Effect of light intensity for adhesives on shear bond strength to dentin

Koichi SHINKAI¹, Shiro SUZUKI² and Yoshiroh KATOH¹

¹Department of Operative Dentistry, School of Dentistry at Niigata, The Nippon Dental University, 1-8 Hamaura-cho, Niigata, 951-8580, Japan
²Department of Prosthodontics, School of Dentistry, University of Alabama at Birmingham, 1919 7th Avenue, South Birmingham, AL 35294-0007, USA
Corresponding author, Koichi SHINKAI; E-mail: shinkaik@ngt.ndu.ac.jp

This study evaluated the effect of light intensity on the shear bond strength (SBS) of two self-etch adhesive systems: SI-R20401 (an experimental two-step) and Fluoro Bond Shake One (a commercial one-step bonding system). The adhesive systems were applied to the flat dentin surfaces of extracted human teeth according to manufacturers’ instructions. Light intensities used for the adhesive systems were 100, 300, and 500 mW/cm². A resin composite paste was placed and polymerized for 40 seconds with 600 mW/cm² of light intensity after each bonding procedure. Specimens were subjected to SBS test with a 1.0 mm/minute crosshead speed. Data were statistically analyzed using ANOVA, followed by Bonferroni post hoc test. Two-way ANOVA showed no significant differences in the effects of the adhesive system, light intensity for applied adhesive, and the interaction between them. Based on the results and limitations of this study, it was concluded that light intensity showed no significant effects on the SBS of the two self-etch adhesive systems.

Key words: Light intensity, Self-etch adhesive, Shear bond strength

INTRODUCTION

Nowadays, self-etching primer systems have been prevalently used for the adhesion of resin composites to the tooth substrate. These newly developed adhesive systems have shown adequate bond strength to both enamel and dentin. However, several studies have reported that self-etching primer systems possess technique sensitivity related to bonding procedures, which consist of a series of consecutive steps. These bonding steps are namely — dissolution of the smear layer, decalcification of superficial dentin under the smear layer, priming of exposed dentinal collagen, and then adhesive monomer diffusion into the collagen network. Consequently, application time and amount of self-etching primer, air-blowing variables for applied primer or adhesive, and light intensity for applied adhesive seem to be important factors which influence the bond strength of self-etching primer systems. Our previous study suggested that air-blowing suitable for self-etching one-step (all-in-one adhesive) systems depended upon the type of solvent contained in the system.

Further on factors that influence bond strength, there are several studies which reported on the effect of light intensity for filled resin composites on their bond strengths to tooth substrates. Benetti et al. investigated the influence of variations in the curing rate of resin composites on the bond strength of resin composites to dentin, and concluded that only power density — but not curing mode, resin composite material, nor mode of aging — significantly affected shear bond strength.

There are also several studies which reported on the effect of light curing of the bonding agents prior to the placement of resin composites on the shear bond strength to tooth substrates. In a study by Leevailoj et al., they evaluated the effects of photo-polymerizing the bonding agents, prior to placement of both dual- and self-cured resin composites, on shear bond strength to dentin. Results showed that light activation of the bonding agents prior to applying the resin composites led to significantly higher shear bond strength of the resin composites to dentin, compared to no light activation. Similarly, Chapman et al. compared the shear bond strengths of three self-etching bonding agents to enamel and dentin, with the bonding agents either being pre-cured before or co-cured together with the application of the resin composite. Results showed that pre-polymerization of self-etching adhesives before polymerization of the resin composite produced greater bond strength to dentin.

However, only few studies have reported on the effect of light intensity for applied adhesives on their bond strengths to dentin. According to Yamamoto et al., the dentin bond strengths of four self-etching adhesive systems were affected by the curing light intensity. With a view to further clarifying the irradiance-dependent properties of light-cured resin adhesive systems, this in vitro study evaluated the effect of light intensity for applied adhesives on the shear bond strength (SBS) of two self-etching systems.
to extracted human dentin. The null hypothesis of this study was that the light intensity for applied adhesives would not affect the bond strength of resin composites to dentin.

MATERIALS AND METHODS

Materials used
Materials used in this study are listed in Table 1. SI-R20401 (Shofu Inc., Kyoto, Japan), an experimental two-step self-etch adhesive system, and Fluoro Bond Shake One® (Shofu Inc., Kyoto, Japan), one-step self-etch adhesive system, were used in this study. SI-R20402G (Shofu Inc., Kyoto, Japan), an experimental composite paste, was used for SI-R2041, and Beautifil® (Shofu Inc., Kyoto, Japan) was used for Fluoro Bond Shake One. Currently, SI-R20401 is available on the market under the trade name of FL-Bond II®, and SI-R20402G is available on the market under the trade name of Beautifil II®.

Tooth specimens
Extracted human premolars (n=30) were used in this study. After informed consents were obtained from the patients under a protocol approved by the institutional review board of The Nippon Dental University School of Life Dentistry at Niigata, the teeth were collected. The teeth were cleaned and stored in 0.01% thymol solution at 4°C until use. The buccal surfaces were ground flat to dentin with 120-grit silicon carbide paper (Carbimet, Buehler Ltd., Lake Bluff, IL, USA) using a polishing machine (Lewel Specimen Polisher, Kasai Co. Ltd., Yokohama, Japan) under water irrigation. The ground surfaces were then finished with wet 600-grit silicon carbide paper. After which, the specimens were randomly divided into six groups of five specimens each.

Bonding procedures
A double-sided adhesive tape (0.12 mm thickness) with a 6-mm-diameter opening was attached to the flat dentin surface to define the bonding area. After a laminated paper was peeled from the attached adhesive tape, a semi-transparent acrylic tube (3 mm inner diameter, 2 mm height) was placed on the adhesive tape. Each adhesive system was applied to the dentin surface through the tube according to one of the following bonding procedures.

With the experimental two-step adhesive system, SI-R20401, Primer was applied to the dentin surface. The surface was left undisturbed for 20 seconds, and then medium air-blowing was applied for five seconds. Bond was placed, and mild air-blowing was applied to the surface of Bond for three seconds. After which, the bonding layer was photopolymerized for 10 seconds.

With Fluoro Bond Shake One, a mixture of an equal amount of Bond A and Bond B was applied to the dentin surface, and the surface was left undisturbed for 20 seconds. Mild air-blowing was applied for three seconds, and then the bonding layer was photopolymerized for 10 seconds.

Air-blowing intensity was measured on a scale with electric balance when air stream was blown vertically over the electric balance at a distance 1 cm away. Medium air-blowing was measured as 10 g

Table 1 Adhesive systems and resin composites used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Material Type</th>
<th>Lot Number</th>
<th>Main Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI-R20401 (FL-Bond II) Primer</td>
<td>Adhesive system</td>
<td>090529</td>
<td>Carboxylic acid monomer, Phosphonic acid monomer, Water, Solvent, Initiator</td>
</tr>
<tr>
<td>SI-R20401 (FL-Bond II) Bond</td>
<td>Adhesive system</td>
<td>090525</td>
<td>S-PRG filler, UDMA, TEGDMA, HEMA, PI</td>
</tr>
<tr>
<td>Fluoro Bond Shake One Bond A</td>
<td>Adhesive system</td>
<td>010601</td>
<td>Glass filler, Acetone, Water</td>
</tr>
<tr>
<td>Fluoro Bond Shake One Bond B</td>
<td>Adhesive system</td>
<td>010601</td>
<td>HEMA, 4-AET, Bis-GMA, Acetone, PI</td>
</tr>
<tr>
<td>SI-R20402G (Beautifil II)</td>
<td>Composite resin</td>
<td>11041101</td>
<td>Bis-GMA, TEGDME, UDMA, S-PRG filler, MF glass filler, Ultra-fine filler, PI</td>
</tr>
<tr>
<td>Beautifil</td>
<td>Composite resin</td>
<td>010529</td>
<td>Bis-GMA, TEGDME, S-PRG filler, MF glass filler, Ultra-fine filler, PI</td>
</tr>
</tbody>
</table>

S-PRG filler: Surface reaction type Pre-reacted glass ionomer filler
MF glass filler: Multifunctional glass filler based on fluoroboric aluminosilicate glass
Bis-GMA: 2, 2-Bis[4-(2-hydroxy-3-methacryloxypropoxy) phenyl]propane
UDMA: Urethane dimethacrylate
TEGDMA: Triethylene glycol dimethacrylate
HEMA: hydroxyethyl methacrylate
4-AET: 4-acryloxyethyl trimellitate
PI: Photoinitiator

*All materials were manufactured by Shofu Inc.
Photopolymerization of adhesives
Light intensities used for photopolymerizing the bonding layer were 100, 300, and 500 mW/cm² for each adhesive system. Each resin composite paste was bulk-filled into the acrylic tube and then photopolymerized for 40 seconds with 600 mW/cm² of light intensity.

A modified Candelux (J Morita Corp., Tokyo, Japan) was used as a light intensity-changeable light curing unit. After storing the specimens in distilled water at 37°C for 24 hours, the roots were removed with a diamond point (Bur No. 105R, ISO size 22, Shofu Inc., Kyoto, Japan) and the pulps were removed.

Shear bond strength test
Each specimen was fixed in a holder ring using a self-curing resin (Unifast Trad, GC Corp., Tokyo, Japan). The holder ring fixed with the specimen was attached to a testing device (Bencor Multi-T, Danville Engineering Co., San Ramon, CA, USA) and mounted in a tabletop material tester (EZ Test, Shimadzu Corp., Kyoto, Japan). Shear testing was carried out at a crosshead speed of 1 mm/minute.

Data were subjected to two-way ANOVA with adhesive system and light intensity as independent factors, followed by Bonferroni post hoc test to compare the differences in SBS among the experimental groups (p<0.05).

RESULTS
Table 2 shows the results of the SBS tests. With SI-R20401 (FL-Bond II), the SBS mean values of specimens photopolymerized with light intensities of 100, 300, and 500 mW/cm² were 13.9, 12.9, and 14.7 MPa respectively. With Fluoro Bond Shake One, the SBS mean values of specimens photopolymerized with light intensities of 100, 300, and 500 mW/cm² were 12.0, 11.5, and 10.6 MPa respectively. Two-way ANOVA showed no significant differences in the effects of the adhesive system (F=3.5, p=0.0717), light intensity for applied adhesive (F=0.1, p=0.8861), and the interaction between them (F=5.0, p=0.6848).

Table 3 shows the failure mode results as observed by SEM. With SI-R20401 and at a light intensity of 100 mW/cm², mixed failure was predominantly exhibited — except for two specimens which had cohesive failure in the bonding resin. With Fluoro Bond Shake One and at a light intensity of 100 mW/cm², mixed failure was predominantly exhibited — except for one specimen which had cohesive failure in the bonding resin. At 300 mW/cm², all SI-R20401 and Fluoro Bond Shake One specimens exhibited mixed failure. At 500 mW/cm²,
both SI-R20401 and Fluoro Bond Shake One specimens predominantly exhibited mixed failure—except for one specimen from each adhesive system showing adhesive failure between the bonding resin and the resin composite.

DISCUSSION

Several studies have reported that photopolymerization of the adhesives before placement of resin composites increased the bond strength to dentin\(^{12,13}\). It was said that co-curing of adhesives at the same time with the resin composites resulted in insufficient dentin bond strength, which consequently could not withstand the stresses arising from the polymerization shrinkage of resin composites\(^{15}\). Conversely, pre-curing of the adhesive rendered a fortified bonding layer, thereby resulting in increase in dentin bond strength.

In general, photopolymerizing adhesive systems are polymerized by photoirradiation to produce a bonding layer before placement of the resin composite. Depth of the cavity may affect the degree of polymerization of adhesive resins because light energy gradually attenuates through the resin composite. In clinical situations, cases such as deep cavity floor and pulp cavity wall of non-vital teeth may obtain insufficient light energy. It is noteworthy that in light-cured resin composites, the photoinitiator used requires a certain amount of light energy to produce a certain number of free radicals\(^{15}\). To overcome this problem, an extension of irradiation time or use of high-light-intensity curing units might be effective in increasing the degree of polymerization of adhesive resins\(^{15}\). However, an extension of irradiation time is tantamount to prolonged operation time, and that irradiation with high light intensity elevates the temperature during adhesive polymerization\(^{16,17}\). Against this backdrop of controversies and concerns, it was therefore important to investigate the effect of light intensity for the polymerization of adhesive resins on dentin bond strength of resin composites.

Results of this study revealed that light intensity for adhesive polymerization did not affect the bond strength to dentin. There were no significant differences in dentin bond strength among the light intensities for each adhesive system. The results showed that the degree of light intensity (100, 300, or 500 mW/cm\(^2\)) used for adhesive polymerization showed no correlation with the dentin bond strengths of both adhesive systems. The dentin bond strengths of the self-etch adhesive systems when photopolymerized at a low light intensity (100 mW/cm\(^2\)) was similar to those at higher light intensities (300 and 500 mW/cm\(^2\)). Based on the results obtained, the null hypothesis that light intensity for applied adhesives would not affect the bond strength of resin composites to dentin was accepted.

On the other hand, Yamamoto et al.\(^{14}\) reported that the dentin bond strength of self-etch adhesive systems when photopolymerized at a low light intensity (150 mW/cm\(^2\)) was significantly lower than those photopolymerized at higher light intensities (300, 600, and 900 mW/cm\(^2\)). Their results showed that bond strength did not decrease in a linear manner with increase in light intensity, which were different from the results of the present study.

Apart from light intensity, the polymerization degree of adhesives may also influence the bond strength to dentin. In a study that investigated the effects of light irradiance and source on the photopolymerization of three commercial dental adhesives, the photopolymerization efficiency of the commercial adhesives varied as a function of light source and distance\(^{18}\). In light of this result, the light source may also be an important factor which affects the bond strength to dentin.

Thickness of an adhesive layer depends on the air-blowing intensity applied after adhesive application. In this study, mild air-blowing was applied to the adhesive for three seconds, thereby yielding a considerably thin bonding layer within 100 μm. With such a thin layer of bonding resin, one is predisposed to think that light intensity will not affect the degree of polymerization. In addition, it is quite likely that the degree of polymerization in the bonding resin layer was increased due to photoirradiation of the resin composite placed over the bonding resin. In this study, resin composite paste placed into the acrylic tube (2 mm height) was photopolymerized for 40 seconds with 600 mW/cm\(^2\) of light intensity. Although the energy density of this photoirradiation would be reduced at the adhesive interface, it would have had certain effect on both the resin composite and bonding resin. Therefore, this second photoirradiation to the adhesive through the resin composite might have caused an increase in dentin bond strength for the specimens pre-irradiated at a lower intensity for adhesive polymerization.

An oxygen-inhibited layer exists on the surface of photopolymerized adhesive resins\(^{19}\). A thick oxygen-inhibited layer on a cured adhesive layer is believed to result in both good adaptation of resin composite and high bond strength\(^{20,21}\). It should be mentioned that a high degree of conversion of the adhesive layer is also needed to provide durable bonding. In a study by Kim et al.\(^{22}\), the thicknesses of both the oxygen-inhibited layer and cured adhesive layer were measured with FT-NIR spectroscopy as a function of light-curing time to evaluate their effect on bond strength. Their results showed that excessive irradiation of the adhesives increased the degree of conversion, but decreased the thickness of
oxygen-inhibited layer. With a thin oxygen-inhibited layer, defects were observed at the interface between the adhesive layer and the resin composite as well as between the hybrid layer and the adhesive layer\(^{664}\). On the other hand, with a thick oxygen-inhibited layer, free radicals from the overlying resin composite might diffuse into the unreacted monomers in the oxygen-inhibited layer, thereby resulting in chemical bonding between the polymerized adhesive layer and the overlying resin composite during photopolymerization. In light of the results obtained, it was suggested that photopolymerizing adhesives should be polymerized according to the irradiation time recommended by the manufacturer.

Pertaining to the conversion of adhesive resins, a more relevant parameter to consider besides light intensity is the total energy measured in mJ/cm\(^2\). This is because in light-cured adhesive resins, the photoinitiator requires a certain amount of light energy to be activated\(^{665}\). In this study, the irradiation time for the adhesives was fixed at 10 seconds for each light intensity. When the light intensity of 100 mW/cm\(^2\) was irradiated for 10 seconds, the adhesives received a total light energy of 1000 mJ/cm\(^2\). From the results of this study, it was speculated that the light energy of 1000 mJ/cm\(^2\) helped to maintain an appropriate thickness of the oxygen-inhibited layer. However, as mentioned above, it was possible that post-irradiation, owing to the photopolymerization of resin composite, compensated for the degree of photopolymerization in the oxygen-inhibited layer. Consequently, even the dentin bond strength of specimens applied with low light intensity for adhesive polymerization was increased.

On the technique sensitivity of dentin adhesive systems, it is desirable to achieve high bond strength to minimize the effects of clinical variables which affect optimal bonding. Therefore, a self-etch adhesive system which maintains a high bond strength to dentin with low-intensity irradiation is desirable. The self-etch adhesive systems used in this study demonstrated that the dentin bond strength obtained with low light intensity was similar to those obtained with higher light intensities. Nonetheless, it warrants another study that uses other adhesive systems to firmly conclude the effect of light intensity on dentin bond strength. On the influence of post-irradiation of the adhesive layer—owing to the photoradiation of resin composite placed over the bonding resin, further studies are also encouraged to investigate the effect of light intensity on dentin bond strength in this context.

## CONCLUSION

Based on the results and the limitations of the photo-irradiation conditions used in the present study, it was concluded that the light intensity for adhesive photopolymerization had no significant effects on the shear bond strength of the two self-etch adhesive systems.

## REFERENCES