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Light-curing efficiency of dental adhesives by gallium nitride violet-laser diode determined in terms of ultimate micro-tensile strength

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Abstract. The purpose of this study was to evaluate whether violet-laser diode (VLD) can be used as light-curing source. The ultimate (micro-)tensile strength (μ TS) of three adhesives was determined when cured by VLD in comparison with curing by two different types of commercial LED light-curing units. One VLD (VLM 500) and two LED units (Curenos and G-Light Prima) were used to cure the adhesive resin of the two-step self-etch adhesives Clearfil SE Bond, Tokuso Mac Bond II, and FL-Bond II. A 0.6-mm thick acrylic mould was filled with adhesive resin and cured for 60 s. After 24-h water storage, specimens were trimmed into an hourglass shape with a width of 1.2 mm at the narrowest part, after which the μ TS was determined ($n = 10$). In addition, the light transmittance of each adhesive was characterized using a UV-vis-NIR spectrometer. No significant difference in curing efficiency between VLD and LED were observed for both Tokuso Mac Bond II and FL-Bond II ($p > 0.05$). For Clearfil SE Bond, the μ TS of VLD-cured specimens was higher than that of the specimens cured by the LED Curenos unit ($p < 0.05$). Spectrometry revealed that this marked difference must be attributed to a different light transmittance of Clearfil SE Bond for visible blue light versus for the lower area of UV and visible violet light. In conclusion, A GaN-based violet laser diode can be used as light-curing source to initiate polymerization of dental resins.

Keywords: Violet laser diode, light-curing unit, dental adhesive, ultimate tensile strength, UV-vis-NIR spectra, photo-initiator

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1. Introduction

Both dental patients and dentists profit from the further advancement of dental composites and dental adhesive technology. Bonding the restorative material directly to the remaining sound tissue preserves tooth tissue, eventually prolonging the lifetime of teeth. In addition, besides restoring a tooth's function and anatomy, a composite-layering technique enables dentists to nearly invisibly reconstruct teeth, thereby fulfilling an increasing patient's demand [1,2].

Handling of composite by the dentist is facilitated much by the ability to enable composite to set 'on command'. In this regard, also light-curing units have evolved greatly. Blue light-emitting diode (LED) technology was invented by Nakamura et al. in 1991 [3], and the first LED light-curing units became commercially available in 2000 [4,5]. Their wavelength emission ranges between 450 and 500 nm [6], which closely fits the absorption spectrum of the camphorquinone-amine (CQ-amine) photo-initiator system that is mostly used in dental light-curing materials. This CQ-amine photo-initiator absorbs blue light with a wavelength peak at 468 nm. In addition, units can be cordless, when they are equipped with a rechargeable battery, and therefore today's LED-curing devices are portable and very practical [7]. For all these reasons, LED units currently replace the traditional quartz-tungsten-halogen (QTH) light-curing units [8].

However, due to the enhanced demand for dental esthetics, 'bleaching-white' composites have been developed to match very light shades. In order to avoid composite yellowing by residual (yellow-colored) CQ, alternative photo-initiators with different absorption spectra were added to dental composites and adhesives [9–16]. Unlike CQ-amine, those photo-initiators possess an absorption peak at a wavelength between the ultraviolet-A and visible violet region (360–420 nm) [10,13,17,18], and are therefore not compatible with blue-LED light-curing units.

In 1996, Nakamura et al. also invented the gallium nitride (GaN) violet-laser diode (VLD) [19], which was soon utilized widespread as light source for high-speed and multilayer recording systems such as Blu-ray Disc and HD-DVD systems [20]. This VLD technology also appears promising for medical applications, such as to cut soft tissue [21], for laser acupuncture [22], tooth whitening [23], and the detection of dental caries [26]. One has also suggested that the wavelength of violet-laser diode is suitable to cure dental adhesives/composites that include an CQ-amine alternative photo-initiator system.

The purpose of this study was to evaluate the effect of VLD as light-curing source on the ultimate (micro-)tensile strength (μ TS) using three commercial dental adhesives, in comparison with that of two different types of commercial LED light-curing units. The null hypothesis tested in this study was that VLD does not cure dental adhesives more effectively than LED light-curing units.

2. Materials and methods

2.1. Light-curing sources

The three light-curing sources tested in this study are listed in Table 1. VLM 500 (Sumitomo Electric Industries, Co., Ltd., Yokohama, Japan) is a VLD device. The VLD module consists of seven fiber-coupled violet-laser diodes and bundled silica-based optical fibers. The emission spectrum of the VLM 500 unit, as determined using a spectrometer (USB-4000, Ocean Optics, Dunedin, FL, USA) at 0.2 nm intervals, is shown in Fig. 1. The unit has a stimulated emission peak at 409.1 nm with an emission range of 402–416 nm and a full-width-at-half-maximum of 7 nm. It produces continuous waves at an output power ranging from 10 mW to 1 W. The light of VLM 500 (Sumitomo Electric Industries) was delivered

Table 1
Light sources used in this study

Light source (manufacturer)	λ_{\max} (nm)	Curing mode	Power density (mW/cm ²)	Time (s)	Diameter (mm)	Total energy (J)
VLM 500 (Sumitomo Electric Industries, Yokohama, Japan)	409.1	–	1150	60	8	137.3
Curenos (Shofu, Kyoto, Japan)	462.9	–	1550	60	8	185.1
G-Light Prima (GC, Tokyo, Japan)	402.9, 462.9	Normal	1150	60	8	137.3

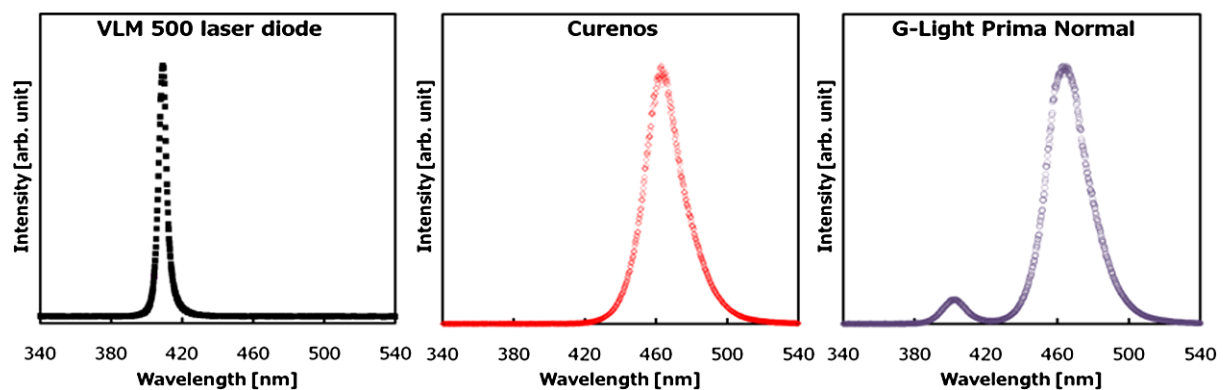


Fig. 1. Spectral characters of curing light source used in this study. (Colors are visible in the online version of the article; <http://dx.doi.org/10.3233/BME-2012-0682>.)

via an optical fiber with a core diameter of 800 nm, and was used at a distance of 16 mm from the tip of the fiber to the surface so that an irradiated surface of 8 mm in diameter was obtained [27].

As controls, two different types of LED light-curing units were used. The unit Curenos (Shofu, Kyoto, Japan) is a high-power blue-LED light-curing unit, while the unit G-Light Prima (used in ‘Normal’ mode; GC, Tokyo, Japan) is a high-power light-curing unit that contains both blue and violet LEDs (Table 1 and Fig. 1) [14]. A tapered light probe with a 8-mm diameter was connected to both the Curenos and G-Light Prima unit.

The light power of all light sources tested was measured using a laser powermeter (Ophir Optics, Jerusalem, Israel), and the power density and total energy value calculated (Table 1). The power output of VLM 500 was adjusted to the same power density (1150 mW/cm²) of the G-Light Prima unit.

2.2. Specimen preparation and μ TS measurement

The three dental adhesives tested in this study are shown in Table 2. Specimen preparation and the procedure of μ TS testing are schematically drawn in Fig. 2. An acrylic frame with a 9-mm inner diameter and a 0.6-mm height was attached onto a glass slide; the mould was then filled with the adhesive resin of the adhesives tested and pressed with a second glass slide with a 1-mm thickness, prior to being light-cured for 60 s through the top glass slide using the light sources tested. The cured adhesive disc was then carefully removed from the acrylic frame, and immersed in 37°C tap water for 24 h, while shielded from light. After water storage, the discs were trimmed into an hourglass shape with a width of

Table 2
Dental adhesives used in this study

Product	Manufacturer	Components	Batch No.
Clearfil SE Bond (Bond)	Kuraray Medical, Kurashiki, Japan	MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photoinitiator, aromatic tertiary amine, silanated colloidal silica	1323AA
FL-Bond II	Shofu, Kyoto, Japan	S-PRG filler, UDMA, TEGDMA, HEMA, others (photoinitiator)	040942
Tokuso Mac-Bond II	Tokuyama Dental, Tokyo, Japan	MAC-10, HEMA, Bis-GMA, TEGDMA, camphorquinone	026029

Notes: Bis-GMA: bisphenol A diglycidyl methacrylate, MAC-10: 11-methacryloyloxy 1,1'-undecanedicarboxylic acid, MDP: 10-methacryloyloxydecyl dihydrogenphosphate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, S-PRG filler: surface reaction type pre-reacted glass-ionomer filler.

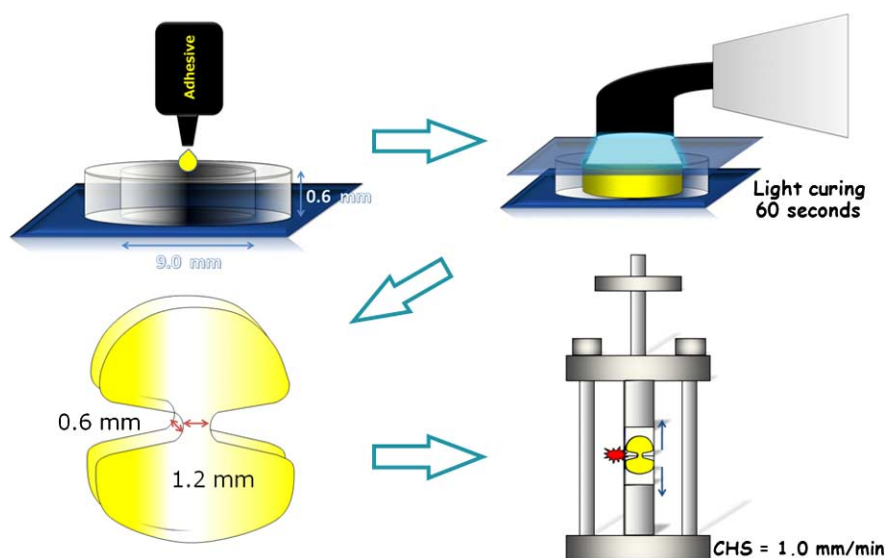


Fig. 2. Schematic illustrating specimen preparation and μ TS testing. (Colors are visible in the online version of the article; <http://dx.doi.org/10.3233/BME-2012-0682>.)

about 1.2 mm at the narrowest part, using a 1:5 high-speed motor handpiece (Ti-Max X95L, Nakanishi Inc., Kanuma, Tochigi, Japan) equipped with a super-fine cylindrical diamond bur (Diamond Point SF 114, Shofu) under continuous water spray. The width and thickness of the narrowest specimen part was measured by a digital caliper (Mitutoyo, Tokyo, Japan) to the nearest 0.001 mm. Thereafter, the microspecimens were attached to a Bencor Multi-T device (Danville Engineering, San Ramon, CA, USA) with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Tochigi, Japan), and the μ TS measured with a universal testing machine (Tensilon RTC-1150-TSD, Orientec, Tokyo, Japan) at a cross-head speed of 1.0 mm/min. The μ TS was expressed in MPa, as derived from dividing the imposed force (N) by the specimen area at the narrowest part (width \times thickness in mm^2).

Finally, the data were analyzed using two-way ANOVA and Tukey's HSD at a 5% level using IBM SPSS 18 statistical software (SPSS Inc., Chicago, IL, USA).

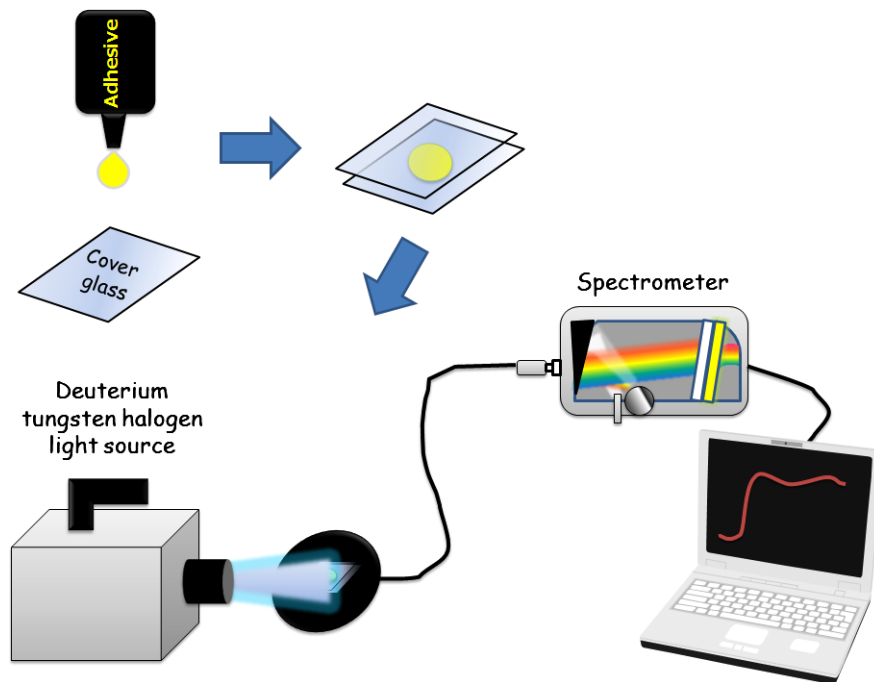


Fig. 3. Schematic illustrating the study set-up regarding the spectral analysis of dental adhesives. (Colors are visible in the online version of the article; <http://dx.doi.org/10.3233/BME-2012-0682>.)

2.3. Spectral analysis of light transmittance through the dental adhesives

The study protocol of the spectral analysis of dental adhesives is schematically shown in Fig. 3. A deuterium tungsten halogen light source (DH-2000, Ocean Optics, Dunedin, FL, USA) was connected to a UV-vis-NIR spectrometer (USB4000, Ocean Optics) using two optical fibers separated by a sample holder. One drop of the unfilled resin was placed onto a thin glass plate (thickness 0.15 mm; Matsunami Glass Ind., Ltd., Kishiwada, Osaka, Japan), covered with another glass plate, and gently compressed using finger pressure. Immediately afterwards, each resin sample was placed in the sample holder and irradiated by the deuterium tungsten halogen light (DH-2000; Ocean Optics). The light-transmission spectra through each adhesive were acquired in 0.2 nm increments.

3. Results

3.1. μ TS

The mean μ TS is graphically presented for each experimental group in Fig. 4. Two-way ANOVA revealed that both the 'adhesive' ($p < 0.001$) and the 'light-curing unit' ($p = 0.002$) significantly influenced the μ TS, while the interaction between both variables appeared not significant ($p = 0.123$). No significant difference in μ TS was found among the three light-curing sources for both FL-Bond II and Tokuso Mac Bond II ($p > 0.05$). On the other hand, the μ TS obtained using the VLM 500 VLD unit was significantly higher than that obtained with the Curenos LED unit for Clearfil SE Bond ($p = 0.032$). No

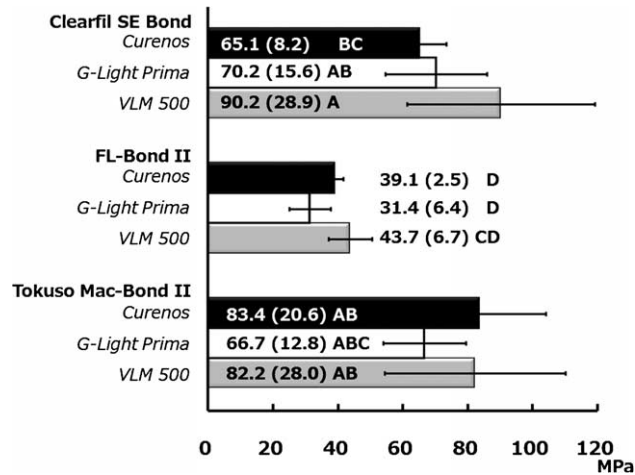


Fig. 4. Ultimate micro-tensile strength (μ TS) per group. Results with the same letter are not statistically significantly different (Tukey HSD test; $p > 0.05$).

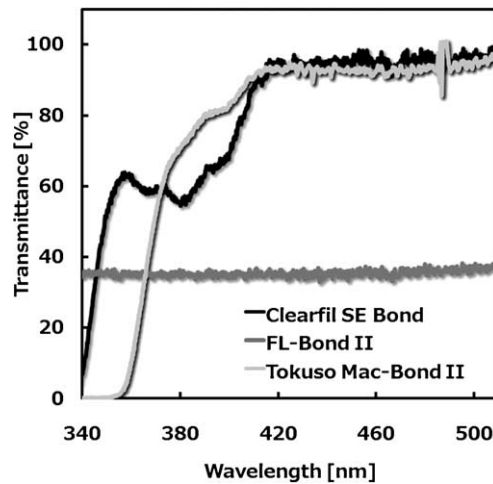


Fig. 5. Light-transmittance spectra of each adhesive.

significant difference in μ TS was found between the Curenos and G-Light Prima (Normal mode) LED units, and between the G-Light Prima (Normal mode) LED and the VLM 500 VLD units ($p > 0.05$).

Comparing the adhesives, ANOVA revealed that the μ TS of FL-Bond II was significantly lower than that measured for both Clearfil SE Bond and Tokuso MacBond II ($p < 0.001$). There was no significant difference in μ TS between the adhesive resin of Clearfil SE Bond and Tokuso MacBond II ($p = 0.868$).

3.2. Spectral analysis

Light-transmittance spectra of each adhesive are shown in Fig. 5. For Clearfil SE Bond, the spectrum was distinctive with three inverse peaks at 366, 381 and 396 nm (asterisks). The reduction rates of light transmittance at 409 nm (emission peak of VLD) and at 463 nm (emission peak of LED) were 12.8% and 4.7% for Clearfil SE Bond, 64.5% and 65.3% for FL-Bond II, and 9.7% and 8.1% for Tokuso Mac-Bond II, respectively.

4. Discussion

The purpose of this study was to evaluate whether commercially available dental adhesives can be cured by a violet-laser diode (VLD) light source. The curing efficiency was determined in terms of ultimate micro-tensile strength (μ TS) of the adhesives. This method has recently been employed as an assessment to measure the mechanical strength of a dental adhesive [28–32], since it can be correlated directly to the micro-tensile bond strength to tooth tissue, differ to the Young's modulus and nano-indentation value [33].

The high-power LED light-curing unit Curenos has been reported to emit light following a spectrum in the visible blue-light region that is similar with, but narrower than that emitted by a conventional quartz–tungsten–halogen (QTH) light-curing unit [14,34]. Although the power density of the LED Curenos unit was higher than that of the VLM 500 unit, the μ TS of the adhesive resin of Clearfil SE Bond was significantly lower when cured by the LED Curenos unit than by the VLM 500 unit ($p < 0.05$). The results indicated that Clearfil SE Bond can be cured more efficiently by VLD than by blue-LED light, by which the null hypothesis tested must be rejected. Following the spectral analysis, the reduction in light transmittance at 463 nm was 2.7 times smaller than that at 409 nm. Reduction in light transmittance reflects the total amount of light absorbance, reflection, and scattering. This finding may indicate that Clearfil SE Bond absorbs violet light more efficiently than blue light.

On the other hand, the μ TS of Clearfil SE Bond was not significantly different when cured by the VLM 500 unit than by the LED G-Light Prima unit ($p > 0.05$). The G-Light Prima unit utilizes a combination of four LEDs, of which three emit blue light and one violet light [14]. This type of light-curing unit is categorized as a 'third-generation' LED light-curing unit, and has been produced to be also compatible with the absorption range of CQ-amine alternative photo-initiators [10,35]. Since Clearfil SE Bond can absorb violet light efficiently, this type of light-curing unit might thus be more efficient than a solely blue-LED light-curing unit.

Following the spectral analysis, Clearfil SE Bond showed three inverse absorption peaks at 366, 381 and 396 nm. This typical peak appearance is similar to that of the Lucirin TPO photo-initiator system [10,13,17,18,36,37]. This possibly indicates that Lucirin TPO is included in the adhesive resin of Clearfil SE Bond as supplementary photo-initiator. Moreover, the bond strength of Clearfil SE Bond cured by a blue-LED light-curing unit has been reported to be significantly lower than when cured by a QTH light-curing unit [38]. By the broader emission spectrum of QTH, the possibly included CQ-amine alternative photo-initiator (with an absorption peak at the low 400-nms wavelength range) must be reached better.

For both the adhesives FL-Bond II and Tokuso Mac Bond II, no difference in reduction of light transmittance was recorded between 409 and 463 nm. However, the spectrum character of each adhesive differed significantly. Indeed, the light transmittance of FL-Bond II was below 40% at each wavelength, and the spectrum did not show any particular shape. FL-Bond II contains specific pre-reacted glass-ionomer filler, being referred to as S-PRG [39]. This S-PRG filler was included in the adhesive with the objective to release and recharge fluoride [40]. However, the presence of this filler may also reduce the transparency of the adhesive itself. The lower light transmittance of FL-Bond II may therefore be ascribed to scattering and/or reflection of light caused by the relatively high opacity of the adhesive.

According to the manufacturer's information, both FL-Bond II and Tokuso Mac-Bond II contain a CQ-amine photo-initiator system, but no alternative photo-initiator. It has been reported that the absorption degree of CQ at 410 nm was only 1/5.4 of that at 470 nm [41]. Regardless of the difference in absorption, the μ TS of both FL-Bond II and Tokuso Mac Bond II when cured using the VLM 500 unit was almost similar as when the adhesives were cured with the LED Curenos and G-light Prima light-curing units. In

our previous study, the μ TS of a 1% CQ-containing unfilled resin that was cured with the LED G-Light Prima unit used in 'PL mode' (only emission by the violet LED with an output of 52 mW/cm²), was significantly lower than when the resin was cured with the LED Curenos unit and the LED G-Light Prima unit used in 'Normal mode' [42]. However, the μ TS did not differ when the resin was cured either using the VLM 500 unit, the LED Curenos unit or the LED G-Light Prima unit used in 'Normal mode' [42]. These data therefore suggested that the lower absorption coefficient of CQ for violet light can be compensated by increasing the power density.

In order to use violet-laser diodes for light-curing dental materials, further study is definitely needed to determine the most optimal conditions, as for example the power density, irradiation time and photo-initiator concentration. Furthermore, since the emitted wavelength is near the ultra-violet region, any potential side-effects of this laser as medical device should be evaluated as well.

5. Conclusion

A GaN-based violet-laser diode can be used to initiate polymerization of dental resins as alternative to blue-light LED curing. In particular, this laser could cure adhesives like Clearfil SE Bond that contain besides the common CQ-amine photo-initiator system an alternative photo-initiator that is sensitive to the low 400-nms wavelength range, more effectively than a blue-light LED unit.

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