Original Article

Dentin Bond Strength of Two One-Bottle Adhesives after Delayed Activation of Light-Cured Resin Composites

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Abstract:

Objective: Adverse surface interactions between one-bottle adhesives and chemical-cured composites may occur with delayed light activation of light-cured composites. The purpose of this study was to assess the Effects of delayed activation of light-cured composites on shear bond strength of two one-bottle adhesives with different acidity to bovine dentin.

Materials and Methods: Flat dentin surface was prepared on sixty-six bovine incisors using 600 grit carbide papers. Prime&Bond NT, and One-Step adhesives and resin composite were applied in six groups: 1) immediate curing of the composite, 2) the composite was left 2.5 minutes over the cured adhesive before light activation, 3) prior to delayed activation of the composite, the cured adhesive was covered with a layer of nonacidic hydrophobic porcelain bonding resin (Choice 2) and cured immediately. After thermocycling, shear bond strength (SBS) test was performed using a universal testing machine at 1 mm/min crosshead speed. Data were analyzed with Friedmans two-way Non-parametric ANOVA.

Results: The SBS of delayed activation of Prime&Bond was significantly lower than immediate activated ($P<0.05$). Decrease in the SBS of One-Step was not statistically significant after delayed activation. The SBS of delayed activation of Prime&Bond and One-Step with an additional resin layer was significantly higher than delayed activation ($P<0.001$).

Conclusion: The bond strength of Prime&Bond might be compromised by the higher acidity of this adhesive during the 2.5 minutes delayed activation of light-cured composite. Addition of a layer of hydrophobic resin compensated the effect of delayed activation and improved the bond strength.

Key Words: Composite Resins; Dentin-Bonding Agents; Prime and Bond NT; Dental Bonding

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INTRODUCTION

Adding hydrophilic monomers to adhesive resins has made adhesion of resin to dentin possible. Infiltration and polymerization of these monomers into a wet mineral depleted dentinal surface may lead to the formation of a hybrid layer. Adhesive systems were originally applied in three separate steps of etching, priming and resin bonding. These systems have been developed into simplified versions with a combination of components. In the fifth generation (one-bottle) adhesives, primer and adhesive resin have been combined for simpler and faster application [1,2].

Some investigations have recently found the evidences of chemical intervention with po-

lymerization of resin composite using the simplified adhesives [3,4]. For a long time, dentists have assumed that the resin composites have a good bond with dentin adhesives when air inhibition layers are present on the surface of the cured adhesives, and any weak links are between adhesives and dentin [4]. However, the incompatibility between some simplified-step adhesives (one-bottle) and chemical-cured composites has recently been reported [3,4,5].

Decrease in microtensile bond strength was found to be inversely proportional to the acidity of these adhesive systems. Moreover, a linear relationship was observed between the pH value of the adhesives and their bond strength using the chemical-cure composites [4].

The incompatibility problem was thought to be caused by the interaction of unpolymerized acidic resin monomers from the adhesive inhibition layer with the nucleophilic tertiary amine that is commonly employed in chemical-cured resin composites [6]. The proposed mechanism of incompatibility between acidic resin monomers and chemical-cured composites is due to the slower rate of polymerization of chemical-cured composites, which is not the case with rapidly polymerized light-cured composites. As a result, there would be no time for acid-base interaction. In addition, the ternary amines used in light-cured composites are generally less nucleophilic than those utilized for chemical-cured composites [3,4,6].

Compared with two-step self-priming adhesives, single-step self-etch adhesives are more acidic because of their self-etching potentionals [7]. Therefore, these adhesives are more susceptible to show incompatibility, since the ternary catalysts are absent in these adhesives [6,8,9].

Incompatibility between single-step self-etch adhesives with light-cured composites has recently been reported, when prior to light activation the latter was left on top of the cured adhesive layer for a period of time [10].

This study examined the effect of delayed activation of light-cured composites on dentin bond strength of two one-bottle adhesives with different acidities; Prime&Bond NT (pH= 2.68), and One-Step (pH=4.60) [4].

MATERIALS AND METHODS

Sixty-six extracted intact bovine lower incisors (stored in 1% thymol solution) were selected for this experimental study. Flat middle dentin surfaces were prepared using 600 grit silicon carbide papers under running water. The specimens were mounted in acrylic molds and randomly divided into six groups.

Prime&Bond NT (Dentsply DeTrey, Konstanz, Germany) and Spectrum TPH composite (Dentsply DeTrey, Konstanz, Germany) were used in the first three groups, and One-Step (Bisco Inc., Schamburg, IL,USA) and AElite composite (Bisco Inc., Schamburg, IL) for other three groups, according to the manufacturer's instructions. Resin composites were bonded on the dentinal surface using a cylindrical split mold with a height of 4 mm and surface diameter of 2 mm. Light-curing was performed using a halogen light-curing unit (Coltolux, Coltene/Whaledent Inc, Switzerland) with the light intensity of 500 mW/cm2.

In groups 1 and 4, Prime&Bond NT, and One-Step bonding materials were used as control groups with immediate light activation of the composite. In groups 2 and 5, after application of Prime&Bond NT and One-Step bonding materials, the first 1mm increment of the composite was applied over the cured adhesive and left in a dark place for 2.5 minutes prior to the light activation.

In groups 3 and 6, after light-curing the adhesives, an additional HEMA-free, hydrophobic layer of porcelain bonding resin (Choice 2; Bisco, USA) was applied and cured immediately before the 2.5 min delayed light activation of composites.

All the specimens were stored in distilled water for 24 hours at room temperature and then thermocycled for 500 cycles at 5°C and 55°C.

Table 1. Descriptive statistics of shear bond strengths (MPa) and types of fracture for the six tested groups.

	Groups	Shear bond strength (MPa)				Fracture
		Max	Min	Mean	SD	
1	**Prim&Bond NT (PB)**	35.6	13.3	20.6^{A}	6.6	4A, 3Cc, 2Cd, 2M
2	**PB (delayed activation)**	15.6	5.4	10.7^{B}	3.3	8A, 2Cc, 1M
3	**PB (delayed activation + resin)**	36.2	16.8	24.3^{A}	6.1	3A, 3Cc, 2Cd, 3M
4	**One-Step**	38.8	13.5	26.2^{AB}	9.1	2A, 6Cc, 3M
5	**One-Step (delayed activation)**	28.8	12.9	17.3^{B}	4.6	5A, 2Cc, 1Cd, 3M
6	**One-Step (delayed activation + resin)**	47.1	22.8	35.6^{A}	7.7	1A, 6Cc, 2Cd, 2M

A=Adhesive ; Cd=Cohesive in dentin ; Cc=Cohesive in composite ; M=mixed

Shear bond strength (SBS) was measured using a universal testing machine (Instron model 4302, Germany) at a crosshead speed of 1 mm/min. The mode of failures was determined by means of stereomicroscope (X20) Collected data were analyzed using Friedmans two-way Non-parametric ANOVA ($P<0.05$).

RESULTS

Descriptive statistics of SBS values in MPa for six tested groups are presented in Table 1. Mean values and standard deviations of SBS are shown in Figure 1. Bond strength of delayed light-activation Prime&Bond NT (group 2) recorded 10.7 (SD=3.3) MPa which was significantly lower than that of the immediately activated group with the SBS of 20.6 (SD=6.6) MPa ($P<0.05$).

There was a significant difference between bond strength of Prime&Bond NT with delayed activation and the delayed activation after applying the additional layer of hydrophobic resin ($P<0.001$); the bond strength increased from 10.7 (SD=3.3) MPa to 24.3 (SD=6.1) MPa with the addition of a layer of hydrophobic resin. However, the bond strength of Prime&Bond NT with immediate activation did not show statistically significant difference with delayed activation, and addition of hydrophobic layer of porcelain bonding resin ($P=0.8$).

Using One-Step adhesive, showed no statistically significant difference between the bond strength of immediate activation and delayed activation with the mean values of 26.2 (SD=9.1) and 17.3 (SD=4.6) respectively ($P=0.3$). Immediate activation and delayed activation with additional layer of resin also showed no significant difference ($P=0.7$). However, the bond strength of One-Step after delayed activation with an additional hydrophobic resin showed a statistically significantly increase compared to the delayed activation without porcelain bonding resin ($P<0.001$).

Stereomicroscopic observation (Table 1) revealed four fracture types including: adhesive; cohesive in dentin; cohesive in composite; and mixed (adhesive and cohesive).

With increasing the SBS, the fracture mode was more cohesive in dentin or composite instead of adhesive fracture.

DISCUSSION

The effect of delayed activation of light-cured composite in reducing dentin bond strength of one-step self-etch adhesives has previously been reported [6,11-13].

Tay et al [10] reported that the microtensile bond strength of two all-in-one adhesives (Prompt L-Pop and One-Up Bond F) decreased with increasing the delay time (0, 2.5, 5, 10, and 20 minutes) in light activation of the composite [10].

In this study, after 2.5 minutes delayed light activation, a significant decrease in SBS of Prime&Bond NT was observed; whereas the decrease in bond strength of One-Step was not statistically significant. This difference could be attributed to the different pH values of two

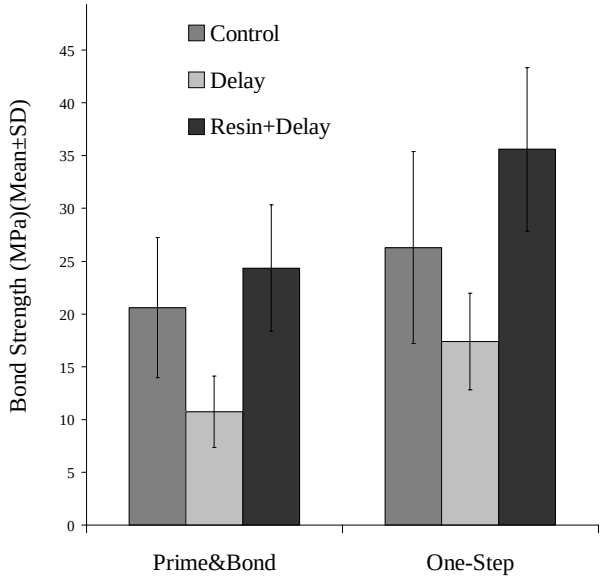

Fig 1. Share bond strength in six groups.

adhesives; Prime&Bond NT (pH=2.68) is more acidic than One-Step (pH=4.60) [4].

Chemical incompatibility or acid-base reaction, which occurred during 2.5 minutes delayed light activation, may have caused the considerable decrease in bond strength of Prime&Bond NT adhesive. In fact, deactivation of tertiary amines of the light-cured composite by the acidic resin monomers of the adhesive could be responsible for the observed results.

Sanares et al [4] reported that microtensile bond strength of One-Step adhesive was significantly higher than Prime&Bond NT when the adhesives were used with chemical-cured composites. This difference was reported to be due to the difference in the acidity of two adhesives [4]. Since the setting time of chemical-cured composite is about 3 minutes, in the present study when polymerization of light-cured composite was delayed for 2.5 minutes, adequate time would be available for acid-base interaction between Prime&Bond NT with the higher acidity and nucleophilic catalyst of the light-cured composite.

Another factor, which may contribute to the difference of incompatibility between these two adhesives and light-cured composite with delayed activation, is permeability. Some studies showed that single-bottle adhesives act as a permeable membranes after polymerization due to their ionic, hydrophilic groups which are originally present in the adhesive monomers, and the absence of an additional coating of hydrophobic bonding resin [5,14-16]. As a result, water can be diffused from the hydrated dentin toward the intermixed zone between the adhesive and the uncured composite. These water droplets not only reduce the bonded surface area, but also they may increase the interfacial stress [16].

The effect of the permeability of one-step self-etch adhesives and osmotic blistering of water droplets along the composite-adhesive interface in decreasing the bond strength to hydrated deep dentin was reported when these adhesives were used with chemical-cured or delayed activation of light-cured composite [6,11-13,17]. Delayed light-activation had no adverse effect when these adhesives were bonded to completely dehydrated dentin and similarly when the processed composite was used as the bonding substrate [13].

For simplified-step adhesives, it seems that the higher concentrations of ionic and acidic groups increase water sorption and hydrophilicity of the adhesive. This may also hasten the water permeability [18].

In this study, water permeability and adverse chemical interaction were explained by virtue of the more acidic potential of Prime&Bond NT compared to One-Step adhesive. Bonding was performed on the middle dentin where permeability may have play less important role, comparing to the deeper dentin, in incompatibility between the adhesive and composite.

This study also demonstrated that the addition of a layer of non-acidic and hydrophobic resin placed over the Prime&Bond NT surface could compensate the decreasing effect of delayed light activation on the bond strength. Place-

ment of a porcelain bonding resin increased the bond strength in a way that, it had no significant difference with the control group (immediate activation).

If Prime&Bond NT is covered by a layer of solvent-free, hydrophobic resin and immediately light cured, the adverse acid-base reaction may be eliminated and water permeability of the adhesive layer could be reduced. In addition, this hydrophobic layer is completely compatible with the hydrophobic resin composite. The beneficial effect of the additional resin layer has been reported for single-step self-etching adhesives [9,13,19,20].

In the present study, when One-Step adhesive was used, delayed light activation resulted in compromised bond strength, which was not statistically significant. However, with an additional coating of hydrophobic resin of the same manufacturer (Bisco), bond strength was significantly increased. Therefore, the additional layer of resin not only eliminated the risk of any incompatibility between adhesive and composite, but also it could increase the thickness of the adhesive layer that resulted in the considerable improvement of the bond strength.

Increased thickness of the adhesive layer has been shown to reduce polymerization shrinkage stress and may improve stress distribution during testing. Both may have contributed to the higher bond strength observed [19,21]. However, this higher bond strength compared to the control group was not significant. In addition, the hydrophobic resin covering from the same manufacturer converted One-Step adhesive into a conventional three-step adhesive which produced the higher bond strength [2,22].

In agreement with this result, Zheng et al [23] reported that microtensile bond strength of Clearfil Liner Bond 2 improved with an increase in the thickness of the adhesive layer. This may be due to the improvement of stress distribution during the experiment [23].

CONCLUSION

The result of this study revealed that the pH value or acidity of one-bottle adhesives could affect dentin bond strength after delayed light activation of resin composite.Compared with One-Step adhesive, the low pH of Prime&Bond NT during 2.5 minute delayed light activation might cause the incompatibility with light-cured composites which lead to the decrease of bond strength to the dentin.

Moreover, addition of a layer of more hydrophobic unfilled solvent-free resin with neutral pH especially from the same manufacturer might produce higher bond strengths to the dentin. However, more in vivo studies are required to confirm these *in vitro* observations.

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