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Effects of radiant exposure and wavelength spectrum of light-curing units on chemical and physical properties of resin cements

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Objectives: In this study, we evaluated the influence of different radiant exposures provided by single-peak and polywave light-curing units (LCUs) on the degree of conversion (DC) and the mechanical properties of resin cements. ***Materials and Methods:*** Six experimental groups were established for each cement (RelyX ARC, 3M ESPE; LuxaCore Dual, Ivoclar Vivadent; Variolink, DMG), according to the different radiant exposures (5, 10, and 20 J/cm^2) and two LCUs (single-peak and polywave). The specimens were made (7 mm in length × 2 mm in width × 1 mm in height) using silicone molds. After 24 hours of preparation, DC measurement was performed using Fourier transform infrared spectrometry. The same specimens were used for the evaluation of mechanical properties (flexural strength, FS; elastic modulus, *E*) by a three-point bending test. Data were assessed for normality, after which two-way analysis of variance (ANOVA) and *post hoc* Tukey's test were performed. ***Results:*** No properties of the Variolink cement were influenced by any of the considered experimental conditions. In the case of the RelyX ARC cement, DC was higher when polywave LCU was used; FS and *E* were not influenced by the conditions evaluated. The LuxaCore cement showed greater sensitivity to the different protocols. ***Conclusions:*** On the basis of these results, both the spectrum of light emitted and the radiant exposure used could affect the properties of resin cements. However, the influence was material-dependent. (*Restor Dent Endod* 2016;41(4):271-277)

Key words: Degree of conversion; Light-curing unit; Mechanical properties; Resin cements

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Introduction

For many years, the halogen lamp was the main light source used for curing dental resin composites. These devices produce light through a thin tungsten filament at high temperatures,[1,2] emitting a broad spectrum of wavelengths. However, some factors might jeopardize the performance of these light-curing devices and thereby promote light degradation.[3] Therefore, other technologies, such as light-emitting diodes (LEDs), have been introduced as alternatives to the use of the halogen lamp. Semiconductors produce light after receiving an electric current,[1,4] consuming less radiant exposure and having lower degradation than the halogen light devices.[3] In the first generation, LEDs used in dental procedures had a reduced irradiance, which allowed the curing of resins without heating. However, despite their capacity to cure the resin materials, the efficiency of the first-generation LEDs was reduced as compared to the quartz-tungsten halogen lamps.[5] Thus, the second-generation LEDs were developed, emitting a narrow spectrum of wavelengths similar the first-generation LEDs, but with higher irradiance allowing better polymerization of dental resins.[5]

To improve the polymerization of dental resins, due to the availability of materials with different compositions related to monomers and initiators, polywave LEDs have been introduced.[6] These LEDs have the similar capacity of conventional single-peak devices to activate camphorquinone (CQ); however, they present a great advantage since these units emit a broad spectrum of wavelengths, allowing the activation of alternative photoinitiators, such as phenyl-1,2-propanedione (PPD, 393 nm),[7] bisacylphosphine oxide (BAPO, 370 nm),[8] or monoacylphosphine oxide (MAPO, 380 nm).[9]

Material polymerization can be related to the light source,[3,10] and this characteristic is linked to the clinical performance of the materials.[11-13] As previously demonstrated, radiant exposure, irradiance, wavelength, and distance of the light-curing tip can influence the degree of conversion (DC) of resin materials.[11,14-16]

Minimally invasive dentistry has resulted in an increased number of adhesive procedures[17] involving dental adhesives, resin composites, and resin luting agents. The latter materials cited might be classified by the mode of polymerization, such as chemical, light, or dual curing (presenting both chemical and light-curing modes in the same product).[18-20] With respect to the light-curing process, CQ is the most commonly used photoinitiator in dental resins, presenting an absorption peak at 470 nm, corresponding to the blue light spectrum.[6,21] However, because of the yellow appearance of CQ, alternative initiators have been inserted into the resin agents to obtain nearly white materials,[6] allowing the restoration of bleached teeth.

It is important to understand the influence of the current LEDs on commercial resins, since such an understanding allows clinicians to proceed in the best possible way when using different materials and devices, and establishing an effective protocol for each luting agent. Therefore, the aim of this study was to evaluate the influence of different radiant exposures of single-peak and polywave LEDs on the DC, elastic modulus (*E*), and flexural strength (FS) of resin cements. The null hypothesis of the present study was that the spectrum of wavelengths and different radiant exposures would not influence the chemical and physical properties of the resin cements tested.

Materials and Methods

Specimen preparation for degree of conversion analysis and three-point flexural testing

In the present study, three luting agents were used (RelyX ARC, 3M ESPE, St. Paul, MN, USA; Variolink, Ivoclar Vivadent, Schaan, Liechtenstein; LuxaCore Dual, DMG Chemisch Pharmazeutische Fabrik GmbH, Hamburg, Germany). The experimental groups were established according to the light-curing units (LCU) used and the radiant exposures tested (Table 1). In the present study, we used a single-peak LED (Bluephase 16i, Ivoclar Vivadent) and a polywave LED (Bluephase G2, Ivoclar Vivadent). The output power (mW) of the LCUs was measured using a calibrated power meter (Ophir Optronics, Har Hotzvim, Jerusalem, Israel). The light irradiance was determined by dividing the output power by the tip area (mW/cm^2). Spectral distributions were obtained using a computer-controlled spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA) with a 0.35 nm resolution linear array detector, over a wavelength range of 340 to 1,100 nm. The spectral distribution and irradiance data were integrated using the Origin 6.0 software (OriginLab, Northampton, MA, USA) and are shown in Figure 1.

Eight specimens were prepared for each group according to the manufacturer's instructions. For the evaluation of DC, *E*, and FS, a silicone mold was used for preparing a bar-shaped specimen with the following dimensions: 7 mm in length × 2 mm in width × 1 mm in height.[11] These sample dimensions were adapted for the microflexural test to enable single-step polymerization instead of several light activations at different points, according to the ISO 4049 specifications.

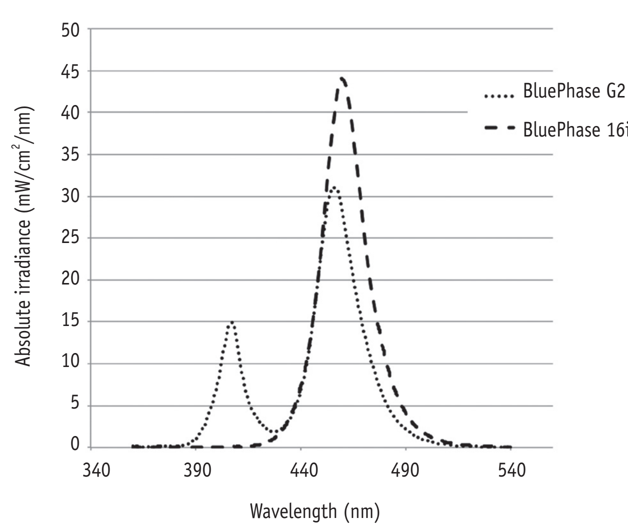

Figure 1. Distribution of wavelengths emitted by the two light-emitting diode sources evaluated in the present study.

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Prior to light activation, a microscope glass slide (0.15 mm thick) was placed over the mold in an attempt to obtain a flat sample surface and avoid the formation of an oxygen-inhibited layer. The resin cements were irradiated immediately after the manipulation, according to the groups with the respective LCU, from the surface of the specimens. After curing, the glass slide was removed and the specimens were stored in light-proof vials for 24 hours at 37℃.

Analysis of degree of conversion

After 24 hours of storage, DC was measured for five specimens by using a Fourier transform infrared spectroscope (FTIR, Spectrum 100 Optica, PerkinElmer, Cambridge, MA, USA), equipped with an attenuated total reflectance (ATR) device with a horizontal ZnSe crystal (Pike Technologies, Madison, WI, USA). The specimens were kept in contact with the horizontal face of the ATR cell.

A preliminary reading for the uncured material was recorded under the following conditions: frequency range of 1,665 – 1,580 cm^{-1}, 32 scans, resolution of 4 cm^{-1}, and Happ-Genzel apodization in the absorbance mode. DC was calculated using a baseline technique based on the band ratios of 1,638 cm^{-1} (aliphatic carbon-to-carbon double bond) and 1,608 cm^{-1} (aromatic component group) as an internal standard between the polymerized and the uncured samples.[22]

Three-point bending test

After the DC analysis, the specimens were subjected to a three-point bending test to measure the FS and *E* at a crosshead speed of 0.5 mm/min in a universal testing machine (Instron model 4411, Instron Corp., Canton, MA, USA). Prior to the test, the dimensions of each specimen were recorded using the Bluehill 2 software (Instron Corp.), which calculated the *E* (GPa) and FS (MPa) values according to the specimen dimensions and tension.

Statistical analysis

The homogeneity and homoscedasticity of the values obtained were analyzed, after which the data for the DC, *E*, and FS of the resin cements were analyzed using two-way analysis of variance (ANOVA), with 'radiant exposure' and 'LCU' as the main variables. All *post hoc* multiple comparisons were performed using Tukey's test. The statistical significance was set at $\alpha = 0.05$. The resin cements were not compared.

Results

Mechanical properties

1. Elastic modulus

The cements Variolink and RelyX ARC were not influenced by the variables tested. In the case of LuxaCore, the effects of the variables 'LCU' and 'radiant exposure' were statistically significant ($p = 0.0001$ and $p = 0.0013$, respectively). However, the interaction effect between the factors was not significant ($p = 0.1723$). The polywave LED promoted higher values of *E* than the single-peak LED. The radiant exposure of 5 J/cm^2 resulted in lower *E* values ($p < 0.05$), and the doses of 10 and 20 J/cm^2 promoted similar results, superior to those of 5 J/cm^2 ($p < 0.05$, Figure 2).

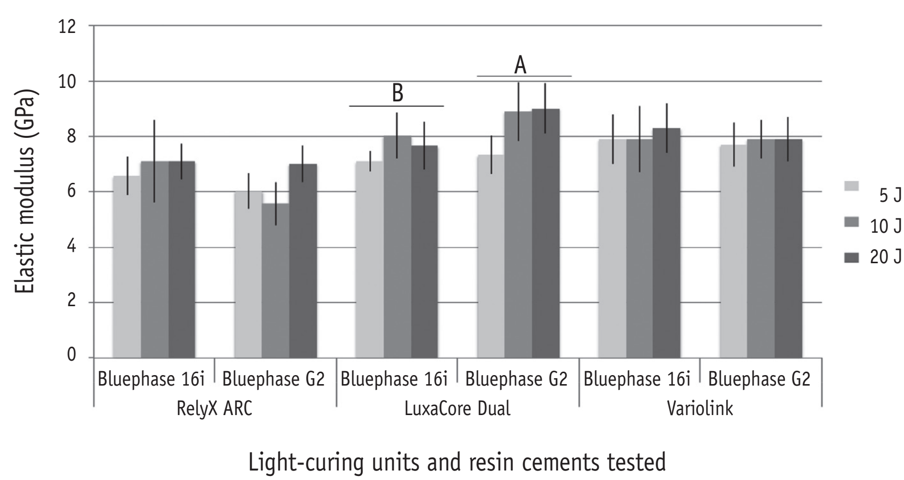

Figure 2. Elastic modulus (*E*, GPa) of the specimens, according to the experimental groups. Capital letters and lines indicate the difference between the light-curing units irrespective of the radiant exposure used (two-way analysis of variance [ANOVA] and Tukey's test, $\alpha = 0.05$). Distinct letters indicate the statistically significant differences. *The resin cements were not compared.

2. Flexural Strength

From the viewpoint of FS, the cement Variolink was not influenced by the experimental conditions. For LuxaCore and RelyX ARC, the effect of the variable 'LCU' was significant (p = 0.0142 and p = 0.0053, respectively), with higher values obtained with the single-peak LED (p < 0.05, Figure 3). The interaction between the factors was not significant for any resin cement.

3. Degree of conversion

The results of DC are presented in Figure 4. As for the mechanical properties, the resin cement Variolink was not influenced by the different experimental conditions. In the case of the LuxaCore dual-cure cement, the effect of the radiant exposure was significant (p = 0.0360), promoting a higher DC with 10 and 20 J/cm^2 and lower values obtained with 5 J/cm^2 (p < 0.05). Only the DC of RelyX ARC was influenced by LCU, with a higher conversion obtained with the polywave LED (p = 0.0270). The interaction between the factors was not significant for any resin cement.

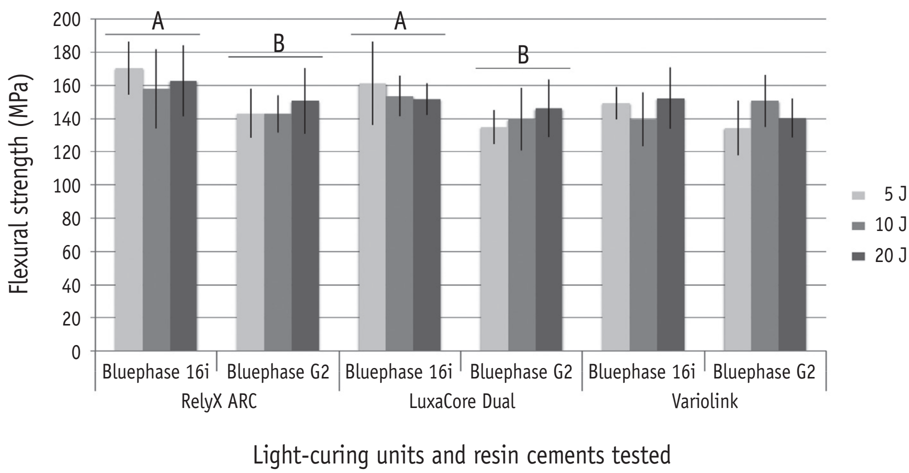

Figure 3. Flexural strength (MPa) of the specimens, in accordance with the light-curing unit and the energy density evaluated. Capital letters and lines indicate the difference between the light-curing units irrespective of the radiant exposure used (two-way analysis of variance [ANOVA] and Tukey's test, α = 0.05). Distinct letters indicate the statistically significant differences. *The resin cements were not compared.

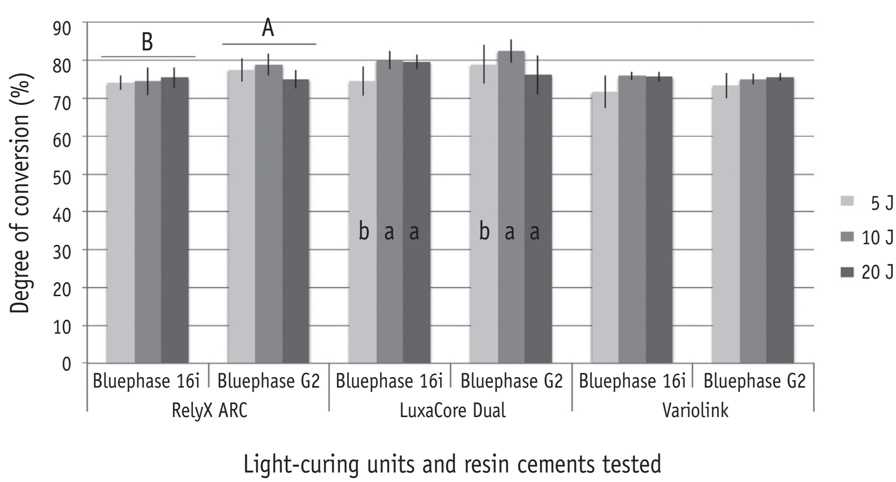

Figure 4. Degree of conversion (%) of the specimens, according to the light-curing unit and the energy density evaluated. Capital letters and lines indicate the difference between the light-curing units irrespective of the radiant exposure used (two-way analysis of variance [ANOVA] and Tukey's test, α = 0.05). Lower-case letters compare the radiant exposures. Distinct letters indicate the statistically significant differences. *The resin cements were not compared.

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Discussion

In clinical practice, several systems are available for the cementation of indirect restorations and/or intraradicular posts. Dual-cure resin cements are more commonly used because of their suitable conversion and mechanical properties, presenting both chemically and light-activated cures.[23,24] Therefore, the wavelength of the LCU and the radiant exposure applied can influence several properties of these materials.

Three radiant exposures were evaluated in this study, from 5 J/cm^2 to the one 4 times higher (20 J/cm^2). Despite the fact that the power of the LCU used in clinical practice is high, these reduced powers were analyzed since some situations, such as post-fixation or cementation of indirect restorations, can drastically reduce the light that reaches the resin materials. On the basis of the obtained results, the null hypothesis was partially rejected, since all the considered resin cements were influenced by the experimental conditions, except for the Variolink cement.

According to the study protocol, the resin cements were not compared, as the aim of this study was to find the best protocol for each material, not the best material. Although the numerical results obtained for the three cements tested were similar, statistical comparisons among them might cloud the main analyses, masking results, since the compositions of the materials differ significantly and can influence all the results.

The mechanical properties of the Variolink system, as well as the DC, were not influenced by the experimental conditions. The system based on bisphenol a-diglycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA) exhibited the same behavior between after curing with 20 J/cm^2 and after curing with just 5 J/cm^2. Despite the large amount of Bis-GMA (around 60 - 70 wt%), the presence of the agent TEGDMA can reduce the viscosity of the system,[25,26] allowing a high-viscosity monomer[25] to obtain the high DC observed (70 - 75%) and improving the mechanical properties of the material even with reduced radiant exposure. It should be noted that this cement and the LCUs tested belong to the same manufacturer. Factors such as initiators and co-initiators,[7,27,28] as well as the selected filler particles and the respective refractive indices[29] can influence the polymerization of the system positively, favored by the characteristics of the LCU, such as wavelength spectrum emitted and the beam profile, resulting in a relatively high compatibility between the resin system and the LCU.

In the case of RelyX ARC, the polywave LED promoted a higher DC. According to the manufacturer's specifications, there is no mention of alternative photoinitiators other than CQ in the composition of the cement. In addition, although the peak of the second-generation LED coincides with the absorption peak of the CQ (468 nm), the broad-spectrum wavelength emitted by the polywave LED can have more excited molecules of the photoinitiator, favoring monomer conversion and thereby improving the DC. However, the FS values of the specimens cured by the polywave LED were lower than those with the single-peak unit probably due to the different polymer network formed because of the differences in the wavelength emission and irradiance among the LCUs.

The resin cement LuxaCore DC was the most influenced by the experimental conditions. *E* increased when this cement was cured by the polywave LED. However, compared with the FS, the single-peak LED exhibited higher results. The single-peak LED may have promoted the formation of a polymer with slightly reduced crosslink chains, through a lower reaction speed, because of the narrow wavelength and the amount of photoinitiator excited, forming a more flexible polymer with higher FS. The radiant exposure was an important factor relevant to the DC of LuxaCore. Higher values were obtained after exposure to 10 and 20 J/cm^2, with lower values at 5 J/cm^2, showing that this cement requires at least 10 J/cm^2 for optimal performance, with respect to not only mechanical properties but also the DC.

The present study demonstrated that the radiant exposure and the wavelength spectrum might influence the chemical and mechanical properties of resin cements. This was not true only for Variolink, which presented similar properties for all the radiant exposures tested, even a low radiant exposure (5 J/cm^2). As discussed, the same manufacturer produces this resin cement and the LCUs analyzed, and this factor can be relevant in the selection of products, as compatibility between the devices and products seems to be important for achieving the best possible performance.

The influence of the light source on resin materials has been demonstrated in other studies.[30-32] However, in the present study, the authors would like to test and establish a suitable protocol for the resin cements tested and indicate the best dose and device to be used according to the material. Despite the fact that the materials tested have not been described in the composition of any initiator other than CQ, the use of a polywave LED unit can allow the activation of a higher number of molecules of the CQ sensitizer, because of the broad spectrum of absorption of this initiator. This point along with the addition of alternative initiators in other commercially available resin materials explains the fact that new LCUs present a polywave peak to guarantee the curing of any resin material present in the market.

The results of this study are important not only for clinicians but also for manufacturers as they demonstrate the importance of adding, in the instructions for use of the materials, information on the wavelength spectrum as well as the minimal radiant exposure required to obtain the best possible performance, thereby alerting users to the possible flaws in the procedure if the recommendations are not followed.

Conclusions

According to the results of the present study, both the wavelength spectrum and the radiant exposure can influence the materials tested. A relatively low radiant exposure (5 J/cm^2) can form a polymer with inferior mechanical properties only in the case of the LuxaCore resin cement. Further, the properties of the resin cement Variolink were not influenced by the experimental conditions tested, showing that the influence is material-dependent.

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Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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