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Effect of Time and Polymerization Cycle on the Degree of Conversion of a Resin Composite

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Clinical Relevance

Initial and final resin composite properties will depend on the photo-activation condition applied.

SUMMARY

The aim of this investigation was to verify the influence of 3 light curing units on the degree of conversion, using different irradiation conditions: 1) manufacturers' recommended times of photo-activation, 2) standardizing total energy density among the units and 3) standardizing energy density at the 450-490 nm wavelength range among the units and the effect of these irradiation conditions on the post-cure. Three light curing units were used: halogen, light emitting diodes (LED) and xenon plasma. Seven groups were tested (n=6). Twenty-four hours after the photo-activation procedures, half of the composite specimens were submitted to Fourier Transformed Infrared Spectroscopy analysis. The other half was analyzed after 1 month. The results were submitted to 2-way ANOVA and Tukey's test (5%). Twenty-four hour analysis revealed that the second set of irradiation conditions produced a similar degree of conversion among the LCUs. After 1 month, the conversion values were statistically higher for 20 seconds of halogen exposure (increased from 46.78 to 49.66%), 20 seconds of LED exposure (from 46.20 to 51.15%), 30 seconds of LED exposure (from 48.29% to 50.68%) and 3 seconds of PAC exposure (from 42.57 to 51.39%). The initial degree of conversion and post-cure depended on the photo-activation condition applied.

INTRODUCTION

Light-cured composites are set via exposure to light of a certain wavelength and an intensity that initiates the

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Table 1: *LCU Characteristics*

LCU	Total Irradiance (mW/cm²)	Specific Irradiance (mW/cm²)	*Irradiation Time (seconds)	*Total Energy Density (mJ/cm²)	Specific Energy Density (mJ/cm²)
QTH	669	357	20	13,380	7,140
PAC	1818	1516	3	5,454	4,548
LED	453	325	20	9,060	6,500

**Using the time of photo-activation as indicated by the manufacturers.*

generation of free radicals that propagate polymerization, causing material hardening.[1-2] This light can be emitted by light curing units (LCUs), such as conventional quartz tungsten halogen (QTH) LCUs, xenon plasma arc (PAC) LCUs and diode (LEDs) LCUs.[3] Studies[4,5] have analyzed the degree of conversion (DC) promoted by photo-activation methods. DC is an important parameter in determining the final physical, mechanical and biological properties of photo-activated composites,[6] since it has been demonstrated that resin composite properties tend to improve as the degree of conversion attained during photo-polymerization is increased.[7] For example, increased cure may result in a lower amount of uncured, potentially leachable monomer, leading to a more biocompatible restoration.[8] Moreover, uncured functional groups can act as plasticizers, reducing the mechanical properties of the composite.[9]

It has been claimed that the DC of dental composites may be energy density (light irradiance vs time of exposure) dependent,[10-11] and an equivalent DC may be achieved by applying a lower light irradiance for a longer time.[10,12-14]

The majority of studies that standardize energy density use the total irradiance values emitted by LCUs. However, one factor should be taken into consideration: in current dental composites, camphorquinone (CQ) is typically used as a visible, light-activated free radical photo-initiator.[15-16] Camphorquinone presents an absorption peak at a wavelength of 468 nm. Thus, the closer the curing unit's wavelength is to this absorption peak, the greater its ability to activate camphorquinone. According to Nomoto,[17] the highest adequate wavelength range for exciting CQ is the 450-490 nm range. Only wavelengths of around 470 nm are strongly absorbed by this photo-initiator, and light outside this region is less effective. Therefore, photo-polymerization also depends on the quality (wavelength) of light emitted by the LCU.[17]

Another factor that should be taken into account is that the photo-polymerization of multifunctional monomers exhibits a post-curing effect, since monomer conversion is never complete and the polymer usually contains considerable quantities of remaining/unreacted double bonds.[18] The post-curing reaction depends on the amount of initiating radicals generated by the irradiation step, storage temperature and reactivity of the polymers.[19]

The aim of the investigation was to verify the influence of 3 LCUs on the initial degree of conversion and post-cure, using different irradiation conditions. Three irradiation conditions were tested: 1) using the time of photo-activation as indicated by the manufacturers of the LCUs and composite; 2) maintaining a constant energy density (ED) value among the LCUs and 3) maintaining a constant ED at the 450-490 nm wavelength range (specific ED) among the LCUs.

METHODS AND MATERIALS

The restorative composite Filtek Z250, shade A3, batch #1BA (1400A2) (3M/ESPE Dental Products, St Paul, MN, USA), was used in this study.

Three LCUs were used: a QTH LCU (XL 2500, 3M/ESPE), a PAC LCU (Apollo 95E, DMD, Westlake Village, CA, USA) and an LED LCU (Ultrablue Is, DMC Equipamentos LTD, São Carlos, Brazil).

Analysis of the Spectral Distribution and Light Irradiance of the LCUs

The power (mW) of the 3 LCUs was measured with a power meter (Ophir Optronics Inc, Danvers, MA, USA). The diameter of the tips was measured with a digital caliper (Mitutoyo, Kanagawa, Japan) to determine the tip areas and calculate the total irradiance values (mW/cm²) by dividing the power (mW) by the area (cm²). With these data, standardization of the energy density was possible based on the total irradiance through different exposure times of photo-activation (Table 1).

The spectral distributions of the LCUs (Figures 1, 2 and 3) were obtained using a spectrometer (USB 2000, Ocean Optics, Dunedin, FL, USA). The total intensity data and spectral distribution of the LCUs were tabulated using the software Origin 6.1 (OriginLab Corp, Northampton, MA, USA) to obtain, by numerical integration, specific light irradiance at the 450-490 nm wavelength range. Thus, with these data, the specific energy density (at the 450-490 nm wavelength range) was standardized using different exposure times of photo-activation.

Table 1 shows energy density values when calculus was used based on total irradiance values or irradiance values at the 450-490 nm wavelength range, using the manufacturers' time exposure. Since QTH LCU demonstrated the highest energy density values in both cases, this source was used as a control.

Seven photo-activation treatments were accomplished:

1. Twenty seconds of photo-activation by QTH (manufacturer's recommendations)–CONTROL.
2. Three seconds of photo-activation by PAC (manufacturer's recommendations).
3. Twenty seconds of photo-activation by LED (manufacturer's recommendations).
4. Photo-activation of 7.5 seconds by PAC (irradiation time necessary to equal PAC energy density with QTH energy density, 13.380 mJ/cm^2, based on the total irradiance values).
5. Thirty seconds of photo-activation by LED (irradiation time necessary to equal LED energy density with QTH energy density, 13.380 mJ/cm^2, based on the total irradiance values).
6. Five seconds of photo-activation by PAC (irradiation time necessary to equal the PAC energy density with QTH energy density at the 450-490 nm wavelength range–7.140 mJ/cm^2).
7. Twenty-two seconds of photo-activation by LED (irradiation time necessary to equal LED energy density with QTH energy density at the 450-490 nm wavelength range–7.140 mJ/cm^2).

Degree of Conversion Analysis

The composite was applied in a circular elastomer mold (3-mm inner diameter and 2-mm in height) in a single increment. Composite specimens 2-mm thick were used to ensure uniform, maximum polymerization. The composite was then covered with a polyester strip and digitally pressed. For photo-activation procedures, the curing tips were positioned close to the elastomer mold/restorative composite. All photo-activations procedures were taken in a temperature-controlled room with a constant temperature of 23°C ± 0.5°C. Six specimens were prepared for each group. Half were DC-tested 24 hours after dry storage at room temperature in a dark container (n=3) and the other half were tested 1 month after the same storage conditions (n=3).

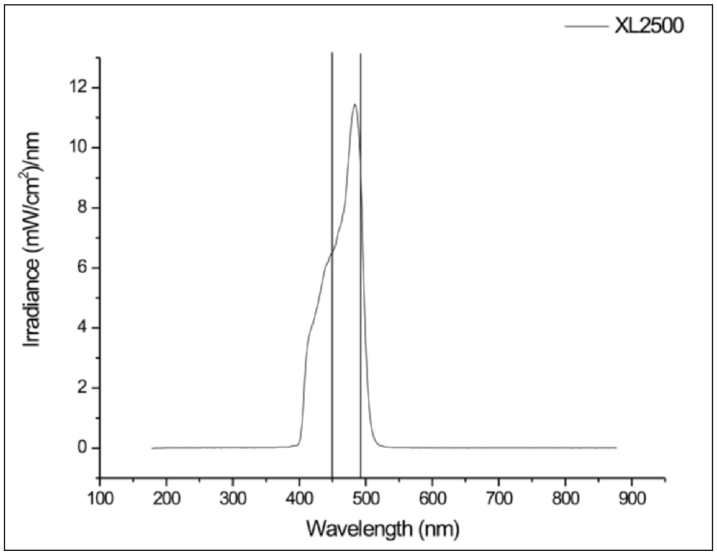

Figure 1: *Spectral distribution of light emitted by the XL2500 QTH LCU.*

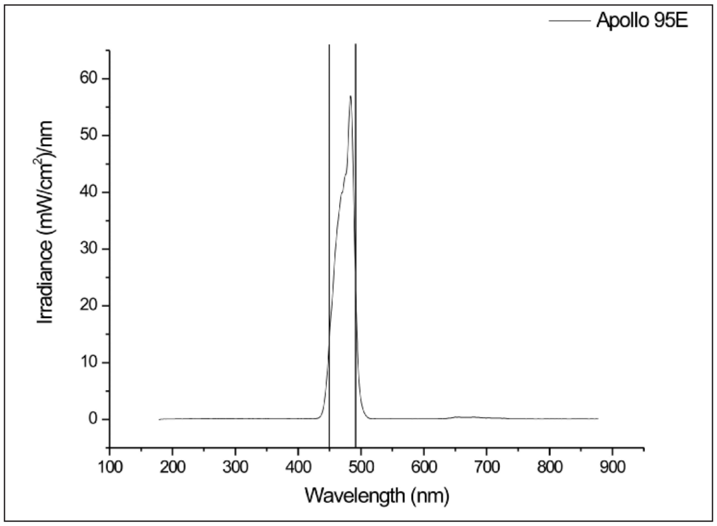

Figure 2: *Spectral distribution of light emitted by the Apollo 95E PAC LCU.*

The DC was measured by Fourier Transformed Infrared Spectroscopy (FTIR) with a Shimadzu 8300 spectrometer (Shimadzu, Tokyo, Japan). The polymerized samples were triturated in a grail and agate pistil containing mineral oil (Nujol, Shering-Plough, Rio de Janeiro, Brazil), forming an emulsion that was placed between 2 disks of NaCl. For each sample, a spectrum was collected in the range of 3000 cm^{-1} to 500 cm^{-1}, starting with 32 scans that had a resolution of 4 cm^{-1} in absorbance. A spectrum of the non-polymerized composite was also obtained and used as a reference for calculation of the conversion degree. Considering the peak intensity of absorbency of the C=C aliphatic connections (1638 cm^{-1}) and, considering the internal pattern of the aromatic connections C-C (1608 cm^{-1}), the degree of conversion was established according to the following formula:

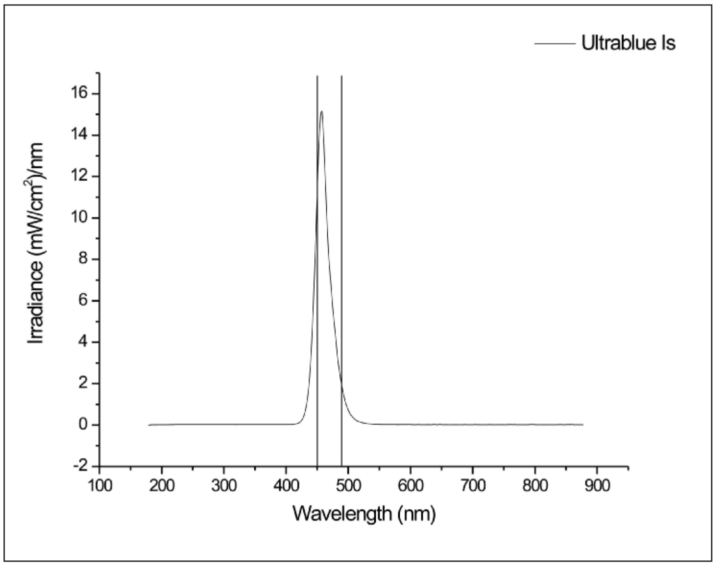

Figure 3: *Spectral distribution of light emitted by the Ultrablue Is LED LCU.*

$$\text{Residual double connections (\%)} = \frac{\text{absorbance in 1638 cm}^{-1}/\text{absorbance in 1608 cm}^{-1}\ \text{(Polymerized)} \times 100}{\text{absorbance in 1638 cm}^{-1}/\text{absorbance in 1608 cm}^{-1}\ \text{(Non-Polymerized)}}$$

Degree of conversion (%) = 100–Residual double connections (%)

The degree of conversion data were submitted to 2-way ANOVA (photo-activation methods and storage time), and the means were compared by Tukey's test (5% of significance level).

RESULTS

Table 2 shows the DC values promoted by photo-activation methods after 24 hours and 1 month.

After 24 hours, using the exposure time recommended by the manufacturers, QTH (20 seconds) and LED (20 seconds) were not statistically different (p>0.05). However, they produced statistically higher DC values (p<0.05) than PAC (3 seconds). Maintaining a constant total energy density (QTH 20 seconds/LED 30 seconds/PAC 7.5 seconds), the DC, promoted by the sources, were not statistically different (p>0.05). Using the time necessary to keep a constant energy density at the 450-490 nm wavelength range, the DC values promoted by the QTH (20 seconds) did not differ statistically from the values promoted by LED (22 seconds) and PAC (5 seconds). However, LED (22 seconds) and PAC (5 seconds) were statistically different (p<0.05).

After 1 month using the exposure time recommended by the manufacturers, DCs, promoted by the sources, were not statistically different (p>0.05). Moreover, keeping a constant total energy density, QTH (20 seconds) not differ statistically from LED (30 seconds) and PAC (7.5 seconds).

However, LED (30 seconds) and PAC (7.5 seconds) were statistically different (p<0.05). Keeping a constant specific energy density, QTH (20 seconds) and LED (22 seconds) were not statistically different (p>0.05). However, they produced statistically higher DC values (p<0.05) than PAC (5 seconds) (p<0.05).

Comparing DC values after 24 hours and 1 month for each method, DC was statistically higher (p<0.05) after 1 month for the QTH, LED (20 seconds) and LED (30 seconds) and PAC (3 seconds) methods. The light activation method of LED (22 seconds), PAC (5 seconds) and PAC (7.5 seconds) did not statistically increase DC values after 1 month (p>0.05).

DISCUSSION

The DC values verified in this study agree with those in the literature. It is difficult to compare DC values among different research, since the remaining double bonds may depend on several factors. In general, the DC values of composites range between 43 and 75%.[20] Using the composite Z250, 2-mm thick, Tarle and others[21] found minimum DC values of 53.8% when PAC Apollo 95E was used for 3 seconds. Moreover, these authors found maximum DC values of 60.6% and 63.5% when QTH Elipar II was used for 20 and 40 seconds, respectively. Through FT-Raman spectroscopy, Emami and others[22] found DC values around 65% for top surfaces of Z250 when using a QTH LCU. Using 1 QTH LCU (VIP) and the composite Z250, Vandewalle and others[23] found DC values in the occlusal surface that ranged between 53.4% and 57.4% by application of 6,000 and 3 x 24,000 mJ/cm^2 of energy density, respectively.

The results in this experiment should not be extrapolated, since they do not represent the performance of a class of LCUs. Care should be taken, because spectral distribution and the light intensity emitted by the LCUs can vary according to the manufacturers and models and, in many cases, among sources of the same model.[24] Moreover, there are significant differences in values of penetration of the activating light into the material,[25] and the resin matrix composition could also affect polymerization behavior.[26]

After 24 hours, using the time exposure recommended by the manufacturers, QTH (20 seconds) and LED (20 seconds) produced statistically higher DC values than PAC (3 seconds) (p<0.05). These findings are probably due to the differences in total energy densities emitted by LCUs, where 3 seconds of PAC exposure promoted the lowest total energy density (5,454 mJ/cm^2) and the lowest energy density (4,548 mJ/cm^2) at the 450-490 nm wavelength range (Table 1).

The DC values promoted by QTH and LED (20 seconds) were not statistically different (p>0.05) when the time exposure recommended by the manufacturers was used. This may be due to the fact that LED LCUs emit a narrow light wavelength that correlates with the spectral absorbency range of camphorquinone.[27] Table 1 shows the total intensity and specific intensity emitted by each LCU. The specific intensity emitted by QTH represents only 53.36% of the total intensity when compared with the specific intensity emitted by the LED, which represents 71.74% of the total intensity value. Thus, the total energy density emitted by the LED represents only 67% of the total energy density emitted by the QTH. However, the specific energy density emitted by the LED represents 91% of the specific energy density.

Maintaining a constant total energy density, QTH (20 seconds), LED (30 seconds) and PAC (7.5 seconds) produced DC values that were statistically similar. Thus, all LCUs had the same curing effectiveness. However, this did not occur when energy densities at the 450-490 nm wavelength range were kept constant. DC values promoted by QTH (20 seconds) were not statistically different from the LED (22 seconds) and PAC (5 seconds) degree of conversion values. However, LED (22 seconds) and PAC (5 seconds) promoted DC values that were statistically different ($p<0.05$). Thus, this method does not produce a constant curing efficiency for all sources, possibly due to the fact that CQ may absorb radiation in the range of 400-550 nm.[16] The most efficient wavelength range may be the 450-490 nm wavelength range;[17] however, regions around this wavelength range could be important. Therefore, it may be suggested that future studies should use a higher wavelength range to standardize the energy density.

Post-cure occurs because some portions of the radicals generated during composite polymerization are trapped within the heterogeneous network and can persist for extended periods.[28] With time, trapped radicals can encounter pendant groups, resulting in additional conversion. These radicals were trapped, because, as the polymerization reaction progresses, both propagation and termination reactions become diffusion limited[26] and, since the creation of a highly cross-linked network polymer limits the mobility of the reacting system, the cure stops prior to all double bonds being consumed.[26] Auto-deceleration describes the fact that, as polymerization continues, mobility of the pendant groups becomes lower and propagation becomes diffusion-controlled. Thus, final conversion is controlled by diffusion limitations of the reacting groups and not by the amount of uncured monomers.[29]

The post-cure process can be facilitated by the application of heat, which increases mobility within the polymeric network. This allows free monomer and pendant chains to encounter the remaining radical sites and react.[7,30] Moreover, with elevated temperatures, auto-deceleration could be delayed.[29] Trujillo, Newman and Stansbury[31] evaluated the temperature effect on immediate and final conversion of 3 resin composites activated by 3 different LCUs (QTH, LED and PAC) at 2 different temperatures—room temperature and 54.5°C—and found that the elevated cure temperature clearly increased the values of both the immediate and final conversion for resin composites with all of the light curing units tested. For this reason, in this study, the samples were maintained at room temperature. Therefore, differences between the 24-hour and 1-month DC values verified in this study represent only the effect of the LCUs' activation.

Additional cures were statistically significant ($p<0.05$), with the use of QTH (20 seconds), LED (20 seconds) and LED (30 seconds) and PAC (3 seconds) when compared with DC after 24 hours and 1 month for each method (Table 2). Interestingly, after 1 month, 3 seconds of PAC exposition (the method that provided the lowest 24 hour DC value) promoted a significant increase in DC value (from 42.57% to 51.39%). This could be related to the fact that, in spite of the high light intensity promoted by the PAC source, the energy den-

Table 2: *Degree of Conversion (DC) Values Promoted by the Photo-activation Methods*

Photo-activation Method	*24 Hours DC %	*1 Month DC %
LED 22 seconds/450-490 nm wavelength range	49.32 (1.20) **A, a**	50.07 (1.39) **A, ab**
LED 30 seconds/total energy density	48.29 (1.32) **B, ab**	50.68 (1.84) **A, a**
QTH 20 seconds/control	46.78 (1.38) **B, abc**	49.66 (0.53) **A, ab**
LED 20 seconds/manufacturers' recommended time	46.20 (1.78) **B, bc**	51.15 (0.57) **A, a**
PAC 7.5 seconds/total energy density	46.11 (0.61) **A, bc**	47.63 (0.95) **A, bc**
PAC 5 seconds/450-490 nm wavelength range	44.83 (0.32) **A, cd**	46.06 (0.63) **A, c**
PAC 3 seconds/manufacturers' recommended time	42.57 (1.30) **B, d**	51.39 (0.71) **A, a**

**Mean values followed by different small letters in the column differ statistically for the photo-activation methods by the Tukey´s Test at the 5% level ( ) Standard Deviation. Mean values followed by different capital letters in the line differ statistically for the storage period by the Tukey´s Test at the 5% level ( ) Standard Deviation.*

sity (total and specific) emitted by this method was the lowest, thus, the initial cure was the also the lowest. If the initial conversion is too low, then sufficient mobility exists,[31] and this increased mobility allows the reaction to continue for longer periods of time before propagation becomes diffusion-limited, resulting in longer molecular chains with higher flow characteristics.[29] Asmussen and Peutzfeldt[9] stated that an initially slow cure might favor the formation of a relatively linear polymer, because a slow polymerization start is associated with relatively few centers of polymer growth, resulting in few crosslinks. However, linear polymer is more susceptible to softening than a more crosslinked polymer at the same DC value.[9] Moreover, chain polymerization of multifunctional monomers leads to the formation of pendant double bonds, which can react with propagating radicals to form primary cycles, secondary cycles or crosslinks.[26,32-33] Cyclization will promote higher local conversion, since it does not decrease mobility of the system as much as cross-linking. However, cyclization can lead to a reduction in effective crosslinking density, with a reduction in composite properties.[33]

With LED (22 seconds), PAC (5 seconds) and PAC (7.5 seconds), DC values after 1 month were not statistically different from those observed after 24 hours (p>0.05). With an LED exposure of 22 seconds, this probably occurs because this method provided a higher initial DC value and, according to Halvorson and others,[14] with increased conversion, there are greater diffusion limitations.

The methods PAC (5 seconds) and PAC (7.5 seconds) did not present any statistically significant increase in DC data (p>0.05), probably because these methods provided high energy density values for composites with high light intensities, creating a fast polymerization. Faster polymerization may generate shorter chain lengths than slower polymerization.[6] However, a shorter chain length may be associated with increased crosslinking,[6,14] and this highly crosslinked polymer develops a network that restricts mobility of the reacting constituents.[34] This study's findings agree with previous studies which have demonstrated that limiting conversion was found when maximum intensity was applied.[35-36]

CONCLUSIONS

This study showed that, depending on the irradiation condition applied, composites could present significant differences in initial post-cure and DC values .

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