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Original

# Effect of exposure time on curing efficiency of polymerizing units equipped with light-emitting diodes

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**Abstract: A study was conducted to evaluate the top and bottom hardness of two composites cured using polymerizing units equipped with light-emitting diodes [LED] (LEDemetron; Elipar FreeLight, Coltolux LED) and one quartz-tungsten halogen device [QTH] (Optilux 501) under different exposure times (20, 40 and 60 sec). A matrix mold 5 mm in diameter and 2 mm in depth was made to obtain five disc-shaped specimens for each experimental group. The specimens were cured by one of the light-curing units (LCUs) for 20, 40 or 60 sec, and the hardness was measured with a Vickers hardness-measuring instrument (50 g/30 sec). Data were subjected to three-way ANOVA and Tukey's test ($\alpha$ = 0.05). LED LCUs were as effective as the QTH device for curing both composites. A significant increase in the microhardness values were observed for all light LCUs when the exposure time was changed from 20 sec to 40 sec. The Z250 composite showed hardness values that were usually higher than those of the Charisma composite under similar experimental conditions. LED LCUs are as efficient for curing composites as the QTH device as long as an exposure time of 40 sec or higher is employed. An exposure time of 40 sec is required to provide composites with a uniform and high Knoop hardness when LED light-curing units are employed. (J. Oral Sci. 49, 19-24, 2007)**

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## Introduction

Composites represent a class of materials widely used in restorative dentistry because of patient demands for better aesthetics with low cost. Although light-cured composites are excellent for aesthetic procedures, both the physical and chemical properties of filled composites are directly related to the conversion of monomers to polymers. Low conversion rates lead to degradation, substance loss and fracture, and marginal breakdown, thus limiting the lifespan of the composites (1).

Adequate polymerization of composite restorative materials is fundamental for obtaining optimal physical and chemical properties, as well as for ideal clinical performance. At present, four types of polymerization sources are available: quartz-tungsten halogen bulbs (QTH), plasma-arc (PAC) lamps, argon-ion lasers and light-emitting diodes (LED).

Halogen bulb-based light-curing units are currently the most commonly used means of curing dental composites. They emit light within a wide range of the visible spectrum, and band-pass filters are required to limit the wavelength between 370-550 nm (2). This makes these light-curing units suitable for activation of several photo-initiators. However, as the spectral irradiance is lower at the blue end of the visible range and high at the red end, a considerable amount of heat is generated, requiring the use of cooling fans. Another disadvantage of these light sources is that halogen bulbs have a limited effective lifetime of about 40 - 100 h (2).

Argon-ion laser and plasma-arc units emit high power densities. The former emit light at distinct frequencies and do not require any filter, while plasma-arc units require filters to block undesirable wavelengths. The main disadvantage of these two light-curing sources is that they are quite expensive compared to the most commonly used halogen bulb-based units, and therefore for reasons of economy they have not found wide acceptance.

An alternative curing device that has been recently investigated in an attempt to overcome the problems of a halogen lamp is the solid state light-emitting diode [LED] (3-5). LEDs used to polymerize composites operate at a wavelength of around λ 470 nm and a bandwidth of about λ 20 nm, and therefore have the spectral purity for highly efficient curing of dental resins. Another advantage of LEDs is that the most common initiator of the polymerization reaction, the diketone camphorquinone, has its maximum absorption at 470 nm (2). Because of these advantages, LEDs have been broadly advertised as an alternative curing source for dental composites.

The first generation of LED curing lights, which often contained multiple LEDs, had a relatively low power output, and did not perform as well as conventional QTH lights (5-8), especially when used to polymerize resins containing certain co-initiators in addition to camphorquinone (9,10). New-generation LED lights are now readily available. These light sources deliver a greater power output than the first units released onto the market, and may therefore offer better performance and shorter curing times (11). So far, few studies have attempted to evaluate the relationship between exposure time and curing efficiency for this new generation of LED light sources, which is essential in order to grasp their effectiveness for polymerization of composites under different exposure times. Therefore the present investigation was conducted for this purpose.

## Materials and Methods

Two light-cured composites were evaluated: Charisma (Shade SL, Heraues Kulzer, Wehrheim, Germany) and Z250 (Shade B0.5, 3M ESPE, St. Paul, MN, USA). The composition of each material is detailed in Table 1.

One QTH and three LED light-curing units were used: the QTH unit Optilux 501 (OP, Kerr Corp., Orange, CA, USA) and the LED units Elipar Free Light (FL, 3M/ESPE, Seefeld, Germany), LEDemetron (LE, Kerr Corp., Orange, CA, USA) and LED Coltolux (CL, Coltène Whaledent, Cuyahoga Falls, OH, USA). The light-curing units were all used in standard mode (continuous, constant light intensity). Technical details of the halogen and LED light-curing units are detailed in Table 2.

For each material, disc-shaped specimens (5 mm diameter and 2 mm thickness) were prepared using metal matrix molds. These molds were placed on flat glass plates on top of acetate strips and then filled in bulk with resin-based composites. The composite was covered with an acetate strip and gently pressed with another glass plate against the mold to extrude excess material. The top glass slide was removed and the specimens were then irradiated through the acetate strip with one of the four light-curing sources detailed in Table 2 for 20, 40 or 60 sec.

Immediately after light-curing, the cover glasses were removed from the mold and the lower surfaces were marked with a pen and stored in the dark in distilled water at 37°C for 24 h. Five specimens were prepared for each experimental group.

Indentations were made with an HMV-2 microhardness tester (Shimadzu, Kyoto, Japan) using a 50-g load and a dwell time of 30 sec. Eight measurements were made on each side of the specimen and the top and bottom mean values of the Vickers hardness number (VHN) were calculated. It has been suggested that acceptable polymerization of a given composite has occurred when the test specimen's bottom surface hardness is at least 80% of the top surface hardness (12). The data from each composite were analyzed by three-way analysis of variance and Tukey's multiple comparison test to evaluate the effects of light-curing units, exposure time and location on the mean VHN for each material ($\alpha = 0.05$).

Table 1 Composition of the restorative materials

| Composite (Manufacturer) | Monomers | Filler type | Batch number |
|---|---|---|---|
| Filtek Z250 (3M ESPE) | UDMA, Bis-EMA, and TEGDMA | Zirconium silicate; average particle size: 0.6 μm; 60 vol% | 4BF |
| Charisma (Heraeus Kulzer) | Bis-GMA and TEGDMA | Barium glass and silica; average particle size: 0.7; 60 vol% | 010133 |

UDMA: urethane dimethacrylate; Bis-EMA: bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-GMA: bisphenol A di (glycidyl methacrylate)

Table 2 Technical details of the light curing units*

| Code | Light curing unit (Manufacturer) | Type | Wavelength range (nm) | Light intensity (mW/cm²) |
|---|---|---|---|---|
| OP | Optilux 501 (Kerr) | QTH | 400-515 | 700 |
| FL | Elipar Free Light (3M ESPE) | LED | 440-490 | 400 |
| LE | LEDemetron (Kerr) | LED | 450-470 | 800 |
| CL | Coltolux LED (Coltène Whaledent) | LED | 450-470 | 1400 |

*: manufacturer information

## Results

The means and standard deviations for both composites under the experimental conditions used in this study are shown in Tables 3 and 4. The interaction Light curing unit × Exposure time × Location ($P < 0.0001$) and the interactions Light curing unit × Location ($P < 0.0001$) and Exposure time × Location ($P < 0.0001$) were statistically significant for the Charisma composite (Table 3). For Z250, the interactions Light curing unit × Exposure time ($P = 0.0017$), Light curing unit × Location ($P < 0.0001$) and Exposure time × Location ($P < 0.0001$) were statistically significant (Table 4).

The top VHN values for the Charisma composite were higher than the bottom values when the exposure time was set at 20 sec, regardless of the light-curing unit employed. For the Z250 composite, the top VHN values was higher than the bottom measurements for all exposure times used, regardless the light curing-unit employed. The VHN values for Z250 were usually higher than those for Charisma under similar experimental conditions.

With regard to the bottom surface, a significant increase in the microhardness values was observed for all light-curing units when the exposure time was changed from 20 sec to 40 sec. However, a further increase in the exposure time from 40 sec to 60 sec did not result in significant microhardness improvements, except when the Optilux 401 was used with Charisma composite.

The hardness ratio (bottom/top) for Charisma was better than 80% under all experimental conditions, except when the Elipar Free Light and LEDemetron devices were employed with an exposure time of 20 sec For Z250, a hardness ratio of 80% was achieved only with the Optilux 501 at 60 sec and with the LEDemetron at 40 and 60 sec.

## Discussion

Hardness has been shown to be a good indicator of conversion of double bonds (13-15), and was therefore used in the present study as an indirect measurement of conversion. Although there were small differences in hardness between the light sources, the four light-curing units showed similar performance as long as a minimum exposure time of 40 sec was used.

LED technology has advanced significantly since blue LEDs were originally adopted for curing of dental composites (7). In contrast to previous generations of LED lights, which used an array of LEDs with low light intensity (3,7), newer LED devices, such as the LEDemetron and Coltolux LED, usually use a high-intensity blue LED containing a larger semi-conductor crystal, which increases both the illuminated area and the light intensity. The higher light intensity of the newer LED devices along with their narrow spectral output makes them similar or even more efficient than conventional QTH light-curing units (16-19).

At the surface, even with short exposure times, all of the light-curing units provided enough energy to cure all composites due to the lack of light attenuation caused by overlying composite, as has already been demonstrated by previous authors (15,20).

High light intensity helps to maintain camphoroquinone (CQ) in the triplet excited state, thus allowing this photosensitizer to react with a co-initiatior (amine) enhancing the formation of free radicals, which initiates polymerization (20). As the thickness of the composite increases, the number of photons available to raise CQ to the activated state is limited by absorption and scattering factors associated with the overlying resin. This reduces the probability of collision of CQ with an amine. Although

Table 3 Means and standard deviations of Vickers Hardness Number (VHN) of Charisma composite in each experimental condition and bottom-top hardness ratio

| | Exposure time (sec) | Top | Bottom | % Bottom/top |
|---|---|---|---|---|
| Optilux 401 | 20 | 42.1 ± 1.1 b | 33.8 ± 4.6 d,e | 80.3 |
| | 40 | 41.6 ± 2.0b,c | 32.6 ± 1.1 d,e | 78.4 |
| | 60 | 46.2 ± 0.7 a | 41.5 ± 2.5 b,c | 89.2 |
| LED Coltolux | 20 | 38.2 ± 1.0 c | 31.2 ± 1.3 e,f | 81.6 |
| | 40 | 40.5 ± 0.9 b,c | 40.9 ± 0.5 b,c | 100.1 |
| | 60 | 41.6 ± 0.1 b,c | 40.4 ± 1.5 b,c | 97.1 |
| Free Light | 20 | 38.2 ± 0.9 c | 19.5 ± 0.9 g | 51.0 |
| | 40 | 39.6 ± 0.8 b,c | 32.2 ± 2.1 d,e | 81.3 |
| | 60 | 40.8 ± 0.6 b,c | 32.7 ± 1.2 d,e | 80.1 |
| LEDemetron | 20 | 39.6 ± 0.3 b,c | 27.9 ± 1.1 e,f | 70.5 |
| | 40 | 40.9 ± 1.0 b,c | 37.1 ± 1.0 c,d | 90.7 |
| | 60 | 41.8 ± 0.6 b | 36.3 ± 3.0 c,d | 86.8 |

Different letters indicate statistically different means.

Table 4 Means and standard deviations of Vickers Hardness Number (VHN) of Z250 composite in each experimental condition and bottom-top hardness ratio

| | Exposure time (sec) | Top | Bottom | % bottom/top |
|---|---|---|---|---|
| Optilux 401 | 20 | 59.2 ± 1.7 b,c,d | 37.8 ± 3.1 f | 66.9 |
| | 40 | 62.0 ± 1.6 a,b,c | 48.0 ± 2.9 d,e | 77.4 |
| | 60 | 62.3 ± 2.1 a,b,c | 52.6 ± 2.4 d,e | 84.4 |
| LED Coltolux | 20 | 60.9 ± 1.6 a,b,c | 32.3 ± 3.2 f | 53.0 |
| | 40 | 63.1 ± 0.5 a,b | 44.5 ± 2.3 e,f | 70.5 |
| | 60 | 64.2 ± 1.3 a,b | 49.3 ± 1.3 d,e | 76.8 |
| Free Light | 20 | 60.8 ± 0.8 a,b,c | 24.8 ± 3.3 g | 40.7 |
| | 40 | 65.7 ± 1.4 a | 44.6 ± 6.0 e | 67.8 |
| | 60 | 65.0 ± 1.2 a,b | 50.2 ± 5.9 d,e | 77.2 |
| LEDemetron | 20 | 60.2 ± 0.5 b,c | 37.3 ± 2.9 f | 61.9 |
| | 40 | 61.4 ± 2.9 a,b | 49.4 ± 1.7 d,e | 80.5 |
| | 60 | 61.7 ± 1.3 a,b | 54.6 ± 1.2 d | 88.4 |

Different letters indicate statistically different means.

LCUs have an irradiance ranging from 400 (FL) to 1400 (CL), a 20-sec exposure time did not provide bottom hardness values as high as those observed at the top surface. In fact, the total energy density delivered to the composites during a 20-sec exposure was rather low for the OP (14 J/cm$^2$), FL (8 J/cm$^2$) and LE (16 J/cm$^2$), compared with the total energy density indicated for polymerization of composites, which is within the range 18 - 24 J/cm$^2$ (21,22).

According to Nomoto et al. (23), when the total energy density, represented by the product of light intensity and exposure time, is kept constant, the depth of cure and polymerization conversion of composites is similar, regardless of the light intensity and exposure time. However, this was not the case for light-cure of Z250 by the CL device: a 20-sec exposure (1400 mW/cm$^2$) provided an energy density of 28 J/cm$^2$, which was higher than the minimum required for polymerization of a 2-mm-thick composite (21,22). One possible reason for this finding is that different light-curing devices are affected by the focusing effect of the emitted light (24). Although all QTH and LED devices show a decrease of light intensity as the distance from the light tip is increased, the rate of decrease is not similar for all devices (24). The aforementioned study have found that devices with a stronger light-focusing effect (due to larger entrance and smaller exit surface areas), such as turbo light guides, show a greater decrease of power density (24). Although we did not examine the focusing effect of the light-curing units, it is likely that the CL device could show stronger light-focusing, which would reduce light diffusion in deep areas of the composite. However, this hypothesis requires further investigation.

To compensate for this decreased light diffusion potential, due to either a strong focusing effect or low light intensity, the exposure time can be increased, providing enhanced opportunity for an excited CQ molecule to collide with an amine, thus creating a free radical (20). In the present study, when the bottom hardness values for each light curing were compared with one another, higher hardness means were observed when the duration of exposure was equal to, or higher than, 40 sec.

It has been suggested that the bottom to top VHN ratio should be higher than 80% for adequate in-depth polymerization (12). A bottom-to-top VHN of 80% corresponds to a bottom-to-top conversion of 90%, i.e., 90% of maximum conversion possible at the composite's top surface (15). The VHN ratios recorded in the present study exceeded the above threshold limit for the Charisma composite, except when two light-curing devices (FL and LE) were used for 20 sec. For the Z250 composite, the VHN ratio exceeded 80% only with an OP at 60 sec and a LE at 40 and 60 sec. However, these ratios only allow comparison of bottom-to-top ratios for individual lights, and conclusions can be misleading if they are not adequately interpreted (11). For example, the VHN was 41.5 at the bottom and 46.2 at the top for Charisma when light-cured with the OP at 60 sec, in which case the bottom hardness would be an acceptable 89.2% of the top hardness. Under the same experimental conditions, the KHN was 52.6 at the bottom and 62.3 at the top for Z250, making the bottom hardness also acceptable (84.4%) when compared with the top hardness, and thus suggesting that the LCU produced equal results for both composites. However, Charisma itself is softer at the top surface, being equivalent to only 74.1% of the top hardness of Z250.

If the deepest layers of composite restorations are not adequately cured, the elastic modulus at the bottom will be lower than that at the surface. This can increase the flexure of the material under masticatory forces, leading to open margins or fracture of the bulk of the restoration.

Higher hardness values were obtained with Z250 light-cured with the four devices, as also observed in previous studies (25-30). However, comparison of bottom-to-top ratios for the two composites suggests that more light attenuation occurred with Z250. It can be hypothesized that the top surface of this composite hardens faster, and thus the optical properties of the overlying composite are altered in such a way that more light-scattering and absorption can occur, reducing the in-depth light diffusion.

Another hypothesis can be applied to the organic matrix. These materials have similar filler loading and size; however the Z250 composite contains UDMA. UDMA-based resins have been shown to be more reactive than Bis-GMA-based composites (31). The higher conversion level may have also been related to the partial substitution of the relatively stiff and hydrogen-bonded Bis-GMA molecules with the longer and more flexible Bis-EMA molecule. The Z250 composite might contain a higher percentage of photo-initiators than other materials, thus increasing the VHN values (32). However, the amount of photo-initiators included in the composition of the materials is not stipulated by the manufacturers, thus preventing us from making further comparisons. According to Park et al. (33), composites from 3M ESPE are based on a three-component initiator system (CQ, tertiary amine and iodonium salt) (34), and therefore iodonium salt may play an important role in increasing the curing efficiency of the composite (33).

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