are summarized.

8. Interfacial Analysis

SEM Evaluation of the Resin-Adhesive Interface

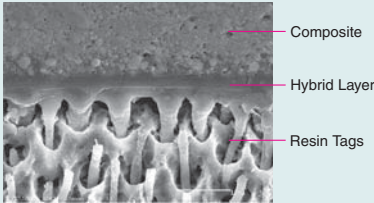
Authors: Dr. Patricia Pereira, Chapel Hill, USA
Reference: Unpublished Data

During the development of Adper Prompt, Dr. Patricia Pereira used SEM analysis to characterize the quality of the resin-tooth interface. Bonded enamel and dentin assemblies were prepared with all the adhesives in a similar way to the specimens that were used for microtensile bond testing. Test specimens were polished with wet silicon carbide papers and diamond pastes to high gloss. They were further subjected to acid/base treatment with 10% phosphoric acid and 6% sodium hypochlorite, gold sputter coated and morphology observed under the SEM. The thickness of the hybrid layers were measured at 5,000x magnification at three different points of five different specimens and means calculated.

Examples of the results are presented on the facing page. Note the excellent adaptation of the adhesive to both enamel and dentin. Note also the resin pattern exposed in the enamel sample, indicative of an excellent initial etch pattern as provided by Adper Prompt.

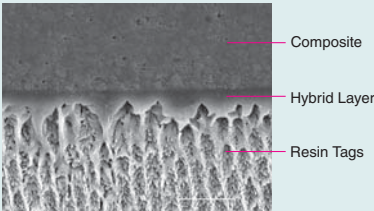
Interfacial Analysis of Adper Prompt

Dentin



Adhesive	Hybrid
Adper Single Bond	4.3 µm
Clearfil SE	0.6 µm
Adper Prompt	1.9 µm

Enamel

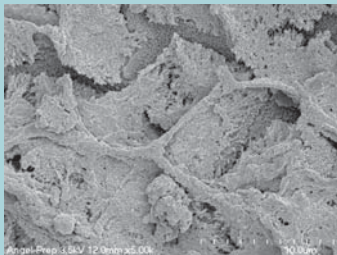


Adhesive	Hybrid
Adper Single Bond	4.5 µm
Clearfil SE	1.0 µm
Adper Prompt	1.2 µm

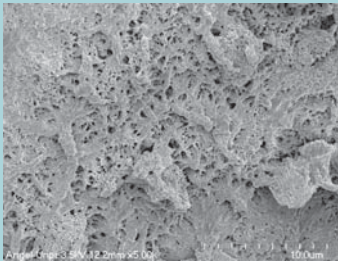
8. Interfacial Analysis

Enamel Etch Patterns; Cut and Uncut Enamel

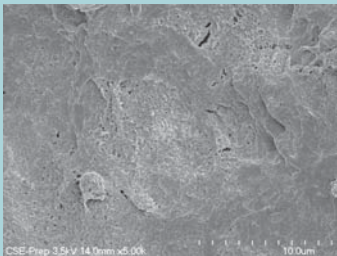
Adper Prompt; Prepared Enamel



Adper Prompt; Unprepared Enamel



Clearfil; Prepared Enamel



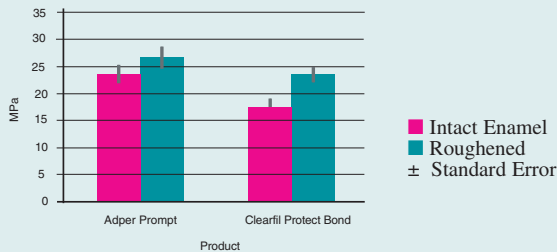
Clearfil; Unprepared Enamel



Dr. Jorge Perdigao, University of Minnesota, used scanning electron microscopy to evaluate the etch patterns of cut and uncut human enamel. Products evaluated were Adper™ Prompt Adhesive and Clearfil SE. The lower pH inherent with Adper Prompt adhesive appeared to provide deeper etch patterns on both substrates.

It should be noted that Adper Prompt adhesive is indicated for use on uncut enamel while Clearfil SE is not.

Microtensile Bond to Enamel



Adper™ Prompt™

8. Interface Analysis

SEM Observation of the Resin-Dentin Interface formed by Various Self-Etching Priming Adhesive Systems

Authors: T. Maseki¹, A. Shiota², T. Kimishima³, Y. Nara¹, and L. Dogon¹,
¹The Nippon Dental University, Tokyo, Japan, ²Harvard School of Dental Medicine, Boston, USA

Reference: IADR 2003, Göteborg, Sweden, #1959

Objective: Resin adhesive systems have been improved, and simplified the pretreatment method. Self-etching resin adhesive systems have been developed recently and widely used in clinical situations. The purpose of this study was to investigate the resin-dentin interface formed by various self-etching priming adhesive systems under FE-SEM observation.

Methods: Coronal dentin surfaces of extracted human third molars were polished with a series of SiC paper to 800 grit. The polished sound dentin surface was partially coated with an etching protector belt-likely, in order to investigate the position of the hybrid layer relative to the untreated dentin surface. 32 dentin specimens were treated by 2 all-in-one systems, **Adper Prompt Self-Etch Adhesive (AP; 3M ESPE)**, **One-up Bond F (OB; Tokuyama)**, and 2 two-step self-etching priming adhesive systems, **OptiBond Solo Plus Self Etch (OS; sds KERR)**, **Clearfil SE Bond (CS; Kuraray)** according to the manufacturer's instructions. Those specimens were sectioned vertically to the resin-dentin interface and polished with aluminum polishing powder. The polished specimens were treated with 10% hypochlorite solution at 60 sec and then observed under FE-SEM (Hitachi S-4000).

Results: The mean values of the thickness of the hybrid layer were **OB**; 0.78(0.12), **SE**; 0.90(0.19), **OS**; 1.56(0.24), **AP**; 2.14(0.29) μ m(SD). There was a significant difference in the thickness of the hybrid layer between **AP** and **OB**, **SE**, **OS** at $p < 0.01$, and also between **OS** and **OB**, **SE** at $p < 0.01$ (ANOVA and q-test).

Conclusion: It could be considered that the properties of self-etching priming adhesive systems, i.e., decalcification, permeation, have influence on the resin-dentin interface.

Adper™ Prompt™

8. Interfacial Analysis

SEM Observation of Bonding Interface of One-step Adhesive Systems to Root Dentin

Authors: T. Kaneko, S. Imazato, A. V Kaneshiro, Y. Takahashi, and S. Ebisu, Osaka University Graduate School of Dentistry, Japan

Reference: IADR 2003, Göteborg, Sweden, #0856

Objective: The purpose of this study was to investigate the bonding interface of one-step self-etching adhesive systems to sound or caries-affected root dentin by morphological observation with scanning electron microscopy (SEM).

Methods: Freshly-extracted human teeth were used. The cavities were prepared on sound root dentin or by removing root caries, and composite filling was performed using Adper Prompt (3M ESPE, AD), Reactmer Bond (Shofu, RE) or One-Up Bond F (Tokuyama, ON). Two-step self-etching system Clearfil SE Bond (Kuraray) served as controls. One half of the teeth in each group was sectioned longitudinally through the center of the restoration immediately after composite placement. The sectioned surface was fixed in half-Karnovsky's solution, polished and observed under SEM. The interfacial morphology of the other half of the specimens was examined after being stored in water at 37°C for 1 day.

Results: Similar to the control adhesive, gap-free adhesion with structural continuity to sound or caries-affected root dentin was observed for the specimens of AD irrespective of storage time. For RE and ON, although one-day storage specimens showed perfect adhesion, bonding was broken in some specimens when examined immediately after restoration probably due to the sample preparation procedure.

Conclusion: The results indicate that AD is able to produce stable adhesion while the systems which involve glass-ionomer reaction in bonding process form fragile interface at an early stage. This work was supported in part by a Grant-in-Aid for Scientific Research (13470402) from the Japan Society for the Promotion of Science.