

## Influence of different shades and LED irradiance on the degree of conversion of composite resins

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**Abstract:** The aim of this study was to evaluate the degree of conversion (DC) of two composite resins with different shades that were light cured by light-emitting diodes (LEDs) of different irradiances. Specimens (5 mm × 2 mm) were prepared with a nanofilled (Filtek Supreme – A2E, A2D, and WE) or microhybrid resin (Opallis – A2E, A2D, and EBleach Low) and were randomly divided into 12 groups (n = 5 each) according to the composite resin and light-curing unit (Elipar FreeLight 2, 1250 mW/cm<sup>2</sup>; Ultralume 5, 850 mW/cm<sup>2</sup>). After 24 h, the DC was measured on two surfaces (top and bottom) with Fourier Transform infrared spectroscopy (FTIR). Data were statistically analyzed with two-way ANOVA and Tukey test ( $\alpha = 0.05$ ). Statistical differences among the surfaces were observed in all experimental conditions, with higher values on the top surface. The microhybrid resin presented the highest DCs for shades A2E and A2D on the top surface. The LED with higher irradiance promoted better DCs. Taken together, the data indicate that the shade of a composite resin and the irradiance of the light source affect the monomeric conversion of the restorative material.

**Descriptors:** Composite Resins; Polymerization; Curing Lights, Dental.

### Introduction

To promote an adequate and natural appearance, composite resins are available in a variety of colors and shades; however, this characteristic can make the restorative procedure challenging for clinicians.<sup>1</sup> Composite resin shades vary substantially among manufacturers.<sup>2</sup> Despite having the same designation (e.g., A2 or B1), characteristics like color and translucency can differ according to the composite type, because the composition and inorganic content influence the optical properties of these materials.<sup>3</sup>

As light passes through a translucent material, it is dispersed, such that an object cannot be viewed clearly through the material. In other words, translucency is an intermediate state between complete opacity and transparency. Composite resins with different translucencies can present distinct behaviors with regards to the degree of conversion (DC), due to light dispersion in the materials.

Different light sources can be used to stimulate photoinitiators to begin free radical formation and start the curing process.<sup>4-6</sup> Efficient curing depends on the light-curing irradiance and wavelength emitted,<sup>7,8</sup> such

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that the spectrum irradiated by the device is higher than the absorption spectrum of the photoinitiator.<sup>9</sup> Despite having different levels of irradiance than halogen lamps (QTH), light-emitting diodes (LEDs) present irradiance concentrated around 470 nm, coincident with the absorption peak of camphorquinone.<sup>8,10</sup> In some situations, this characteristic favors the improved DC of dental adhesives.<sup>11</sup> However, the influence of different irradiances on the DC of composite resins is questionable.

As manufacturers introduce more options for composite resins, with different shades and compositions to promote better performance and characteristics similar to those of the natural tooth, studies are necessary to evaluate the influence of these parameters on composite behavior.

The aim of this study was to evaluate the DC of composite resins (microhybrid and nanofilled) of different shades that were light cured by LEDs with distinct irradiances. The null hypothesis was that differences in DC would not be observed among the shades and types of composite resins, and that monomer conversion would not be influenced by the LEDs tested.

## Methodology

Two composite resins were used in the current study:

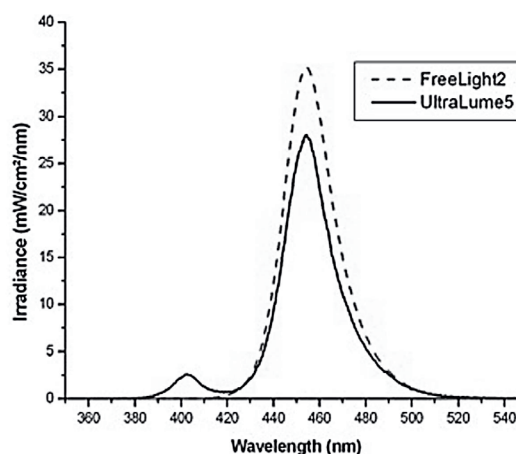
- a nanofilled resin (Filtek Supreme XT) and
- a microhybrid resin (Opallis).

The commercial names, composition, and manufacturers of the resins used are listed in Table 1.

Specimens were divided into groups (n = 5 each) according to the light-curing irradiance (Elipar™ Freelight 2 [EF], 3M ESPE, St. Paul, USA, 1250 mW/

cm<sup>2</sup>; Ultralume 5 [UL], Ultradent Products Inc., South Jordan, USA, 850 mW/cm<sup>2</sup>) and shades (Supreme – A2E, A2D, and WE; Opallis – A2E, A2D, and Ebleach Low). The experimental groups are listed in Table 2. The wavelength spectra and peak emissions of tested LEDs are presented in Figure 1.

Specimens were prepared with a metallic matrix (5 mm Ø and 2 mm height). Composite resin was inserted in a single increment. A mylar strip and 500 g weight were placed over the mold and left for 20 s, to allow for better accommodation of the composite. Specimens were light cured according to the manufacturers' instructions (20 s with Filtek Supreme A2E/A2D/WE and Opallis A2E/ Ebleach Low; or 40 s with Opallis A2D) with the light-curing unit corresponding to each group. Specimens were stored for 24 h at 37 °C under light-protected and dry



**Figure 1** - Light spectrum profiles emitted by the light-curing units. Elipar Freelight 2 presented an emission spectrum between 415 and 520 nm. Ultralume 5 presented a spectrum similar to that of Elipar Freelight 2; however, its wavelength spectrum was extended, with initiation at 385 nm and a peak at 405 nm. Both light-curing units had a peak at 454 nm.

**Table 1** - Composition of the composite resins used in the present study.

Composite	Composition	Manufacturer
Filtek Supreme XT	Bis-GMA, Bis-EMA, UDMA, TEGDMA Filler: 59.5% by Wt - silane treated ceramic (65-75% by Wt) and silane treated silica (5-15% by Wt) – size: 20 and 75 nm	3M ESPE, St. Paul, Min., USA
Opallis	Bis-GMA, Bis-EMA, TEGDMA Filler: 57% by Wt - silane treated ceramic (65-75% by Wt) and silane treated silica (5-10% by Wt) – size: 3 µm	FGM Produtos Odontológicos Ltda, Joinville, SC, Brazil

Bis-GMA (Bisphenol A Diglycidyl Ether Methacrylate), Bis-EMA (Bisphenol A Polyethylene Glycol Diether Dimethacrylate), TEGDMA (Triethylene Glycol Dimethacrylate), UDMA (Diurethane Dimethacrylate).

conditions. After this period, both the top and bottom surfaces were polished with abrasive papers of decreasing grit (400, 600, and 1200, Buehler Ltd., Lake Bluff, USA) to analyze monomer conversion.

The DC of the composite resin was measured on the bottom and top surfaces of each specimen. Measurements were performed with Fourier Transform infrared spectroscopy (FTIR - Spectrum 100

Optica; PerkinElmer, USA), equipped with an attenuated total reflectance (ATR) device, in a room with controlled temperature and humidity. The DC (%) was evaluated in the absorbance mode with a baseline technique<sup>12</sup> and was traced by the Spectrum program (Spectrum 100 Optica; PerkinElmer, USA). The DC was calculated with an equation considering the intensity of the C=C stretching vibration (peak height) at 1638 cm<sup>-1</sup> and, as an internal standard, with symmetric ring stretching at 1608 cm<sup>-1</sup>.

The DC results were statistically analyzed by split-plot two-way ANOVA and Tukey test ( $\alpha = 0.05$ ) with the SAS 9.1 software package (SAS Institute, Cary, USA).

## Results

Table 3 shows the results obtained in the present study. A significant interaction was found between the variables “composite resin” and “light source” ( $p < 0.01$ ). Specimen tops showed better DC values than specimen bottoms. The nanofilled composite resin presented lower DC values than the microhybrid resin in all situations, except for the A2E shade (bottom), which presented similar results. The high-irradiance LED (Elipar) showed better DC values than the third-generation LED (Ultralume 5).

**Table 2** - Experimental groups according to the light-curing unit, type, and shade of composite resin.

Composite resin	Light curing unit	Shades
Micro-hybrid	EF	A2E
		A2D
		EBleach Low
Micro-hybrid	UL	A2E
		A2D
		EBleach Low
Nanofilled	EF	A2E
		A2D
		WE
Nanofilled	UL	A2E
		A2D
		WE

**Table 3** - Percent means (standard deviations) of the degree of conversion, according to the light-curing unit, type, and shade of the composite resin and surface evaluated.

Light curing unit	Composite resin and shades	Surface		
		Top	Bottom	
EF	Micro-hybrid EBleach Low	59.3 (3.7) Ba	52.2 (3.6) Ab	
	Micro-hybrid A2D	63.5 (1.4) ABa	54.0 (2.0) Ab	
	Micro-hybrid A2E	66.3 (10.3) Aa	55.4 (3.7) Ab	@
	Nanofilled WE	53.2 (2.8) Ca	47.9 (2.3) Bb	
	Nanofilled A2D	53.3 (4.7) Ca	37.7 (1.3) Cb	
	Nanofilled A2E	55.9 (1.3) Ca	49.3 (3.8) ABb	
UL	Micro-hybrid EBleach Low	57.7 (1.6) Ba	55.4 (2.4) Ab	
	Micro-hybrid A2D	62.9 (2.5) ABa	51.5 (2.1) Ab	
	Micro-hybrid A2E	64.7 (1.1) Aa	54.4 (0.9) Ab	*
	Nanofilled WE	49.7 (5.9) Ca	44.9 (5.1) Bb	
	Nanofilled A2D	51.5 (8.1) Ca	31.8 (2.7) Cb	
	Nanofilled A2E	55.5 (1.8) Ca	47.9 (3.1) ABb	

Different letters indicate statistically significant difference (two-way ANOVA/Tukey's Test,  $p < 0.05$ ). Upper- and lower-case letters compare resin on columns and surfaces on lines, respectively. \*/@ indicate differences among the light-curing units.

## Discussion

According to the current results, the resin shade and LED irradiance can affect the DC of the composite resin. Therefore, the null hypothesis of this study must be rejected.

The specimen bottoms (2 mm height) presented lower DC values than the tops. This result can be explained by the increased distance between the light-curing tip and bottom of the composite, which attenuates light activation at the bottom surface and compromises the extent of curing.<sup>13,14</sup> Similarly, Beun *et al.*<sup>15</sup> showed a progressive reduction in DC depending on the specimen thickness when either QTH or LED was used.

The microhybrid resin promoted better conversion than the nanofilled resin. According to some studies, the DC is influenced by the organic content, because characteristics such as reactivity and monomer mobility are related to the formation of polymeric chains.<sup>16,17</sup> The higher amount of filler present in the nanofilled resin could influence the results by promoting a lower DC.

Nanofilled composites are characterized by the wide distribution of nanosized filler particles. This characteristic improves the mechanical properties of the resin. Nevertheless, due to the higher amount of filler, a reduction in the organic matrix can be observed in the interfacial region between the particles. This reduction alters the DC, which is directly related to the concentration of monomer content.<sup>18</sup> In addition, a reduced light intensity reaches the photoinitiator, due to the proximity of the fillers. As a result, the formation of free radicals (which are responsible for the curing process) is reduced.

No differences were observed among the nanofiller resins in terms of the shades on the top surfaces. However, EBleach Low (which was developed to restore bleached teeth) showed lower DC values with the microhybrid resins. It can be speculated that EBleach Low presents an alternative photoinitiator, because camphorquinone has a yellow coloration.<sup>19</sup> Alternative initiators are activated at different wavelengths that are not provided by the LEDs used, due to the short spectrum of light that they emit, which compromises the curing efficacy of this material.<sup>20</sup> Because manufacturers are usually reluc-

tant to reveal the exact compositions of their products,<sup>21</sup> it is not possible to determine whether this situation explains the current results completely.

Greater DC values and similar results were observed among the microhybrid resins on the specimen bottoms. However, the nanofilled resin A2D showed a reduced DC compared to the other shades. As described above, use of high amounts of filler can reduce the formation of free radicals and compromise the DC.<sup>18</sup> The high amount of filler and reduced translucency of this resin likely interfered with the passage of light through the material and decreased the monomeric conversion. This problem was not observed with the microhybrid resin, probably because of the extended curing time applied to shade A2D. The manufacturer's instructions were followed rigidly, because manufacturers generally recommend the best way to use their materials to obtain optimal results. Nevertheless, use of the extended curing time could have promoted improved monomer conversion of the tested nanofilled resins.

Use of high-irradiance LED (1250 mW/cm<sup>2</sup>) resulted in the best DC values. Despite having narrow wavelength spectra (between 415 and 520 nm), the LEDs tested presented a peak emission of around 454 nm (Figure 1). This peak emission is near the absorption peak of camphorquinone (470 nm), which was the photoinitiator used in the resins evaluated. Because the two LEDs displayed similar wavelength spectra, the high irradiance of Elipar FreeLight was most likely responsible for the obtained results. The third-generation LED used (Ultralume 5) has auxiliary LEDs, which expanded the emitted wavelength spectrum<sup>22</sup> (Figure 1). However, due to the proximity of the light source to the specimen at the moment of curing, the advantage of the auxiliary LEDs was not fully realized. As a result, the optimal performance of this LED was restricted.

In conclusion, not only the light source, but also the intrinsic properties of the resin (e.g., translucency, amount, type and size of filler), influence material polymerization. To obtain the best DC, when using resins with low translucency, an increase in curing time (mainly at the bottom of the increment) should be considered.

## Conclusion

The DC of composite resin is influenced by the light source, with high-irradiance LEDs promoting better DC values. Specimen bottoms presented lower DC values in all situations tested, and the nano-filled resin presented lower means of DC. The shade can influence the DC of the top of microhybrid resins and the bottom of nanofilled resins.

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