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Original

Influence of light intensity on dentin bond strength of self-etch systems

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Abstract: The purpose of this study was to investigate the influence of light intensity on dentin bond strengths of four self-etch adhesive systems. The light intensities used to polymerize specimens were controlled at levels of 150, 300, 600, and 900 mW/cm^2. The two-step self-etch adhesive systems Imperva Fluoro Bond and Mac Bond II, and the one-step self-etch systems Fluoro Bond Shake-One and One-Up Bond F Plus were used with their corresponding light-cured resins. Labial surfaces of lower bovine incisors were ground with #600 grit SiC paper to expose the dentin. The dentin surfaces were treated according to each manufacturer's instructions and bonded with resin composites. A shear bond strength test was performed and the data were analyzed by one-way ANOVA followed by Newman-Keuls multiple comparison at a level of 0.05. Statistical analysis of the data indicated that light intensity affected the dentin bond strengths of the adhesive systems tested. Significantly lower bond strengths were obtained by exposure to 150 mW/cm^2, and there were no differences between the bond strengths obtained at 600 and 900 mW/cm^2 for all the adhesive systems used. Further research will be required to clarify the irradiance-dependent properties of light-cured resin adhesive systems. (J. Oral Sci. 48, 21-26, 2006)

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Introduction

Visible light-cured resin has been accepted as an esthetic restorative for anterior and posterior dental lesions because of its esthetic advantages, ease of use, improved bonding to tooth structure, and enhanced mechanical properties. The main advantage of a visible light-curing system is its easy handling, allowing a clinician to manipulate materials for long periods while still having a rapid cure available on demand (1). Visible light-cured resins usually employ photosensitized initiators with visible light around 470 nm wavelength to activate polymerization (2). The spectral distribution around the absorption peak wavelength of the photosensitizer is an important factor in the cure of light-cured resin (3). In addition to the proper wavelength of visible light, sufficient intensity from the curing unit is needed to excite the photoinitiator. The curing pattern of light-cured resin has several disadvantages that may compromise its ability to achieve an excellent seal along the cavity wall, such as the direction and speed of polymerization shrinkage, depth of cure, and polymerization contraction stresses (4,5). However, the output intensity of curing units has been developed so as to promote the greatest intensity in order to cure the deeper parts of a resin restoration as well as reduce the time of polymerization (6).

To successfully place a light-cured resin restoration, certain criteria have to be met. The most important of these are a combination of optimal speed of polymerization, good characteristics of flow, complete polymerization and a high shear bond strength (7). Many studies have shown that the tooth/restoration interface of light-cured resin composites can be improved by curing the material at a slower rate and at a lower light intensity (8-10). The reason for this is that slower polymerization allows for a better flow of the material. This also causes less tension within the material, resulting in improved marginal adaptation. However, to ensure a successful restoration, it is also important to obtain sufficient surface hardness to ultimately ensure favorable physical properties of the restoration. Therefore, a sufficient period of high-intensity irradiation of the restoration is necessary (11,12).

The purpose of this study was to investigate the influence of light intensity on dentin bond strength of four commercially available self-etch adhesive systems. The null hypothesis tested was that reduction of light intensity would not significantly reduce the shear bond strengths of the self-etch adhesive systems.

Materials and Methods

The light generator used in this study was an Optilux 501 (Demetron/ Kerr, Danbry, CT, USA). It was plugged into a variable transformer in order to change the intensity of the light output. The light intensity used to polymerize specimens was controlled at levels of 150, 300, 600, and 900 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/Kerr)

The spectral distributions of the curing unit were determined using a computer-controlled spectroradiometer (LI-1800, Li-Cor, Lincoln, NE, USA) as described previously (13). This device has three major components, a filter wheel, a holographic grating monochromator, and a silicon detector with an autoranging amplifier. Light entering the device through the fiber optic probe enters the monochromator after passing through a filter wheel which eliminates second order harmonics. Scans were done for the same input voltages used when measuring light intensity. The spectral distribution for each input voltage was determined from the average of two data scans.

The adhesive systems used in this study are listed in Table 1, and their application procedures are shown in Table 2. The two-step self-etch adhesive systems Imperva Fluoro Bond (FB, Shofu, Kyoto, Japan) and Mac Bond II (MB, Tokuyama Dental, Tokyo, Japan), the one-step self-etch systems Fluoro Bond Shake-One (FSO, Shofu) and One-Up Bond F Plus (OBP, Tokuyama Dental) were used with their corresponding light-cured resins, Beautifil for FB and

Table 1 Self-etch adhesive systems tested

Adhesive system (Code)	Primer (Lot No.) Main component	Adhesive (Lot No.) Main component	Manufacturer
[Two-step system]			
Imperva Fluoro Bond (FB)	FB Primer (A: 060060, B: 060076) A: Catalyst, water B: 4-AET, 4-AETA, HEMA	FB Bond (060070) 4-MET, HEMA, filler, UDMA, TEGDMA, CQ, PI	Shoufu Inc.
Mac-Bond II (MB)	Primer (A: 0251, B: 013) A: MAC-10, HEMA, acetone, isopropyl alcohol, phosphate monomer B: Ethanol, water	Bonding Agent (0181) MAC-10, HEMA, PI, bis-GMA, TEGDMA	Tokuyama Dental
[One-step system]			
Fluoro Bond Shake-One (FSO)		Adhesive (A, B: MS-13) PRG, fluoroaluminosilicate glass, 4-AET, 4-AETA, bis-GMA initiator, water, solvent	Shoufu Inc.
One-Up Bond F Plus (OBP)		Adhesive (A, B: 551F-2) MAC-10, HEMA, MMA, multifunctionl methacrylic monomer fluoroaluminosilicate glass, water, aryl borate catalyst	Tokuyama Dental

4-AET: 4-acryloyloxyethyl trimellitate, 4-AETA: 4-acryloyloxyethyl trimellitate anhydride, HEMA: 2-hydroxyethyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethyleneglycol di-methacrylate, CQ: *dl*-camphorquinone, MAC-10: 11-methacryloxy-1,1-undecan dicarboxylic acid, bis-GMA: 2, 2bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]phenyl propane

Table 2 Application protocols of self-etch adhesive systems

Code	Application protocol
FB	Mix equal amounts of primers A and B. Apply to dentin for 20 s. Adhesive is then applied and light irradiation delivered for 10 s.
MB	Mix equal amounts of bond agents A and B. Apply to dentin for 20 s. Adhesive is then applied and light irradiation delivered for 10 s.
FSO	Mix equal amounts of bond agents A and B. Apply to dentin for 20 s. Briefly air-dry and expose to light irradiation for 10 s.
OBP	Mix equal amounts of the bond agents A and B until a pink homogeneous liquid mixture is obtained. Apply to dentin for10 s with agitation and expose to light irradiation for 10 s.

FSO, and Palfique Estelite Σ for MB and OBP.

Mandibular incisors extracted from 2-3-year-old cattle and stored frozen for up to 2 weeks were used as a substitute for human teeth. After removing the roots with a low-speed saw (Isomet, Buheler, Lake Bluff, IL, USA), the pulps were removed, and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of the bovine incisors were ground on wet 240-grit SiC paper to a flat dentin surface. Each tooth was then mounted in cold-curing acrylic resin (Resin Tray II, Shofu) to expose the flattened area and placed into tap water to reduce the temperature rise from the exothermic polymerization reaction. The final finish was accomplished by grinding on wet 600-grit SiC paper. After ultrasonic cleaning with distilled water for 1 min to remove the excess debris, these surfaces were washed and dried with oil-free compressed air.

A piece of double-sided adhesive tape (Nichiban, Tokyo, Japan), which had a 4-mm diameter hole, was firmly attached to define the adhesive area of the dentin for bonding. The adhesive was applied on the dentin surface according to the manufacturers' instructions. A Teflon (Sanplatec, Osaka, Japan) mold, 2.0 mm high and 4.0 mm in diameter was used to form and hold the restorative resin on the dentin surface. Resin composite was condensed into the mold and cured for 30 s. The finished specimens were transferred to distilled water and stored at 37°C for 24 h.

Ten specimens per group were tested in a shear mode using a shear knife edge testing apparatus in a universal testing machine (Type 4204, Instron, Canton, MA, USA) at a cross-head speed of 1.0 mm/min. Shear bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area. After testing, the specimens were examined in an optical microscope SZH-131 (Olympus, Tokyo, Japan) at a magnification of 10 × to define the location of the bond failure (14). The type of failure was determined based on the percentage of substrate-free material: adhesive failure, cohesive failure in resin and cohesive failure in dentin.

The results were analyzed by calculating the mean shear bond strength (MPa) and standard deviation for each group. Statistical analysis was carried out to show how the bond strengths were influenced by air-drying times. The data for each group were subjected to analysis of variance (ANOVA) followed by Newman-Keuls multiple comparison at a level of 0.05 within each adhesive system. The statistical analysis was carried out with the Sigma Stat software system (Ver. 3.1, SPSS, Chicago, IL, USA).

Results

The spectral distribution characteristics of the curing unit at various light intensities are shown in Fig. 1. The wavelength position of the peak on the curve was almost the same among the different light intensities.

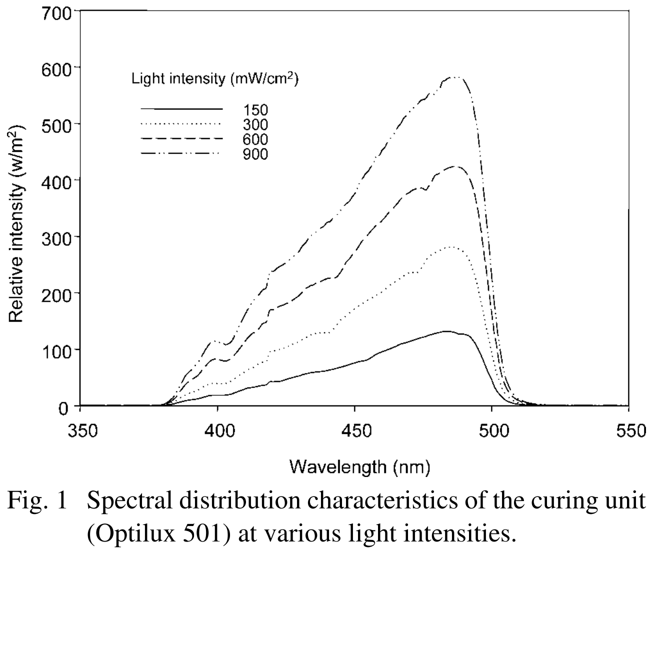

Fig. 1 Spectral distribution characteristics of the curing unit (Optilux 501) at various light intensities.

The influences of light intensity on the dentin bond strengths of the self-etch adhesive systems are shown in Table 3, where the results of the statistical analysis are shown with alphabetic characters. A significantly lower bond strength than that obtained at 900 mW/cm^2 was produced by exposure to 300 mW/cm^2 in FB and MB, and to 150 mW/cm^2 in FSO and OBP. The pattern of decreasing bond strength differed between the one- and two-step self-etch adhesive systems, and each test material had a threshold light intensity required for bond strengths obtained with a light intensity of 900 mW/cm^2.

After testing, the specimens were examined in an optical microscope to locate the bond failure site. Generally, the failure mode was found to be cohesive within resin, and/or partially in dentin for the groups that showed a mean bond strength exceeding 10 MPa. The failure patterns seemed to depend on the light intensity. Irradiation with light at a lower intensity resulted in increased adhesive failure at the dentin surface because of lower bond strength.

Discussion

Light-cured adhesive is polymerized with light irradiation to make an adhesive layer on the dentin surface. As with other light-cured materials, a reduction in light intensity may impair polymerization of the adhesive. When applying an adhesive to a cavity, the depth of the cavity floor may affect its polymerization because the light intensity diminishes with the distance from the light tip end (15). A prolonged irradiation time might be effective for improving bond strength in such situations, but as the distance increases, a point would be reached at which longer exposure times no longer compensated for the reduced intensity. Thus, it is important for clinicians to monitor their curing unit frequently to ensure that adequate light intensity has been maintained (16,17).

Light curing has mainly targeted a fixed exposure time but at different light intensities. The light intensity is measured in mW/cm^2, while a more relevant parameter to consider seems to be the total energy measured in mJ/cm^2 (18). The reason is that the photosensitized initiator used in a light-cured resin requires a certain amount of quantum energy (light energy) (19). Thus, by assuming that a fixed energy level produces a certain number of free radicals, one should achieve the same conversion with a low-intensity lamp as with a high-intensity lamp (20,21). This assumption should be correct as long as the energy input, or the number of useful photons, is the same. In general, the data from the present study showed that decreased light intensity resulted in a lower bond strength for all of the bonding systems used, but bond strengths did not decrease in a linear manner with decreasing light intensity.

The polymerization reaction of light-cured resins is faster than that of self-cured composites, which leads to the development of higher setting stresses than in self-cured resins (22). Such marginal gaps and subsequent micro-leakage may cause marginal staining, postoperative sensitivity and secondary caries. In addition, cavity-wall gap formation may lead to pain on biting and failure of adhesion by repeated occlusal loading. Furthermore, the maximum stress generated at the cavity wall in light-cured resin restorations is twice as large as that for self-cured resin restorations (23).

The variation in sensitivity of bond strength may be due to variation in the concentration of photosensitizers contained in the adhesives used. Furthermore, differences in material composition are also an important variable to consider. Photopolymerization of dimethacrylate dental resins is a complex process that exhibits diffusion-controlled

Table 3 Influence of light intensity of the curing unit on dentin bond strengths of self-etch adhesive systems

Code	Light intensity (mW/cm^2) 150	300	600	900
Two-step systems				
FB	11.8 (2.1)[a]	14.3 (3.3)[a,b]	16.4 (3.1)[b]	16.7 (2.7)[b]
MB	10.6 (1.6)[c]	13.9 (1.4)[c,d]	15.8 (2.3)[d]	16.7 (2.6)[d]
One-step system				
FSO	11.2 (2.7)[e]	14.4 (3.1)[f]	14.3 (2.8)[f]	14.9 (2.6)[f]
OBP	10.3 (2.3)[g]	14.1 (2.9)[h]	14.5 (3.0)[h]	14.5 (2.8)[h]

Values with the same letter in each adhesive system are not significantly different ($P > 0.05$).

kinetics and heterogeneous network growth (24). The initiator system of OBF contains a dye-sensitizer, a co-initiator and a borate derivative. The energy transfer reaction from the dye-sensitizer to the co-initiator takes place upon light irradiation to place the co-initiator in an excited state. Following this, the polymerizable radical species is formed by the reaction of the borate derivative with the activated co-initiator containing hydrogen ions derived from the dye-sensitizer as well as acidic functional monomers (25). Though these materials have been used for decades as the matrix of composite materials, questions remain about their polymerization behavior.

The effect of light intensity on composite properties has been investigated previously. In earlier work, the degree of conversion as a function of depth did not change for 8-mm-thick composite samples when the light intensity was decreased by a factor of eight, as long as the total energy (light intensity x exposure time) remained constant (26). Similarly, the flexural strength and fracture toughness for four common dental composites were found to be unaffected by the light intensity when a constant energy level was supplied (27). In addition, the polymerization shrinkage strain of two dental composites was shown to be a linear function of conversion, regardless of the light intensity used (28). All of these studies suggest that light intensity does not significantly affect the material properties of dental composites. Since the light intensities utilized in these studies generally ranged from 150 to 900 mW/cm^2, the same range of power density for the curing unit was employed in this study.

Conversion of methacrylate functionalized dental restorative materials via photoinitiated polymerization is dependent upon several parameters. Monomer formulation has been shown to impact the conversion of unfilled resins and resin-based composite (29). Even with the most reactive monomers, the fraction of reacted functional groups is significantly less than unity due to the highly cross-linked structure of the developing polymer. Conversion is also dependent upon the rate of polymerization and the exposure time. Since the former is impacted by the radiant intensity absorbed by the photoinitiator, the irradiance of the curing source and its spectral distribution become critical variables. The efficiency of the photoinitating system and oxygen quenching also affect the polymerization rate. Of all these variables, the light intensity of the curing unit and the exposure time are of particular interest since they, in practice, are amenable to manipulation by the clinician. Considering the technique sensitivity of adhesive systems, it is desirable to use a material that will achieve high bond strength with minimum concern for the clinical variables that diminish bond strength. Thus, adhesive systems that will cure with low light intensity exposure are desirable.

The dependence of bond strength on the exposure time and intensity of light-cured resins has been a topic of considerable investigation. Of particular interest is determining the bond strength of these materials under conditions of equivalent radiant energy (dose) by adjusting the irradiance (light intensity) and exposure time. Establishing a reciprocal relationship between these two parameters would add significance to the analysis of bonding properties as a function of radiant energy rather than as two separate variables. Further studies will be needed to investigate the irradiance-dependent properties of newly developed light-cured resin adhesive systems.

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