

**3M** ESPE

A Collection of Scientific Results



**Adper<sup>TM</sup>**  
**Prompt<sup>TM</sup>**

*Self-Etch Adhesive*

In-Vivo Clinical Studies,  
In-Vitro Research, Reviews  
January 2002 — March 2005

# Adper™ Prompt™ L-Pop™



# Adper™ Prompt™ L-Pop™



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.

Adper Prompt Self-Etch Adhesive System is available as vial dose or unit-dose (L-Pop), depending on countries' registration.

■ Study sites

# Adper™ Prompt™ L-Pop™

## 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,

A handwritten signature in black ink, appearing to read "O. Gasser". The signature is stylized and written in a cursive-like font.

Dr. Oswald Gasser  
Global Technical Director 3M ESPE

# Adper™ Prompt™ L-Pop™

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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 you find summaries of clinical investigations on 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.

Parameter	Baseline (n = 40)	6 Months (n = 40)		12 Months (n = 40)		
		Alpha	Bravo	Alpha	Bravo	Charlie
<b>USPHS-valuation [%]</b>	Alpha	Alpha	Bravo	Alpha	Bravo	Charlie
<b>Marginal integrity</b>	100	100	0	92.5	5	0
<b>Marginal discoloration</b>	100	97.5	2.5	97.5	2.5	0
<b>Integrity of tooth</b>	100	97.5	2.5	82.5	17.5	0
<b>(Change in) sensitivity</b>	100	100	0	95.0	2.5	2.5
<b>Comments of patients</b>	100	100	0	97.5	2.5	0

# Adper™ Prompt™ L-Pop™

## 1. Clinical Results (In-Vivo)

### Three Year Clinical Performance of “All-in-one” Prompt L-Pop Self-Etch Adhesive

**Authors:** Manhart J., Huth K., Glomb C., Stueckgen D., Neurer P., Flessa H.-P., Hickel R.

**Reference:** IADR 2002, Cardiff, Wales, 25.-28.09.2002

**Objective:** The aim of this study was to assess clinical relevant data of class I fillings during a 3 years interval.

**Methods:** 25 patients were treated with 40 Class I fillings using Prompt L-Pop and the compomer material Hytac™ (3M ESPE). Marginal integrity, marginal discoloration, integrity of the tooth, Sensitivity were assessed baseline and after 6,12 and 24 months. Clinical parameters were evaluated according to modified USPHS criteria:

Parameters	Valuation method
Marginal integrity	Visually, probe
Marginal discoloration	Visually
Integrity of tooth	Visually, probe
(Change in) sensitivity	CO <sub>2</sub> -probe
Comments of patient	Questioning of patient

#### Results:

		24 Months (n = 34)				36 Months (n = 40)			
	Delta	Alpha	Bravo	Charlie	Delta	Alpha	Bravo	Charlie	Delta
	2.5	88.2	8.8	0	2.9	80.0	17.5	0	2.5
	0	100	0	0	0	100	0	0	0
	0	82.4	17.6	0	0	85.0	15.0	0	0
	0	97.1	0	2.9	0	97.5	0	2.5	0
	0	97.1	2.9	0	0	95.0	5.0	0	0

**Conclusion:** The self-etching adhesive Prompt L-Pop showed very good clinical results over a 3 years period. For the criteria marginal integrity, marginal discoloration and sensitivity predominantly alpha scores (USPHS-System) were reached.

# Adper™ Prompt™ L-Pop™

## 1. Clinical Results (In-Vivo)

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

1

**Authors:** C. Munoz<sup>1</sup>, J. Dunn<sup>1</sup>, J. Fundingsland<sup>2</sup>, and R. Richter<sup>3</sup>, <sup>1</sup>Loma Linda University, CA, USA, <sup>2</sup>3M ESPE, St. Paul, MN, USA, <sup>3</sup>3M ESPE, Seefeld, Germany

**Reference:** IADR 2004, Honolulu USA, #0541

**Objective:** 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
<b>Alpha</b>	72	75	100	95	100	95	100	90	100	100
<b>Bravo</b>	28	20	0	0	0	0	0	10	0	0
<b>Charlie</b>	0	5	0	0	0	5	0	0	0	0
<b>Delta</b>	0	0	0	5	0	0	0	0	0	0

- 1) Categories marginal adaptation (MA), adhesive retention (AR), and secondary caries (SC), were unchanged from baseline, with the exception of one restoration that was lost at the two-year recall.
- 2) Marginal discoloration (MD) showed a slight decrease in marginal discoloration.
- 3) No sensitivity (SE) 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.

**Conclusion:** The Prompt adhesive system displayed very good performance in all criteria (marginal adaptation, adhesive retention, marginal discoloration) 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.

# Adper™ Prompt™ L-Pop™

## 1. Clinical Results (In-Vivo)

### Adper™ Prompt™ Self-Etch Adhesive One-Year Clinical Report

**Authors:** Browning W., Medical & Dental University of Georgia in Augusta, USA

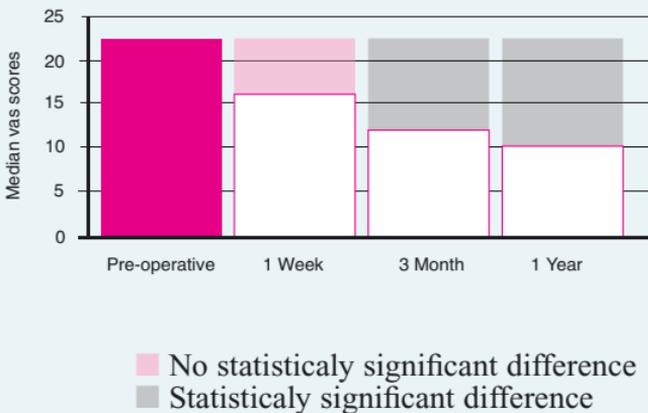
**Reference:** unpublished data

**Objective:** The aim of the study was to evaluate post-operative sensitivity. Evaluations were done pre-operatively, at one week, three months and one year using cold-water stimulus.

**Methods:** Two-hundred and nine restorations were placed in a general practice setting by four practitioners: 108 Adper Prompt self-etch adhesive and 101 Adper Single Bond adhesive. The majority of restorations were Class II. The patients scored their response on a Visual Analog Scale (VAS). The VAS was a 100mm line which ranged from “most severe pain” at one end to “no pain at all” at the other; a low numeric score indicated low pain. The restorations were also evaluated for clinical performance at baseline and one year by two examiners. Median sensitivity scores to cold stimulus at  $p < 0.05$  for both Adper Prompt self-etch adhesive and Adper Single Bond adhesive.

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#### Sensitivity to Cold Stimulus



**Results:** There was a statistically significant reduction in sensitivity from pre-operative to three months, and pre-operative to one year for both Adper Prompt self-etch adhesive and Adper Single Bond adhesive.

**Conclusion:** From over 200 posterior restorations placed by four general practitioners, both dentin bonding systems showed good clinical performance and low levels of post-operative sensitivity.

# Adper™ Prompt™ L-Pop™

## 2 Adhesion to Enamel and Dentin

This chapter presents results on 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 no 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 shows test results from product comparisons. Following this section you find test that were conducted as the formulation of Prompt was modified to the current product Adper Prompt Adhesive. One study shows testing of adhesives to primary dentin. The chapter ends with some SEM evaluations.

# Adper™ Prompt™ L-Pop™

## 2. 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

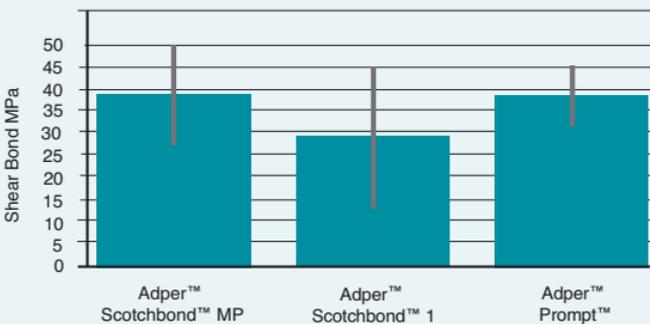
**Objective:** The aim of this study was to evaluate the in vitro bond strength to dentin of three adhesive systems.

**Methods:** Thirty freshly extracted human teeth were mounted in acrylic molds and the facial surfaces were grounded 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	Adper™ Scotchbond™ 1	Adper™ Prompt™
Mean	39.2	29.16	38.48
SD	11.89	15.81	6.83
ANOVA		p < 0.05*	p = 0.0871

#### Shear Bond to Dentin



**Conclusion:** The multi-bottle Scotchbond Multi-Purpose showed significantly (p < 0.05) higher bond strengths compared to Scotchbond 1. No differences were found between the group Scotchbond Multi-Purpose and Adper Prompt L-Pop. The self-etch adhesive Adper Prompt L-Pop showed the lowest standard deviation value.

# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Bond Strength of Resin Adhesive Systems to Enamel and Dentin

**Authors:** T. Kimishima<sup>1</sup>, Y. Nara<sup>1</sup>, T. Eguro<sup>1</sup>, T. Maseki<sup>1</sup>, and I.L. Dogon<sup>2</sup>, <sup>1</sup>Nippon Dental University, Tokyo, Japan, <sup>2</sup>Harvard University, Forsyth Institute, Boston, MA, USA

**Reference:** IADR 2004, Honolulu USA, #3118

**Objective:** 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.

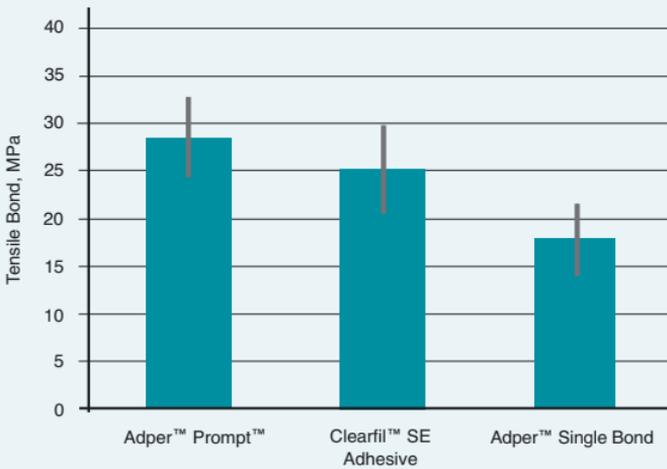
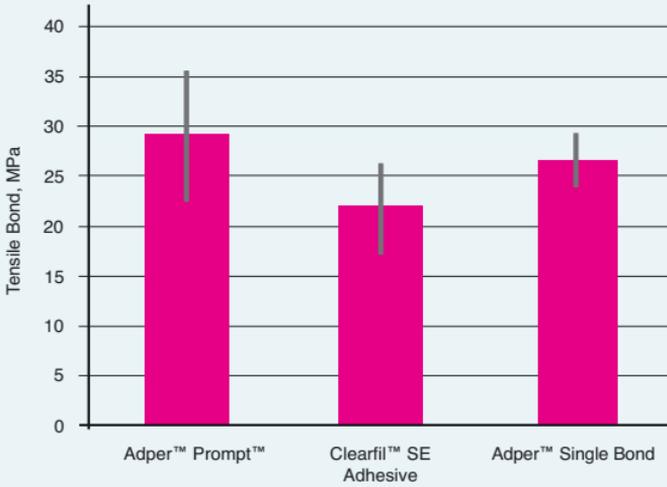
**Results:** Mean values (standard deviation) of EBS/DBS in MPa were; AL; 28.92 (6.57) / 28.39 (4.23), SE; 21.64 (4.56) / 25.00 (4.59), SB; 26.47 (2.72) / 17.68 (3.64). [EBS ]; AL was statistically higher than SE (p < 0.05). [DBS ]; SB was lower than SE (p < 0.05) and AL (p < 0.01). EBS was higher than 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 enamel bond strength of each system showed equivalent in value, Single Bond (SB) seemed to have better bonding quality, compare to the other systems. On the other hand, Adper Prompt L-Pop (AL) and Clearfill SE (SE) dentin bond strength were higher than Single Bond (SB); however the quality of bonding were equivalent among the three systems.

# Adper™ Prompt™ L-Pop™

## Immediate Tensile Bond to Enamel and Dentin

2



■ Enamel  
■ Dentin

# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

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

**Authors:** S. Horiuchi<sup>1</sup>, F. Nagano<sup>1</sup>, W. Sasakawa<sup>1</sup>, Y. Nakaoki<sup>1</sup>, T. Ikeda<sup>1</sup>, S. Uno<sup>1</sup>, H. Sano<sup>1</sup>, Y. Shimada<sup>2</sup>, T. Nikaido<sup>2</sup>, and J. Tagami<sup>2</sup>, <sup>1</sup>Hokkaido U Dent, Sapporo, Japan, <sup>2</sup>Tokyo Medical & Dental University, Graduate School, Faculty of Dentistry, Japan

**Reference:** IADR 2004, Honolulu USA, #0238

**Objective:** 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.

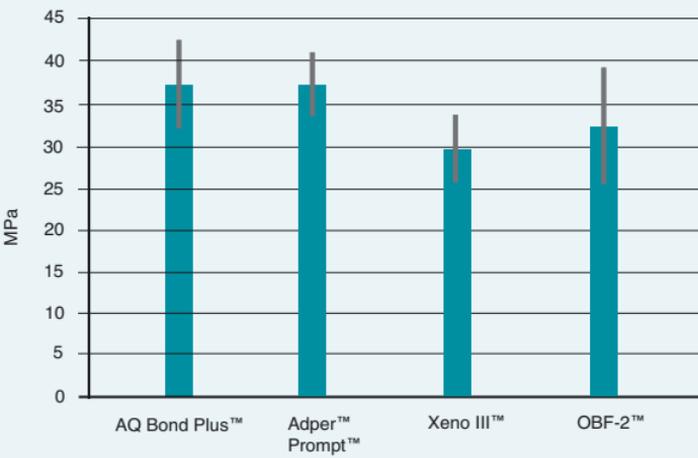
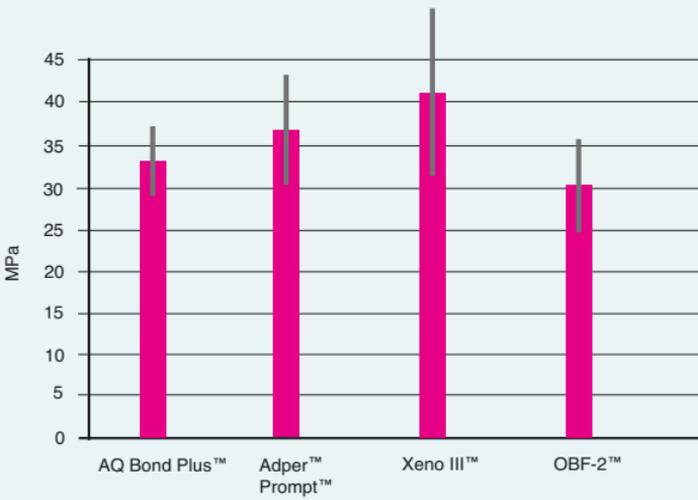
**Conclusion:** The all-in-one adhesives used in this study showed the equivalent bond strength both for enamel and dentin.

(This work was supported by grant-in-aid for Scientific Research, # 15390573 from the Ministry of Education, Culture, Sport, Science and Technology, Japan)

# Adper™ Prompt™ L-Pop™

## Micro-shear Bond Strengths

2



■ Enamel  
■ Dentin

# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Microtensile Strength of Five Different Adhesive Systems

**Authors:** R.F. Mazuri, E.M. Souza<sup>1</sup>, S. Vieira<sup>1</sup>, S. Ignacio<sup>1</sup>, and J.R. Saad<sup>2</sup>, <sup>1</sup>Pontificia Universidade Catolica do Parana, Curitiba, Brazil, <sup>2</sup>Unesp Araraquara Dental School, Brazil

**Reference:** IADR 2003, Göteborg, Sweden, #0336

2

**Objective:** 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).

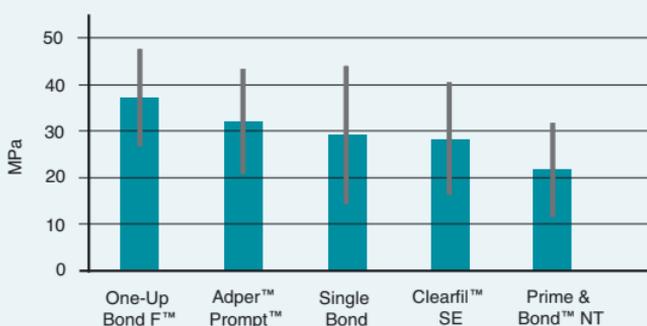
**Methods:** Ten freshly extracted human teeth were transversely wet-cut using a diamond disk in order to expose the occlusal dentin surface. Then the adhesive systems were applied according to the manufacturer’s instructions and the teeth were restored with composite resin Z100 (3M ESPE) and stored 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 a universal testing machine at a cross head speed of 1 mm/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



# Adper™ Prompt™ L-Pop™

## 2. 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.

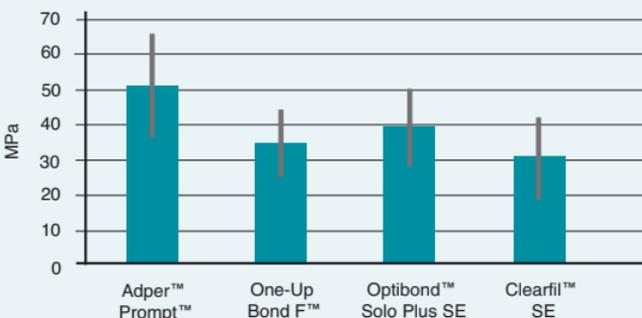
**Methods:** Twenty human molars were transversally cut, polished to 600-grid. Teeth were randomly assigned to one of the groups: Adper™ Self Etch, 3M ESPE (AD) and One-Up Bond F™ – Tokuyama (OU) as self-etching adhesives; Clearfil™ SE Bond, Kuraray (SE) and Optibond™ Solo Plus-Self Etch, Kerr (OP) as 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. 0.35 mm<sup>2</sup> (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.

2

### Adhesion to Dentin



# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Bond Strength of All-in-one Adhesive System to Enamel and Dentin

**Authors:** Y. NARA, Y. NAGAKURA, T. NITTA, Y. ITO, T. KIMISHIMA, T. MASEKI, and I.L. DOGON, Nippon Dental University, Tokyo, Japan, Harvard University, Boston, MA, USA

**Reference:** IADR 2005, #2943

2

**Objective:** Recently various types of all-in-one self-etch adhesive system have been developed and widely applied to clinical treatment. The purpose of this study was to examine the tensile bond strength (TBS) of all-in-one self-etch adhesive systems to cervical enamel and dentin.

**Methods:** Four systems on the market, Adper™ Prompt™ L-Pop™ Self-Etch Adhesive (APL, 3M ESPE), Brush&Bond™ (B&B, Sun Me-dical), G-Bond™ (GBN, GC) and One Up Bond F Plus™ (OBP, Tokuyama), and two experimental systems, SI-IB551 (SIB, Shofu) and SSB-200 (SSB, Kuraray), were used. Standardized V-shaped cavity having an occlusal enamel bevel was prepared in the buccocervical region of 72 extracted human premolars. The cavities were pretreated clinically with the six systems according to the manufacturer's instructions. The TBS of the systems to beveled enamel (E, n = 12) and gingival dentin wall (D, n = 12) were measured with a custom-made in vivo/vitro bi-use portable ad-hesion tester (JDR,78,SI,#3001,1999). The data were statistically analyzed using ANOVA and Tukey's q-test.

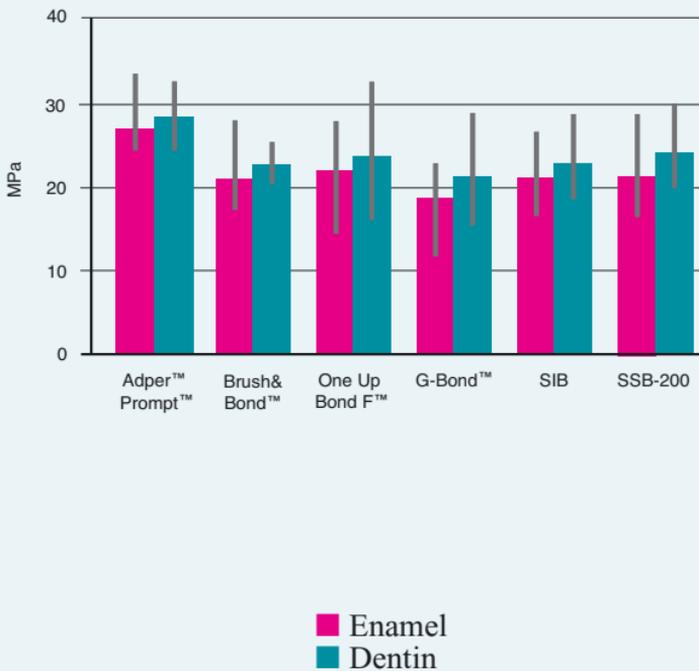
**Results:** The mean TBS (s.d.) in MPa to E/D were APL; 26.9 (6.1)/28.5 (3.5), B&B; 21.2 (6.2)/23.2 (2.6), GBN; 18.8 (4.5)/21.7 (7.0), OBP; 22.3 (5.0)/24.3 (7.7), SIB; 21.4 (5.0)/23.5 (4.9) and SSB; 21.6 (7.0)/25.0 (5.1). The TBS was influenced significantly by the difference in systems at  $p < 0.01$ . The TBS of APL was greater than those of GBN, B&B and SIB at  $p < 0.05$ . There was a significant difference in the TBS to E between APL and GBN, but the value to D did not vary with the systems. The TBS based on the six systems to D was significantly greater than the value to E at  $p < 0.05$ .

**Conclusion:** The TBS to both cervical enamel and dentin did vary with the six all-in-one self-etch adhesive systems used in this study. The difference in the TBS among the systems was found obviously in enamel, but not recognized in dentin. It seemed that the dentin bonding of the systems was superior to the enamel bonding.

# Adper™ Prompt™ L-Pop™

2

Tensile bond to enamel and dentin



# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Changes to Original Prompt™ and Studies to Validate Improvement

2

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 which ensures reproducible dentin adhesion. 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.



Prompt™	HEMA phosphates Photoinitiator	Water FI-complex
Adper™ Prompt™	HEMA phosphates <b>Bis-GMA</b> Photoinitiator	Water <b>HEMA</b> <b>Methacrylate modified polyalkenoic acid</b>



# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Microtensile Adhesion Comparison of Prompt™ and Adper™ Prompt™

**Authors:** Dr. Bart vanMeerbeek, Leuven, Netherlands

**Reference:** Unpublished data

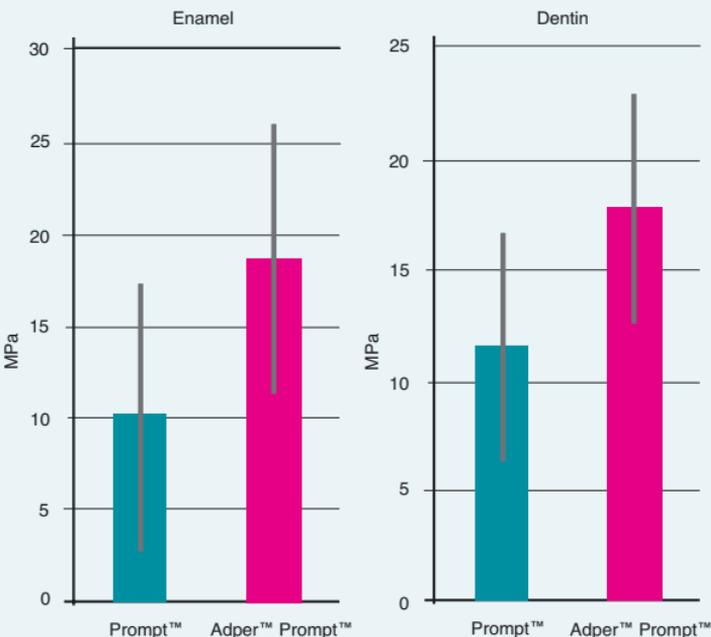
**Objective:** Compare the adhesion of the original Prompt™ and the revised Adper™ Prompt™.

**Methods:** Early in the development phase of the Adper Prompt system, Dr. Van Meerbeek used the microtensile approach.

**Conclusion:** 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.

2

#### Microtensile Bond to Enamel and Dentin



# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

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

**Authors:** T. KIMISHIMA<sup>1</sup>, Y. NARA<sup>1</sup>, S. OSHITA<sup>1</sup>, T. MASEKI<sup>1</sup>, T. SUZUKI<sup>1</sup>, I. KIZUKI<sup>1</sup>, H. TANAKA<sup>1</sup>, and L. DOGON<sup>2</sup>, <sup>1</sup>The Nippon Dental University, Tokyo, Japan, <sup>2</sup>Harvard School of Dental Medicine, Boston, USA ANMERKUNG kontrolliert

**Reference:** AADR 2003, San Antonio USA, #1630

**Objective:** 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 2.0 mm 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 SE (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).

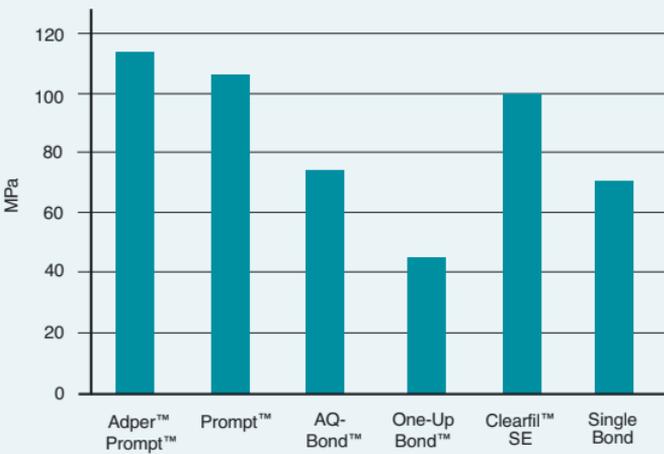
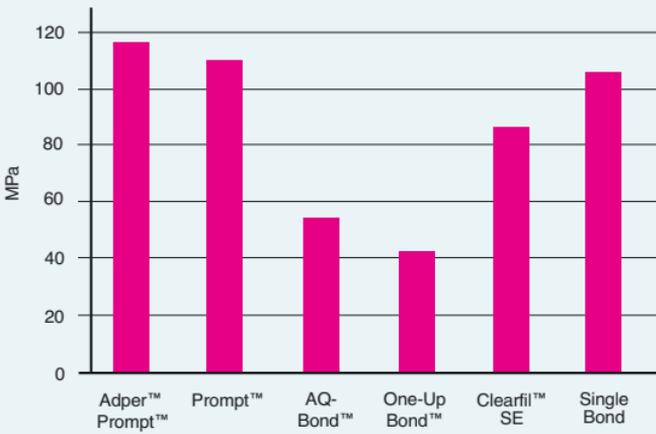
Tensile bond strength of Adper Prompt L-Pop was higher than that of Clearfil SE. Adper Prompt L-Pop and Prompt L-Pop were on a higher statistical level than Single Bond, One-Up Bond F and AQ Bond. Adper Prompt L-Pop, Prompt L-Pop and Clearfil SE showed high performance in both enamel and dentin bond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

**Conclusion:** Adper Prompt L-Pop, Prompt L-Pop and Clearfil SE Bond showed high performance in both enamel and dentine bond strength test.

# Adper™ Prompt™ L-Pop™

## Bond Strength

2



■ Enamel  
■ Dentin

# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### Bond Strengths of Self and Separate-Etching Adhesive Systems

**Authors:** O. TULUNOGLU<sup>1</sup>, I. TULUNOGLU<sup>2</sup>, and N. HERSEK<sup>2</sup>, <sup>1</sup>Gazi Universitesi, Ankara, Turkey, <sup>2</sup>Hacettepe University, Ankara, Turkey

**Reference:** 2004 Continental European Division of the IADR, # 0263

2

**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 (1,5 mm 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.

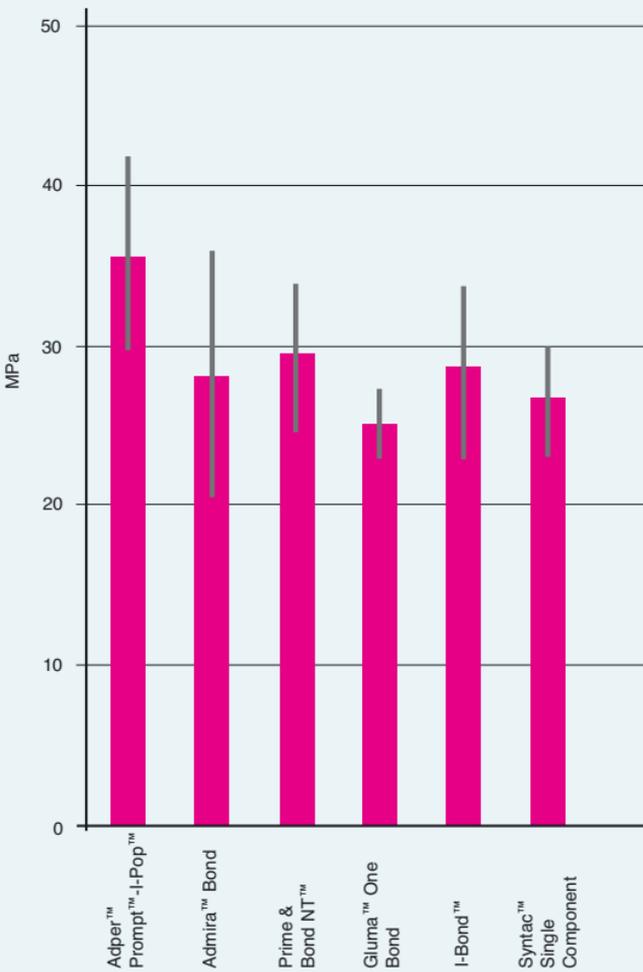
**Results:** The values of shear bond strengths for test groups were from higher to lower respectively as: Adper Prompt-L-Pop > Prime & Bond NT > I-Bond > Admira Bond > Gluma One Bond > Syntac Single Component. However, only the differences between Prompt-L-Pop vs. Syntac groups (U = 0, p = 0.000) and 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.

# Adper™ Prompt™ L-Pop™

## Shear Bond to Primary Dentin

2



# Adper™ Prompt™ L-Pop™

## 2. Adhesion to Enamel and Dentin

### SEM Evaluation of the Resin-Adhesive Interface

**Authors:** Dr. Patricia Pereira, Chapel Hill, USA

**Reference:** Unpublished data

2

**Objective:** During the development of Adper™ Prompt™, Dr. Patricia Pereira used SEM analysis to characterize the quality of the resin-tooth interface.

**Methods:** 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.

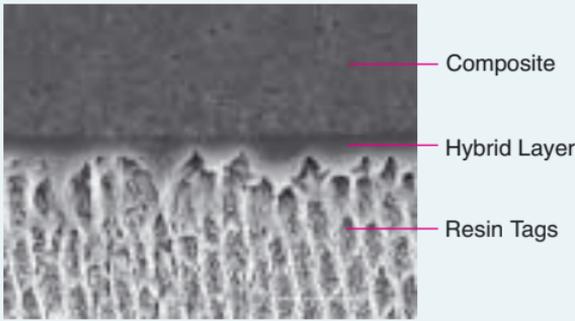
**Results:** Examples of the results are presented on the facing page.

**Conclusion:** 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.

# Adper™ Prompt™ L-Pop™

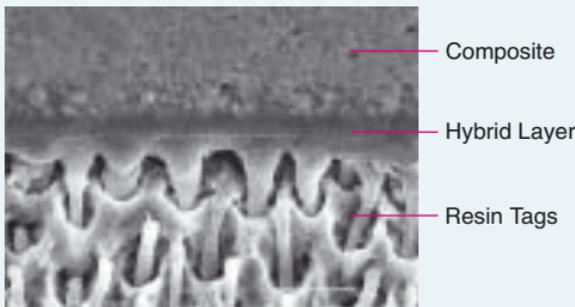
## Interfacial Analysis of Adper™ Prompt™

Enamel



Adhesive	Hybrid Layer
Adper™ Single Bond	4.5 $\mu\text{m}$
Clearfil™ SE	1.0 $\mu\text{m}$
Adper™ Prompt™	1.2 $\mu\text{m}$

Dentin



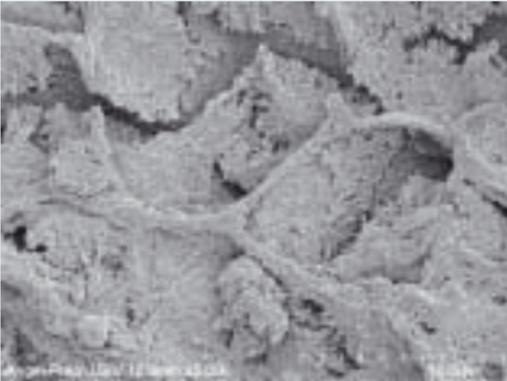
Adhesive	Hybrid Layer
Adper™ Single Bond	4.3 $\mu\text{m}$
Clearfil™ SE	0.6 $\mu\text{m}$
Adper™ Prompt™	1.9 $\mu\text{m}$

# Adper™ Prompt™ L-Pop™

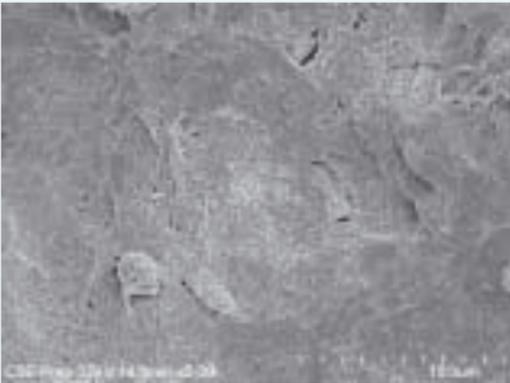
## 2. Adhesion to Enamel and Dentin

### Enamel Etch Patterns; Cut and Uncut Enamel

Adper™ Prompt™; Prepared Enamel



Clearfil™; Prepared Enamel



**Objective:** Dr. Jorge Perdigao, University of Minnesota, evaluated the etch patterns of cut and uncut human enamel.

**Methods:** Products evaluated were Adper™ Prompt™ Adhesive and Clearfil™ SE.

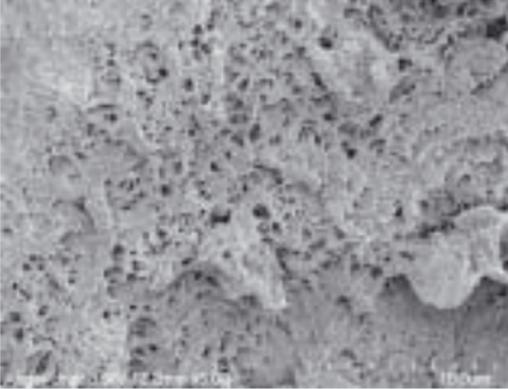
**Results:** The lower pH inherent with Adper Prompt adhesive appeared to provide deeper etch patterns on both substrates.

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

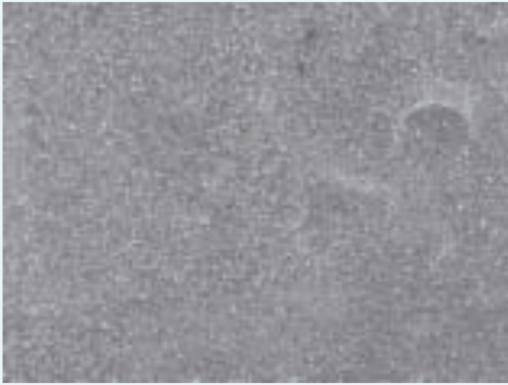
# Adper™ Prompt™ L-Pop™

2

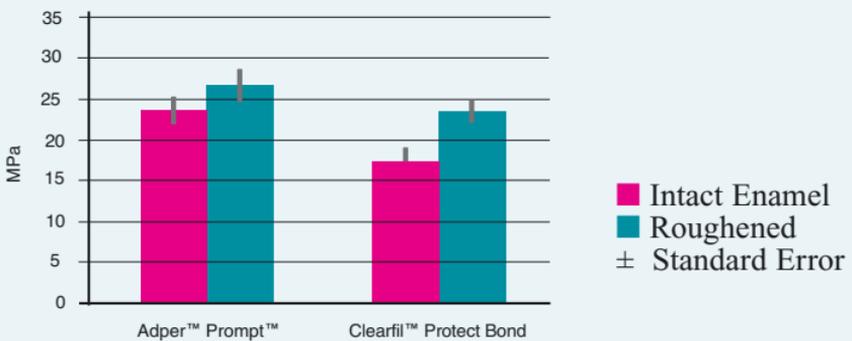
Adper™ Prompt™; Unprepared Enamel



Clearfil™; Unprepared Enamel



## Microtensile Bond to Enamel



# Adper™ Prompt™ L-Pop™

## 3 Marginal Integrity

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.

# Adper™ Prompt™ L-Pop™

## 3. 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:** IADR 2003, San Antonio USA, #1276

**Objective:** 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™ + Filtek™ Supreme	7	3	6	4
Single Bond + Filtek™ 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.

**Conclusion:** The new restorative system (Adper Prompt L-Pop and Filtek Supreme) resulted in enamel and dentin marginal sealing comparable to total-etch adhesives.

# Adper™ Prompt™ L-Pop™

## 3. Marginal Integrity

### Microleakage at Gingival and Occlusal Margins

**Authors:** Dr. Yoichiro Nara, Tokyo, Japan

**Reference:** Unpublished data

**Objective:** 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.

**Methods:** 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 kfg 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.

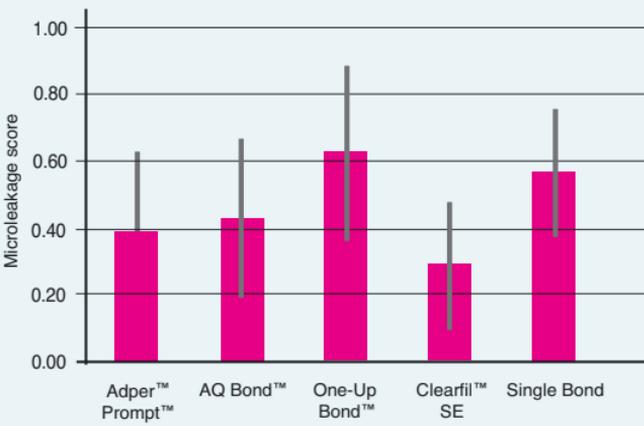
**Results:** Results are summarized in the adjacent figures.

**Conclusion:** Adper Prompt compared favorably to both 5th and 6th generation products in the ability to resist microleakage.

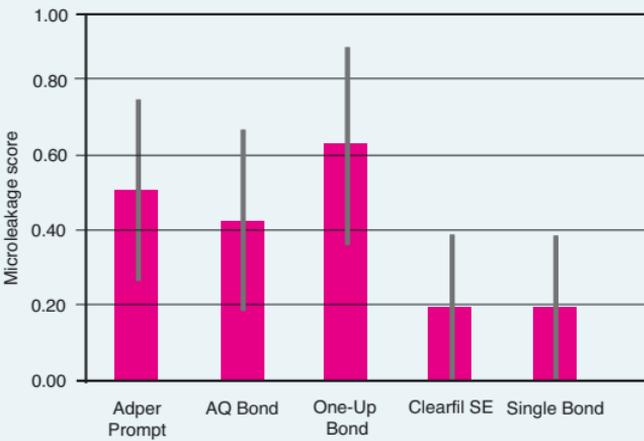
# Adper™ Prompt™ L-Pop™

3

## Gingival Microleakage



## Occlusal Microleakage





# Adper™ Prompt™ L-Pop™

## Optimize Clinical Use

## 4

Other variables besides adhesion can be decisive 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 you find several studies on 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.

# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

### 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.

**Methods:** All the components of the manufacturers product – closed adhesive 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.

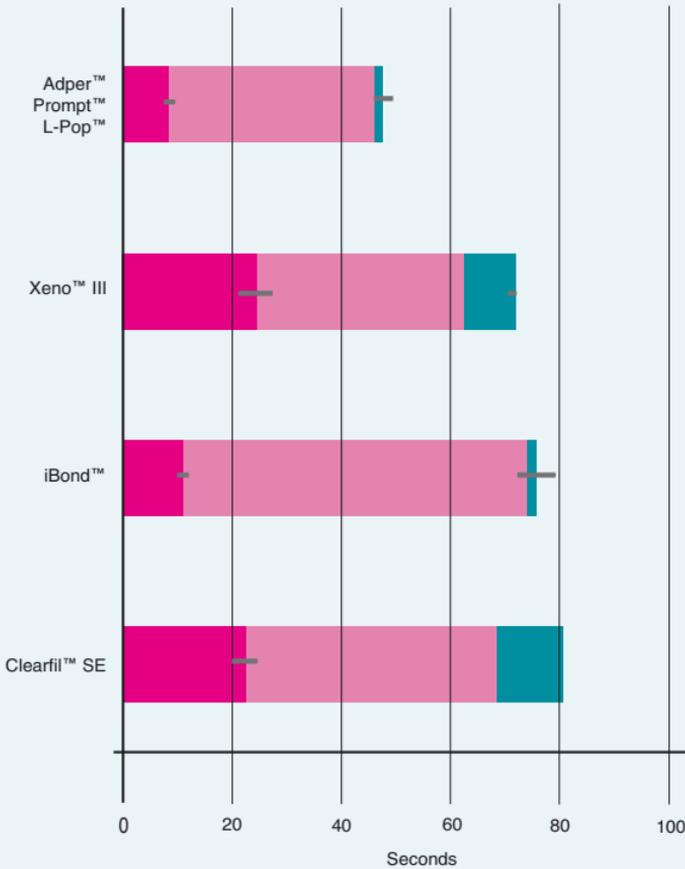
**Results:** The total working time (sec.) is the sum of the mean values of (Set-up time  $\pm$  standard deviation / Application time / Clean-up time  $\pm$  standard deviation) A: Adper™ Prompt™ L-Pop™ (3M ESPE)  $(8.8 \pm 0.87 / 38 / 1.7 \text{ a} \pm 0.65) = 48.5$ ; B: Clearfil™ SE Bond (Kuraray)  $(22.7 \pm 2.23 / 46 / 12.2 \pm 3.41) = 80.9 + \text{disinfection}$ ; C: iBond™ (Heraeus Kulzer)  $(11.3 \pm 1.07 / 63 / 1.9 \text{ a} \pm 0.67) = 76.2$ ; D: XENO™ III (Dentsply)  $(24.8 \pm 3.16 / 38 / 8.8 \pm 1.64) = 71.6 + \text{disinfection}$ . 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 Adper Prompt L-Pop and iBond. Adper Prompt L-Pop resulted in the shortest total working time of 48.5 seconds while Clearfill SE Bond resulted in the longest working time of 80.9 seconds.

# Adper™ Prompt™ L-Pop™

## Total Working Time

4



- Set-Up
- Application
- Clean-Up

# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

### Microtensile Bond Strengths of Sealants to Unprepared Enamel

**Authors:** J. PERDIGÃO<sup>1</sup>, J. FUNDINGSRAND<sup>2</sup>, S. DUARTE, Jr.<sup>3</sup>, and M.M. LOPES<sup>1</sup>, <sup>1</sup>University of Minnesota School of Dentistry, Minneapolis, USA, <sup>2</sup>3M/ESPE Dental Products, St Paul, MN, USA, <sup>3</sup>University of Minnesota, Minneapolis, USA

**Reference:** IADR 2003, Göteborg, Sweden, #0863

**Objective:** 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).

**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 (Filtek™ Z250, 3M ESPE) to provide a gripping surface. Specimens were cut in X and Y directions in sticks with section of  $0.7 \pm 0.1 \text{ mm}^2$  and tested in an Instron at 1mm/min at 24 h.  $\mu\text{TBS}$  data in MPa were analyzed with one- and two-way ANOVA/ Tukey's (superscript letters,  $p < 0.05$ ).

4

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

# Adper™ Prompt™ L-Pop™

**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.

**Conclusion:** Adper Prompt L-Pop 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 Adper Prompt L-Pop in 1 coat cured prior to sealant application is not recommended.

(This project was supported by 3M ESPE).

## Sealant Adhesion with Adper™ Prompt™



# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

### 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

**Objective:** To evaluate and compare the shear bond strength to superficial dentin of two different dentin adhesives, using two different application techniques. In this study, the effect of a second application of several adhesive systems was measured.

**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 Filtek™ 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.

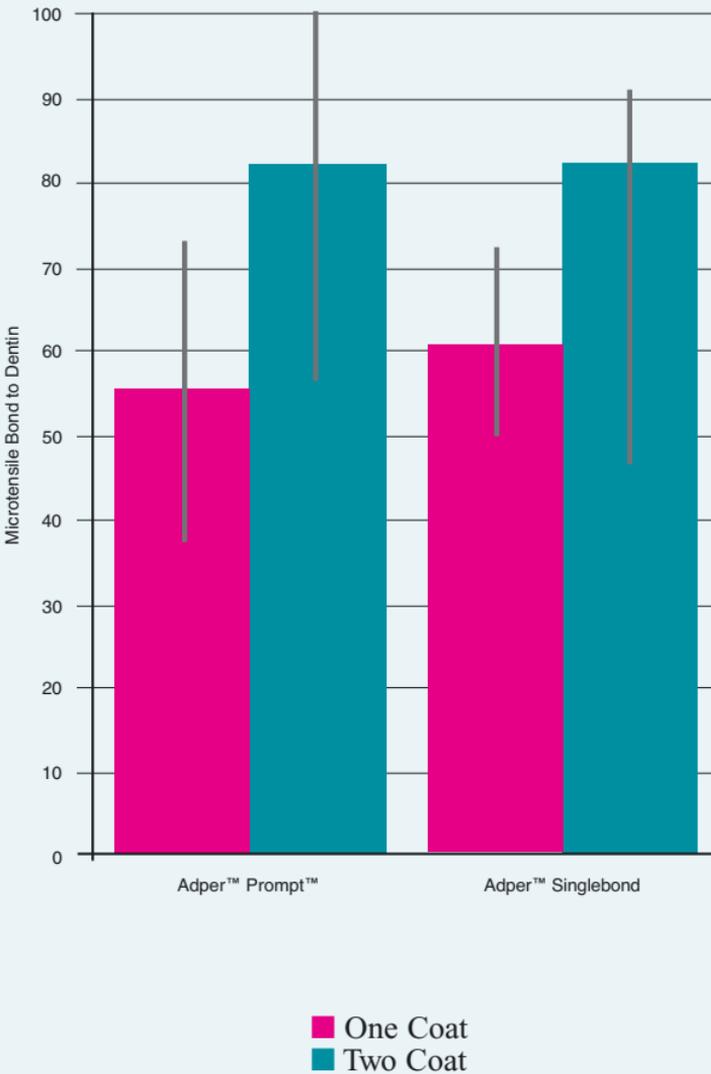
**Results:** Means and standard deviation in MPa using the suggested application technique were Adper Prompt 54.8 (17.6) and Singlebond 60.9 (11.3), and using a second coat the results were Adper Prompt 81.9 (25.8) and Singlebond 81.9 (35.8). If the application technique is changed, an increase in the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

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

# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

### Effect of Application of Second Adhesive Layer



# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

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

**Authors:** Y. Nakaoki<sup>1</sup>, F. Nagano<sup>1</sup>, S. Horiuchi<sup>1</sup>, W. Sasakawa<sup>1</sup>, T. Ikeda<sup>1</sup>, S. Inoue<sup>1</sup>, S. Uno<sup>1</sup>, H. Sano<sup>1</sup>, T. Ide<sup>2</sup>, Y. Shimada<sup>2</sup>, T. Nikaido<sup>2</sup>, and J. Tagami<sup>2</sup>, <sup>1</sup>Hokkaido U Dent, Sapporo, Japan, <sup>2</sup>Tokyo Medical & Dental University, Japan

**Reference:** IADR 2004, Honolulu USA, #0029

**Objective:** 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). In this study, the effect of a second application of several adhesive systems was measured.

**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. 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.

	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.

**Conclusion:** 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.

(Supported by grant-in-aid for Scientific Research, # 15390573 from the Ministry of Education, Culture, Sport, Science and Technology, Japan).

# Adper™ Prompt™ L-Pop™

## 4. Optimize Clinical Use

### 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

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

**Methods:** 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. Self etch adhesives caused less high-tissue irritation scores.

# Adper™ Prompt™ L-Pop™

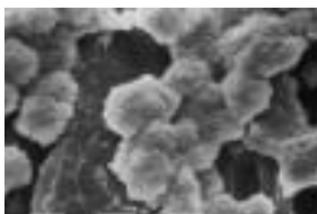
## 4. Optimize Clinical Use

### Effect of Adper™ Prompt™ Adhesive™ on Oral Bacteria

**Authors:** Dr. Susanne Kneist, Jena, Germany

**Reference:** Unpublished data

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



*S. Mutans*

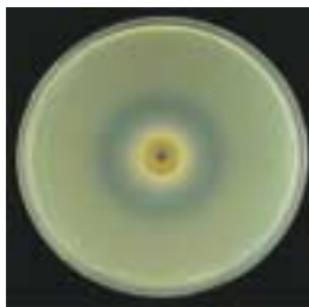
**Methods:** The 8 strains of bacteria; *A. naeslundii*, *A. odontolyticus*, *S. sanguis*, *S. mutans*, *S. sobrinus*, *S. salivarius*, *L. casei*, *L. delbrueckii* ss *lactis*; were cultured under anaerobic conditions in Balmelli bouillon at 37°C. Strain suspension was suspended in liquefied balmelli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

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 mixed adhesive on S. Mutans.*



*Effect of cured adhesive on S. Salivarius.*

# Adper™ Prompt™ L-Pop™

## Desensitization of Hypersensitive Root Surfaces 5

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.

# Adper™ Prompt™ L-Pop™

## 5. Desensitization of Hypersensitive Root Surfaces

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

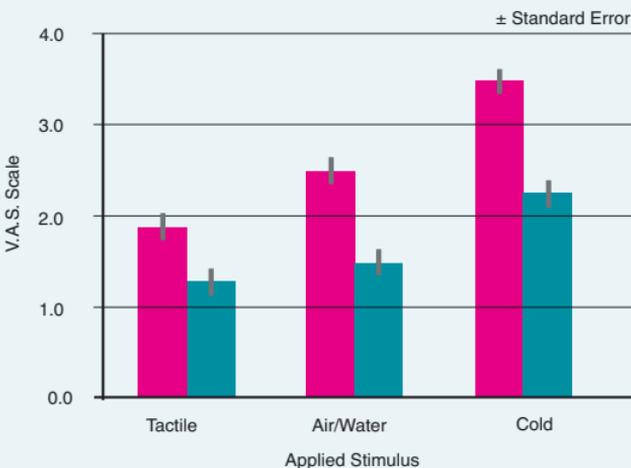
**Reference:** Unpublished data

**Objective:** 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.

**Methods:** The randomized, controlled clinical study was conducted with Dr. G Kugel and Dr. R Perry, Boston, MA. 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.

5

### Effect of Adper™ Prompt™ on Root Surface Hypersensitivity



# Adper™ Prompt™ L-Pop™

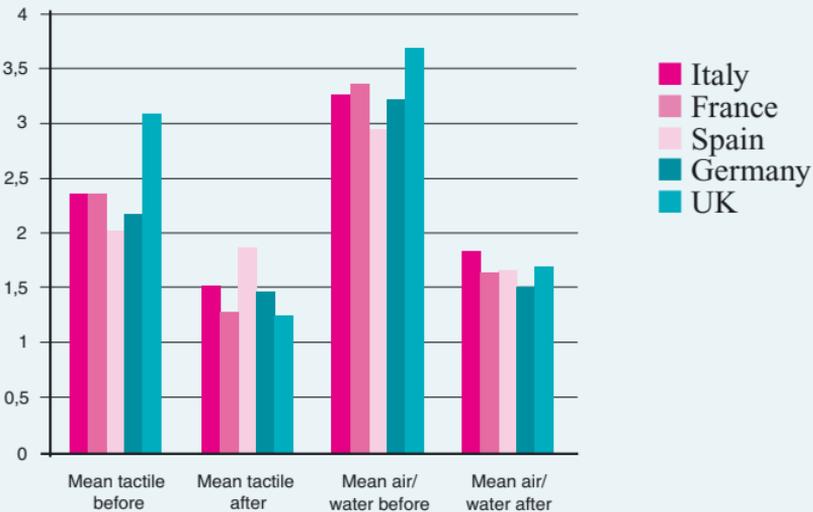
## 5. Desensitization of Hypersensitive Root Surfaces

**Results:** 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.

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

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Effect of Adper™ Prompt™ on Root Surface Hypersensitivity



## Adper™ Prompt™ L-Pop™

## 5. Desensitization of Hypersensitive Root Surfaces

## Effect of Dentin Desensitizing Agents on Dentin Permeability After Thermocycling

**Authors:** K.-A. HILLER, S. VRANA, M. DAUBNNER, and G. SCHMALZ, University of Regensburg, Germany

**Reference:** IADR 2005, Baltimore, #0298

**Objective:** Previous studies have shown a reduced permeability of dentin after application of dentin desensitizing agents.

The purpose of this study was to compare the reduction of dentin permeability using dentin desensitizing agents after thermocycling.

**Methods:** 52 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 200  $\mu\text{m}$  thickness. Dentin permeability (hydraulic conductance,  $L_p$ ,  $\text{m}^3/\text{Ns}$ ) using aqua bidest at 1.4 m  $\text{H}_2\text{O}$  was measured for each specimen at baseline, after treatment, and after thermocycling (5°C-55°C, 5000 cycles, 60 s/cycle) using a split-chamber apparatus connected to a commercially available measurement unit (Flodec, DeMarco Engineering SA, Geneva). Materials were applied according to manufacturers' instructions. The slices served as their own controls. Materials tested comprised Gluma™ Desensitizer (Heraeus Kulzer, GD), Seal&Protect™ (Dentsply/DeTrey, SP), SuperSeal™ (Phoenix Dental Inc., SS), and Adper™ Prompt™ L-Pop™ (3M ESPE, AL). 6 slices without treatment were used as additional controls. Test parameter  $L_p\text{-REL}$  was the percentage of fluid flow before and after thermocycling (100% =  $L_p$  before treatment). Mann-Whitney test ( $p \leq 0.05$ ) was used to evaluate differences between groups (10–13 samples).

**Results:** The median  $L_p$  before treatment varied between 1.7–2.9e-10  $\text{m}^3/\text{Ns}$  and were statistically not different. Median  $L_p\text{-REL}$  (25-75% Quantiles) values were:

Material	$L_p\text{-REL}$ (%) before thermocycling	after thermocycling
No material	107(97-157)	361(256-536)
GD	103(40-116)	146 (37-323)
SP	81(49-99)	464(200-1030)
SS	56(0-130)	654(116-795)
AL	54(2-99)	80(8-123)

Thermocycling significantly increased the permeability. No statistical influence of materials before thermocycling was found, but after thermocycling. Before thermocycling, SP, SS, and AL, and after thermocycling, GD and AL reduced  $L_p$  significantly. After thermocycling only AL caused reduced permeability.

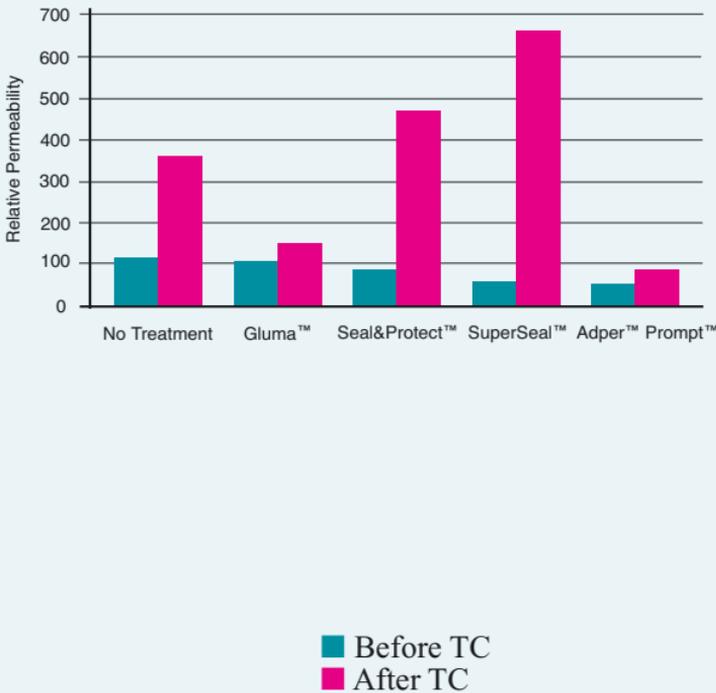
**Conclusion:** Thermocycling had an effect on hydraulic conductance, depending on dentin desensitizing agents.

# Adper™ Prompt™ L-Pop™

## 5. Desensitization of Hypersensitive Root Surfaces

5

Effect of several treatments on dentin permeability



# Adper™ Prompt™ L-Pop™

## 5. Desensitization of Hypersensitive Root Surfaces

### Sealing effects of adhesives to protect demineralization on root surface

**Authors:** S. IMAZATO<sup>1</sup>, S. EBISU<sup>1</sup>, and A.W.G. WALLS<sup>2</sup>, <sup>1</sup> Osaka University, Suita, Japan, <sup>2</sup> Newcastle University, Newcastle Upon Tyne, United Kingdom

**Reference:** IADR 2005, Baltimore, #0523

**Objective:** The aim of this in vitro study was to examine the ability of an all-in-one adhesive to seal and inhibit demineralization on root surface in comparison with a protective sealant.

**Methods:** The root specimens cut from human extracted molars were covered with nail varnish, leaving a window of approximately 4 mm x 4 mm. Adper™ Prompt™ (3M ESPE, AD) or Seal & Protect™ (Dentsply/DeTrey, SP) was applied to the window, and the specimens were immersed in acetate buffer at pH 5.5. After 4 weeks of storage at 37° C, the presence of demineralized area was evaluated using a dissecting microscope and a contact microradiography after sectioning the specimen through the center of the window. Five specimens were tested for each material. The additional specimens, to which AD or SP was applied, were sectioned longitudinally and cementum-adhesive interface was observed using a scanning electron microscopy (SEM).

**Results:** Demineralization of root surface by acid attack was inhibited by application of both materials. All of the AD specimens exhibited inhibition, while demineralization patterns were observed for some of the SP specimens in which very thin resin layer was seen on the surface. SEM analysis demonstrated formation of hybridized layer of cementum with resin components after application of both materials in addition to coverage of the root surface by resin.

**Conclusion:** The sealing ability of the all-in-one adhesive AD on root surface is greater than that of SP, although both materials showed protection against demineralization under the present experimental condition.

(This work was supported by a Grant-in aid for Scientific Research (16390545) from the Japan Society for the Promotion of Science and the 21st Century COE at Osaka University Graduate School of Dentistry supported by the Ministry of Education, Culture, Sports, Science and Technology).



# Adper™ Prompt™ L-Pop™

## 5. 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

**Objective:** 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.

**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,  $L_p, m^3/Ns$ ) using aqua bidest at 0.7 m H<sub>2</sub>O, 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 H<sub>2</sub>O 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  $L_p$  after treatment (100%= $L_p$  before treatment). Mann-Whitney Test ( $p \leq 0.05$ ) was used to evaluate differences between groups (10 samples).

**Results:** The median  $L_p$  before treatment varied between 1.01–2.02E-10m<sup>3</sup>/Ns for the 12 groups. Median P-RED[%] of  $L_p$  (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)

# Adper™ Prompt™ L-Pop™

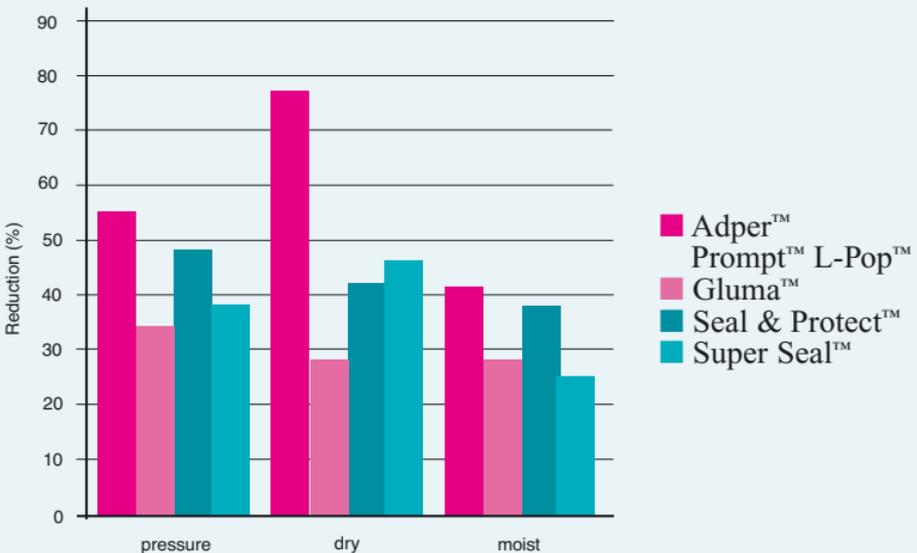
## 5. Desensitization of Hypersensitive Root Surfaces

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.

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

5

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



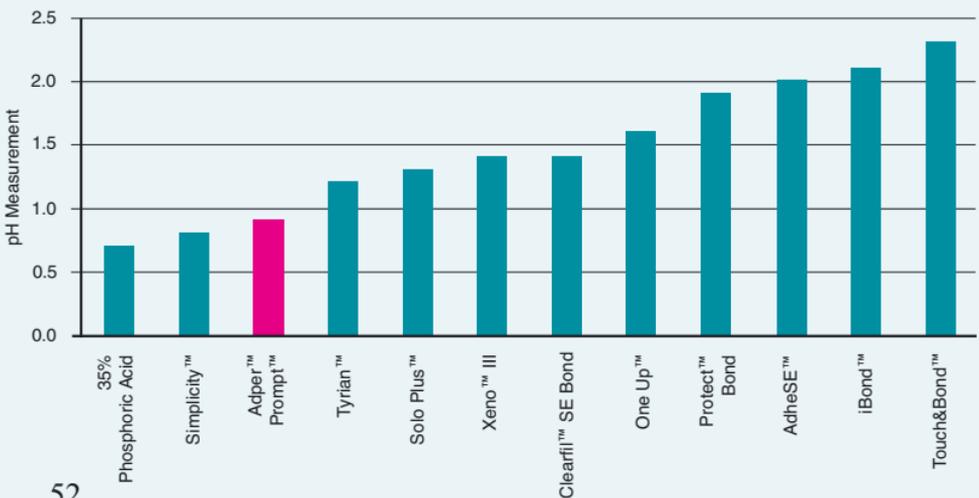
# Adper™ Prompt™ L-Pop™

## 6 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. The low pH of self etching adhesives ensures a good etching pattern on cut and especially 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



# Adper™ Prompt™ L-Pop™

## 6. Bonding Sealants

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

**Authors:** F.R. TAY<sup>1</sup>, S.H.Y. WEI<sup>1</sup>, D.H. PASHLEY<sup>2</sup>, and R.M. CARVALHO<sup>3</sup>,  
<sup>1</sup>The University of Hong Kong, China, <sup>2</sup>Medical College of Georgia, Augusta, USA, <sup>3</sup>University of São Paulo, Bauru, Brazil

**Reference:** IADR 2003, Göteborg, Sweden, #0718

**Objective:** 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.

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

# Adper™ Prompt™ L-Pop™

## 6. Bonding Sealants

### Microleakage of Sealants Bonded with Adper™ Prompt™ Self-Etch Adhesive

**Authors:** Dr. Robert Feigal, Ann Arbor, USA

**Reference:** Unpublished data

**Objective:** 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.

**Methods:** 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.

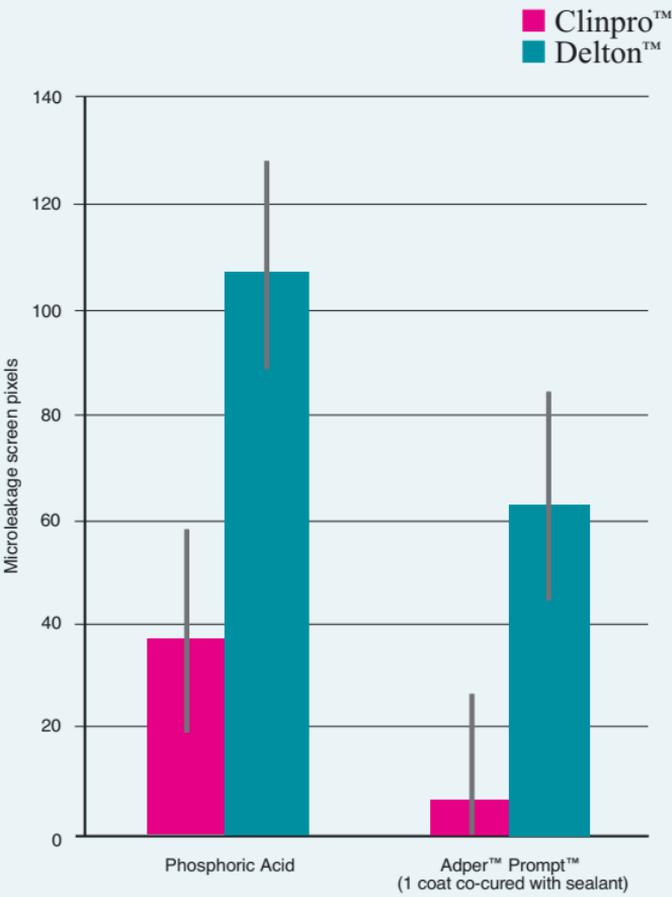
The measurements were made on an Image Pro Plus program as microns of leakage from the external margin on each side of the sealant to the half-way point through the sealant interface. 6 measurements of buccal margin leakage and 6 measurements of lingual surface leakage were averaged for the mean leakage 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.

**Conclusion:** Pretreatment with Adper prompt for the placement of light-cure sealants leads to significantly less microleakage than pretreatment with phosphoric acid.

# Adper™ Prompt™ L-Pop™

## Adper™ Prompt™: Sealant Microleakage



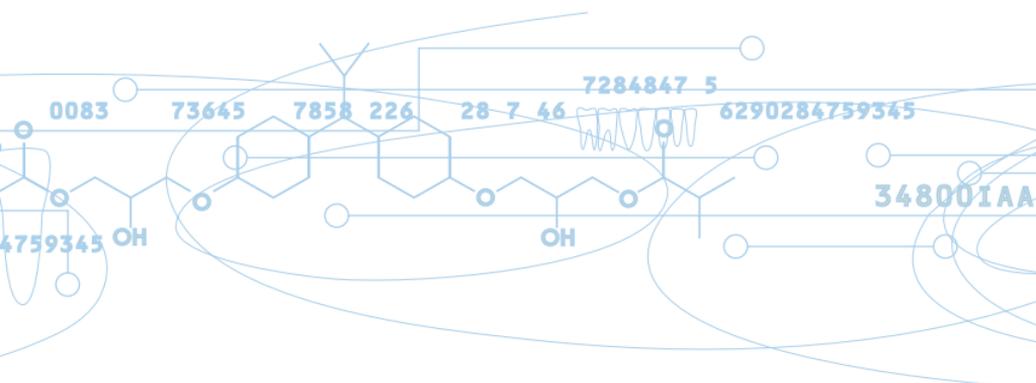
\*Note:

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

6







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