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Effect of Light Activation Mode on the Incompatibility Between One-bottle Adhesives and Light-cured Composites: An *In Vitro* Shear Bond Strength Study

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Clinical Relevance

Low dentin bond strength has been previously documented when one-bottle adhesives are used with self-cured resin-based composites. Low dentin bond strength may also occur when one-bottle adhesives are used with light-cured resin-based composites that are activated by soft-start or pulse-delay curing strategies.

SUMMARY

This study examined the effect of different light activation modes for light-cured resin-based composites on the shear bond strength to dentin of two one-bottle adhesives with differing acidity. In this experimental study, a flat middle dentin surface was prepared on 110 extracted sound molars using a 600-grit polish paper. The teeth were then randomly divided into 10 equal groups (n=11). One-Step Plus (OS) and Prime & Bond NT (P&B NT) were used according to the manufacturer's instruction with their respective composite (Aelite, Spectrum TPH) applied and cured using five different light-activation modes:

1. **Conventional (CO): 600 mW/cm^2 (40 seconds)**
2. **Soft-Start I (SSI): 100 mW/cm^2 (10 seconds) 600 mW/cm^2 (30 seconds)**

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3. Soft-Start II (SSII): 200 mW/cm^2 (10 seconds), 600 mW/cm^2 (30 seconds)

4. Pulse-Delay I (PDI): 100 mW/cm^2 (3 seconds), 3-minute delay, 600 mW/cm^2 (37 seconds)

5. Pulse-Delay II (PDII): 200 mW/cm^2 (3 seconds), 3-minute delay, 600 mW/cm^2 (37 seconds)

After 24 hours storage in distilled water at room temperature, a shear bond strength (SBS) test was performed using an Instron machine at 1 mm/minute and the results were recorded in MPa. Statistical analysis included two-way ANOVA and Tukey HSD ($p<0.05$).

The highest SBS (MPa) was shown in the OS conventional group (19.62 ± 2.21) and the lowest SBS was shown in P&B NT, PDII (5.93 ± 1.79). In each group of five curing modes, the mean SBS for P&B NT was significantly lower than OS: conventional mode—P&B NT (17.27 ± 1.98) vs OS (19.62 ± 2.21); SSI–P&B NT (10.84 ± 2.82) vs OS (13.09 ± 1.24); SSII - P&B NT (14.78 ± 1.63) vs OS (18.79 ± 1.57); PDI–P&B NT (5.93 ± 1.79) vs OS (11.97 ± 2.59) and PDII–P&B NT (11.82 ± 1.24) vs OS (16.00 ± 1.62) ($p<0.001$ for all comparisons).

For each of the adhesives, the ranking of SBS was as follows: CO>SSII>PDII>SSI>PDI, with the two-paired comparisons of curing modes being significantly different ($p<0.05$).

The results of the current study indicated that the bond strength of P&B NT might be compromised by the higher acidity of this adhesive compared to OS during each curing mode, especially PDI. The conventional mode was least affected by the acidity of the adhesive.

INTRODUCTION

Adhesive systems were originally formulated with separate etching, priming and adhesive for a specific resin. These systems have developed into simplified versions for simpler and faster application.[1-2]

In commercially available one-bottle adhesives, an adhesive resin and the primer component, hydrophilic and acidic monomers, which have carboxylic or phosphoric acid functional groups, are combined. This combination results in a low and different pH for these adhesives.[3] These adhesives, which accompany light-cured resin-based composites (RBCs), have been widely used in clinical practice. Unlike conventional adhesives that utilize an intermediate resin layer to bond the primed dentin to restorative RBCs, uncured acidic resin monomer in the oxygen-inhibited layers of one-bottle adhesives are in direct contact with the RBCs.[4]

Clinicians have always assumed that RBCs bond well to the oxygen-inhibited layer of cured adhesives. However, it is known that the interaction between acidic resin monomers and the basic tertiary amines of RBCs result in incomplete polymerization of the self-cured RBCs.[3,5-7]

Light–cured RBCs utilize photochemical redox systems that also involve the use of tertiary amines. However, the rate of initiation of free radicals in light-cured RBCs is much faster than in self-cured RBCs. Thus, the competition for nucleophiles between the two reactions is in favor of the generation of free radicals, and the acid-base reaction is suppressed. Additionally, the amines in light-cured RBCs are less reactive with acids when compared to self-cured RBCs.[3,8-9]

Nevertheless, incompatibility between light-cured RBCs and single-step, self-etch adhesives has been reported when the latter were left on top of the cured adhesives for a period of time before light-activation.[10] Therefore, there was an exponential decline in bond strength with an increasing delay time for 0, 2.5, 5, 10 and 20 minutes before light activation. When the cured adhesive was covered with a layer of non-acidic bonding resin, a 20-minute delay time had no detrimental effect on bond strength.[10]

Although the properties of modern RBCs have been improved, polymerization shrinkage still remains a major problem in light curing restorations. This can create contraction stress, which may disrupt the bond to cavity walls.[11] During polymerization, the elastic modulus of the composite is developed. Prior to reaching the gel-point, the slip of molecules to new orientations, known as flow, compensates for the polymerization contraction. After the gel-point, the composite becomes rigid and flow ceases, with polymerization inducing significant stress at the composite-tooth bond.[12-13]

The speed of the polymerization reaction has a great effect on the generation of stress. When the polymerization reaction proceeds more slowly, as in the case of self-cured RBCs, more time is available for flow to compensate for polymerization contraction. An increase in polymerization time can occur for light-cured RBCs by pre-polymerization at a low light intensity followed by final cure at high intensity (soft-start polymerization).[14-16]

To allow more time for composite flow, a pulse-delay curing mode has been suggested. For example, polymerization is started with a short irradiation time of three seconds at low intensity; after a three-minute delay, curing is completed with a high light intensity.[14,17]

To date, almost nothing is published about the effect of adverse acid-base reactions on the bond strength of one-bottle adhesives to dentin with different light-activation modes.

Therefore, the current study evaluated the relationship between different acidities of two one-bottle adhesives and the composite light activation mode by determining the shear bond strength to dentin. The modes of light-activation included two soft-start and two pulse-delay curing methods. The null hypothesis tested was that the shear bond strength of each one-bottle adhesive to dentin is not affected by different light-activation modes of the RBC.

METHODS AND MATERIALS

One-hundred and ten extracted sound human molars were stored in a 1% chloramine T solution for two weeks, then in distilled water. The teeth were used within three months after extraction. After removing the roots, the teeth were mounted in cold-curing acrylic resin. The occlusal enamel was removed by using a diamond saw (Letiz, 1600, Germany) under running water, to expose flat mid-coronal dentin. The dentin surfaces were polished with silicon carbide paper using an Abramatic polisher machine (LOH Tronic HLP 300, Germany) under water-cooling.

After ultrasonic cleaning, washing and drying, adhesive tape was used to define the bonding area. The specimens were randomly divided into 10 groups of 11 teeth each. In the first five groups, One-Step Plus (OS) was used in combination with Aelite composite (BISCO Inc, Schaumburg, IL, USA) and Prime & Bond NT (P&B NT, Dentsply DeTrey, Konstanz, Germany) was used in combination with Spectrum TPH composite (Dentsply DeTrey) in the other five groups. All of the materials were used according to manufacturer's instructions (Table 1).

pH Measurements

The pH value of two adhesives was determined using a digital pH meter (Metrohm, model 744, Metrohm Schweiz AG, Zofingen, Switzerland) at room temperature in a dark room with a special red light. The measurements were performed on approximately 10 drops of each adhesive and the values were read after 15 seconds, when the pH reading was stable.

The composite was placed on the cured adhesive using a cylindrical split mold with a height of 2.5 mm and surface diameter of 2 mm in two increments of 1 mm and 1.5 mm.

For each adhesive, five light curing modes were employed for curing the first layer of composite. Group 1, conventional (CO), involved irradiation at 600 m W/cm^2 for 40 seconds as the control group. In Group 2, the soft-start I (SSI) method used an initial low intensity (100 mW/cm^2 for 10 seconds) followed by a final cure at a high intensity (600 mW/cm^2 for 30 seconds). Group 3, the soft-start II (SSII) method, was similar to SSI, with the exception of a higher initial intensity (200 m W/cm^2). Group 4, pulse-delay I (PDI), used an initial low intensity (100 mW/cm^2 for three seconds) followed by a waiting time of three minutes and a final cure at high intensity (600 m W/cm^2 for 37 seconds). Group 5, pulse-delay II (PDII), was similar to SSI, with the exception of a higher initial intensity (200 m W/cm^2).

A commercial light-cure unit that allowed for independent command over time and intensity (Variable Intensity Polymerizer [VIP], BISCO Inc) was employed for this study, allowing the manual selection

Table 1: *Adhesive pH and Application Protocol for Materials Used in the Current Study*

Material/Batch #	Adhesive	Composition pH	Application Protocol	Manufacturer
Gel etchant 7 JT		35% phosphoric acid	Etch for 15 seconds. Rinse for 10 seconds, leaving dentin moist	3M Dental Products, Seefeld, Germany
Prime & Bond NT 0509001773	2.68	PENTA, UDMA, T-resin, D-resin, Silicone dioxide nanofiller, photo-initiators, stabilizer, acetone, cetylamine hydrofluride	Apply the adhesive to saturate the surface, reapply if necessary. Leave the surface undisturbed for 20 seconds, gently air dry for at least five seconds. Light cure for 10 seconds.	Dentsply DeTrey, Konstanz, Germany
One-step plus 0600003549	4.61	bis-GMA, BPDM, HEMA, P-dimethylamiobenzoic acid, acetone, glass fillers	Shake the bottle for 3-5 seconds before use. Apply two consecutive coats, agitate 20 seconds, gently air dry. Light cure for 10 seconds.	BISCO Inc, Schamburg, IL USA

PENTA: dipenta-erythritol phosphorous acid ester
UDMA: urethane dimethacrylate
T-resin: cross-linking agent
D-resin: small hydrophilic molecule
bis–GMA: bispheyl glycidyl–methacrylate
BPDM: biphenyl dimethacrylate
HEMA: 2 hydroxyethyl methacrylate

Table 2: *Shear Bond Strength (Mean ± SD) to Dentin and Fracture Mode*

Group	Code		Mean ± SD (MPa)	Fracture (A/Cc/Cd/M)*
1	CO	OS	19.62 ± 2.21^{A}	2 / 4 / 4 / 1
2	SSI	OS	13.09 ± 1.24^{B}	5 / 2 / 2 / 2
3	SSII	OS	18.79 ± 1.57^{C}	2 / 3 / 4 / 2
4	PDI	OS	11.97 ± 2.59^{D}	6 / 2 / 1 / 2
5	PDII	OS	16.00 ± 1.62^{E}	4 / 3 / 1 / 3
6	CO	P&B NT	17.27 ± 1.98^{F}	3 / 4 / 3 / 1
7	SSI	P&B NT	10.84 ± 2.82^{G}	6 / 2 / 1 / 2
8	SSII	P&B NT	14.78 ± 1.63^{H}	5 / 1 / 2 / 3
9	PDI	P&B NT	5.93 ± 1.79^{I}	8 / 2 / 0 / 1
10	PDII	P&B NT	11.82 ± 1.17^{J}	6 / 2 / 0 / 3

A different superscript in the column of mean values indicates statistically significant different values
**A: Adhesive, Cc: Cohesive in composite, Cd: Cohesive in dentin, M: Mixed*

of curing time and intensity. The intensities for each light-curing mode were checked with the built-in radiometer before use.

All specimens were stored in distilled water for 24 hours at room temperature and then loaded in shear to failure in a universal testing machine (Instron model 4302, Instron Corp, Canton, MA, USA) at a crosshead speed of 1 mm/minute.

The shear bond strength was recorded in MPa and the data were analyzed with two-way ANOVA and Tukey's HSD post hoc tests at a significance level of 0.05.

After testing, the fracture modes were evaluated, using a stereomicroscope (Carl Zeiss, Inc, Thornwood, NY, USA) at 10x and classified according to the predominant mode of fracture including: I) adhesive, II) cohesive in dentin, III) cohesive in composite and IV) mixed, a combination of adhesive and cohesive (Table 2).

RESULTS

The pH measurements of the two adhesives showed that the pH of P&B NT was 2.68 and and that of OS was 4.61 (Table 1).

The mean shear bond strengths and standard deviations of the 10 experimental groups are shown in Table 2. Two-way ANOVA revealed that the bond strength results were significantly influenced by the adhesive type ($p<0.0001$) and composite curing mode ($p<0.001$). The interaction of these two factors was also statistically significant ($p<0.05$).

The highest and lowest bond strengths (MPa) were achieved using OS in conventional curing (19.62 ± 2.21) and P&B NT with PDI (5.93 ± 1.79), respectively. In each of the five curing modes, the bond strength of P&B NT was significantly lower than OS ($p<0.001$).

When the bond strengths obtained via different curing modes were compared using the Tukey's test for each individual adhesive, significant differences were observed for all two-paired comparisons: CO > SSII > PDII > SSI > PDI ($p<0.05$).

Fracture analysis revealed that most of the fractures of OS and P&B NT in conventional curing and in OS in SSII were cohesive in dentin or composite, and in P&B NT in the PDI group, they were adhesive. In the other groups, all four modes of fracture were observed.

DISCUSSION

The bond strengths of each of the adhesives were significantly different in the five curing modes of light-cured composites. Thus, the null hypothesis of the current study was rejected. The objective of this study was not to evaluate the effect of relieving the contraction stress developed during composite light curing by flow with different light activation modes on the dentin bond strength of two one-bottle adhesives. Therefore, only flat dentin surfaces with a high degree of ability to relieve shrinkage strain were used for bonding. Therefore, the composites had relatively unrestricted flow for the relief of polymerization shrinkage stress. While the polymerizing composite is constrained by adhesion to cavity walls, its contraction is limited, resulting in more stress. The bond strength to dentin is affected by compatibility between the adhesive and RBC.[3,5,7-8,10,18]

The results of the current study showed that the bond strengths of two one-bottle adhesives were affected by five curing modes. In both adhesives, the lowest bond strength was observed in the PDI group and the highest was presented in the conventional group. The mean bond strength of OS was reduced by almost one-half when conventional curing was substituted for PDI curing, while that of P&B NT was reduced to more than one-third. Also, in each of the five curing modes, the bond strengths of P&B NT were significantly lower than OS. This difference could be attributed to the different pH values of the two adhesives. Therefore, P&B NT with a 2.68 pH value is more acidic when compared to OS with a pH of 4.61.

Nevertheless, the different RBCs used could have influenced the results and it may not be just the pH differences; elastic modulus of the RBCs used could have been a significant factor. Since the aim of the current study was to test the entire system, not just its parts, each adhesive was used with its manufacturer's respective composite. The results from the current study were consistent with a study by Sanares and others,[3] who reported microtensile bond strength in the P&B NT/self–cured composite subgroup, which was significantly lower than the one-step/self-cured composite subgroup, although the bond strengths of these adhesives used with a light–cured composite were not significantly different. However, Swift and others[5] reported that the bond strength of P&B NT was significantly lower than One-Step with a light-cured composite. The results of the Swift and others[5] study were consistent with the current results for the conventional curing groups. In a recent study, no difference in bond strength between a self-cured and a light-cured composite bonded with One- Step to dentin was reported.[18]

On the other hand, in PDI curing, the energy density of the initial light-activation (the intensity x exposure time) was not sufficiently effective to initiate a polymerization reaction.[17,19] Thus, during the three-minute waiting period, uncured composite remained in contact with uncured acidic monomer from these adhesives. Therefore, sufficient time would be available for the acid-base interaction. Although there are no free ions in the absence of water or a high dielectric medium, neutralization of the amines by an acidic monomer in these water-free adhesives can still occur.[9]

The free radical polymerization of light-cured resins requires a co-initiatior (tertiary amine photoreductant) in combination with a photoinitiator, such as comphorquinone (CQ).[20] Deactivation of this amine would negatively affect polymerization of the surface of the composite in contact with the adhesive and the air-inhibition layer of the adhesive. The lower degree of polymerization of resin monomers can cause a reduction in bond strength.

It has been reported that the adhesive functional monomers affect the polymerization of benzoyl peroxide/amine or even CQ/amine catalysts, resulting in poor polymerization.[3,21]

The organophosphates (such as PENTA in P&B NT) are much more aggressive than carboxylated methacrylate monomers (such as biphenyldimethacrylate in OS) in inhibiting resin polymerization.[9]

When a tertiary amine photoaccelerator was substituted as an alternative photoaccelerator, such as trimethyl barbituric acid or dibutyl tin dilaurate, it is possible to overcome the acid-base incompatibility.[22]

It was reported that delayed light activation of light-cured RBCs and dual cured resin cements can cause a decrease in the dentin bond strength of simplified step adhesives.[10,23]

Improper polymerization of light-cured RBCs was reported at the junction of the single-application self-etch adhesives and the composite, even when the composites were placed and cured immediately after light curing these adhesives.[24]

Another possible factor contributing to the incompatibility was the inherent permeability of one-bottle adhesives to water from the underlying dentin, as water may transport through the cured hydrophilic adhesive layer to the bonding interface during the delay time, because of the presence of a hypertonic adhesive air-inhibition layer. This osmotically-induced water movement was observed on hydrated deep dentin with a one-bottle adhesive after delayed light activation of the composite.[7] Moreover, Hashimoto and others demonstrated outward evaporative water flux after air drying the adhesive to remove solvents before light curing the adhesive, irrespective of the tooth vitality status.[25] Therefore, a water blister may become trapped beneath the uncured or partially cured hydrophobic composite, resulting in decreasing bond strength. Further study is needed to confirm this possibility.

In the current study, the bond strength of two adhesives was significantly higher for PDII curing as compared to PDI. However, both of these curing modes were still significantly lower than conventional curing. The degree to which light-cured RBCs polymerize is proportional to the light intensity and exposure time. In PDII as compared to PDI, despite doubling the initial light intensity, the exposure time is the same and very short. Also, in SSI and SSII, low initial intensity may not have activated a sufficient number of initiator molecules to start an adequate polymerization reaction, especially at the composite surface in contact with adhesive due to light scattering. The low starting intensities might have also resulted in negative effects caused by delayed curing.[26-27] Ruggeberg and others[28] showed that an adequate cure of RBC was not provided below a level of 233 mW/cm^2. Thus, in SSI and SSII curing, there was the possibility for an acid-base interaction to occur during the 10-second initial light curing.

It was reported that different curing modes may result in similar degrees of cure but with different polymer networks with respect to cross-linking density.[29-30] In the current study, it is possible that SS and PD curing may produce a lower cross-linked polymer. It seems that differences in composite behavior and rate of polymerization during the polymerization reaction when light curing was done by SS or PD curing modes could have enabled the adhesive to intervene in the final polymerization of the bonding interface to different degrees. The results of the current study could be complemented with other studies that consider the degree of conversion measurements of the bonding interface layer.

Additionally, further research should be carried out to determine the simultaneous negative effect of acid-base incompatibility and the beneficial effect of reducing polymerization stress using SS and PD curing on the bond strength of acidic adhesives to dentin in composite restorations with a high C-Factor.

CONCLUSIONS

Within the limits imposed in the experimental design, the following conclusions may be drawn. The dentin bond strength of one-bottle adhesive using light-cured resin-based composites might be compromised if the primer/adhesive has a higher acidity. Additionally, delayed light activation modes using low initial intensity, such as pulse delay and soft start curing methods, reduce bond strength. Immediate and adequate light curing of the first layer of composite is recommended.

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