

## Adhesive Bond Strengths Using Self- and Light-Cured Composites

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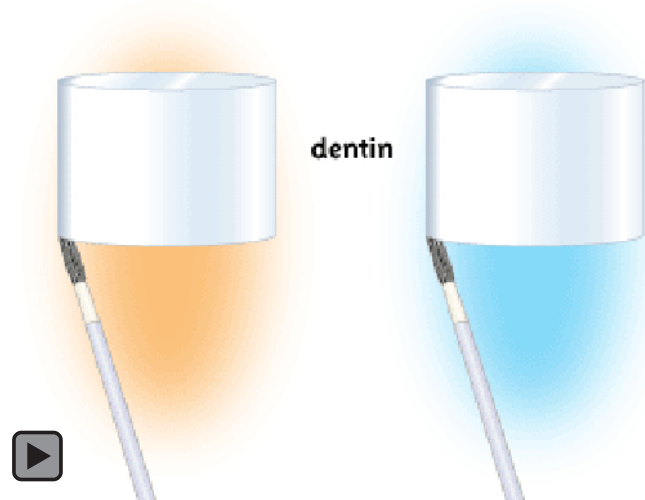
### Abstract

**Aim:** To evaluate the microtensile bond strength ( $\mu$ TBS) of four adhesive systems to dentin, using self- and light-cured resin composites.

**Methods and Materials:** Crowns of human molars were separated from the roots, and the occlusal surfaces were ground to obtain flat superficial dentin. Three etch-and-rinse adhesives—All-Bond 2, One-Step Plus, and OptiBond Solo Plus—and one self-etching primer system, Peak SE, were evaluated. Each adhesive group was divided into two subgroups according to the type of resin composite used. A self-cured (Bisfil 2B) or light-cured (Filtek Supreme Plus) resin composite build-up was incrementally inserted to the dentin after each adhesive system was applied. The bonded specimens were stored in water for 24 h and sectioned into beams. Microtensile testing was done, and the data were subjected to ANOVA and Fisher's PLSD test.

**Results:** The  $\mu$ TBS of All-Bond 2 and One-Step Plus was not affected by the type of resin composite used ( $p=0.3131$  and  $p=0.1562$ , respectively). The  $\mu$ TBS of OptiBond Solo Plus was significantly reduced when used with self-cured resin composite ( $p<0.0001$ ). Peak SE formed no bond of self-cured resin composite to dentin.

**Conclusions:** Some adhesives do not effectively bond self-cured resin composite to dentin.



**Clinical Significance:** Incompatibility between adhesives with low pH and certain self-cured resin composites can cause clinical debonding of restorations.

**Keywords:** Laboratory research, microtensile bond strength, self-cured, light-cured, resin composite

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## Introduction

Several dentin bonding agents have been developed and used with varying degrees of success since the introduction of the total-etching technique by Fusayama three decades ago.<sup>1</sup> From the reliable three-step etch-and-rinse systems to the largely unproven “all-in-one” systems, dentin bonding agents have proven to work well in many clinical situations but have been shown to be less effective in others. One area of concern is the compatibility between some dentin bonding agents and self- and dual-cured resin composites.<sup>2-11</sup>

Three-step etch-and-rinse adhesive systems were introduced approximately 15 years ago and have been shown to be clinically reliable.<sup>12-14</sup> However, their high bond strengths and successful clinical performance record often are overlooked in the quest for simpler materials. The potential for saving time by simplifying the clinical technique appears to be the primary reason for the excellent acceptance of simplified systems among clinicians. These have replaced their three-step predecessors in the majority of clinical applications.

The first simplified dentin bonding agents developed were the etch-and-rinse one-bottle systems.<sup>15</sup> Those materials combine primer and adhesive resin in a single solution, with separate etching. For many of these systems, the dentin must be left “moist” after rinsing away the etchant.<sup>16</sup> The more recently developed self-etching systems have overcome this problem as no separate etching step is required. For this reason, self-etching systems seem to be less technique sensitive when compared to the previous generation of these materials.<sup>17,18</sup>

While both etch-and-rinse one-bottle and self-etching systems can be effectively used with light-cured resin composites, some have shown incompatibility with self- and dual-cured materials. This limitation seems to relate to the high hydrophilicity of some of the adhesives and to an incompatibility between adhesive system and resin composite. Uncured acidic resin monomers in the oxygen-inhibited layer of simplified adhesive systems interact with the tertiary amines involved in the polymerization of self- and dual-cured resin composites, preventing a proper polymerization of the resin composite in contact

with the adhesive. There is evidence of a direct correlation between the pH of adhesive systems and mean bond strengths between adhesives and resin composites.<sup>2-11</sup>

The present study was designed to compare the microtensile bond strength ( $\mu$ TBS) of a three-step etch-and-rinse adhesive, two etch-and-rinse one-bottle adhesives, and one self-etching primer system to dentin using both a light-cured and self-cured resin composite material. The adhesives tested have pH values ranging from < 1.0 to 7.<sup>19,20</sup> The hypothesis tested was that the pH of an adhesive has an effect on the compatibility between the adhesive and resin composite, i.e., adhesives with low pH are incompatible with the self-cured resin composite.

## Methods and Materials

Twenty-four extracted human molars were used in this study. All teeth were debrided and examined to ensure that they were free of defects. They were disinfected in 0.5 % chloramine solution for one week, and then stored in distilled water until used in the study.

Crowns were separated from the roots using an Isomet (Buehler Ltd., Lake Bluff, IL, USA) diamond saw under running water. The occlusal surfaces of the crowns were ground mechanically (Isomet 1000, Buehler Ltd.) under running water with 600-grit silicon carbide paper to obtain a flat dentin surface. Specimens were randomly assigned to eight groups of three teeth each to be treated with All-Bond 2 (three-step etch-and-rinse, control), One-Step Plus or OptiBond Solo Plus (etch-and-rinse one-bottle adhesives), or Peak SE (self-etching system). Each tooth rendered nine beams, for a total of 27 beams per subgroup. Each adhesive was used to bond both Filtek Supreme Plus (light-cured resin composite) and Bisfil 2B (self-cured resin composite) to dentin. See Table 1 for detailed information about the materials.

Bonding procedures were performed as per manufacturers' instructions. For all etch-and-rinse groups, the dentin was conditioned with Ultra-Etch (35% phosphoric acid, Ultradent) for 15 s and a moist technique for bonding was used by blotting the dentin with a KimWipe (Kimberly-Clark Corporation, Irving, TX) laboratory tissue.

**Table 1. Materials manufacturers, main components, and batch numbers.**

Material	Manufacturer	Composition	pH	Batch #
All-Bond 2	Bisco, Inc. Schaumburg, IL	Primer A: Acetone, ethanol, NTG-GMA Primer B: Acetone, ethanol, BPDM D/E Resin: Bis-GMA, UDMA, HEMA	7.0	0600009055 0600009058 0600009206
One-Step Plus	Bisco, Inc. Schaumburg, IL	BPDM, Bis-GMA, HEMA, acetone, fluoroaluminosilicate glass filler	4.6	0600011603
OptiBond Solo Plus	Kerr Corporation Orange, CA	Bis-GMA, HEMA, GDMA, GPDM, ethanol, filler (fumed SiO <sub>2</sub> , barium aluminoborosilicate, Na <sub>2</sub> SiF <sub>6</sub> )	2.8	2722122
Peak Self-Etch	Ultradent Products, Inc. South Jordan, UT	Proprietary, not available from manufacturer	<1.0	PQSE34 PQT07
Filtek Supreme Plus (A2B)	3M ESPE St. Paul, MN	Silanated silica and zirconia, Bis-EMA6, UDMA, Bis-GMA, TEGDMA		20070201/200703 12
<p><b>Monomer Abbreviations:</b> Bis-EMA: Ethoxylated bisphenol A glycol dimethacrylate; Bis-EMA6: Bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol A diglycidyl methacrylate; BPDM: Biphenyl dimethacrylate; GDMA: glycerol dimethacrylate; GPDM: glycerol phosphate dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; NTG-GMA: N-tolyglycine glycidyl methacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate.</p> <p>Source: MSDS of materials posted online on manufacturers' websites and Van Landuyt KL</p>				

For the All-Bond 2 groups, Primers A and B were mixed and five coats were applied with a disposable brush. The primer was dried for at least 5 s to drive off residual water and excess solvent. A thin layer of D/E Resin was brushed on and light-cured for 20 s.

In the One-Step Plus groups, two generous coats of the adhesive were applied and gently agitated for 10–15 s. Excess solvent was removed with compressed air for 5 s, and the adhesive was light-cured for 10 s.

Specimens in the OptiBond Solo Plus groups were treated by applying the adhesive with a brush-tip applicator with a light brushing motion for 15 s. The adhesive was gently air-thinned for 3 s. The adhesive was light-cured for 20 s.

Peak SE was applied to dry dentin for 10 s. After drying the primer for 3 s, Peak LC Bond Resin was applied using a brush-tip applicator for 10 s.

The adhesive layer was air-thinned for 10 s and light-cured for 20 s.

After completion of the bonding procedure, a resin composite buildup of either a light-cured resin composite—Filtek Supreme Plus—or a self-cured resin composite—Bisfil 2B—was made. Filtek Supreme Plus was placed in two 2-mm increments, with each light-cured for 40 s. The self-cured Bisfil 2B resin composite was applied to the treated dentin and left undisturbed for ~ 5 min until curing. All visible light-curing procedures were accomplished using an Optilux 501 unit (Kerr Demetron) at a minimum intensity of 500 mW/cm<sup>2</sup>.

Following polymerization, specimens were stored in distilled water for 24 h at 37°C. Specimens were sectioned into ~ 1-mm<sup>2</sup> beams, according to the “non-trimming” technique proposed by Shono et al.<sup>21</sup> and tested for  $\mu$ TBS with a tabletop tester (EZTest, Shimadzu Corporation, Tokyo, Japan) using a Ciucchi jig at a crosshead speed of 1

**Table 2. Twenty-four-hour mean microtensile bond strengths (MPa + SD) of adhesives to dentin.**

	Filtek Supreme Plus	Bisfil 2B
All Bond 2 <sup>®</sup>	54.7 ± 21.3 AB	60.8 ± 23.3 Aa
One-Step Plus <sup>®</sup>	54.6 ± 26.0 AB	65.1 ± 27.8 Aa
OptiBond Solo Plus <sup>®</sup>	68.2 ± 21.0 Aa	12.9 ± 11.6 Bb
Peak SE <sup>®</sup>	53.2 ± 26.5 b	0.0 ± 0.0*

Buildups were done with Filtek Supreme Plus (light-cured) or Bisfil 2B (self-cured). Uppercase different superscript letters indicate significantly different means within rows. Lowercase different superscript letters indicate significantly different means within columns. (n = 27.)

\* This group was not included in the statistical analysis given the results obtained.

mm/min.<sup>22</sup> The  $\mu$ TBS values were expressed in MPa, which were calculated by dividing the peak break by the cross-sectional area of the bonded interface.

The data were analyzed by two-way ANOVA, with adhesive systems and type of resin composite as the two main factors. For each combination of adhesive and type of resin composite, one-way ANOVA was applied. Where appropriate, Fisher's PLSD test was used (Analyse-It, Analyse-It Software, Ltd., Leeds, England) at the 95% confidence level.

## Results

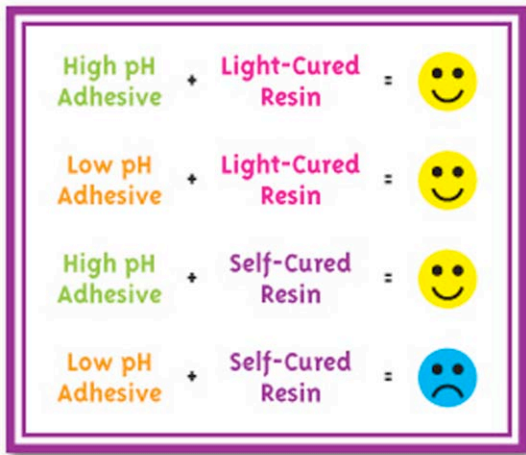
Bond strength data are summarized in Table 2.

The mean  $\mu$ TBS for the All-Bond 2 and One-Step Plus groups were similar for light-cured and self-cured resin composite ( $p=0.3131$  and  $p=0.1562$ , respectively). However, the use of self-cured resin composite significantly reduced the  $\mu$ TBS of OptiBond Solo Plus ( $p<0.0001$ ) and Peak SE. In fact, Peak SE could not be tested with Bisfil 2B due to debonding during specimen preparation. While OptiBond Solo Plus has the highest mean  $\mu$ TBS when tested with Filtek Supreme Plus, it had a significantly lower mean  $\mu$ TBS than All-Bond 2 and One-Step Plus when restored with Bisfil 2B ( $p<0.0001$ ). The results of the two-way ANOVA analysis with pooled data showed that the effects of both adhesive system and resin composite were significant in the  $\mu$ TBS results.

## Discussion

Bonding of the two materials with the lowest pH values (Peak SE and OptiBond Solo Plus) was compromised by the use of self-cured resin composite. In contrast, bonding of the two adhesive systems with the highest pH values was not affected by the type of resin composite used. Therefore, the hypothesis that the pH of the adhesive systems has an effect on the compatibility between adhesive and resin composite was accepted.

Incompatibilities between simplified adhesive systems and self- and dual-cured resin composites have been described in the literature several times during the last decade. Simplified adhesive systems are highly hydrophilic, allowing moisture to diffuse across their cured structure. This moisture is drawn from the dentin by osmotic gradient generated by prolonged contact of the uncured resin composite with the adhesive. This event has been shown to interfere with the interface between adhesive and resin composite, as has the inherent acidity of simplified adhesive systems. The latter has been called a true incompatibility whereby uncured acidic monomers present in the oxygen-inhibited layer of the adhesive interact and consume initiators within the resin composite. This interaction is believed to prevent the tertiary amine co-initiator from generating free radicals required for adequate polymerization of the resin composite layer in contact with the adhesive.<sup>2-11</sup>



Of the adhesives used, All-Bond 2 and One-Step Plus were the only materials to properly couple with Bisfil 2B. While the former is a three-step etch-and-rinse adhesive, the latter is an etch-and-rinse one-bottle adhesive. For All-Bond 2, the adhesive resin layer applied as the last step is relatively hydrophobic, has a neutral pH, and should neither interfere with resin polymerization nor permit fluid flow. Other studies have reported  $\mu$ TBS for All-Bond 2 to dentin varying from 41 to 50 MPa when bonded to light-cured resin composites,<sup>23-25</sup> and to be higher to dual- than to light-cured resin composite.<sup>26</sup> Those are in agreement with our study where All-Bond 2 showed a  $\mu$ TBS of ~ 55 MPa to Filtek Supreme Plus and ~ 61 MPa to Bisfil 2B.

It should be noted that All-Bond 2 was not used with Pre-Bond (Bisco, Inc.), which can be mixed with D/E Resin to make the adhesive dual-cured. Therefore, the effect of converting the adhesive to a dual-cure on the bond of All-Bond 2 to Bisfil 2B is unknown. Of course, All-Bond 2 performed very well even in the light-cured mode, so this might be a moot point. When clinically tested, the combination of All-Bond 2 and Bisfil 2B has had 88% success in the bonding of IPS Empress restorations during a seven-year follow-up.<sup>27</sup> Likewise, One-Step Plus has also been successfully used with light-cured resin composites with  $\mu$ TBS reported in the literature ranging from 31 to 47 MPa.<sup>25,28-30</sup> No *in vitro* or *in vivo* report on the compatibility of One-Step Plus to self- or dual-cured resin composites could be found in the dental literature.

High bond strength values also were obtained for OptiBond Solo Plus when bonded to dentin coupled with Filtek Supreme Plus (~ 68 MPa).

This mean  $\mu$ TBS is considerably higher than what has been recently published (14–39 MPa).<sup>31-33</sup> However, OptiBond Solo Plus did not perform well when used along with Bisfil 2B. An incompatibility to that self-cured resin composite was evident as the  $\mu$ TBS was ~ 20% of its light-cured counterpart (~ 13 MPa). This probably can be explained by the low pH of this material, which can interfere with the adhesive/resin composite interface as previously explained. OptiBond Solo Plus Activator (Kerr Corporation), which is an unfilled resin designated to initiate a chemical cure when mixed with OptiBond Solo Plus, was not used as it has been shown in some studies to have minimal to no positive effect on the  $\mu$ TBS of OptiBond Solo Plus to self-cured resin composites.<sup>8,11,34</sup>

Peak SE, which has the lowest pH value among the materials tested, performed well with Filtek Supreme Plus and was incompatible to Bisfil 2B. A recently published article by Brackett et al. has reported  $\mu$ TBS of Peak SE to dentin of 80 MPa.<sup>35</sup> To the best of our knowledge, there is no report on bond strength of Peak SE to self- or dual-cured resin composites.

The effect of the permeability of certain adhesive systems on the bonding of resin composites was not evaluated in this study. Clinically, permeability might have an effect on adhesion of some of the materials tested. The application of several coats of adhesive when using etch-and-rinse one-bottle systems or the use of a hydrophobic adhesive resin after self-etching adhesive systems might help prevent water flux through the adhesives and has been previously recommended.<sup>31</sup>

To solve the so called true incompatibility problem, i.e., the negative effect that acidic monomers have on the polymerization of self- and dual-cured resin composites, deprotonization of the acidic monomer with an admixed anion exchange compound added to the intermediate bonding layer<sup>36,37</sup> might help.

The addition of an anion exchange compound to self-cured resin composites has been studied by the addition to the aromatic tertiary amine dimethyl-p-toluidine component of an experimental self-cured resin composite. This seems to counteract the neutralization of the aromatic tertiary amine by deprotonization of the acidic monomer from the adhesive. Its ability to



scavenge hydrogen ions appears to be strong and takes place quickly, superseding the adverse effects of oxygen inhibition.<sup>36,37</sup> Further research focusing on the elimination of the incompatibility between self- and dual-cured resin composites and simplified acidic adhesive systems is needed.

## Conclusions

Incompatibility of self-cured resin composites and low-pH light-cured adhesives adversely affects bonding of those materials to dentin.

## Clinical Significance

Incompatibility between adhesives with low pH and certain self-cured resin composites can cause clinical debonding of restorations.

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