Materials Research. 2015; 18(2): 240-244
DOI: http://dx.doi.org/10.1590/1516-1439.267514

Wavelength of Experimental LEDS: Hardness, Elastic Modulus, Degree of Conversion and Temperature Rise of a Microhybrid Composite

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Received: January 27, 2014; Revised: March 4, 2015

The aim of this study was to evaluate the effect of different peak wavelengths (450nm, 468nm and 490nm) of experimental LEDs on hardness, elastic modulus, degree of conversion and temperature rise of a microhybrid resin composite – Venus® (Heraeus Kulzer). Hardness and elastic modulus were determined by nanoindentation technique (n=5), degree of conversion was measured by FTIR (n=5) and temperature rise was measured with a thermistor (n=30). Data were submitted to ANOVA and multiple comparisons tests ($\alpha=0.05$). Mechanical properties and degree of conversion ($p<0.001$) were superior on the top surfaces of the specimens. 468nm showed the highest mechanical properties values. There was no statistical difference in the degree of conversion ($p=0.51$) and in temperature rise ($p=0.06$) among all LEDs. Hardness and elastic modulus were influenced by LED´s wavelength, whereas degree of conversion and temperature rise were not influenced.

Keywords: *laboratory research, composite resin, mechanical properties, degree of conversion, temperature rise, LED, wavelength*

1. Introduction

Composites are expected to reach a high degree of curing for adequate clinical performance[1]. When inadequately polymerized, composites can have their biological, physical and mechanical properties compromised[2,3]. As a result, marginal degradation and discoloration[4], postoperative sensitivity and pulp irritation may occur[1,5]. The efficiency of polymerization depends on several factors, including those related to the material itself in terms of its composition[1,6], and those related to the light-curing unit (LCU), such as light intensity, spectral distribution, thermal emission and exposure time[7].

LED (Light Emitting Diode) LCUs are gradually replacing halogen LCUs because they have a longer lifetime, and the light flux is not compromised with time[4,8]. A suitable photoactivation process is of great importance to emit radiant energy at a specific wavelength[9,10]. The LEDs' spectral range is narrow and light is emitted with a wavelength near 470nm[3,8], matching the camphorquinone's peak[3,11,12]. Compared to halogen LCUs, LEDs thermal emissions are minimal[8]. This is of great clinical importance since excessive heat can be hazardous to the pulp[13].

One of the methods used to assess whether a composite is properly cured is a study of its hardness. Nanoindentation is one of the methods that can be used to determine not only the hardness but also the elastic modulus of the composite through curves of applied load and penetration on the specimen surface[14,15,16]. The efficiency of polymerization can also be evaluated by studying the degree of conversion (DC),[4] which can be reached by Fourier Transform Infrared Spectroscopy (FTIR)[3,4,17,18].

Several studies have been conducted focusing on the relationship between the energy density of LED LCUs and the degree of polymerization of composites[17,19,20]. However, there is little data about the performance of experimental LED LCUs with different wavelength peaks evaluated through DC, hardness and the elastic modulus of the cured composite. Moreover, authors found LED LCUs whose wavelength peak is below the optimum value for activation of camphorquinone[21,22].

The aim of this study was to evaluate the effect of different peak wavelengths of experimental LED LCUs on the hardness, elastic modulus, degree of conversion and temperature rise of a composite. Our hypothesis is that

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468nm peak wavelength will present the best values for the assessed properties.

2. Material and Methods

For this study, a Bis-GMA based microhybrid resin composite – Venus® (Heraeus Kulzer, GmBH, Wehrheim, Germany), A3 colour (batch 010125) was used.

A cylindrical steel mould of 4 mm in diameter and 2 mm thick was placed on a polyester strip and a glass slide was used to prepare the specimens. The mould was filled with the composite, and another polyester strip followed by another glass slide were placed over the filled mould. This process ensured the specimens were of a constant thickness and a standard distance from the tip of the curing light.

Using experimental LED LCUs (MMOptics, São Carlos, SP, Brazil) specifically developed for this study, the composites were photopolymerized immediately after their insertion into the mould. The power density of these LED LCUs was 350mW/cm^2 and the energy density applied was 21 J/cm^2. LED LCUs wavelength calibrations were obtained from a spectrophotometer (USB 4000, Ocean Optics) which showed 450nm (L450), 468nm (L468) and 490nm (L490).

After curing, the specimens were removed from the mould and marked on their top surface. The specimens were stored for 48 hours in a dark container, at relative humidity of 100% at 37 °C. Before hardness and DC tests, the specimens were polished metallographically using silicon carbide discs of decreasing abrasiveness (400, 600, 800, 1200 grit). For the final polishing, special soft discs with diamond suspensions of decreasing grit size (6µm, 3µm, and 1µm) were used, combined with a diamond paste. The specimens were then washed in running water to remove any residual particles. Ten specimens were prepared for each experimental LED, i.e. five for nanoindentation and five for FTIR. Temperature variation measurements were performed on 30 of the specimens prepared for each experimental LED.

Nanoindentation was used to assess the hardness and elastic modulus of the top and bottom surfaces of the specimens. Twenty-five indentations were performed on each surface of each specimen using a diamond Berkovich geometry indenter on a Nanoindenter XP (MTS System Corporation, Oak Ridge, TN, USA). Each indentation comprised a full loading and unloading cycle, with a maximum applied load of 400 mN applied for 30 seconds. The hardness and elastic modulus were calculated from the load curves versus penetration by the Oliver and Pharr method[23].

The DC of the specimens was measured by FTIR in reflectance mode, using an attenuated total reflectance accessory (ATR) on a spectrometer (Spectrum One B, Perkin-Elmer, Beaconsfield, Bacon, UK), using 32 scans with a resolution of 4 cm^{-1} in the range of 4000 to 400 cm^{-1}. Following that, the band from 1570 to 1670 cm^{-1} was scanned again to achieve better resolution in the region of interest. Spectral analysis was performed using Spectrum One software (Perkin-Elmer, Beaconsfield, Bacon, UK). Spectra were acquired from the top and bottom surfaces of the specimens.

The DC was determined by comparing the relative amount of aliphatic carbon double bonds (1638 cm^{-1}) to the aromatic double bonds (1609 cm^{-1}) of the polymerized and non-polymerized phases. The DC was calculated by the following Equation 1:

$$DC(\%) = \left(1 - \frac{\left[\begin{array}{c} abs(C=C\ aliphatic)/ \\ abs(C=C\ aromatic) \end{array} \right] of\ polymer}{\left[\begin{array}{c} abs(C=C\ aliphatic)/ \\ abs(C=C\ aromatic) \end{array} \right] of\ monomer} \right) \times 100 \quad (1)$$

Temperature variation was recorded from a thermistor connected to a multimeter. Temperature measurements started immediately after the resin composite (20 ± 0.5 °C) was inserted in a teflon mould, and thereafter it was obtained every 2.5 seconds until it was polymerized for 60 seconds when the final temperature was recorded. The initial temperature was deducted from the final temperature in order to obtain the temperature variation (ΔT). All measurements were taken in controlled temperature environment (20±1 °C).

The mean values of hardness and elastic modulus of the top and bottom surfaces were subjected to two-way ANOVA, full factorial design, and Games-Howell parametric testing for multiple comparisons, considering the heterogeneous variances. The level of significance was 5%. Statistical analysis was performed using SPSS 18.0 for Windows (SPSS Inc, Chicago, IL, USA).

The mean DC values of the top and bottom surfaces were subjected to two-way ANOVA, full factorial design, and Tukey HSD parametric testing for multiple comparisons, considering homogeneous variances. The level of significance was 5%.

The temperature variation data was submitted to one-way ANOVA criterion. The level of significance was 5%.

3. Results

The mean values of hardness, elastic modulus and hardness of the ratio between the bottom and top (B/T) surfaces of the light-cured resin composite with different LED LCUs are shown in Table 1. The top surface presented higher values for the hardness and elastic modulus compared to

Table 1. Mean values (standard deviation) of hardness (GPa), elastic modulus (GPa) and hardness of ratio between the bottom and top (B/T) of evaluated resin composites.

	LED	Top	Bottom	B/T (%)
Hardness	450 nm	0.73 (0.03) aA	0.44 (0.02) aB	61 (0.03) a
	468 nm	0.76 (0.04) bA	0.71 (0.01) cB	94 (0.06) c
	490 nm	0.76 (0.02) bA	0.50 (0.03) bB	66 (0.04) b
Elastic Modulus	450 nm	13.09 (0.37) aA	9.25 (0.39) aB	
	468 nm	13.87 (0.58) bA	13.04 (0.42) cB	
	490 nm	13.70 (0.44) bA	10.78 (0.49) bB	

Mean values followed by different lowercase in column and different uppercase in line exhibit significant differences, with a significance level of 5%.

Table 2. Mean values (standard deviation) of DC (%) of evaluated resin composite.

LED	Top	Bottom
450 nm	69.40 (6.06) aA	28.91 (0.36) bB
468 nm	64.40 (6.18) aA	27.04 (3.67) bB
490 nm	63.33 (2.92) aA	26.96 (1.06) bB

Mean values followed by different lowercase in column and different uppercase in line exhibit significant differences, with a significance level of 5%.

Table 3. Mean increase (standard deviation) of temperature (degree Celsius) of each LED tested.

LED	Temperature variation (°C)
450 nm	1.89 (0.27) a
468 nm	1.71 (0.36) a
490 nm	1.87 (0.36) a

Mean values followed by same lowercase do not exhibit significance differences, with a significance level of 5%.

the bottom surface ($p < 0.001$). When only top surfaces were considered, L450 provided the lowest values for the hardness and elastic modulus ($p < 0.001$). There was no significant difference between L468 and L490 on both the hardness ($p = 0.87$) and elastic modulus ($p = 0.10$). Considering the bottom surfaces, L468 generated the highest hardness and elastic modulus. Regarding B/T's hardness, L468 provided the highest value, followed by L490 and L450.

The mean values and standard deviation of DC are shown in Table 2. DC was significantly higher on the top surface compared to the bottom surface ($p < 0.001$), for all LED LCU. There were no significant differences among all LED LCUs tested on the top and bottom surfaces ($p = 0.51$).

Regarding temperature variation, there was no significant difference among the LEDs LCU tested ($p = 0.06$) (Table 3).

4. Discussion

The effectiveness of composite restorative procedures is directly dependent upon its polymerization. As curing equipments are fundamental in achieving this goal, several studies have focused on the irradiance of LEDs LCUs[17,19,20]. However, other variables such as the wavelength emitted by LED LCUs and temperature variation are also relevant in determining the efficiency of polymerization of resin composites.

In this study, different wavelength peaks influenced the polymerization of a resin composite differently. The lowest hardness and elastic modulus values were found on composite's bottom surface. Previous studies had also found similar results[12,21,24,25]. During photopolymerization, there is a considerable reduction of irradiance in deeper regions through the bulk of the composite, due to absorption and scattering of light by the resin matrix and particles filler[25]. This decrease in light results in reduced photons emission, interfering in the material's curing, and resulting in lower values for the mechanical properties tested.

The effectiveness of polymerization cannot only be measured by the hardness of the composite's top surface. Hardness of the bottom is mostly affected by light intensity, and is therefore considered a more accurate parameter for evaluating the effectiveness of curing[24], and consequently the performance of the LCUs. Considering only the bottom surface, L468 generated the highest results for hardness and elastic modulus. Since the energy density was kept constant for the three LED LCUs, we could suggest the influence of the light's wavelength on the results. Previous studies report that not only the irradiance but also the wavelength of the emitted light has a direct effect on the degree of polymerization of resin composites[12,21]. According to Nomoto[11], the polymerization of composites is affected by light wavelength, 470 nm being the most efficient because it maximizes the camphorquinone activation[1,10,19], which is the most commonly present photoinitiator in the composition of composites[26]. If the composite photoinitiator does not absorb enough photons at an appropriate wavelength, polymerization may be impaired[11]. In this study, the blue light in different parts of the absorption spectrum of the camphorquinone produced different levels of curing efficiency. The wavelength closer to the absorption peak was more effective in polymerization, reflected by a higher hardness and elastic modulus. These same results were found in earlier studies[27,28]. Comparing different photoinitiators, Price et al.[12] also reached the same conclusions. The LEDs LCU with a wavelength peak near 470 nm polymerized the composite more efficiently when the photoinitiator was camphorquinone.

The B/T ratio of L468 demonstrates that the wavelength peak coinciding with the maximum spectral absorption of camphorquinone meant that there was a better composite depth of curing[29] . To considerer a composite's depth of cure to be adequate, the B/T ratio should be greater than 80%[30]. On the other hand, Torno et al.[21] do not consider that the B/T ratio alone is a good method to evaluate the polymerization effectiveness[21]. That is because if the composite is not properly cured but has a bottom hardness value similar to the top hardness value, the B/T ratio can be greater than 80%, and as such the deficient polymerization will be considered suitable.

Despite the low irradiance of experimental LEDs, the DC at the top of the composite was within the acceptable pattern, that is between 55 and 75%[31]. Emami et al.[32], Peutzfeldt & Asmussen[17] and Gritsch et al.[22] showed that lower power densities can be compensated for by increasing the photopolymerization time, which increases the energy density, and thus induces a higher degree of polymerization. They justify this finding by the kinetics of polymerization, i.e. lower power densities and longer periods of time slow down the formation of a rigid polymer chain, which allows for more efficient polymerization. Continuous exposure to light helps maintain the activation of the camphorquinone molecules near to the surface[24], increasing the material's degree of polymerization on top surface[33]. On the other hand, studies demonstrate that very high power densities applied in a shorter time do not increase the DC of composites[32,34]. The DC on the bottom surface of the composite was lower, which is consistent with other studies[19,31,32]. The DC was not influenced by the light wavelengths, either on the top or on the bottom of the resin composite.

This study did not find any association between hardness and DC. Although some studies have reported an association between hardness and DC[35,36], others do not agree with this association[20,37]. Ferracane[36] and Obici et al.[37] justify this result based on the fact that the mechanical properties of a resin composite are much more dependent on crosslinks density and on the quality of the polymer chain formed during polymerization reaction than in the DC itself. Increasing the exposure time of the composite to light can result in longer polymer chains with fewer crosslinks, thus not reducing the DC but only affecting its mechanical properties. Ferracane[36] also adds that an absolute value for DC cannot predict an absolute value of hardness for all composites. Obici et al.[37] conclude their study by pointing out that we should be cautious when hardness is considered as an indicator of DC. In some instances, composites with similar DC can present crosslinkings with different densities, which in turn can affect hardness.

The temperature variation generated by LED LCUs was low. The low power density of experimental LED LCUs can explain this finding, in agreement with results of other studies[2,13,27,38]. Studies show that the higher the irradiance, the greater the heat generated by LCUs[38,39,40]. The authors also argue that LED LCUs have a narrow light spectrum and this causes the lower heat emission[13,38]. Not only are the emitted light spectrum and irradiance involved with a temperature increase, but also the exposure time, cavity depth[40] and chemical composition of resin composites[21].

5. Conclusions

Hardness and elastic modulus were affected by the LED LCUs' wavelength, with the highest values for 468nm. However, both DC and temperature variations were not affected by the LED LCUs' wavelengths evaluated.

Acknowledgements

The authors thank the resin and LCUs manufacturers for providing the materials, the Nanostructured Materials Laboratory (Physics Department, UFPR) and the Polymer Laboratory (Materials Sciences and Engineering Program, UDESC) for their assistance.

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