Dental composite depth of cure with halogen and blue light emitting diode technology

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Objectives To test the hypothesis that a blue light emitting diode (LED) light curing unit (LCU) can produce an equal dental composite depth of cure to a halogen LCU adjusted to give an irradiance of 300 mWcm⁻² and to characterise the LCU's light outputs.

Materials and methods Depth of cure for three popular composites was determined using a penetrometer. The Student's *t* test was used to analyse the depth of cure results. A power meter and a spectrometer measured the light output.

Results The spectral distribution of the LCUs differed strongly. The irradiance for the LED and halogen LCUs were 290 mWcm⁻² and 455 mWcm⁻², when calculated from the scientific power meter measurements. The LED LCU cured all three dental composites to a significantly greater (P < 0.05) depth than the halogen LCU.

Conclusions An LED LCU with an irradiance 64% of a halogen LCU achieved a significantly greater depth of cure. The LCU's spectral distribution of emitted light should be considered in addition to irradiance as a performance indicator. LED LCUs may have a potential for use in dental practice because their performance does not significantly reduce with time as do conventional halogen LCUs.

Curing of dental composites with blue light was introduced in the 1970s. The source of blue light is normally a halogen bulb combined with a filter, so that blue light in the 410 nm – 500 nm region of the visible spectrum is produced. Light in this range of wavelengths is the most effectively absorbed by the camphorquinone photoinitiator² that is present in the resin component of light activated dental composites. The light causes excitation of the camphorquinone, which in combination with an amine produces free radicals. This results in polymerisation of resin monomers at the molecular scale. Macroscopically, the dental composite hardens, typically after light exposure times ranging from 20 s to 60 s.

The blue light is delivered to the dental treatment area using various types of light guides. These guides may be fused rigid glass fibre bundles or moulded polymer guides. Some guides use a flexible pipe containing a transparent liquid to transmit the light.

Although halogen bulb based light curing units (LCUs) are most commonly used to cure dental composites, this technology has inherent drawbacks. Halogen bulbs have a limited effective lifetime

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REFEREED PAPER Received 12.10.98; accepted 18.02.99 © British Dental Journal 1999; 186: 388–391 of around 50 hours.³ The bulb, reflector and filter degrade over time due to the high temperatures produced, leading to a reduction in light output. The result is a reduction of the LCU's effectiveness to cure dental composites.⁴ The clinical implication of this for the dentist is a negative effect on the physical properties of composites with an increased risk of premature failure of restorations. Martin *et al.*⁵ and others^{4,6} have shown that many halogen LCUs used by dental practitioners do not produce their optimum power output. A reduced output of LCUs is due to a lack of maintenance, such as changing the filter and/or the halogen bulb from time to time, and checking the LCU's irradiance. The lower effective limit of irradiance for halogen technology based LCUs used in dental practice has been suggested to be 300 mWcm⁻².^{7,8} Some halogen LCUs available presently exceed an irradiance of 1000 mWcm⁻².

Recently, other curing methods such as laser and xenon arc sources have been used in clinical practice with the advantage of a reduced curing time. These devices have a more complex construction and are more costly compared with halogen sources. In addition, lasers require stringent additional safety precautions.

Light emitting diodes (LEDs), such as those encountered as indicators in car dashboards, have lifetimes of over 10,000 hours and undergo little degradation of light output over this time, a distinct advantage when compared with halogen bulbs. In addition, LEDs require no filters to produce blue light. LEDs are very resistant to shock and vibration, and their relatively low power consumption makes them suitable for portable use. Low power blue LEDs based on silicon carbide technology have been available for many years. 10 With a power output of 7 μW per LED, however, these were too weak to be considered for curing resin-based materials. In 1995, more powerful 3 mW blue LEDs based on gallium nitride technology were developed. 11 This improvement represented a more than 400-fold increase in power, compared with silicon carbide technology. The spectral output of these blue LEDs falls mainly within the absorption spectrum of the camphorquinone photoinitiator (400 nm-500 nm) of most dental composites.

Based on these developments one of the authors proposed blue light emitting diodes for curing composites in dentistry. ¹² The long lifetime and consistent light output of LEDs compared with halogen sources promise the dentist the potential for sustained quality of curing.

In a study in 1996, blue light produced by 61 LEDs with a typical peak wavelength of 450 nm, was focused with a lens to a spot size of approximately 8 mm onto dental composite samples. ¹³ An irradiance of 100 mWcm⁻² was produced by this method and compared with a halogen LCU also adjusted to give an irradiance of 100 mW cm⁻². No significant difference in depth of cure or Knoop hardness values was found in the samples cured with the halogen or the LED LCU, respectively. In a later study, ¹⁴ a 61 LED source using 470 nm typical peak wavelength LEDs was used. This LED source produced a depth of cure significantly larger than the halogen and a 450 nm

LED source, although all the sources were adjusted to give an irradiance of 100 mWcm⁻². The degree of monomer conversion to polymer assessed by Fourier transform infrared (FTIR) spectroscopy was found to be greater using the 470 nm LEDs than the halogen source at the same irradiance (100 mWcm⁻²).

In the present study, we aimed to assess the performance of a LED LCU that produces irradiance relevant to clinical applications. We aimed to accomplish similar depths of cure of dental composites, to those obtained with a halogen LCU having a minimum level of irradiance of 300 mWcm $^{-2}$.7,8

Materials and methods

A Coltolux 4 halogen LCU (Coltene/Whaledent Inc, Mahwah NJ, USA), was used in this study. The light assembly from this unit was used separately from the base power unit. This enabled a variable bench power supply unit to be connected to adjust the output of the halogen lamp. A second variable bench power supply was used to power the halogen LCUs cooling fan. Figure 1 shows the light curing handpiece of the Coltolux 4 LCU.

The Coltolux 4 halogen LCU has a built-in radiometer with a digital readout to display the irradiance in mWcm⁻² for the LCU's 8 mm diameter light guide tip. The variable power supply was used to adjust the irradiance of the halogen unit to give a reading of 300 mWcm⁻² on this radiometer. To ensure a constant power output over the whole curing period, the unit was allowed to reach a stabilised light output. The unit was taken to have stabilised when two consecutive irradiance measurements differed by no more than 5 mWcm⁻². A radiometer reading of the halogen LCU was taken before and after each composite sample was cured, and checks made that both readings were 300 mWcm⁻².

An LED LCU shown in figure 2 containing 25 blue LEDs (Nichia Chemical Industries Ltd, Anan, Japan) was constructed. ^{15–17} The light from this LCU was concentrated using a polymer optical taper having a diameter of 6 mm at the end directed to the composite samples and 15 mm at the end directed to LED arrangement. An optical taper uses the principle of total internal reflection to propagate light, in the same manner as an optical fibre. A tapered construction, however, concentrates light entering the wider end and gives a higher power density, (power per unit area) at the narrow end that is directed towards the composite. A valid reading from the Coltolux 4 radiometer could not be taken for the LED LCU, as this radiometer is calibrated for 8 mm light guides. No fan cooling was necessary for the LED LCU owing to the small amounts of heat generated during the experiments.

The power output for both LCUs was measured on a Coherent 210 power meter (Coherent Ltd., Cambridge, UK) which uses a thermopile detector. The light guides were brought in direct contact with the surface of the detector when recording the measurement. The spectra of both sources were also measured using a MS127i imaging spectrograph with an Instaspec IV CCD array detector (LOT Oriel, UK). This method is more comprehensive than using a commercial dental radiometer that is designed for practice use with one specific halogen LCU only, as the distribution of light output across the spectrum can be measured.

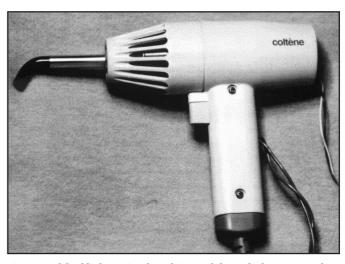


Fig. 1 Modified light curing handpiece of the Coltolux 4 LCU. The diameter of the fibre light guide is 8 mm.

Three types of composite (3M, St Paul, MN, USA) were used. Silux Plus shade U (filler particle size 0.01–0.09 microns and filler content 40% by volume), P50 shade U (filler particle sizes 0.2–6 microns and filler content 77% by volume) and Z100 MP shade A3.5 (filler particle size 0.01–3.5 microns and filler content 66% by volume). This range of composites was chosen to represent materials that are used clinically in both anterior and posterior restorations.

A stainless steel mould with a diameter of 4 mm and depth of 6 mm was filled with the composite. The curing times recommended by the manufacturer are 40 s, 60 s and 40 s for each of the three composites respectively, and six samples of each composite were cured either with the halogen LCU or with the LED LCU, for these times. A digital stopwatch was used to time the curing cycle.

A penetrometer similar to that suggested by Harrington and Wilson, ¹⁸ was used to measure the depth of cure. This penetrometer consists of a 0.5 mm needle connected rigidly to a weight of 1250 g. A digital dial gauge accurate to 0.01 mm rests on this assembly and is zeroed prior to each measurement. When the weight and needle are lifted and the assembly lowered onto the surface of the soft uncured portion of the composite sample, the needle penetrates this soft (uncured) composite until it reaches the harder, cured layer. This measurement is done from the bottom side (away from the light source) of the sample. The depth of cure can be read directly from the dial gauge. Although conventional microhardness measurements may give additional information, the depth of cure measurement method used in the present study is simple but precise and can be performed quickly. This penetrometer method of measuring the depth of cure has potential advantages over the ISO 4049 scrape test for depth of cure measurements. The degree of force applied is reproducible, and is determined by the weight of the penetrometer, rather than subjective judgement of the operator scraping away uncured composite for each sample.

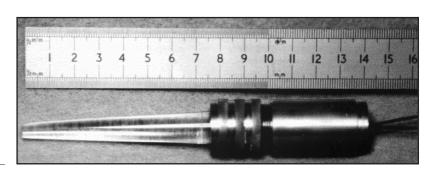


Fig. 2 LED LCU used in this study. The LED LCU contains 25 LEDs and uses a tapered optical light guide. The diameter of the light guide at the end directed at the composite samples is 6 mm.

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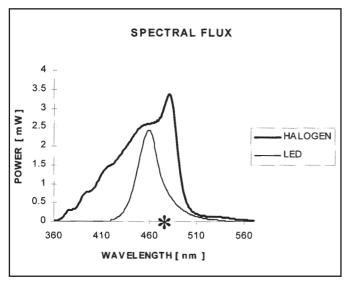


Fig. 3 Spectral flux of both the halogen (bold curve) and the LED (fine curve) LCU used in this study. Note the relatively sharp peak of the LED LCU around 470 nm. The flux of the halogen LCU exhibits a broader distribution than the LED LCU. The peak of the light absorption of the camphorquinone photoinitiator is at 468 nm denoted by an asterisk on the x-axis.

Results and statistical analysis

The spectra for both the halogen and LED LCUs are displayed in figure 3. The area under each plot represents the power outputs (flux) of the LCUs. The distribution of this power over the spectrum is the spectral power output (spectral flux). This represents the optical power output from each LCU in milliwatts at each wavelength. The y-axis of the graph in figure 3 was calibrated from the power meter measurements for each LCU. Figure 3 shows the spectrum for wavelengths ranging from 360 nm–570 nm. The proportion of total flux for both the halogen and LED LCUs lying outside this range was negligible and is, therefore, not shown. The flux from the LED LCU peaks at 460 nm and is concentrated over a much narrower wavelength band than for the halogen LCU.

The readings for the halogen and LED LCUs are displayed in Table 1. The total optical power output (flux) measured with the power meter for the halogen lamp was 229 mW and for the LED LCU was 82 mW. The power output of the LED LCU was therefore 36% of the halogen LCU. Between 410 nm and 500 nm, the output dropped to 195 mW for the halogen LCU and 78 mW for the LED LCU. In this spectral region, the LED LCU gave 40% of the output of the halogen LCU. Ninety-five per cent of the total output for the

LED LCU lies between 410 nm–500 nm compared with 85% for the halogen LCU.

The power output values were converted into power density (irradiance) values in mWcm⁻², for the area of the halogen LCU light guide tip (8 mm diameter) and the area of the LED LCU light guide tip (6 mm diameter). These values are displayed in figure 4. The change in appearance of the plots relative to figure 3 is due to the halogen and LED LCUs having different size lightguide tips. The halogen LCU produced an irradiance of 455 mWcm⁻² with the LED LCU producing 290 mWcm⁻². The LED LCU therefore produced 64% of the irradiance of the halogen LCU. Between 410 nm–500 nm, these values dropped to 388 mWcm⁻² for the halogen LCU, and to 276 mWcm⁻² for the LED LCU. In this bandwidth, the LED LCU produced 70% of the irradiance produced by the halogen LCU. The irradiance produced by the halogen LCU measured by the Coltolux 4 radiometer designed for surgery use was 300 mWcm⁻².

The results of the depth of cure measurements are shown in Table 2. For each group of the three composites, the Student's t test was used to compare the LED and halogen LCU results. For each composite, the depth of cure was significantly greater, P < 0.05, for the LED LCU than for the halogen LCU. This increase in depth of cure with the LED LCU was around 0.2 mm in each type of composite. The depth of cure values were about 1.5 mm greater for the Z100MP composite than for the other two composites for both LCUs.

Discussion

The light output from both units is different in several respects. Not all wavelengths of the emitted light are useful for the composite's curing process. Cook found the light between 410 nm–500 nm was the most effective. Between 410 nm–500 nm the LED LCU has 40% of the power output of the halogen LCU. Due to the smaller light guide tip, however, the irradiance produced by the LED LCU is 70% of the halogen LCU. Ninety-five per cent of the total flux for the LED LCU lies between 410 nm–500 nm.

The spectral distributions for the light sources within the effective range of 410 nm–500 nm also differ strongly. The flux from the LED LCU peaks at 460 nm and is concentrated over a much narrower wavelength band than for the halogen LCU. This may be expected as LEDs produce light by electroluminescence, the radiative recombination of an electron and hole in a semiconductor p-n junction to give a photon. The physical characteristic of the so-called band gap of the semiconductor used determines the wavelength of these photons. A wide bandgap material produces high-energy photons towards the blue region of the visible spectrum, while a narrow bandgap material produces lower energy photons in the red region.

Halogen lamps produce light by incandescence, whereby a filament is heated and causes the excitation of atoms over a wide range

Table 1 LCU flux and irradiance for the LCUs used in this study and for an LED LCU used earlier by Fujyabasi. Note the difference between the irradiance reading for the halogen LCU measured with the power meter (388 mWcm⁻²) and the Coltolux 4 radiometer (300 mWcm⁻²).

	Total flux measured on power meter [mW]	Total flux between 410–500 nm [mW]	Irradiance [mWcm ⁻²]	Irradiance between 410–500 nm [mWcm ⁻²]	Irradiance measured on Coltolux 4 radiometer [mWcm ⁻²]
Halogen LCU 8 mm Ø light guide	229	195	455	388	300
LED LCU 6 mm ∅ light guide	82	78	290	276	N/A
Fujiyabashi ¹⁴ Halogen LCl 8 mm ∅ light guide	J –	50	-	100	-
Fujyabashi ¹⁴ LED LCU 8 mm Ø light beam	-	50	-	100	_

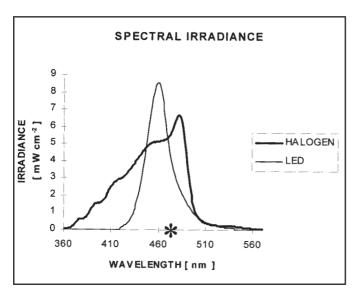


Fig. 4 Spectral irradiance of both the halogen (bold curve) and the LED (fine curve) LCU used in this study. The curves take the different diameters of the LCU's light guides (8 mm for the halogen LCU and 6 mm for the LED LCU) into consideration. The peak of the light absorption of the camphorquinone photoinitiator is at 468 nm denoted by an asterisk on the x-axis.

Table 2 Results of the depth of cure measurements for three common dental composites. The LED LCU cured the composites significantly deeper than the halogen LCU adjusted to 300 mWcm⁻². The data presented in the table are means (±SD) of six specimens for each test.

	Depth of cure	Depth of cure	Depth of cure
	[mm]	[mm]	[mm]
	Silux (U) 40s	P50 (U) 60s	Z100MP (3.5) 40s
Halogen Unit	3.44 ± 0.05	3.44 ± 0.07	4.93 ± 0.03
LED Unit	3.64 ± 0.02	3.61 ± 0.06	5.14 ± 0.07
Probability	P < 0.05	P < 0.05	P < 0.05

of energy levels producing a very broad spectrum. A filter is therefore required to restrict the emitted light to the blue region of the spectrum required for curing.

In figure 4, it can be seen that the LED LCU has a higher irradiance in the region of the peak absorption for camphorquinone (ie 468 nm). This may account for the greater depth of cure observed for the samples cured with the LED LCU. It has been shown that blue light in different parts of the absorption spectrum of camphorquinone has a different effectiveness, and that light near to the absorption peak is more effective at curing. ¹⁹

The Coltolux radiometer used to determine the halogen LCUs 300 mWcm⁻² gave a different reading from the value calculated from the power meter and spectrometer readings. It has been previously reported that LCUs producing an adequate depth of cure can be classified as good with one radiometer and poor with another.²⁰

Fujibayashi *et al.* 14 also found an LED source producing the same irradiance as a halogen source produced a significantly greater depth of cure than the halogen source. The results from the present study suggest that an LED LCU producing a lower irradiance than a halogen LCU can also produce a greater depth of cure.

The composites used in this study are of similar shades but different compositions. The composition of a composite has been shown to affect the depth of cure, since smaller filler particles scatter the light more than large filler particles.²¹ Light attempting to penetrate

small particle composites, therefore, has a more difficult task to penetrate the deeper regions of the material and greater irradiances or exposure times are required to cure the composites adequately. The ratio of filler relative to resin is also important. The higher the proportion of filler, the more difficult it is for the light to penetrate the composite.

The results showed that an LED source is capable of a significantly greater depth of cure for three different types of composite than a halogen LCU adjusted to an irradiance of 300 mWcm⁻² on a commercial dental radiometer. Depth of cure, however, is only one of many tests that can be applied to light cured composites. Other mechanical tests need to be conducted to determine whether LED cured composites perform in the same way as conventionally cured composites. Depth of cure is a significant first step as this depends on the quantity of useful blue light energy that can be applied to a given volume of composite in a reasonable time. Future research will also aim toward comparisons of LED LCUs with halogen LCUs operating at normal light intensities. As blue LED technology continues to improve, LED curing will become a useful adjunct to existing curing methods.

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- Bassiouny M A, Grant A A. A visible light-cured composite restorative. Br Dent J 1978; 145: 327-330.
- 2 Cook W D. Spectral distribution of dental photopolymerization sources. J Dent Res 1982; 61:1436-1438.
- 3 Cayless M A, Marsden A M. Tungsten halogen lamps. In *Lamps and lighting* 3rd ed. pp 169-182 London: Edward Arnold Ltd, 1983.
- 4 Barghi N, Berry T, Hatton C. Evaluating intensity output of curing lights in private dental offices. *J Am Dent Assoc* 1994; 25:992-996.
- 5 Martin F E. A survey of the efficiency of visible light curing units. *J Dent* 1998; 26: 239-243.
- 6 Miyazaki M, Hattori T, Ichiishi Y, Kondo M, Onose H, Moore B K.Evaluation of curing units used in private dental offices. *Oper Dent* 1998; 23: 50-54.
- 7 Caughman W F, Rueggeberg F A, Curtis J W Jr. Clinical guidelines for photocuring restorative resins. J Am Dent Assoc 1995; 126: 1280-1286.
- 8 Shortall A, Harrington E. Guidelines for the selection, use, and maintenance of visible light activation units. *Br Dent J* 1996; **181**: 383-387.
- 9 Haitz R H, Craford M G, Weissman R H. Light Emitting Diodes. In Bass M (ed) Handbook of optics. 2nd ed. pp 12.1-12.39, McGraw Hill Inc, 1995.
- 10 Nakamura S, Mukai T, Senoh M. High-Power GaN P-N Junction bluelight-emitting diodes. *Jpn J Appl Phys Lett* 1991; 30: L1998-L2001.
- Nakamura S, Senoh M, Iwasa N, Nagahama S. High-power InGaN single-quantum-well-structure blue and violet light-emitting diodes. *Appl Phys Lett* 1995; 67: 1868-1870.
- 12 Mills R W. Blue light emitting diodes an alternative method of light curing? *Br Dent J* 1995; 178: 169 Letter.
- 13 Fujibayashi K, Ishimaru K, Kohno A. A study on light activation units using blue light-emitting diodes. *J Jap Dent Pres Acad* 1996; **39**: 180-188.
- 14 Fujibayashi K, Ishimaru K, Takahashi N, Kohno A. Newly developed curing unit using blue light-emitting diodes. *Dent Jap* 1998; 34:49-53.
- 15 Mills R W, Jandt K D. LEOS IEEE Newsletter 1998; 12: 9-10.
- 16 Mills R W, Jandt K D. UK Pat app GB 9720443.2 (1997).
- 17 Mills R W, Jandt K D. UK Pat app GB 9806046.0 1 (1998).
- 18 Harrington E, Wilson H J. Depth of cure of radiation-activated materials. Effect of mould material and cavity size. J Dent 1993; 21: 305-311.
- 19 Yearn J A. Factors affecting cure of visible light activated composites. *Int Dent J* 1985; 35: 218-225.
- 20 Hansen E K, Asmussen E. Reliability of three dental radiometers. *Scand J Dent Res* 1993; **101**: 115-119.
- 21 Pearson G J. Aspects of the use and abuse of aesthetic restoratives: 1. Composite Materials. *Dent Update* 1990; 17:103-108.