Evaluation of the influence of light-curing units on the degree of conversion in depth of a bulk-fill resin

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Abstract

Background: It is known that bulk-fill have been widely studied and used by dentists in the clinic. However, the use of light-curing units that do not have the ability to adequately light-cure these materials at the appropriate depth can affect their clinical performance. The aim of this study was evaluating the influence of 5 different light curing units (LCUs) on the degree of conversion (DC) of a bulk-fill resin at depths of 0 to 4 mm and determined the effect of using 20s exposure and 40s.

Material and Methods: Cylinders of composite were made in a stainless steel matrix (n=10). The specimens were exposed from the top surface using 5 LCUs: Valo® Cordless (VA); Radii Plus (RA); Emitter.D (EM), Biolux Plus (BI), Woodpecker® (WO). The emission wavelength and the power density was determined. After the photoactivation, the Raman vibrational modes were calculated taking as reference the peaks at 1,601 (aromatic bonds C=C) and 1,640 cm⁻¹ (aliphatic bonds C=C).

Results: The largest difference in DC in 20s, comparing the values obtained in the first and last layer is for BI, with a variation from 61.24% to 53.86%. Comparing the LCUs, the last layer in 40s DC values are 57.40% (BI), 58.21% (WO), 58.97% (VA), 60.90% (RA) and 62.42% (EM). The higher the dose (J/cm²) and the close the λmax is to the maximum CQ absorption length (λmax ~ 470 nm) the better the DC value.

Conclusions: There was a significant difference in the DC values between the LCUs with increasing depth of the bulk-fill increments. Results indicate significant differences in DC among the different LCUs as well as enhanced DC when using 40s exposure compared to 20s. It is suggested that for DC improvement using lower power photoaactuator increase the exposure time the exposure time should be 20s to 40s.

Key words: Polymerization, Composite Resins, Raman spectroscopy.
Introduction

Clinical use of composite resins has become indispensable in dentistry (1). The clinical versatility of resin requires constant improvements in properties for better performance in long-term restorations, which is reflected in the continued launch of new products on the market (2,3). In 2009, bulk-fill resin was launched and the first material commercially available was Surefil® SDR resin (Dentsply Caulk, Mildford, DE, USA) (3). Recent studies show that the bulk-fill resin has improved mechanical properties (4,5), less polymerization stress and reduced microleakage (6,7). Another advantage would be the possibility of filling the cavity in single increments of up to 4 mm, with minimal polymerization shrinkage during the photoactivation process (8), which means reducing the clinical time and reducing the risk of contamination during the restorative procedure (4).

To do so, one must take into account the type of LCU used, as this may influence the depth of cure of resin composites. The monomer polymer conversion of the resins is directly related to the intensity, wavelength and irradiation time of the LCU (9).

The degree of conversion (DC) directly affects the physical and mechanical characteristics of the composite resin, influencing the durability of the restoration (10). The presence of unconverted carbon double bonds may render the material more susceptible to degradation, promoting reduced color stability and release of substances with potential for toxicity (11). Therefore, an incorrect photoactivation can cause adverse biological reactions, as well as the reduction of mechanical properties (12). In this way, adequate photoactivation is required so that light-curing resins achieve the properties desired by the manufacturer, a basic requirement for long-term predictable clinical success (13). In addition, with the introduction of different LCUs with increasing power, there is a real danger that dentists are not adequately informed about their use, increasing the number of restorative failures (14).

Among the different devices, the spectral radiant power, the active curing tip diameter and the irradiance are different, thus altering the capacity to light-cure the resins (13). Several studies (15-17) have evaluated the curing depth of bulk fill resins, but none have verified the values to their full extent when using different LCUs.

In this context, the objective of this study was to evaluate the DC in depth of the bulk-fill resin Surefil® SDR with different LCUs. The null hypotheses tested were: (1) DC values do not change significantly with increasing depth of restoration; (2) DC in depth of a bulk-fill resin does not depend on the LCU used for photoactivation of the material and (3) DC values do not change significantly with increasing activation time.

Material and Methods

-Experimental Design

The DC in depth was evaluated using a bulk-fill flow resin bulk-fill flow, Surefil® SDRTM Flow (Dentsply Caulk, Mildford, DE, USA), photoactivated in the time of 20 and 40 seconds, with 5 types of light-curing units (LCUs): Valo® Cordless/Ultradent (VA); Radii Plus/SDI (RA); Emitter.D/Schuster (EM), Biolux Plus/Bioart (BI), Woodpecker®/Guilin Woodpecker Medical Instrument (WO). The emission wavelength of each LCU was determined by a linear array spectrometer (VSI40, HORIBA Jobin Yvon, Kyoto, KA, JPN). The power density of each light-curing unit was also determined (407A, Spectra Physics, Mountain View, CA, USA).

Ten specimens were prepared for each group of LCU and for each time totaling 100 specimens. The DC was evaluated by means of Raman spectroscopy after 24 hours.

-Preparation of specimens

For the preparation of the specimens, we used a stainless-steel matrix containing a central hole measuring 2 mm in diameter by 4 mm in height. The matrix was divided in half, with lateral screws, that allow the easy removal of the specimen. The matrix was superimposed on a polyester matrix tape (TDV, Pomerode, SC, BRA) supported on a glass plate and filled with bulk-fill resin. A second polyester tape was placed with light pressure on the matrix in order to avoid excesses, obtain a regular surface and prevent contact with the surface of the light tip (Fig. 1). After the photoactivation at 20 and 40 seconds, each specimen was removed from the matrix and stored in a dry and dark environment at 36ºC for 24 hours.

-Degree of conversion assessment

All specimens were subjected to DC measurement using a confocal Raman microscope (SENTERRA, Bruker)
Optik GmbH, Ettlingen, BW, DEU), 24 hours after preparation. The measurements were taken with an excitation laser wavelength of 532 nm, nominal power of 20 mW, focused on the specimen by a lens of magnitude of 50x, with 3 seconds of integration time, 20 scans and spectral resolution of 9-15 cm⁻¹ in the region between 1,778-419 cm⁻¹. The spectra were corrected by baseline using OPUS software (7.2, Bruker Optik GmbH, Ettlingen, BW, DEU). Twenty points were read along the length of the specimen, from the face irradiated by the LCU.

-Degree of conversion calculation
In order to evaluate the DC of the bulk-fill resin, the Raman vibrational modes were calculated taking as reference the peaks at 1,601 (aromatic bonds C=C) and 1,640 cm⁻¹ (aliphatic bonds C=C). The percentage of unconverted carbon double bonds (% C=C) was determined by the intensity rate of C=C bonds (1,601 cm⁻¹) and C=C bonds (1,640 cm⁻¹) before and after polymerization. The ratio between the two peaks intensity was calculated for both the polymerized and unpolymerized material. The equation used to calculate the DC was:

\[ \text{DC (‰)} = 100 \times (1 - \frac{R_{\text{Polymerized}}}{R_{\text{Unpolymerized}}}) \] (1)

* R = Ratio between peaks 1,640 and 1,601 cm⁻¹

-Statistical analysis
DC are presented as mean ± standard deviation of the mean (SD). After tabulation of the results in a database, they were analyzed using analysis of variance (ANOVA). The Shapiro-Wilk test was used to assess the sample normality pattern. The Tukey test was used to compare the different depths and different photoactivation times. The significance level of \( p < 0.05 \).

**Results**
The emission spectrum of the LCUs evaluated showed that the devices work at a wavelength of 420 to 500 nm, and for VA there is a peak of light emission in the ultraviolet region (Fig. 2), wavelength region that is required to activate camphorquinone photoinitiator present in the SDR resin (13). The average power rating of each appliance is listed in Table 1. Figure 3 illustrates the Raman spectra of the SDR resin before and after polymerization for 20 and 40 seconds. Note the polymerization effect by decreasing peak intensity at 1,640 cm⁻¹; this change is associated with the formation of the polymeric structure.

Table 2 presents the significant differences in DC between photoactivators and depths. Comparing the devices, at the time of photoinitiation of 20s, the first depth layer of the resin presented homogeneous DC values for all LCUs. However, in 1-2 mm BI shows the lowest DC values. From 2 mm, BI, WO and RA presented the lowest values. In 40s time, BI showed statistical differences between all photoactivators at all resin depths. In the last layer, DC values in ascending order are 57.40% (BI), 58.21% (WO), 58.97% (VA), 60.90% (RA) and 62.42% (EM).

In general, within the same photoactivator, the layers presented statistical differences from 3mm. The largest difference in DC in 20s, comparing the values obtained in the first and last layer is for BI, with a variation from 61.24% to 53.86%. In the 40s, the WO photoactivator is responsible for the greatest variation between the first and last layers, with 64.11% and 58.21%, respectively.

![Fig. 2: LCUs emission spectrum.](image)
Influence of LCUs in depth of a bulk-fill resin

Table 1: Specifications of LCUs tested in the study.

| Commercial brand/ Manufacturer       | Wavelength (nm) | λmax (nm) | Power density (W/cm²) | LCU dose 20s (J/cm²) | LCU dose 40s (J/cm²) |
|---------------------------------------|-----------------|-----------|----------------------|----------------------|----------------------|
| Valo® Cordless/Ultradent (VA)         | 375 - 500       | 458       | 1.01                 | 20.2                 | 40.4                 |
| Raddi Plus/SDI (RA)                  | 420 - 495       | 452       | 1.30                 | 26.0                 | 52.0                 |
| Emitter.D/Schuster (EM)              | 420 - 500       | 457       | 1.25                 | 25.0                 | 50.0                 |
| Biolux Plus/Bioart (BI)              | 420 - 490       | 449       | 0.88                 | 17.6                 | 35.2                 |
| Woodpecker®/Guilin Woodpecker        | 420 - 490       | 455       | 0.52                 | 10.4                 | 20.8                 |

Table 2: Mean ± SD for ranges within 1 mm thick increments of the degree of conversion (%) of bulk-fill SDR resin at different depths and different photoactivation times (values measured every 0.2 mm in figure 4 were summed and the average was obtained for each mm depth).

| Depth (mm) | VA          | RA          | EM          | BI          | WO          |
|------------|-------------|-------------|-------------|-------------|-------------|
| 0 - 1      | 62.6±0.6Aa  | 61.2±0.4Aa  | 62.2±0.9Aa  | 61.2±0.7Aa  | 61.1±0.7Aa  |
| 1 - 2      | 62.1±0.7Aa  | 61.2±0.3Aa  | 62.6±0.4Aa  | 60.4±0.3Ab  | 61.0±0.6Aa  |
| 2 - 3      | 60.9±0.5Aa  | 59.5±0.7Bb  | 62.1±0.5Aa  | 57.7±1.2Bb  | 58.9±1.3Bb  |
| 3 - 4      | 58.9±1.8Ba  | 56.4±1.8Cb  | 60.4±1.4Aa  | 53.9±2.9Cb  | 56.9±1.9Ba  |
| 0 - 1      | 62.2±0.7Aa  | 64.3±1.2Aa  | 64.3±1.1Aa  | 61.5±1.3Ab  | 64.1±0.7Aa  |
| 1 - 2      | 63.4±0.6Aa  | 65.3±0.5Aa  | 64.5±0.9Aa  | 60.9±0.9Ab  | 63.3±0.9Aa  |
| 2 - 3      | 62.3±0.5Aa  | 64.8±0.6Aa  | 64.3±0.8Aa  | 59.7±0.9Ab  | 61.3±1.2Aa  |
| 3 - 4      | 59.7±2.6Ba  | 60.9±4.9Ba  | 62.4±4.1Ba  | 57.4±1.9Ab  | 58.2±2.6Bb  |

Within their respective times of photoactivation, capital letters compare the columns (DC between different LCUs) and lowercase letters compare lines (DC in depth) (p < 0.05).

Fig. 3: Peaks of 1,640 cm⁻¹ (aliphatic carbon) and 1,601 cm⁻¹ (aromatic carbon), before and after polymerization along the extension of the specimen. (A) Photoactivated for 20 seconds; (B) Photoactivated for 40 seconds.

Table 2: Mean ± SD for ranges within 1 mm thick increments of the degree of conversion (%) of bulk-fill SDR resin at different depths and different photoactivation times (values measured every 0.2 mm in figure 4 were summed and the average was obtained for each mm depth).

| Depth (mm) | VA          | RA          | EM          | BI          | WO          |
|------------|-------------|-------------|-------------|-------------|-------------|
| 0 - 1      | 62.6±0.6Aa  | 61.2±0.4Aa  | 62.2±0.9Aa  | 61.2±0.7Aa  | 61.1±0.7Aa  |
| 1 - 2      | 62.1±0.7Aa  | 61.2±0.3Aa  | 62.6±0.4Aa  | 60.4±0.3Ab  | 61.0±0.6Aa  |
| 2 - 3      | 60.9±0.5Aa  | 59.5±0.7Bb  | 62.1±0.5Aa  | 57.7±1.2Bb  | 58.9±1.3Bb  |
| 3 - 4      | 58.9±1.8Ba  | 56.4±1.8Cb  | 60.4±1.4Aa  | 53.9±2.9Cb  | 56.9±1.9Ba  |

Within their respective times of photoactivation, capital letters compare the columns (DC between different LCUs) and lowercase letters compare lines (DC in depth) (p < 0.05).
Figure 4 shows the significant differences in CG measured every 0.2 mm at different times. The increase from 20s to 40s in the photoactivation time of the resin results in the increase of DC, with the exception of the VA photoinitiator that maintained the DC similar when comparing the times. The RA obtained the greatest statistical differences comparing the two times of photoactivation.

Discussion
The null hypotheses were rejected because the DC values varied significantly according to the increase in the depth of the material, the LCU and the time of photoactivation. As the light absorption of photoinitiators is essential to improve the efficiency of the photochemical reaction, it is important to select resins with absorption spectra that overlap the emission spectra of the irradiation sources. The literature describes that in order to improve the photoactivation depth of bulk-fill resins, several characteristics have been introduced into their composition (6,16,19), which provides a more uniform conversion of monomers in depth (20-22).

According to the manufacturer, resin has a photosensitive molecule called camphorquinone (CQ) and a new UDMA-based monomer that reduces resin shrinkage stress (10,23). The results showed that the LCUs exhibited 420-490 nm emission peak, coinciding with the maximum absorption peak of the CQ (24). The emission peak in the ultraviolet region found in Valo (Fig. 2) is because it is a polywave device. For bulk-fill resins containing only CQ as a photoinitiator, as in the resin case, the monowave and multywave LEDs have shown the same efficiency (1,24,25). For composites containing CQ associated with alternative photoinitiators, the polywave LED has higher DC because these alternative photoinitiators require shorter wavelengths.
were performed clinically, the distance from the cusps, have presented better results because if the restorations in this study. Because it is an in vitro study, the DC may be inserted, the photoactivation time of the resin and the type of LCU used, in order to increase the clinical life of restorations and decrease sensitivity, microleakage and caries recurrence.

Conclusions
In summary, there was a significant difference in the DC values between the LCUs and with increasing depth of the resin increments. It was also observed that the increase in the time of LCUs results in increased DC of the resin. It is suggested that for DC improvement using lower dose photoactivators increase the exposure time from 20 to 40s.

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Author’s contributions
Fernanda Tsuzuki contributed in the collection of data, analysis, interpretation of data and revising it for critically important intellectual content; Lidiane Castro-Hoshino contributed in the conception, analysis and interpretation of data; Larissa Lopes contributed in the collection of data, analysis and interpretation of data; Francielle Sato contributed to interpretation of data and revising it for critically important intellectual content; Mauro Baesso contributed in the conception and interpretation of data, drafting the article for critically important intellectual content; Raquel Terada contributed in the conception, analysis and interpretation of data, drafting the article for critically important intellectual content and final approval of the version to be published.

Conflict of interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.