

Dentin bond strength of bonding agents cured with Light Emitting Diode

Sun-Young Kim, In-Bog Lee, Byeong-Hoon Cho, Ho-Hyun Son, Mi-Ja Kim, Chang-In Seok, Chung-Moon Um*
Department of Conservative Dentistry, College of Dentistry, Seoul National University

ABSTRACT

This study compared the dentin shear bond strengths of currently used dentin bonding agents that were irradiated with an LED (Elipar FreeLight, 3M-ESPE) and a halogen light (VIP, BISCO).

The optical characteristics of two light curing units were evaluated. Extracted human third molars were prepared to expose the occlusal dentin and the bonding procedures were performed under the irradiation with each light curing unit. The dentin bonding agents used in this study were Scotchbond Multipurpose (3M ESPE), Single Bond (3M ESPE), One-Step (Bisco), Clearfil SE bond (Kuraray), and Adper Prompt (3M ESPE). The shear test was performed by employing the design of a chisel-on-iris supported with a Teflon wall. The fractured dentin surface was observed with SEM to determine the failure mode.

The spectral appearance of the LED light curing unit was different from that of the halogen light curing unit in terms of maximum peak and distribution. The LED LCU (maximum peak in 465 nm) shows a narrower spectral distribution than the halogen LCU (maximum peak in 487 nm). With the exception of the Clearfil SE bond ($P < 0.05$), each 4 dentin bonding agents showed no significant difference between the halogen light-cured group and the LED light-cured group in the mean shear bond strength ($P > 0.05$).

The results can be explained by the strong correlation between the absorption spectrum of camphoroquinone and the narrow emission spectrum of LED. (J Kor Acad Cons Dent 29(6):504-514, 2004)

Key words : LED, Halogen, Dentin bonding agent, Dentin shear bond strength, Spectrum, Camphoroquinone

I. Introduction

The proper performance of a visible light-curing unit (LCU) is important for optimizing the physi-

cal properties of light-activated dental materials. Inadequate polymerization is associated with inferior physical properties, a higher solubility, a retention failure, and adverse pulpal responses caused by the residual unpolymerized monomers¹⁻³. Until now, halogen light curing has been the most widely used method for polymerizing resin-based composites. However, a halogen LCU has inherent limitations. For example, the halogen bulbs have a limited effective lifetime of approximately 100 hours⁴. It has been shown that

* Corresponding author: **Chung-Moon Um**

Department of Conservative Dentistry,
College of Dentistry, Seoul National University
28 Yoengun-dong, Chongro-gu, Seoul, Korea, 110-749
Tel : 82-2-2072-2651 Fax : 82-2-2072-3859
E-mail : umcmoon@snu.ac.kr

※ This study was supported by 04-2002-061, SNUH FUND.

the light delivered by halogen LCU diminishes in intensity with use mainly due to degradation of the halogen bulb and its reflector, blistering and cracking of the internal filter, and damage to the fiber-optic tips used to focus the light on the restorative material⁵. Some reports have shown that many halogen LCUs used by dental practitioners do not produce their optimum power output and that a reduced LCU output is generally the result of a lack of maintenance, such as changing the filter and /or the halogen bulb periodically, and checking the LCU's irradiance⁶⁻⁸.

Recently, solid state light emitting diode (LED) was proposed for curing light-activated dental materials⁹. Instead of the hot filaments used in halogen bulbs, LEDs use junctions of doped semiconductors for generating the lights¹⁰. The spectral output of blue LEDs fall mainly within the absorption spectrum of the camphoroquinone (CQ) photoinitiator, which is used in most dental composites. It was reported that an LED LCU has several advantages compared to a halogen LCU^{11,12}. The most remarkable things are an extended shelf-life and no decrease in output as the bulb ages. Less heat production and an enhanced portability due to the cordless features are also additional advantages of an LED LCU.

Several studies using LEDs to cure composite resin have been reported¹³⁻¹⁶. Unfortunately, little attention has been paid to the performance of LED LCU with a Dentin Bonding Agent (DBA). Therefore, the aim of this study was to compare the dentin shear bond strength of five DBAs, which were irradiated with an LED LCU and a halogen LCU. The hypothesis is that the dentin shear bond strength of a DBA irradiated with a LED LCU of similar intensity are comparable to those of a DBA irradiated with a halogen LCU. Using a precise power meter and a monochromator, the actual intensity and the spectrum through the wavelength of two LCU used in this study were also compared in order to obtain the basis for the optic characteristics for the hypothesis.

II . Materials and methods

1. Optic characteristics

The halogen LCU used in this study was a VIP (Bisco, Schaumburg, IL, USA), which has an adjustable light intensity, with a curved parallel, a fused glass fiber light guide with an 8 mm diameter tip. In this study, a $400 \text{ mW} \cdot \text{cm}^{-2}$ intensity mode was used to adjust to the intensity of the LED LCU. The LED LCU used in this study was an Elipar FreeLight (3M-ESPE, St Paul, MN, USA) with a curved, parallel, fused glass fiber light guide with an 8 mm diameter tip.

The power output for the LCUs was measured using a Molecron PM30 thermopile sensor connected to a Molecron EPM 1000 power meter (Molecron Detector Inc., Oregon, USA). The light guides were brought in direct contact with the detector when recording the measurements. The power output values were converted into power density values in $\text{mW} \cdot \text{cm}^{-2}$, by considering the area of the LCU light guide tip (8 mm diameter). This calculated total intensity was compared with the value reported by the manufacturers and the value measured with a simple hand-held radiometer (Model 100, Demetron, USA).

The spectra of the LCUs were measured using a MS 257 monochromator with an Instaspec IV CCD array detector (Oriol Ins., Stratford, USA). The real Y-axis unit value of each LCU's spectrum was determined through calculations using the total intensity value obtained by Molecron EPM 1000 power meter and the integrated sum of LCU's spectrum. The calculation procedures are as follows:

$$S \cdot Y \text{ nm} = I \text{ mW cm}^{-2}$$

$$Y = I \cdot S^{-1} \text{ mW cm}^{-2} \text{ nm}^{-1}$$

S: Integrated sum from the spectrum of either the halogen LCU or LED LCU

Y: real y-axis unit of the spectrum

I : Total Intensity

Each Y value was applied to the VIP and Elipar FreeLight spectrum, and the resulting spectral intensities of the two LCUs were compared in the range of 410-500 nm and 450-490 nm. The integration of the spectrum was performed using Origin 7.0 (OriginLab, USA).

2. Shear bond strength test

2-1. Specimen preparation

The 120 extracted caries-free human third molars were collected and stored for < 3 months under refrigeration in a 10% formalin solution until needed. The teeth were embedded in prefabricated acrylic molds using a self-curing resin. After waiting 5 minutes for the material to begin an exothermic reaction, the specimens were placed in distilled water. For the bond strength tests, the teeth were sectioned using a low-speed diamond saw (ISOMET, Buehler Co., IL, USA) under water irrigation through the mid-crown to expose the dentin surface. The exposed dentin surface was polished on a 500-grit SiC paper (Rotopol-V, Struers Co., Copenhagen, Denmark).

2-2. Bonding procedure

The prepared specimens were randomly divided into 10 groups of 12 teeth (five groups light-cured with halogen LCU; and five groups light-cured with LED LCU) (Table 2). The DBA was applied according to the manufacturers' protocol (Table 1). Light-curing of the DBA was performed at a distance of 1 mm from the light guide tip. A Teflon-coated metal Iris, 3 mm internal diameter and a 1.5 mm height, was used as a mold for the composite. The iris, which was placed in a holder, was pressed against the treated dentin surface and the cavity was filled with the same manufacturers' composite with the DBA (Table 1), which was then irradiated for 40 seconds with either the VIP or Elipar FreeLight. Light-curing was performed at direct contact with light guide tip and the upper surface of metal iris. The assembly was allowed to sit for an additional 4 minutes, and was then immersed in distilled water at room temperature.

2-3. Shear bond strength test and fracture surface analysis

All the specimens were stored in distilled water at room temperature for 24 hours prior to testing. They were then loaded in shear mode until fracture in a universal testing machine (Instron 4466, Instron Corp., Canton, MA, USA) at a crosshead speed of 1.0 mm/min using a knife-edged chisel that pressed as close as possible to the edge of the metal iris adjacent to the treated tooth surface. The technique of this study is shown schematically (Figure 1). The shear bond strength in Mpa was calculated by dividing the maximum load by the cross-sectional area of the bonded surface.

After the shear bond strength test, each fractured dentin specimen was trimmed and dried in a 37°C oven, and then placed on an SEM stub followed by a gold-sputter-coating and were observed on a SEM (JSM-840, JEOL, Japan) to assess microscopic failure patterns.

3. Statistical analysis

After ascertaining the level of normality using the Kolmogorov-Smirnov test, a student t-test

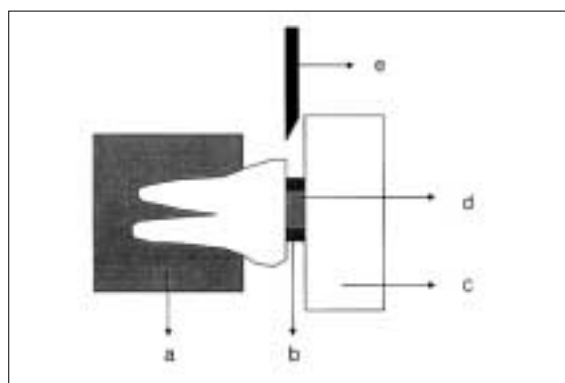


Figure 1. Schematic diagram of the shear bond strength test used in this study.

- a. Embedding resin
- b. Teflon-coated metal iris
- c. Teflon wall
- d. Composite
- e. Load application chisel

Table 1. Manufacturers, components and bonding procedures of the different dentin bonding systems and composites used in this study.

Bonding systems	Components	Bonding procedures	Composite resin
Scotchbond Multipurpose 3M ESPE (MP)	Primer : HEMA, polyalkenoic acid copolymer, water Adhesive : Bis-GMA , HEMA, initiator, amine	Etched with 35% phosphoric acid for 15s, rinsed, lightly dried with a gentle air stream to leave a moist surface, primer applied, dried with gentle air stream, adhesive applied, light-cured for 10s.	Z-100 (A2 shade)
Single Bond 3M ESPE (SB)	HEMA, Bis-GMA, dimethacrylates, polyalkenoic acid copolymer, photoinitiator, ethanol, water	Etched with 35% phosphoric acid for 15s, rinsed, lightly dried with a gentle air stream to leave a moist surface, adhesive applied in two consecutive coats, air-dried, light-cured for 10s	Z-100 (A2 shade)
One-Step Bisco (OS)	HEMA, BPDM, Bis-GMA, photoinitiator, acetone	Etched with 32% phosphoric acid for 15s, rinsed, lightly dried to leave the dentin moist, two coats of adhesive applied with agitation, air dried, light-cured for 10 s	Renew (A2 shade)
Clearfil SE Bond Kuraray (SE)	Primer: MDP,HEMA, hydrophilic DMA, CQ, N,N-Diethanol-p-toluidine, water Adhesive : MDP, Bis-GMA, hydrophobic dimethacrylate, HEMA, CQ, toluidine, silanated colloidal silica	Dried with light air, SE primer applied, wait 20s, evaporate with a light air flow, adhesive applied, gently air blow, light-cured for 10s	Clearfil AP-X (A2 shade)
Adper Prompt 3M ESPE (AP)	Liquid A : methacrylated phosphoric ester, Bis-GMA, initiators based on CQ, stabilizer Liquid B : water, HEMA, polyalkenoic acid, stabilizer	Aggressively mix each one drop from solution A and solution B for 5 s. the mixed adhesive applied and rubbed in the solution with a moderate finger pressure for 15s. gently dried, light-cured for 10s	Z-100 (A2 shade)

Abbreviation: Bis-GMA, bisphenyl-glycidyl-methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; BPDM, Biphenyl Dimethacrylate; DMA, Dimethacrylate; CQ, Camphoroquinone

Table 2. The groups examined in this study; Abbreviation.

	Halogen (VIP)	LED (Elipar Freelight)
Scotchbond Multi-Purpose	H-MP	L-MP
Single Bond	H-SB	L-SB
ONE-STEP	H-OS	L-OS
Clearfil SE Bond	H-SE	L-SE
Adper Prompt	H-AP	L-AP

was used to determine the shear bond strength differences between the halogen light-cured group and the LED light-cured group of each DBA. A two-way ANOVA test was performed to determine the interaction between the LCU and DBA. A frequency test of the fractured surface type was performed with a Fisher's exact test. A Mann-Whitney U-test was used to determine the DC differences between the halogen light-cured group and the LED light-cured group of each DBA. All statistic analyses were conducted at the 95% confidence levels using SPSS software (SPSS 10.0, SPSS Inc., USA).

III. Results

1. Optic characteristics

The power intensity of the LCUs used in this study was shown in Table 3. There was a differ-

ence among the intensities reported by the manufacturer, those measured using a simple hand-held radiometer, and those obtained using a Molecron EPM 1000 power meter and dividing the values by the area of light guide. The output ratio of the halogen LCU to LED LCU according to Manufacturer (approximately 1) and simple radiometer (1.13) was also different from the ratio obtained from the Molecron EPM 1000 power meter (1.72).

The spectral distributions of the LCUs used in this study are shown in Figure 2. The halogen LCU had a maximum peak at 487 nm and showed a broad spectrum. In contrast, the LED LCU had a maximum peak at 465 nm and showed a narrow spectrum around that peak. The area under each curve in Figure 3 represents the total intensity of the respective LCU.

The total intensity and spectral intensity of the LCUs are shown in Figure 3. The total intensity

Table 3. Light power measurement and conversion to intensity.

	Manufacturer (mW cm ⁻²)	Demetron (mW cm ⁻²)	EPM 1000 (mW)	Intensity calculated (mW cm ⁻²)
VIP (Halogen)	400	350	254	505
Elipar Freelight (LED)	About 400	310	148	294

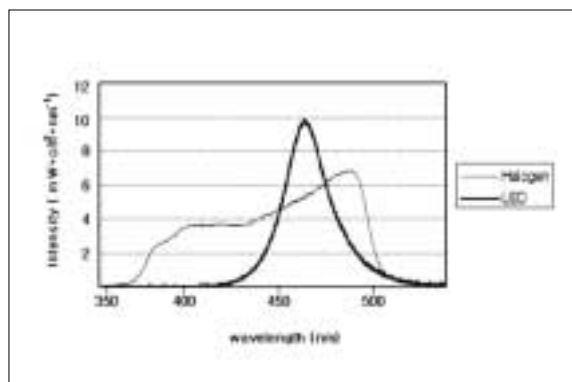


Figure 2. Spectral distributions of the two LCUs.

The LED LCU (maximum peak in 465 nm) shows a narrower spectral distribution than the halogen LCU (maximum peak in 487 nm). The area under each curve means the total intensity of the respective LCU.

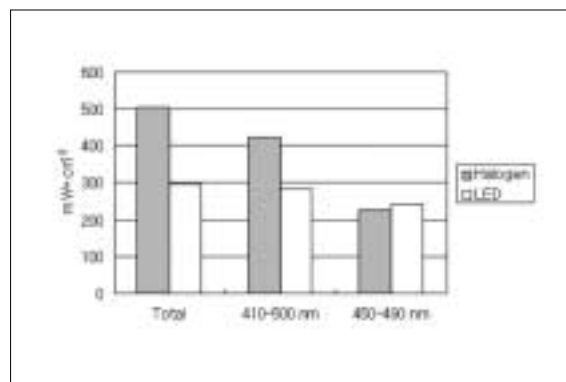


Figure 3. Spectral intensity of two LCUs used in this study.

Intensity of the halogen LCU reduced to 83.8%, 44.9% of the total intensity in 410-500 nm, 450-490 nm, respectively, whereas it was 96.2%, 82.6% for LED LCU.

of the LED LCU was 58.2% of the halogen LCU. In the 410–500 nm range, the output dropped to 423 mW · cm⁻² for the halogen LCU and 283 mW · cm⁻² for the LED LCU. In this spectral region, the LED LCU gave 66.9% of the output of the halogen LCU. 96.2% of the total output for the LED LCU was between 410–500 nm compared to 83.8% for the halogen LCU. The output of the halogen LCU and LED LCU in the range of 450–490 nm dropped to 227 mW · cm⁻² and 243 mW · cm⁻² respectively. 82.6% of the total output for the LED LCU was between 450 nm and 490 nm compared to 44.9% for the halogen LCU.

2. Shear bond strength test

The results of the shear bond strength are summarized in Table 4. MP, SB, OS and AP showed no statistically significant difference between the halogen light-cured group and the LED light-cured group in terms of the mean values of shear bond strengths ($P > 0.05$). For the SE, the shear bond strength of the halogen light-cured group was significantly higher than that of the LED light-cured group ($P < 0.05$). Two-way ANOVA showed that the factors, LCU ($P = 0.018$) and DBA ($P = 0.000$), have a statistically significant effect on the shear bond strength. Their interactions were also significant ($P = 0.009$).

The failure modes of the fractured dentin surface are shown in Figure 4. The fractured dentin surfaces were classified into 3 groups: Type I –

adhesive failure between the bonding resin and the hybrid layer, Type II – mixed failure (adhesive failure between bonding resin and hybrid layer + cohesive failure in bonding resin), Type III – partially cohesive failure in the composite resin. No specific evidence of dentin pull-out was observed in this study. The frequency of the failure modes among the DBAs as well as between the LCUs was similar ($P > 0.05$). SEM views of the fractured dentin surfaces are shown in Figures 5–7. Generally, the Type II mode (Figure 6), or mixed failure occupied a large portion, which was followed by the Type I mode (Figure 5). The Type III mode (Figure 7) occurred in only one case in the H-MP group.

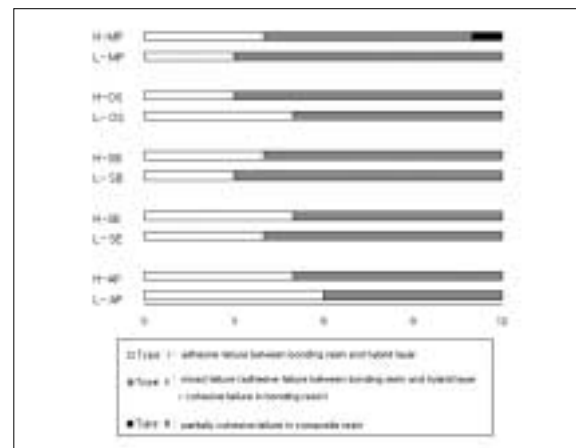


Figure 4. The failure mode.

There was no statistically significant difference in the frequency of the failure modes among the DBAs as well as between the LCUs ($P > 0.05$).

Table 4. Mean shear bond strength values of the bonding agents (MPa) irradiated with the halogen (VIP) and LED (Elipar Freelight).

DBA	Halogen mean \pm s.d	LED mean \pm s.d	p-value
SE	37.60 \pm 7.34 a	25.38 \pm 9.24 A, B	0.002 *
SB	25.32 \pm 8.63 b	26.98 \pm 5.37 A	0.577
MP	24.24 \pm 8.06 b	22.22 \pm 7.62 A, B	0.535
OS	20.91 \pm 8.02 b	21.29 \pm 5.62 A, B	0.892
AP	20.14 \pm 3.69 b	16.81 \pm 5.21 B	0.085

The same letters indicate no significant difference between the groups.

* Means a statistically significant difference.

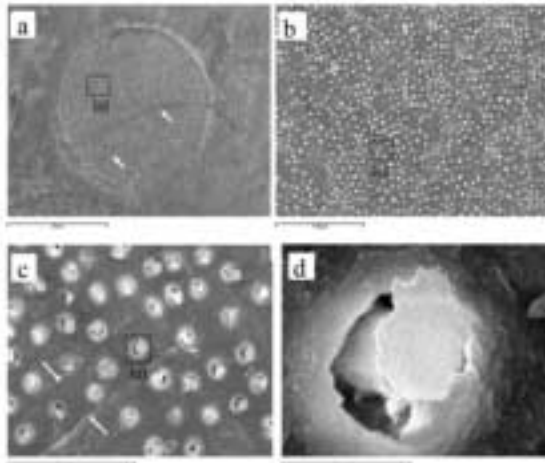


Figure 5. Adhesive failure.

a. Total fractured surface on the dentin side of a H-SE specimen ($\times 23$). b. Higher magnification of the sections of the fractured surface marked (b) in figure 6a ($\times 400$). c. Higher magnification of the sections of the fractured surface marked (c) in figure 6b ($\times 2000$). d. Higher magnification of the sections of the fractured surface marked (d) in figure 6c ($\times 20,000$). Shear force is suggested by the appearance of the fractured resin tag.

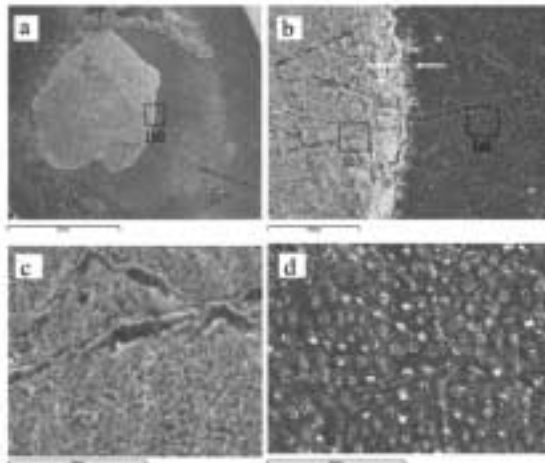


Figure 6. Mixed failure : adhesive failure + cohesive failure within the DBA.

a. Total fractured surface on the dentin side of a L-SB specimen ($\times 23$). b. Higher magnification of the fractured surface marked (b) in figure 7a ($\times 400$). c, d Higher magnification of the sections of the fractured surface marked (c), (d) in figure 7b ($\times 1000$). Figure 7c shows cohesive failure of the bonding resin. Figure 7d shows adhesive failure between the hybrid layer and the bonding resin.

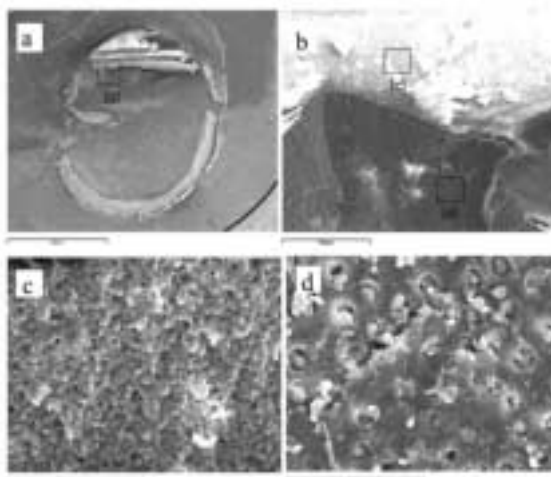


Figure 7. Partially cohesive failure within the composite.

a. Total fractured surface on the dentin side of a H-MP specimen ($\times 23$). Partially cohesive failure in the composite was observed in the upper portion. b. Higher magnification of the sections of the fractured surface marked (b) in figure 8a ($\times 400$). c, d. Higher magnification of the sections of the fractured surface marked (c), (d) in figure 8b ($\times 3000$).

IV. Discussion

It is difficult to compare the optical characteristics of the halogen LCU and the LED LCU based on the intensity reported by the manufacturer and that measured by a simple hand-held radiometer, as shown in Table 3 and Figures 3 and 4. The actual total intensity measured using the Molecron EPM 1000 power meter was different from that of the simple hand-held radiometer. The intensity of the halogen LCU was 1.72 times higher than that of the LED LCU with the Molecron, while it was 1.13 times with the simple radiometer (Table 3). Hansen & Asumussen¹⁷⁾ reported that the LCUs producing an adequate depth of cure could be classified as being good with one radiometer and poor with another. Several authors have also reported the inconsistency of the irradiance with the radiometers used in dental practice^{8,18)}. For example, Leonard *et al.*¹⁸⁾ reported that commercial radiometers should only be used for making a comparison of the initial baseline and the subsequent irradiance values of the LCUs over time. They also stated that the obtained irradiance values should not be considered absolute.

A simple commercial radiometer is also unsuitable for comparing the halogen LCU and LED LCU with a different spectral intensity. The halogen LCU, with a broad spectrum and a higher intensity value, can be overestimated to be superior to a narrow-spectrum LED LCU since the total intensity integrates the spectral intensity of each wavelength. Therefore, a comparison of the spectral intensity matching the absorption spectrum of the photoinitiator will facilitate a more meaningful result. Cook¹⁹⁾ and McCabe & Carnick²⁰⁾ reported the efficient wavelength range for the activation of CQ to be 410–500 nm with a peak of 470 nm. Nomoto²¹⁾ also reported the spectral range of 450–490 nm to be most effective in initiating the polymerization process and an accurate indicator of the light's efficiency. Therefore, the spectral intensity of the halogen LCU and LED LCU obtained in this study were compared in the two ranges, 410–500 nm and 450–490 nm.

The total intensity of the LED LCU was 58.2% of the halogen LCU (Figure 4). Concerning the total intensity, one can assume that the curing capacity of the LED LCU is only 58.2% of the halogen LCU. However, the intensity of the LED LCU was 66.9% of the halogen LCU in the 410–500 nm range, and it was higher by 107% in the 450–490 nm range. Moreover, the maximum emission spectrum of the LED LCU used in this study was 465 nm, which is relatively close to the maximum absorption spectrum of the photoinitiator CQ (Figure 2). This means that the photons emitted from the LED LCU have a higher probability of being absorbed by CQ than those emitted by the halogen LCU. The results in this study suggest that the LED LCU was more efficient in activating CQ in comparison to the halogen LCU in the 450–490 nm range.

Dickens and Milos²²⁾ performed a shear test study of the following designs, a chisel-on-iris, a chisel-on-composite cylinder, and a wire loop on the composite cylinder, and reported that there is a lower incidence of deep dentin failures with the chisel-on-iris design. They suggested the following reasons for the superiority of the chisel-on-iris design: (a) the applied load is closest to the interface, reducing the bending moments and peeling effects, (b) it provides a distributed load over a 180 half circle, thereby avoiding the occurrence of stress concentration at the point of loading, and (c) it does not produce surface flaws that can initiate fracture in the composite material itself. In this study, a modified shear test design from that of Dickens and Milos was used. The iris size was reduced to 3 mm from the original 4 mm and the Teflon wall supported the outer side of the iris (Figure 1). None of the specimens of the designs used in this study resulted in a deep dentin cohesive failure, as shown in Figures 4–7. This corresponds to the results reported by Dickens and Milos. Usually in a conventional shear test, the load applied to the dentin causes a tensile stress condition, resulting a failure path to divert into the dentin, which is a relatively weak in tension²³⁾. This condition leads to dentin cohesive failure. The supporting Teflon wall and the

iris used in this study might have solved the above problem. It is conceivable that the iris and the Teflon wall guided the true shear load as much as possible, which could have reduced the bending moment of the composite. However, the raggedness of the DBA around the metal iris opening, friction between the metal iris surface and the ground tooth surface, and between the metal iris surface and the Teflon wall, should be considered. This raggedness and friction possibly had some influence in increasing the bond strength value. Nonetheless, the "iris-on-chisel supported with the Teflon wall" design is assumed to be valuable because of the reproducing shear bond strength data without a dentin cohesive failure.

The majority of the light-activated composite resins and bonding resins contains CQ as the main photoinitiator. The absorbance of CQ strongly affects the initiation of the polymerization process. Therefore, the two LCUs with a similar intensity in the 450-490 nm range were assumed to have a similar bond strength and DC, because spectral intensity of this 450-490 nm was believed to be the most effective in activating CQ as stated previously. Consistently with the assumption, there was no statistically significant difference in the mean shear bond strength between the halogen light-cured group and the LED light-cured group in all of the DBAs except the SE. In addition, there was also no statistically significant difference in the DC between the halogen light-cured group and the LED light-cured group in all of DBAs.

Several studies have already demonstrated the polymerization effectiveness of the LED LCU with the resin-based composite¹³⁻¹⁶. For example, Fujibayashi *et al.*¹³ reported that in the same irradiance of 100 mWcm⁻², the depth of cure and the degree of monomer conversion of the LED LCU to be significantly higher than that obtained with the halogen LCU. Mills *et al.*¹⁴ compared the depth of cure of the dental composites with the LED LCU adjusted to 290 mWcm⁻² and the halogen LCU adjusted to 300 mWcm⁻². Under these conditions, the LED LCU cured the finefilled, microfilled, midfilled, and hybrid composites of medi-

um shades significantly deeper than the halogen LCU did. Stahl *et al.*¹⁵ compared the flexural strength of the LED LCU with an irradiance of 350 mWcm⁻² and the halogen LCU with an irradiance of 755 mWcm⁻², and reported no statistically significant difference. In a recent study, the LED LCU had only 78% of the irradiance of the halogen LCU, which were 661 mWcm⁻² and 851 mWcm⁻², respectively, and exceeded the curing depth¹⁶.

An explanation for the significantly different mean shear bond strength between the halogen light-cured group and the LED light-cured group in SE is unclear. One possible reason, the existence of an alternative photoinitiator in SE, can be implied. Some photoinitiators, such as 1-phenyl-1,2-propanedione (peak absorption of 410 nm), bisacylphosphine oxide, and triacylphosphine oxide (peak absorptions of 320 to 390 nm) fall outside of the absorption range of CQ and cause curing problems²⁴. In particular, in dentin bonding, the addition of a water-soluble photoinitiator, 2-hydroxyl-3-(3, 4-dimethyl-9-oxo-9H-thioxanthene-2-yloxy)-N,N,N-trimethyl-1-propanaminium chloride (QTX) (peak absorption of 402 nm) to self-etching primers has been reported to be effective in improving the adhesion of the composite resin to dentin²⁵. The use of these photoinitiators is due to a rate-limiting effect of the strong, yellow color of the CQ, which restricts its use: higher concentrations do not improve the curing rate²⁶. QTX was used because of the limitation of CQ but not to initiate the polymerization of the diffused monomers inside the dentin in the presence of water²⁵. For a DBA, which contains an alternative photoinitiator to CQ with a different absorption spectrum, the use of the narrow-spectrum LED LCU, which is mostly effective in activating CQ, can result insufficient polymerization of the bonding resin in comparison to the broad-spectrum halogen LCU. Insufficient polymerization will result in lower mechanical properties. Generally, adhesive resins have favorable mechanical properties when they are cured sufficiently²⁷. Takahashi *et al.*²⁸ reported that the mechanical properties of cured adhesive resins could promote initial bond performance of the

adhesive. This means that the inferior mechanical properties of the bonding resin due to insufficient polymerization can lead to a weaker bond strength. Further study on the dentin shear bond strength in each DBA regarding the light curing source will be necessary to obtain more details.

V. Conclusion

In this study, most of the DBAs irradiated with a LED LCU showed comparable dentin shear bond strength to those irradiated with a halogen LCU, but the L-SE had significantly lower mean dentin shear bond strength than the H-SE. This can be explained by a strong correlation between the absorption spectrum of CQ and the narrow emission spectrum of LED and by a possible existence of alternative photoinitiator.

LED LCU has been introduced in the dental market as an effective LCU for polymerization of composite resin, which is supported by several studies. Unfortunately, its curing effectiveness on the advancing DBAs is still unclear. Therefore, further study on the mechanical properties and clinical performance of the DBAs cured with an LED will be needed in order to confirm its practical availability.

References

- Council on Dental Materials, Instruments, and Equipment. Visible light-cured and activating units. *J Am Dent Assoc* 110(1):100-102, 1985.
- Blankenau RJ, Kelsey WP, Powell GL, Shearer GO, Barkmeier WW, Cavel WT. Degree of composite resin polymerization with visible light and argon laser. *Am J Dent* 4(1):40-42, 1991.
- Ferracane JL, Mitchem JC, Condon JR, Todd R. Wear and marginal breakdown of composites with various degrees of cure. *J Dent Res* 76(8):1508-1516, 1997.
- Rueggeberg FA, Twigg SW, Caughman WF, Khajotia S. Life-time intensity profiles of 11 light-curing units. *J Dent Res* 75:380, Abstr. No. 2897, 1996.
- Friedman J. Variability of lamp characteristics in dental curing lights. *J Esthet Dent* 1(6):189-190, 1989.
- Martin FE. A survey of the efficiency of visible light curing units. *J Dent* 26(3):239-243, 1998.
- Barghi N, Berry T, Hatton C. Evaluating intensity output of curing lights in private dental offices. *J Am Dent Assoc* 125(7):992-996, 1994.
- Miyazaki M, Hattori T, Ichiishi Y, Kondo M, Onose H, Moore BK. Evaluation of curing units used in private dental offices. *Oper Dent* 23(2):50-54, 1998.
- Mills RW. Blue light emitting diodes: Another method of light curing? *Br Dent J* 178:169, 1995.
- Nakamura S, Mukai T, Senoh M. Candela-class high brightness InGaN/AlGaN double heterostructure blue-light-emitting diodes. *Appl Phys Lett* 64:1687-1689, 1994.
- Duke ES. Light-emitting diodes in composite resin photopolymerization. *Compend Contin Educ Dent* 22(9):722-725, 2001.
- Yap AU, Soh MS. Thermal emission by different light-curing units. *Oper Dent* 28(3):260-266, 2003.
- Fujibayashi K, Ishimaru K, Takahashi N, Kohno A. Newly developed curing unit using blue light-emitting diodes. *Dent Jap* 34: 49-53, 1998.
- Mills RW, Jandt KD, Ashworth SH. Dental composite depth of cure with halogen and blue light emitting diode (LED) technology. *Br Dent J* 186(8):388-391, 1999.
- Stahl F, Ashworth SH, Jandt KD, Mills RW. Light-emitting diode (LED) polymerization of dental composites: flexural properties and polymerization potential. *Biomaterials* 21(13):1379-1385, 2000.
- Mills RW, Uhl A, Jandt KD. Optical power outputs, spectral and dental composite depths of cure, obtained with blue light emitting diode (LED) and halogen light curing units (LCUs). *Br Dent J* 193(8):459-463, 2002.
- Hansen EK, Asumussen E. Reliability of three dental radiometers. *Scand J Dent Res* 101(2):115-119, 1993.
- Leonard DL, Charlton DG, Hilton TJ. Effect of curing-tip diameter on the accuracy of dental radiometers. *Oper Dent* 24(1):31-37, 1999.
- Cook, W.D. Spectral distribution of dental photopolymerization sources. *J Dent Res* 61: 1436-1438, 1982.
- McCabe JF, Carnick TE. Output from visible-light activation units and depth of cure of light-activated composites. *J Dent Res* 68(11):1534-1539, 1989.
- Nomoto R. Effect of light wavelength on polymerization of light-cured resins. *Dent Mater J* 16(1):60-73, 1997.
- Dickens SH, Milos MF. Relationship of dentin shear bond strengths to different laboratory test designs. *Am J Dent* 15(3):185-192, 2002.
- Versluis A, Tantbirojn D, Douglas WH. Why do shear bond tests pull out dentin? *J Dent Res* 76(6):1298-1307, 1997.
- Stansbury, J.W. Curing dental resins and composites by photopolymerization. *J Esthet Dent* 12:300-308, 2000.
- Hayakawa T, Kikutake K, Nemoto K. Effectiveness of the addition of water-soluble photoinitiator into the self-etching primers on the adhesion of a resin composite to polished dentin and enamel. *Dent Mater J* 18(3):324-333, 1999.
- Burgess JO, DeGoes M, Walker R, Ripps AH. An evaluation of four light-curing units comparing soft and hard curing. *Pract Periodontics Aesthet Dent* 11(1): 125-132, 1999.
- Koliniotou-Kubia E, Jacobsen PH. The effect of irradiation time on the physical properties of light-cured resins. *Clin Mater* 6(1):21-28, 1990.
- Takahashi A, Sato Y, Uno S, Pereira PN, Sano H. Effects of mechanical properties of adhesive resins on bond strength to dentin. *Dent Mater* 18(3):263-268, 2002.

국문초록

LIGHT EMITTING DIODE로 광조사한 상아질 접착제의 상아질 전단접착강도와 중합률에 관한 연구

김선영 · 이인복 · 조병훈 · 손호현 · 김미자 · 석창인 · 엄정문*

서울대학교 치과대학 치의학과 치과보존학교실

LED 광중합기(Elipar FreeLight, 3M-ESPE)와 할로겐 광중합기(VIP, Bisco)로 광조사한 수종의 상아질 접착제에 대해서 상아질 전단접착강도를 비교하였다. 또한 이번 연구의 광학적 근거를 얻기 위해 두 광중합기의 파장에 따른 광강도와 스펙트럼을 비교하였다. 이번 연구에서 사용된 상아질 접착제는 Scotchbond Multipurpose (3M ESPE), Single Bond (3M ESPE), One-Step (Bisco), Clearfil SE Bond (Kuraray), Adper Prompt (3M ESPE) 이다.

VIP는 487 nm에서 최대정점을 가지는 넓은 스펙트럼의 분포를 보이는 반면에, Elipar FreeLight는 465 nm에서의 최대정점을 중심으로 좁은 스펙트럼의 분포를 보였다. Clearfil SE bond를 제외하고 할로겐과 LED로 광조사한 각 상아질 접착제에서 상아질 전단접착강도값의 유의성 있는 차이는 보이지 않았다 ($P > 0.05$). 이러한 결과는 camphoroquinone의 흡수스펙트럼과 LED의 좁은 영역의 스펙트럼사이에 강한 연관성으로 설명할 수 있다.

주요단어 : LED 광중합기, 할로겐 광중합기, 상아질 접착제, 상아질 전단접착강도, 스펙트럼, Camphoroquinone