

# Bond Strength of One-Step Adhesives under Different Substrate Moisture Conditions

André Luís Faria-e-Silva<sup>a</sup>  
Mayra Melo Fabião<sup>b</sup>  
Ravana Angelini Sfalcin<sup>b</sup>  
Murilo de Souza Meneses<sup>c</sup>  
Paulo César Freitas Santos-Filho<sup>c</sup>  
Paulo Vinícius Soares<sup>c</sup>  
Luís Roberto Martins<sup>d</sup>

## ABSTRACT

**Objectives:** The aim of this study was to evaluate the bond strength of one-step adhesive systems to dry or moist dental substrate.

**Methods:** Thirty human third molars were sectioned into two halves, in the mesio-distal direction, parallel to the long axis of the tooth. Each half was embedded in a polystyrene resin cylinder so that the buccal/lingual surface remained exposed. This exposed surface was abraded to obtain both flat exposed enamel and dentin. The samples were randomly allocated according to the adhesive system (Xeno III, Adper Prompt and iBond) and moisture condition (dry and moist). The substrates were air-dried for 30 s for dry condition, while the moist substrates were re-wet with 2.5 µl of distilled water after drying. After the adhesive procedures, two resin composite cylinders were build-up on dentin and enamel substrates, totaling four per sample. A shear load was applied to the samples at a crosshead speed of 0.5 mm/min until failure. Data were statistically analyzed by three-way ANOVA and the Tukey test ( $\alpha=0.05$ ).

**Results:** The evaluated one-step adhesives showed higher bond strength to dentin than enamel. The iBond presented better bond performance to moist substrate and Xeno III to dry substrate. The moisture condition did not interfere in the performance of Adper Prompt. The Xeno III and iBond presented higher bond strength than the other adhesives to both dry and moist substrates.

**Conclusions:** The moisture condition of substrate interfered in the performance of one-step self-etching adhesives and the best moisture condition was material dependent. (Eur J Dent 2009;3:290-296)

**Key words:** Bond strength; One-step adhesives; Moisture; Self-etching adhesives.

<sup>a</sup> Professor of Dentistry Department, State University of Montes Claros, Brazil.  
<sup>b</sup> Graduate student of Restorative Dentistry Department, State University of Campinas, Brazil.  
<sup>c</sup> Professor of Restorative Dentistry Department, Federal University of Uberlândia, Brazil.

<sup>d</sup> Professor of Restorative Dentistry Department, State University of Campinas, Brazil.

■ Corresponding author: André Luís Faria-e-Silva  
Dentistry Department, State University of Montes Claros, Brazil.  
E-mail: andrelsilva@hotmail.com

## INTRODUCTION

The fundamental principle of bonding to dental hard tissues is based on micromechanical interlocking of the adhesive resin with dentin/enamel surfaces.<sup>1,2</sup> While bonding to enamel depends on the micromechanical retention to the etched substrate, bonding to dentin relies on hybridization with the exposed collagen mesh.<sup>3</sup> The current dental adhesive systems rely on different strategies for bonding to the dental substrates. In etch-and-rinse systems, a conditioner (usually 30-40% phosphoric acid) selectively dissolves the hydroxyapatite crystals and creates spaces for infiltration;<sup>3,4</sup> the acid gel needs to be removed and the dentin kept moist for adhesion.<sup>5,6</sup> However, managing the proper moisture condition is a critical procedure. In order to reduce the technique-sensitivity and simplify the bonding procedures, self-etching adhesive were introduced.<sup>4,7,8</sup>

The concept of self-etching is based on the use of a non-rinse acidic primer that simultaneously etch and penetrate the dental substrates,<sup>9</sup> presenting either one or two application steps. In two-step systems, the priming step is followed by application of free-solvent adhesive resin.<sup>8</sup> Although two-step systems generally present adequate bonding performance, new single-step systems are constantly introduced into the market. These systems are mixtures of acidic resin monomers, additives, solvents and water.<sup>9,10</sup> Water is required to enable dissociation of the acidic monomers responsible for etching.<sup>9</sup> However, excess water might interfere with the polymerization of the adhesives.<sup>11</sup> Thus, solvents, such as ethanol and acetone are added to accelerate water elimination.<sup>9</sup>

Most manufacturers recommend that self-etching adhesive should be applied to dry substrate. Therefore, the water contained in the adhesive needs to be sufficient to ionize the acid monomers without, however, compromising the polymerization process.<sup>9</sup> The first generation single-step self-etching adhesives were presented in two bottles that needed to be mixed before use. This mode of presentation permits water and acidic monomers to be in different bottles, hence the ionization process does not occur before the components of the two bottles were mixed, allowing the addition of a high water content. However, some recently introduced one-step self-etching

systems present only one bottle in order to simplify the procedure by eliminating the mixing of solutions. These systems probably present lower water content and therefore might need water from the substrate for proper ionization.

Despite the adequate performance on dentin, one-step self-etching adhesives present limitation in properly bonding to enamel, probably due to the relatively high pH compared with phosphoric acid.<sup>1,4</sup> Conventional bonding tests usually do not allow the evaluation of dentin and enamel substrates in the same specimen. Shear bond strength tests with reduced bonding area have been widely used because this reduction allows regional mapping and preparing multiple specimens from the same tooth.<sup>12</sup> The experimental setup of this methodology allows both substrates to be evaluated in the same specimen. In addition, the orientation of enamel prisms and dentin tubules obtained with this type of preparation are close to the orientation presented in the lateral walls of tooth cavities in clinical situation.<sup>13</sup>

The aim of this study was to evaluate the bond strength of three one-step adhesive systems to dry or moist dentin and enamel. The null hypotheses tested were: (1) the moisture condition of substrate would not affect the bond strengths; and (2) no significant differences in bond strength between enamel and dentin would be detected.

## MATERIALS AND METHODS

Thirty human third molars stored in 0.05% thymol saline solution for no more than 3 months were used. In order to obtain two halves, the teeth were sectioned to the mesio-distal axis, parallel to the long axis of tooth, using a slow speed diamond saw under water cooling. Each half was embedded in polystyrene resin to facilitate handling, keeping the buccal/lingual surfaces exposed. The surfaces were wet-ground with 320-grit SiC papers (Carburundum, Vinhedo, SP, Brazil) until a flat surface on both enamel and dentin was obtained, with sufficient area to build up resin composite cylinders (1 mm in diameter), as shown in Figure 1. The specimens were rinsed with water and debris were ultrasonically cleansed for 20 min. The dental surfaces were wet-polished with 600-grit SiC papers to standardized the smear layer. Thereafter, the bonding procedures were immediately performed.

The specimens were randomly allocated according to adhesive procedure. The adhesive systems used in this study, with their batch numbers, manufacturers and compositions are listed in Table 1. Each one-step self-etching adhesive was used with two moisture conditions. The substrate surface was extensively air-dried for 30 s with oil-free compressed air. The surface was either kept dry (no water rinsing) or left wet after rinsing it for 10 s with 2.5 µl of distilled water using a pipette (Micropipet, Pipetman, Gilson, NY, USA). For application of Adper Prompt, one drop of each bottles A and B were mixed, applied to dental surface and agitated for 15 s. An additional layer was applied, gentle air-dries and light polymerized for 10 s. For iBond samples, three consecutive coats were applied, left undisturbed for 30 s and then

gently air-dried. This adhesive was light polymerized for 20 s. For application of Xeno III, equal amounts of bottles A and B were mixed, applied to tooth substrates and left undisturbed for at least 20 s. After brief air-drying, the adhesive was light polymerized for 10 s.

After the adhesive procedures, polyvinyl siloxane (Aquasil, Dentsply DeTrey, Konstanz, Germany) molds with a cylinder-shaped orifice (1 mm in inner diameter × 2 mm in height) were individually placed onto the dentin and enamel surfaces. The composite resin Filtek Z-350 (3M ESPE, St. Paul, MN, USA) was used to fill the orifices. The resin composite was light polymerized for 20 s. Light polymerization procedures were performed using the quartz-tungsten-halogen curing unit Optilux 501 (Demetron Kerr, Orange, CA, USA) with approximately 650 mW/cm<sup>2</sup> irradiance. Afterwards, the molds were removed to expose the resin cylinders. Two cylinders were made on both dentin and enamel substrates for each tooth (n = 4 per specimen), as shown in Figure 2.

The embedded specimens were attached to the testing device and each resin composite cylinder was tested on a mechanical testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil). A thin steel wire (0.2 mm in diameter) was looped around each cylinder and a shear load was applied to the base of the cylinder at a crosshead speed of 0.5 mm/min until failure (Figure 2). Shear bond

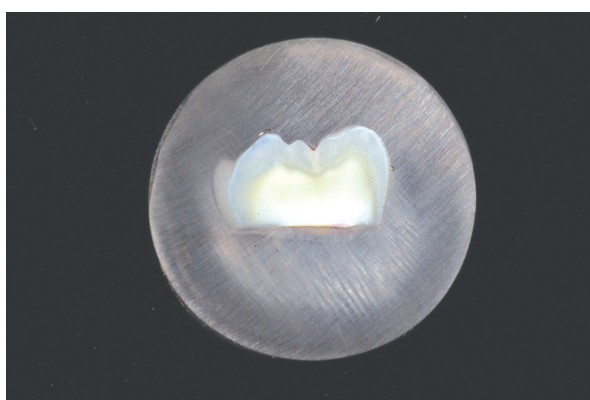


Figure 1. Abraded hemi-section of a human molar, exposing flat dentin and enamel surfaces.

Table 1. Adhesives systems: manufacturer, batch number and composition.

Adhesive system	Manufacturer	Batch number	Composition
Adper Prompt	3M ESPE, St Paul, MN, USA	Liquid A: 238002	Liquid A: Methacrylic phosphates, Bis-GMA, photo-initiator
		Liquid B: 242929	Liquid B: Water, HEMA, polyalkenoic acid polymer, stabilizers
iBond	Heraeus Kulzer, Hanau, Germany	10086	UDMA, 4-META, glutaraldehyde, acetone, water, photo-initiators, stabilizers
Xeno III	Dentsply De Trey, Konstanz, Germany	Liquid A: 0702000544	Bottle A: HEMA, ethanol, water, aerosil, stabilizers (BHT)
		Liquid B: 0702000545	Bottle B: Pyro-EMA, PEM-F, UDMA, CQ, BHT, ethyl-4-dimethylaminobenzoate (co-initiator)

\* Information provided by the manufacturers.

Bis-GMA: bisphenol-A glycidyl dimethacrylate; HEMA: hydroxyethyl methacrylate; UDMA: urethane dimethacrylate; CQ: camphorquinone; 4-META: 4-methacryloyloxyethyl trimellitate anhydride; BHT: butylhydroxytoluene; PEM-F: pentamethacryloyloxyethylcyclohexaphosphazene monofluoride; Pyro-EMA: tetramethacryloyloxyethyl pyrophosphate.

strengths were calculated and expressed in MPa. The average value of the two bonded cylinders for each substrate in the same specimens was recorded as the shear bond strength for that specimen. Data were submitted to three-way ANOVA (dental substrate × moisture condition × adhesive system) and the Tukey test at a 95% confidence level. The fractured specimens were mounted on aluminum stubs, coated with gold (SCD 050, Baltec, Vaduz, Liechtenstein) and evaluated by scanning electron microscopy (JSM-5600LV, JEOL, Tokyo, Japan). The failure modes were classified as follows: Type 1— adhesive failure between bonding agent and substrate; Type 2— cohesive failure within the substrate; Type 3 - cohesive failure within the composite resin; Type 4 - cohesive failure within the adhesive; Type 5— mixed failure.

**RESULTS**

The statistical analysis showed the factors “substrate” (P<.01) and “adhesive system” (P<.05) were significant, as was the interaction between “adhesive system” and “moisture condition” (P<.01). The results of the Tukey’s test for the interaction and substrate are shown in Tables 1 and 2, respectively. In the moist substrate, iBond presented higher shear bond strength than the other adhesives. The opposite was observed in the dry

substrate. There was no difference between the moisture conditions for Adper Prompt. iBond presented higher values in the moist condition, whereas Xeno III showed a better performance on the dry substrate. The evaluated one-step self-etching adhesives showed higher shear bond strength to dentin than to enamel.

The failure modes of the tested samples to enamel and dentin are shown in Figures 3 and 4, respectively. In enamel, there was a predominance of mixed and adhesive failures. Adper Prompt presented some cohesive failures within the adhesive, mainly under the dry substrate condition. For dentin, the cohesive failures within the adhesive were predominant, except for Xeno III applied to moist dentin.

**DISCUSSION**

Irrespective of the moisture condition or adhesive system used, the shear bond strength to enamel was lower than to dentin. Enamel has a higher mineral content than dentin, and it requires a solution with lower pH to etch it.<sup>4,14,15,16</sup> Despite the relatively low pH of the one-step self-etching adhesives used in this study, they may have etched the enamel in a less effective manner compared with dentin. This may have resulted in incomplete penetration of the adhesive resin and creation of a more heterogeneous inter-diffusion zone, reducing in bond strengths. Another hypothesis is that the re-precipitation of calcium phosphates may occur into partially desmineralized interfibrillar spaces, impairing the adhesive penetration.<sup>17</sup> Similar results of better performance of one-step self-etching adhesives bonded to dentin than enamel are reported by Burrow et al<sup>18</sup> using micro-shear testing.

Regarding the moisture conditions of substrates, only iBond presented higher bond strength values when applied on the moist substrates. This



Figure 2. Specimen tested under shear loading.

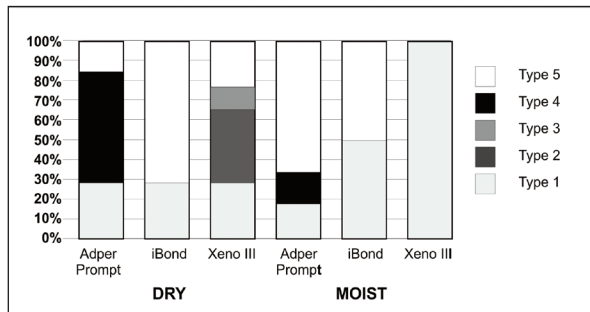


Figure 3. Failure mode of tested specimens in enamel.

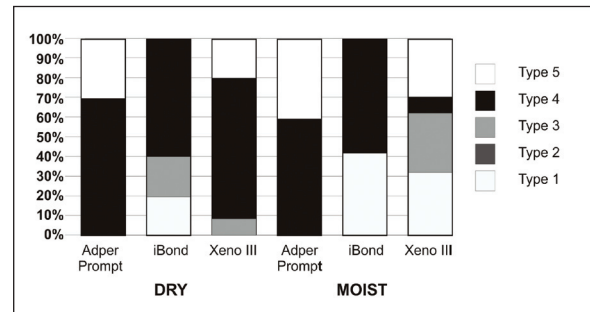


Figure 4. Failure mode of tested specimens in dentin.

result may indicate that this adhesive needs more water to effectively dissociate the acidic monomers and etch the substrate. iBond is presented in a single bottle, which means that the acidic monomers and the solvents (including water) are together in the same solution. This presentation does not allow a high water fraction to be mixed in the solution. Thus, a moist substrate is necessary for a better performance of this system. Furthermore, acetone is used as a co-solvent to aid in eliminating the excess water.<sup>9,19</sup> The presence of acetone may allow the use of iBond on moist substrates, without compromising its polymerization and performance. However, about 60% of the failures occurred cohesively within the adhesive on dentin. The higher intrinsic water content of dentin may increase the water fraction and compromise the effective polymerization of the adhesive.<sup>20</sup>

On the other hand, Xeno III presented the highest values on the dry substrate. This adhesive is presented in two bottles, which are mixed before use. This mode of presentation permits water and the acidic monomers to be in different bottles. Thus, the ionization process does occur before the components of the two bottles are mixed, allowing the addition of a high water content. The possibility of having more water allows this adhesive to efficiently etch both enamel and dentin. Despite presenting ethanol as co-solvent to accelerate water elimination, a higher amount of water, can interfere with the adhesive polymerization and reduce the bond strength.<sup>20-22</sup> In dentin, this effect

was more pronounced and there were cohesive failures within the adhesive, mainly under the dry dentin condition. Again, this probably occurred because of the higher intrinsic water content of this substrate. However, these failures did not occur in the moist substrates. The excess of water may also compromise the diffusion of adhesive into the etched substrate.<sup>23,24</sup> Thus, more mixed and adhesive failures were detected.

Adper Prompt presented similar bond strength under moist and dry conditions. This system, like Xeno III, is presented in two bottles; which allows a high amount of water in its composition. The water content of Adper Prompt seems to be more balanced, and the adhesive presented similar values under both moisture conditions. However, the bond strength values of Adper Prompt were similar to the lowest values obtained with Xeno III (moist) and iBond (dry). Furthermore, it was observed that this adhesive presented more cohesive failures within the adhesive than the others. This can be explained by the absence of a co-solvent, which makes it difficult for the excess water to be eliminated and compromises the performance of this adhesive.<sup>9,25</sup>

The self-etching adhesives were developed in an endeavor to reduce the technical sensitivity of etch-and-rinse adhesive systems. The main limitation of the latter is determining the optimal moisture condition for adhesive application after rinsing dentin to eliminate the phosphoric acid gel.<sup>25</sup> The present study demonstrated that

**Table 2.** Shear bond strength means (SD) in MPA.

Adhesive systems	Moisture conditions	
	Moist	Dry
Adper Pompt	10.86 (2.46) Ab	11.63 (2.43) Ab
iBond	15.73 (5.48) Aa	10.18 (3.62) Bb
Xeno III	10.07 (4.37) Bb	18.73 (6.64) Aa

Means followed by different letters (upper case – row, lower case – column) differ among them by the Tukey test at the 95% confidence level.

**Table 3.** Means followed by different letters (upper case – row, lower case – column) differ among them by the Tukey test at the 95% confidence level.

Substrate	Means (DP)
Dentin	15.19 (6.06) A
Enamel	10.54 (3.47) B

Means followed by different letters differ among them by Tukey's test at the 95% confidence level.

that the moisture condition is also essential for the proper performance of some one-step self-etching adhesives. However, the optimal moisture condition seems to be material dependent and should be investigated in further studies.

## CONCLUSIONS

Based on the results of the present study, it can be concluded that the:

- Xeno III and iBond presented the highest bond strength on dry and moist substrates, respectively.
- The moisture condition did not interfere in the performance of Adper Prompt.
- The one-step adhesives showed higher bond strength to dentin than to enamel.

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□ Bond strength of one-step adhesives

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