#### **Operative Dentistry**

# Marginal microleakage of class V resinbased composite restorations bonded with six one-step self-etch systems

**Abstract:** This study compared the microleakage of class V restorations

bonded with various one-step self-etching adhesives. Seventy class V res-

in-based composite restorations were prepared on the buccal and lingual

surfaces of 35 premolars, by using: Clearfil S<sup>3</sup> Bond, G-Bond, *i*Bond, One Coat 7.0, OptiBond All-In-One, or Xeno IV. The Adper Single Bond etch-and-rinse two-step adhesive was employed as a control. Specimens

were thermocycled for 500 cycles in separate water baths at 5°C and

55°C and loaded under 40 to 70 N for 50,000 cycles. Marginal micro-

leakage was measured based on the penetration of a tracer agent. Al-

though the control showed no microleakage at the enamel margins, there

were no differences between groups (p = 0.06). None of the adhesives avoided microleakage at the dentin margins, and they displayed similar

performances (p = 0.76). When both margins were compared, iBond<sup>®</sup>

presented higher microleakage (p < 0.05) at the enamel margins (median,

1.00;  $Q_3-Q_1$ , 1.25-0.00) compared to the dentin margins (median, 0.00;  $Q_3-Q_1$ , 0.25-0.00). The study adhesives showed similar abilities to seal the margins of class V restorations, except for *i*Bond®, which presented

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lower performance at the enamel margin.

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### Introduction

The management of noncarious cervical lesions is an ongoing problem in preventive and restorative dentistry, due to their multifactorial etiology and increased prevalence.¹ The use of direct resin-based composite as a conventional restorative procedure is still critical since this type of lesion contains simultaneously enamel and cementum/dentin contours. Therefore, class V restorations should employ bonding agents that allow effective interactions with different tissues, simple handling, and quick application, due to the relative difficulty in accessing these cavities and the presence of margins adjacent to the moist gingival tissue.²

One-step systems were developed to minimize the number of clinical steps, while incorporating the primer and adhesive into a single bottle. In addition to reducing the clinical time, these systems reduce the sensitivity of the technique and the risk of errors during application.<sup>3</sup> With the one-step system, the infiltration of adhesive monomers occurs simultaneously with the self-etching process, decreasing the possibilities of discrepancies

between the processes and of unprotected collagen fibers.<sup>4</sup> However, the clinical procedures are likely to be simplified at the expense of bonding performance.<sup>3-6</sup>

Due to their hydrophilic nature, one-step self-etching adhesives accumulate water upon solvent evaporation, degrading faster than hydrophobic multistep adhesives.<sup>5</sup> Although strong one-step self-etching adhesives (pH < 1) contain aggressive primers, their effectiveness of adhesion to the dental substrate may be harmed because esters, such as hydroxyethyl methacrylate, TEGDMA, and ure-thane dimethacrylate, are hydrolytically unstable.<sup>6</sup> This effect may be reduced in mild systems (pH ~ 2); however, these adhesives may produce poorly defined etching patterns and very thin, oxygen-inhibited coatings, resulting in inadequately polymerized adhesive layers.<sup>7</sup>

Clinical microleakage is the major cause of failure for composite restorations and may lead to post-operative sensibility, marginal discoloration, secondary caries, or pulpal inflammation.<sup>8</sup> A widely used *in vitro* method for determining the restoration durability involves the aging of class V restorations by thermal stressing.<sup>9</sup> Changes in the elastic capacity of the restoration elements under occlusal loading may lead to damages in the margins.<sup>10</sup> The assessment of tracer molecule microleakage through the adhesive interface is another commonly used method to evaluate the bonding effectiveness.<sup>8-10</sup>

One-step self-etch adhesives initially showed inferior results compared to multistep systems.<sup>3,4</sup> However, since those studies were performed, improvements have been made in the chemical compositions of the systems and in our understanding of application techniques.<sup>11</sup> Because few well-designed studies are available that liken one-step self-etching adhesives with etch-and-rinse control systems,<sup>11,12</sup> the aim of this study was to compare the marginal microleakage of class V resin-based composite restorations bonded with these systems *in vitro*.

## Methodology

Thirty-five sound human premolars were carefully scaled of calculus, soft tissue, and other debris by using a dental curette and pumice/water slurry.

The teeth were stored at room temperature in 0.1% thymol solution to avoid bacterial development. The source of the teeth (no. 17/2007) and the research protocol (no. 06273/06) were approved by the Ethics Committee of the State University of Ponta Grossa.

Seventy class V cavities (3.0 mm mesial-distal, 2.0 mm occlusal-gingival, and 1.8 mm deep) were made on the buccal and lingual surfaces, with the occlusal margin in enamel and the gingival margin in cementum (dentin). The preparations were made with carbide bur #6 (Beavers Dental, Morrisburg, Canada) and diamond bur #3131 (KG Sorensen Ind., São Paulo, Brazil) in a high-speed hand piece cooled with an air-water spray and using a digital caliper (Mitutoyo, Kanagawa, Japan). The burs were discarded after each preparation.

The specimens were randomly divided into seven groups (n = 10) according to the one-step self-etching system used:

- Clearfil S<sup>3</sup> Bond (Kuraray Medical Inc., Okayama, Japan),
- *i*Bond (Haraeus Kulzer, Armonk, USA),
- G-Bond (GC Corp., Tokyo, Japan),
- One Coat 7.0 (Coltène/Whaledent, Altastätten, Switzerland),
- OptiBond All-In-One (Kerr Corp., Orange, USA), and
- Xeno IV (Dentsply/Caulk, Milford, USA).

The Adper Single Bond 2 (3M-ESPE, Saint Paul, USA) etch-and-rinse two-step adhesive system was used as a control (Table 1).

After bonding, the cavities were each filled with three increments of the microhybrid composite resin Synergy D6 (Coltène/Whaledent, Altastätten, Switzerland) and light-activated for 40 s with 600 mW/cm² by a dental lamp Demetron Optilux 400 (Kerr Corp., Orange, USA). The light intensity was verified every five activations. The teeth were stored at 37°C for 24 h, and the restoration surfaces were finished by using diamond PRO discs (Dental MFG, Aurora, USA).

Teeth were submitted to thermal stressing for 500 cycles in separate distilled water baths at 5°C and 55°C, with dwell and transference times of 15 and 5 s, respectively (El Quip, São Carlos, Brazil). The roots of the teeth were embedded in epoxy resin and

**Table 1 -** Characteristics of the adhesive systems.

Adhesive system	Composition	Application mode			
Adper Single Bond 2	<b>Etchant:</b> 35% H <sub>3</sub> PO <sub>4</sub> ; <b>Adhesive:</b> dimethacrylates, HEMA, polyalkenoid acid copolymer, 5 nm silane treated colloidal silica, ethanol, water, photoinitiator; pH 3.3; <b>Batch:</b> 4BC	Etch for 15 s; rinse for 10 s; blot excess water with a cotton pellet (glistening surface w/o pooling of water); apply 2 consecutive coats of adhesive, by brushing gently on the cavity surface; evaporate solvent by gentle air stream for 5 s; light-cure for 10 s			
Clearfil S <sup>3</sup> Bond	MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, photoinitiators, ethanol, water, silanated colloidal silica; pH 2.7; <b>Batch:</b> 41132	Dry; apply 1 layer of adhesive onto surface cavity w/o agitation for 20 s; evaporate solvent by high-pressure air flow for 5 s; light-cure for 10 s			
G-Bond	4-MET, phosphoric ester-monomer, UDMA, TEGDMA, acetone, water, stabilizer, silica filler, water, photoinitiator; pH 2.0; <b>Batch:</b> 0609051	Shake the bottle; dry; apply 1 layer of adhesive onto surface cavity w/o agitation for 10 s; dry thoroughly under maximum air pressure for 5 s; light-cure for 10 s			
iBond	UDMA, 4-MET, glutaraldehyde, acetone, water, photoinitiators, stabilizers; pH 2.1; <b>Batch:</b> 010084	Shake the bottle; dry; apply 1 layer onto surface cavity; agitate slightly for 20 s; evaporate solvent by air flow for 5–10 s; apply multiple times if cavity surface does not appear shiny; light-cure for 20 s			
One Coat 7.0	HEMA, ethanol, water, photoinitiators, stabilizer; pH 2.0–2.8; <b>Batch:</b> 070205-CK	Shake the bottle; dry; apply 1 layer onto surface cavity; massage for 20 s; gently dry for 5 s to evaporate solvent; light-cure for 10 s			
Optibond All-In-One	GPDM, HEMA, bis-GMA, mono- and di-functional methacrylate, water, acetone, ethanol, photoinitiators, silica filler, sodium hexafluorosilicate; pH 2.5–3.0;  Batch: 454454	Dry; apply 2 layers onto surface cavity; scrub each layer for 20 s each; air dry gently with medium force for 5 s to evaporate solvent; light-cure for 10 s			
Xeno IV	PENTA, mono-, di- and tri-methacrylate resins, cetylamine hydrofluoride, acetone, water, photoinitiators; pH ~ 2.1; <b>Batch:</b> 060915	Dry; apply 2 layers onto surface cavity; scrub each layer for 15 s each; evaporate solvent by gentle stream of air pressure until there is no more flow; light-cure for 10 s			

HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; bis-GMA: Bis-phenol A diglycidylmethacrylate; 4-MET: 4-methacryloxyethyltrimellitate; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; GPDM: glycerol phosphate dimethacrylate; PENTA: dipentaerythritol pentaacrylate phosphate.<sup>17-19,21</sup>

submitted to 50,000 mechanical cycles at 2 Hz each. Teeth were loaded perpendicularly on the center of the occlusal surface by using a steel ball tip 5.1 mm diameter (El Quip, São Carlos, Brazil). Each cycle involved a load variation from 40 to 70 N, according to the lower and higher deflections of a spring system.<sup>9</sup>

The entire tooth surface was sealed with two coats of commercial nail varnish, with a 1-mm-wide border being left around the restoration margins. The teeth were immediately immersed in 50% AgNO<sub>3</sub> solution for 2 h in a dark room. After washing, the specimens were placed in a developer solution and exposed to fluorescent light for 6 h (Eastman Kodak Company, Rochester, USA).<sup>9</sup>

The specimens were sectioned longitudinally through the center of the restoration, from the facial to the lingual surface. Two sections were obtained from each restoration. Microleakage at the enamel and dentin margins was analyzed with an optical microscope (Olympus American Ind., Hamburg, Germany) at 40× magnification. The section with the greatest microleakage was selected for analysis. Marginal microleakage was scored as follows:

- 0: absence of penetration of tracer agent through the enamel or dentin margin;
- 1: penetration to one-third of the cavity;
- 2: penetration to two-thirds of the cavity; and
- 3: penetration to more than two-thirds and in the axial wall of the cavity.9

All of the procedures were performed by a single calibrated examiner. Data were explored with the SPSS statistical program (IBM, Armonk, EUA). Analysis of variance on ranks test was used to compare the microleakage scores of each adhesive system for the enamel and dentin margins independently. The Wilcoxon signed rank test was applied to compare the microleakage scores between the enam-

**Table 2 -** Frequency of microleakage scores at the enamel and dentin marains.

	Score			Central tendency measure					
Adhesive system	0	1	2	3	Mean	SD	Median	Q3 – Q1	
Enamel									
Adper Single Bond 2	10	0	0	0	0.00	0.00	0.00	0.00 - 0.00	
Clearfil S³ Bond	9	1	0	0	0.10	0.32	0.00	0.00 - 0.00	
G-Bond	7	3	0	0	0.30	0.48	0.00	1.00 – 0.00	
iBond	4	4	2	0	0.80	0.79	1.00	1.25 – 0.00	
One Coat 7.0	6	4	0	0	0.40	0.52	0.00	1.00 – 0.00	
Optibond All-In-One	6	4	0	0	0.40	0.52	0.00	1.00 – 0.00	
Xeno IV	7	3	0	0	0.30	0.48	0.00	1.00 – 0.00	
Dentin									
Adper Single Bond 2	7	1	2	0	0.50	0.85	0.00	1.25 – 0.00	
Clearfil S <sup>3</sup> Bond	8	2	0	0	0.20	0.42	0.00	0.25 – 0.00	
G-Bond	6	3	1	0	0.50	0.71	0.00	1.00 – 0.00	
iBond	8	2	0	0	0.20	0.42	0.00	0.25 – 0.00	
One Coat 7.0	9	1	0	0	0.10	0.32	0.00	0.00 – 0.00	
Optibond All-In-One	7	3	0	0	0.30	0.48	0.00	1.00 – 0.00	
Xeno IV	7	3	0	0	0.30	0.48	0.00	1.00 – 0.00	

Q3 - Q1: interquartile interval.

el and dentin margins for each adhesive ( $\alpha = 0.05$ ).

### Results

The frequencies of microleakage scores at the enamel and dentin margins for each adhesive are shown in Table 2. Resin-based composite restorations bonded with Adper Single Bond 2 showed absence of microleakage, and the highest microleakage values were displayed by iBond. Nevertheless, no significant difference was found (p = 0.06). All of the systems appeared to be incapable of sealing the restoration and, thus, preventing microleakage through the dentin margins. At the dentin margins, One Coat 7.0 presented the lowest microleakage values, whereas Adper Single Bond 2 and G-Bond showed the highest microleakage values, however no significant difference was also found (p = 0.76). When comparing the enamel and dentin margins for each adhesive system, *i*Bond presented higher microleakage (p < 0.05) for the enamel than for the dentin margins.

### Discussion

The results from this study are consistent with those of Owens *et al.*, <sup>11</sup> who found no difference be-

tween iBond or OptiBond Solo Plus, a two-step etchand-rinse system. Yazici et al.12 also observed similar microleakage at the enamel margins among Single Bond, Adper Easy One, and G-Bond. Geerts et al.8 showed no difference between Single Bond or AdheSE One, and Manuja and Nagpal<sup>13</sup> also found similar performance between OptiBond All-In-One or iBond. In contrast, Vinay and Shivanna<sup>14</sup> found that Clearfil S<sup>3</sup> Bond presented better sealing compared to Single Bond, iBond®, and G-Bond. Deliperi et al.15 found higher microleakage in restorations adhered with iBond compared to those treated with Prime & Bond NT, a two-step etch-and-rinse system. The latter system also presented lower microleakage at the dentin margins than Clearfil S3 Bond.16 Yaman et al.17 found lower microleakage for XP Bond (a twostep etch-and-rinse system) compared to One Coat 7.0. Owens and Johnson<sup>18</sup> found lower performance at the enamel margins and higher sealing at the dentin margins using Xeno IV and Clearfil S3 Bond, respectively, as compared to *i*Bond and G-Bond.

The performance of Clearfil S<sup>3</sup> Bond may be explained by its alcohol solvent. It has a higher vapor pressure and a low dielectric constant, and produc-

es hydrogen bridges with water, resulting in a better evaporation rate.<sup>3,9,19</sup> Other solvents such as acetone promote higher dehydration and collapse of the collagen network at the dentin margin.<sup>18</sup> Water sorption by the osmotic gradient or phase-separation of system components after acetone evaporation may lead to dentin and enamel blisters. This adhesive also includes a proprietary "Molecular Dispersion Technology," which enables the formation of a two-phase, liquid hydrophilic/hydrophobic component homogenous state at the molecular level. It may result in the reduction and/or loss of water droplets at the interface.<sup>14</sup> Furthermore, the methacryloxy decyl phosphoric acid (MDP) monomer can generate decalcification and bond to ionic calcium and hydroxyapatite.<sup>6</sup>

G-Bond decalcifies the dentin surface only slightly and produces almost no exposure of the collagen fibers. 14 Consequently, an extremely thin adhesive interface is formed, explaining the microleakage results. However, in the "nano interaction zone", the 4-MET monomer may react with hydroxyapatite to form an insoluble calcium compound, resulting in a better seal that is less likely to be enzymatically deteriorated. 7 The low flow rate of the adhesive due to the 4-MET component can result in less water uptake when the adhesive is stored in water. 5 Its properties may also be improved by applying a strong, continuous air blast to remove the solvent prior to polymerization. 4

However, the drying procedures associated with iBond have been regarded as labor-intensive and may not be realistic for some practitioners in a clinical setting.14 These procedures include multiple applications on prepared surfaces and relatively long waiting periods.<sup>18</sup> The lower performance of this adhesive at the enamel margin may be explained by the lack of an apparent etching pattern, the minimal exposure of crystallites in irregular areas on the unground enamel, and the islands of superficially dissolved tissue within areas without dissolution displayed on ground enamel after its application.<sup>4</sup> Adhesives containing 4-MET also may show lower enamel-bonding capacities than MDP-containing adhesives. Therefore, enamel pre-etching with phosphoric acid is recommended to improve adhesion.<sup>20</sup>

Limited evidence is available regarding microleakage with the use of the HEMA-based adhesives One Coat 7.0 and Optibond All-In-One. HEMA improves the miscibility of hydrophobic and hydrophilic adhesive components.<sup>5</sup> It can replace the solvent and keep the less-miscible dimethacrylate monomers in solution, whereas the water molecules in a hydrophilic adhesive remain hydrogen-bonded to the HEMA monomers.<sup>21</sup> Nevertheless, void formation by the increment on hydrophilicity and HEMA presence has frequently been discussed. Optibond All-In-One has two solvents, ethanol and acetone, with different vapor pressures, which also might increase the void formation.<sup>19</sup> A higher concentration of solvent would be necessary for HEMA to be omitted.5 The solvent-monomer balance might be disturbed by evaporation or lack thereof; water droplets and any nonevaporated water remaining can also contribute to void formation;<sup>22</sup> a strong air blowing was shown to have a significant effect on the degree of solvent evaporation and removing water droplets.4 The application of two adhesive layers onto the surface cavity could help to improve the results.7

Adper Single Bond 2 is able to wet and impregnate the etched enamel in an efficient manner comparable to those of three-step systems. However, its efficiency on dentin is lower because the adhesive incompletely diffuses under wet-bonding conditions, and a porous collagen network remains. <sup>6,19</sup> Phase-separation also occurs in the interphase region between the hydrophilic primers and hydrophobic resins, resulting in water absorption. This effect may be justified by the incorporation of a high-molecular-weight polyalkenoic acid and the presence of HEMA. <sup>3</sup> Nevertheless, the incorporation of alcohol as a cosolvent may help to explain its performance. <sup>14</sup>

In addition to the compressive/tensile stresses produced during occlusal loading,<sup>10</sup> thermocycling results in contraction/expansion stresses and accelerated chemical degradation.<sup>3</sup> However, the use of an extremely large number of cycles may be unnecessary, because the stress emerges early on the adhesive interface components. Thus, the use of 500 cycles can induce aging.<sup>23</sup> In this study, the use of a dwell time of 15 s was considered clinically relevant, because a vital tooth will not tolerate extreme temperatures for extended periods.<sup>24</sup> The viscosity, surface tension, functional monomers, pH, water con-

centration, and cohesive strength of adhesives may affect the bonding and explain the microleakage of the adhesives studied.<sup>6,13</sup> The absence of these variables can be considered as study limitations. Other features, such as the type of composite, cavity, and dye tracer, analyzed tooth section, and number of sample, may also have influenced the results.<sup>3</sup>

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### **Conclusions**

No significant differences between the adhesive systems were encountered at the enamel and dentin margins. Comparing all surfaces, *i*Bond showed significantly greater microleakage values than the other adhesives at the enamel margins.

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