

# Microleakage After Thermocycling of 4 Etch and Rinse and 3 Self-etch Adhesives With and Without a Flowable Composite Lining

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## Clinical Relevance

This study indicates that etch and rinse adhesive systems are less prone to microleakage than self-etch systems.

## SUMMARY

**This study evaluated the microleakage of composite fillings prepared with 4 etch and rinse and 3 self-etch adhesive systems after thermocycling. Also evaluated was the potential improvement of cavity sealing when utilizing a low charged resin lining for cavity preparations. Seventy recently extracted teeth were randomly allocated to 7 experimental adhesive systems: two 3-step etch and rinse adhesive systems, Scotchbond**

**Multipurpose (SBMP) and Optibond Solo Plus (OS); two 2-step etch and rinse adhesive systems, referred to as “one-bottle,” Scotchbond 1 (SB1) and Gluma Comfort Bond + Desensitizer (G); and 3 self-etch “all-in-one” adhesives, Adper Prompt-L-Pop (PLP), Xeno III (X-III) and iBond (iB). On each tooth, 2 rectangular cavities were prepared at the cemento-enamel junction: 1 cavity was prepared with adhesive and the hybrid composite and the second was filled with the same adhesive and a thin layer of flowable composite (Filtek Flow) under the resin composite (Z100). All teeth were thermocycled for 800 cycles (5°C-55°C, 30 seconds dwell time). Leakage was evaluated on a 6-point (0-5) severity scale for enamel and dentin on 4 interfaces for each restoration. The results are expressed as means  $\pm$  standard deviation (SD). Microleakage scores were analyzed by means of generalized linear mixed models (GLMM), assuming an ordinal logistic link function. Covariates in the model were: (1) adhesives, (2) fluid composite and (3) interface. The model also accounts for repeated measurements on the various teeth. The authors found that the mean score of microleakage per tooth was significantly higher at the enamel rather than at the dentin**

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interfaces ( $1.21 \pm 0.51$  and  $0.87 \pm 0.48$ ;  $p < 0.0001$ ). In this study, there was no significant difference among the 4 etch and rinse adhesive systems. On the other hand, these adhesives yielded smaller mean scores of microleakage than the 3 self-etch systems (respectively,  $0.85 \pm 0.2$  and  $1.3 \pm 0.5$ ;  $p < 0.0001$ ). Among the self-etch adhesives, microleakage was significantly greater for PLP ( $1.74 \pm 0.46$ ) than for the other self-etch products ( $p < 0.0001$ ), while X-III, an intermediary strong self-etch, was found to be as good as the etch and rinse systems, with a mean score of  $0.97 \pm 0.27$ . In addition, results have also shown that an under layer of flowable composite significantly improved the water tightness of the PLP adhesive restorations ( $p = 0.042$ ). This *in vitro* study concluded that the self-etch adhesives remain less effective than etch and rinse. Nevertheless, X-III, a self-etch adhesive, showed acceptable performance in accordance with this study's 6-point severity scale of microleakage, but this needs to be confirmed in further clinical studies. On the other hand, this study failed to reveal that the addition of a thin layer of fluid composite improved the water tightness of the restoration, except for PLP.

## INTRODUCTION

Adhesive systems will remain in danger of not totally preventing gap formation and microleakage as long as polymerization contraction and dimensional changes of resin composites are not substantially reduced (Goracci, Mori & de Martinis, 1996; Belli & others, 2001). Different methods are used to improve the cavity sealing of composite restorations, such as soft-curing (Davidson & de Gee, 2000; Knezevic & others, 2001), the application of successive increments or composite stratification to respect the C-factor (Tjan, Bergh & Lidner, 1992; Mulleijans & others, 2003), use of a resin-modified glass-ionomer base in a sandwich-technique (Lutz, Krejci & Oldenburg, 1986; Dietrich & others, 1999) and placement of a thin layer of low charged composite lining. This "elastic layer" may absorb shrinkage stresses; however, this still remains under review. Some studies have already shown that such a lining significantly reduces microleakage at the dentin-restoration interfaces (Belli & others, 2001; Civelek & others, 2003; Ferdianakis, 1998; Swift & others, 1996; Tung Estafan & Sherer, 2000), while Chuang and others (2001) have found that this flowable lining reduced voids in the restored interface of a Class II composite cavity but did not significantly improve marginal sealing.

In a recent paper, Van Meerbeek and others (2003) proposed a new classification of adhesive system. The authors also described 3 strategies of bonding: etch and rinse, self-etch and (resin-modified) Glass Ionomer.

With this in mind, the first objective of this study was to compare 7 adhesive systems that fall into 2 of the main strategies of dental adhesion, etch and rinse and self-etch. This study's second objective was to test the potential effect on microleakage after thermocycling samples with a low charged resin lining.

## METHODS AND MATERIALS

Seventy recently extracted third molars, free of decay, were stored in refrigerated saline solution. On each tooth, 2 rectangular cavities ( $h \times w \times l = 2 \text{ mm} \times 2 \text{ mm} \times 3 \text{ mm}$ ) were prepared at the cemento-enamel junction using a cylindrical diamond drill. The margins of the cavities were butt joint, half in enamel and half in cementum. The teeth were randomly and equally allocated to the 7 groups of the tested adhesive systems: Scotchbond Multipurpose (3M ESPE AG, Dental Products, Seefeld, Germany) (SBMP), Optibond Solo Plus (Kerr, European Union Representative, Scafati [SA], Italy) (OS), Scotchbond 1 (3M ESPE AG) (SB1), Gluma Comfort Bond + Desensitizer (Heraeus Kulzer GmbH & Co KG, Hanau, Germany) (G), Adper Prompt-L-Pop (3M ESPE AG) (PLP), Xeno III (Dentsply Detrey GmbH, Konstanz, Germany) (X-III) and iBond (Heraeus Kulzer GmbH & Co KG) (iB). SBMP and OS are 3-step etch and rinse adhesive systems, SB1 and G are 2-step etch and rinse systems and PLP, X-III and iB are 1-step self-etch or "all-in-one" adhesives.

Each adhesive was used according to the manufacturer's instructions. All restorations were performed with the same microhybrid composite (Z100, 3M ESPE AG). All of the products were polymerized with the same halogen lamp (XL 3000, 3M ESPE AG).

On each tooth, after application of the adhesive system on the 2 cavities, 1 was randomly lined with a thin layer (1 mm) of flowable composite (Filtek Flow, 3M ESPE AG). This lining covered the pulpal and lateral walls of the cavity but was not applied about 1 mm from the cavosurface angles. The flowable composite was polymerized for 20 seconds. Finally, both cavities were filled with microhybrid composite in 2 successive oblique layers, with each polymerized for 40 seconds. The preparations were finished with diamond drills and polished with disks (Hawe Neos Dental, Bioggio, Switzerland).

The apexes were then fixed in an auto polymerizing resin (Paladur, Heraeus-Kulzer GmbH & Co KG), and the specimens were immersed in saline solution for 12 weeks. Thereafter, they were thermocycled ( $5^{\circ}\text{C}$ - $55^{\circ}\text{C}$ ) for 800 cycles in 22 hours. After thermocycling, the teeth were immersed in silver nitrate solution (6 hours) and Vitamin C (15 minutes). After immersion, the samples were prepared with 3 grooves in the restoration. The interfaces between the teeth and the composite filling were described in a previous study (Cao & others, 2003). Briefly, these preparations yield-

ed four evaluating surfaces for each preparation, for a total of 560 viewing surfaces, 80 surfaces for each adhesive system. Each sample allowed 1 measure in enamel and 1 in dentin, for a total of 1,120 measures, 160 for each adhesive system.

Each section was examined using 2 methods: (a) magnifying glass under a direct light source and (b) magnification by means of an optic microscope (Carl Zeiss, SAS, Oberkochen, Germany) (2.5x).

Arbitrarily, the evaluation of leakage was made using a 6-point severity scale (Cao & others, 2003):

Score = 0—no leakage

Score = 1—slight leakage up to the enamel-dentin junction or a depth of 0.5 mm on the radicular wall

Score = 2—moderate leakage, up to maximum half of the lateral wall (leakage depth  $\leq$  1 mm)

Score = 3—high leakage over half of the lateral wall (1 mm < leakage depth < 2 mm)

Score = 4—subtotal leakage on the whole of the lateral wall (leakage depth = 2 mm)

Score = 5—total leakage partly or entirely on the pulpal wall of the cavity (leakage depth > 2 mm)

Taking into account that all adhesives permit some leakage, the authors of this study considered that microleakage scores equal or less than 2 after thermocycling were clinically acceptable and that the higher scores of leakage (scores 3, 4 and 5) were responsible for failure of the bonding.

### Statistical Analysis

The results are expressed as means  $\pm$  standard deviations (SD). Microleakage scores were analyzed by means of generalized linear mixed models (GLMM), assuming an ordinal logistic link function. Covariates in the model were: (1) adhesive, (2) fluid composite and (3) interface. The model also accounts for repeated measurements on the various teeth. All results were considered to be significant at the 5% critical level ( $p < 0.05$ ). Statistical calculations were done utilizing the SAS (version 8.2 for Windows) package.

Table 1: Mean Scores of Microleakage for Each Adhesive System and for All Tested Teeth

Adhesive Strategy Adhesive System Score of Microleakage by Tooth ( $\pm$ SD)	
<b>Etch &amp; Rinse</b>	
SBMP	0.76 $\pm$ 0.24
SB1	0.89 $\pm$ 0.16
G	0.93 $\pm$ 0.12
OS	0.84 $\pm$ 0.25
<b>All tested Etch &amp; Rinse adhesives 0.85 <math>\pm</math> 0.20</b>	
<b>Self-Etch</b>	
X-III	0.97 $\pm$ 0.27
iB	1.2 $\pm$ 0.42
PLP	1.7 $\pm$ 0.46
<b>All tested Self-Etch adhesives 1.3 <math>\pm</math> 0.50</b>	
<i>p</i> value <0.0001*	
*The Etch & Rinse strategy is significantly better ( $p < 0.0001$ ) than the Self-Etch approach for dental adhesion.	

## RESULTS

The mean scores of microleakage for each adhesive are shown in Table 1; SBMP gave the best score of microleakage (0.76  $\pm$  0.24), while PLP yielded the worst score (1.7  $\pm$  0.46). Overall, the etch and rinse systems were significantly better than self-etch (0.85  $\pm$  0.20 vs 1.3  $\pm$  0.50,  $p < 0.0001$ ).

The mean scores of microleakage for each adhesive system used with or without an under layer of flowable composite are displayed in Table 2. The results show that the flowable layer did not significantly improve the microleakage scores, except for PLP ( $p = 0.042$ ) and, to a lesser extent, for G ( $p = 0.055$ ).

When all of the experimental results were grouped together, the authors of this study found that the mean score of microleakage was significantly higher at the enamel interfaces (1.21  $\pm$  0.51) than at the dentin interfaces (0.87  $\pm$  0.48) ( $p < 0.0001$ ). The same observation was made in each group of adhesives, with or without flowable composite (data not shown).

To analyze microleakage ordinal scores with respect to all experimental factors, adhesive, fluid composite and interface, a generalized linear mixed model with an ordinal logistic link function was applied to the data obtained from repeated measurements on each tooth. Table 3 shows the levels of statistical significance between adhesive systems adjusting for the other covariates.

The PLP adhesive was significantly different from all the others adhesive systems ( $p < 0.0001$ ). It has also been noted that iB did not differ from X-III ( $p = 0.11$ ), while significant differences were found when iB was compared to the other adhesives. Also, SBMP exhibited significant differences compared to the other tested adhesives except for OS ( $p = 0.64$ ).



## DISCUSSION

It was empirically postulated that leakage scores from 0 to 2 were clinically acceptable results in the case of a composite restoration, which is a moderate leakage, maximum half of the lateral wall (leakage depth  $\leq 1$  mm). Additionally, silver nitrate penetration was used, which is a harsh test of marginal seal, because the size of the silver ions is smaller than bacteria. This suggests that less leakage could occur *in vivo* rather than *in vitro* (Barnes & others, 1993; Swift, Perdigão & Heymann, 1995).

Surprisingly, a statistical difference was found between the 2 interfaces, with a better score at the dentin interface than at the enamel interface. This difference does not appear clinically important for at least 3 reasons: first, the mean scores of microleakage at dentin and enamel remained within acceptable clinical limits,  $1.21 \pm 0.51$  for enamel and  $0.87 \pm 0.48$  for dentin. Second, the observational techniques used were probably not precise enough to detect smaller micrometric differences (1.21 versus 0.87). Third, it seems that the cavity preparation most likely created a short bevel at the curved enamel interface, which may partly explain the slight difference that was observed between both interfaces.

In this study, 2 strategies of dental adhesion were compared: the etch and rinse (SBMP, SB1, G, OS) and

self-etch (X-III, iB, PLP) techniques, according to a recent classification by Van Meerbeek and others (2003). From a biological viewpoint, mechanisms of adhesion are quite different for all of these products. In the case of the etch and rinse strategy, adhesion is essentially micromechanical, with tag formation on both etched enamel and dentin surfaces. In addition, on etched dentin, adhesion depends on diffusion and infiltration of resin within the exposed collagen fibril scaffold. This technique is well documented, and several authors have found it to be a good approach to bond resin to dental tissues (Davidson & Wolters, 2003; Roulet & Degrange, 2000; Swift & others, 1995). However, new products are commonly focusing on simplifying the clinical procedure of adhesion, with manufacturers proposing many self-etch systems (Roulet & Degrange, 2000). This kind of adhesion reduces technique-sensitivity, because there is no etching, no rinse and no risk of desiccating the dentin surface. Another important advantage of the self-etch approach is that infiltration of resin occurs simultaneously with the self-etching process. This technique is more complex to describe, because there are several approaches to self-etching adhesion, depending on the product's pH: strong self-etch (SSE) with  $\text{pH} \leq 1$ , intermediary strong self-etch (ISSE) with  $\text{pH} = 1.5$  and mild self-etch (MSE) with  $\text{pH} = 2$ .

SSE adhesives, such as PLP, act like an etch and rinse system, but their high acidity appears to dramatically weaken the bonding performance. In addition, products of demineralization are not eliminated and are incorporated into the hybrid layer (Van Meerbeek & others, 2003). Therefore, with SSE, adhesion is only mechanical, such as with etch and rinse adhesives. As an etch and rinse adhesive or SSE approaches dentin, the transition of the exposed collagen fibril network to the underlying unaffected dentin is quite abrupt. For MSE (for example Clearfil Bond SE, Kuraray Dental Europe GmbH, Düsseldorf, Germany), the demineralization of dentin and enamel is less important than with the etch and rinse or SSE approaches: exposed hydroxyapatite crystals remain around collagen in dentin and are exposed in enamel. The exposed hydroxyapatite crystals enable more intimate chemical interactions with the functional monomers and help to prevent or retard marginal leakage (Van Meerbeek & others,

Table 2: Influence of the Flowable Composite Used as a Liner Under the Composite Restoration (measure per tooth  $\pm$  SD)

Adhesive System	Flowable Composite		p value
	Yes	No	
<b>Etch &amp; Rinse</b>			
SBMP	$0.76 \pm 0.54$	$0.76 \pm 0.22$	0.50
SB1	$0.88 \pm 0.29$	$0.92 \pm 0.19$	0.37
G	$0.84 \pm 0.26$	$1.0 \pm 0.23$	0.055
OS	$0.80 \pm 0.18$	$0.88 \pm 0.39$	0.29
<b>Self-Etch</b>			
X-III	$0.85 \pm 0.29$	$1.1 \pm 0.56$	0.13
iB	$1.1 \pm 0.45$	$1.3 \pm 0.65$	0.29
PLP	$2.0 \pm 0.82$	$1.5 \pm 0.29$	<b>0.042</b>

Table 3: Comparison of Microleakage Scores Between Adhesive Systems After Adjusting for Interface and Fluid Composite (p-values)\*

Adhesive Strategy		Etch & Rinse				Self-Etch		
		SBMP	SB1	G	OS	X-III	iB	PLP
<b>Etch &amp; Rinse</b>	SBMP	-						
	SB1	<b>0.016</b>	-					
	G	<b>0.0009</b>	0.63	-				
<b>Self-Etch</b>	OS	0.64	0.14	<b>0.046</b>	-			
	X-III	<b>0.013</b>	0.603	0.85	0.084	-		
	iB	<b>&lt;0.0001</b>	<b>0.019</b>	<b>0.028</b>	<b>0.0008</b>	0.11	-	
	PLP	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	-

\*p-values were obtained by means of a generalized linear mixed model (GLMM) assuming an ordinal link function.

2003). For the ISSE approach (X-III and iB), the deepest region of the hybrid layer still contains hydroxyapatite, and the transition of the hybrid layer to the underlying unaffected dentin is more gradual (Van Meerbeek & others, 2003). Mechanical adhesion is less important than with SSE, but there are possibilities of chemical adhesion.

Actually, it seems that the etch and rinse strategy is still the most effective approach to achieving stable, efficient bonding to dental tissues (Asmussen & Peutzfeldt, 2003; Grégoire & others, 2003; Pradelle-Plasse & others, 2001; Roulet & Degrange, 2000; Van Meerbeek & others, 2003). The results from this study support this fact, showing that the etch and rinse technique remains the best adhesion strategy and that 1-bottle tested adhesives (SB1 and G) show acceptable results. Therefore, they may be routinely used to facilitate the clinical procedure of the etch and rinse strategy. In this study, OS permits maximal leakage in less than 1% of the samples. This lack of performance is probably due to a manipulation error, because OS has shown acceptable scores in 96.3% of the cases. In addition, data from the literature showed that this filled ethanol-based adhesive is more resistant to fatigue forces and provides excellent retention (Swift & others, 2001). When the self-etch adhesive systems were tested, it was found that PLP, a SSE adhesive system, yielded the worst results. On the other hand, X-III, an ISSE adhesive, gave the best results of the self-etch strategy and even approached the same results as the etch and rinse systems. However, the results of another ISSE adhesive, iB, were disappointing.

The influence of a thin layer (1 mm) of flowable composite under the hybrid resin restoration was also tested, because several studies have indicated that this flowable resin is able to absorb shrinkage stresses due to the polymerization of the composite placed over it while also reducing microleakage (Belli & others, 2001; Payne, 1999; Tung & others, 2000). Fluid resin composite contains small particles and many resin components, so it is more elastic than the hybrid composite itself (Bayne & others, 1998). In fact, it is sufficiently elastic that it should be able to resist the contraction stresses of the hybrid composite and diminish the presence of gaps and microleakage. Nevertheless, polymerization shrinkage of the fluid composite is greater than that of the hybrid. For some authors, it should induce far more stresses on the adhesive layer (Bayne & others, 1998). However, for other authors, the adhesive systems should be able to resist these stresses and act on this "elastic layer" (Choi, Condon & Ferracane, 2000; Fortin & others, 1994; Kemp-Scholte & Davidson, 1990; Swift & others, 2001) and probably is really active in this system (Belli & others, 2001). On the other hand, even if the addition of a fluid composite did not significantly improve the hermetic nature of the

composite restoration, the fluid composite is able to infiltrate all the irregularities, then reduce voids in the preparation interfaces (Chuang & others, 2001). In this study, for 6 of the 7 tested adhesives with a thin layer of fluid composite, a statistical improvement of hermeticity was not found. The reasons for this might include the fact that these results were already very good without flowable composite and also because of the greater polymerization shrinkage that may have made the use of a fluid composite ineffectual in improving marginal microleakage. Therefore, these results were unable to determine whether this technique is able to significantly reduce microleakage, except for the PLP adhesive, which seems to normally be less effective than the others. Nevertheless, when regression analysis took into account all the covariables (kind of adhesive, fluid composite, type of interface, repeated measurements on the same tooth), the fluid composite did not improve the performance of PLP ( $p=0.24$ ).

## CONCLUSIONS

Etch and rinse adhesive systems are still the best adhesives, showing minimal leakage with 3-step systems, such as Scotchbond Multipurpose, but also with 1-bottle adhesives, including Optibond Solo Plus, Gluma Comfort Bond + Desensitizer and Scotchbond 1. It is still suggested that self-etch adhesive systems remain less effective than etch and rinse systems. Nevertheless, Xeno III, an intermediary strong self-etch adhesive, shows acceptable results; however, more clinical investigations will be required to confirm this performance. On the other hand, Adper Prompt-L-Pop, a strong self-etch, permitted maximal leakage to the pulpal wall in several cases and, for this reason, it seems that this system may be critical. Finally, it was also found that the addition of a thin layer of fluid composite gives no statistical improvement in the majority of adhesives tested.

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