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Original

Curing efficiency of high-intensity light-emitting diode (LED) devices

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Abstract: We evaluated the curing efficiency of 4 high-intensity light-emitting diode (LED) devices by assessing percentage of residual C=C (%RDB), surface microhardness (SM), depth of cure (DC), percentage of linear shrinkage-strain (%LS), and percentage of wall-to-wall contraction (%WWC). The light-curing units tested were a QTH light, the Elipar TriLight (3M/ESPE), and 4 LED devices – the Allegro (Denmat), the Bluephase (Ivoclar/Vivadent), the FreeLight2 (3M/ESPE), and The Cure TC-01 (Spring Health Products). The %RDB was measured by microFTIR spectroscopy. Microhardness measurements (Vickers) were performed at the surface (H0) and at depths of 3 mm (H3) and 5 mm (H5) of cylindrical specimens. Depth of cure was expressed as the ratio of microhardness at each depth, relative to the corresponding surface value (H3/H0 and H5/H0). The bonded disc method was used to evaluate %LS. For the %WWC evaluation, cylindrical resin restorations were imaged by high resolution micro-CT and the %WWC was calculated at depths of 0 mm and 2 mm. There were no statistical differences among the LEDs in %RDB or %LS. The Bluephase and Allegro had the highest SM values. As compared with the other LEDs, the Bluephase and The Cure TC-01 had lower values for depth of cure at depths of 3 mm and 5 mm. There were no significant differences in %WWC among the LEDs at either depth, and the QTH had the lowest %WWC at both depths. (J Oral Sci 52, 187-195, 2010)

Keywords: LED; QTH; light-curing devices; depth of cure; degree of conversion.

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Introduction

Because the demand for esthetic restorative results is growing, the use of light-cured resin composites is increasing. The curing efficiency of light-cured resin composites affects the clinical integrity of resin composite restorations. For this reason, it is important to investigate the factors that control the composite photopolymerization reaction. In addition to the characteristics of materials, light-curing units significantly affect the degree of polymerization in light-activated resin composites.

Until recently, the most commonly used light-curing unit was the quartz tungsten halogen lamp (QTH). Recently, new lamps have been manufactured to improve both curing efficiency and the procedure for photopolymerizing resin composites (1). Currently, 4 main types of polymerization sources are available: halogen bulbs, plasma-arc lamps, argon-ion lasers, and light-emitting diodes (LEDs).

Although the most common method of photopolymerization uses QTH curing units, these units have important disadvantages. The incandescent filament produces heat, which is harmful to pulp integrity (1,2). Although fans are used with QTH units to reduce the temperature increase, this makes the units heavier and less energy-efficient. In addition, halogen lamps produce a wide spectrum of light, so optical filters are necessary. Finally, reflectors, fans, and filters degrade over time and

the effective time of the bulb is therefore limited to between 40 and 100 h of constant use (3-5).

To overcome the aforementioned drawbacks of QTH curing units, blue LEDs were introduced in the late 1990s (6). First-generation blue LEDs had a narrow spectral output (approximately 470 nm), which coincides with the absorbance of the most frequently used initiator, camphorquinone; power density ranged from 160 to 400 mW/cm^2. LEDs depend on electroluminance; therefore, there is lower heat production and less power consumption (7). Moreover, there is no need for fans. As a result, battery-powered cordless lights have been developed. The lifetime of the LED is much greater – approximately 100,000 h – without significant degradation of light flux over time (3-5).

Many studies have attempted to evaluate the properties and polymerization characteristics of resin composites cured with LED curing units (7-11). The majority of these compared LEDs to QTH units with power densities from 300 to 800 mW/cm^2, and found that resin composites polymerized with LEDs had inferior mechanical properties to composites produced with QTH.

Recently, new LED curing units were introduced with higher light intensities, ranging from approximately 500 to 1,400 mW/cm^2. To achieve increased power and a wider spectrum, a higher number of more powerful LEDs are used. The main reason for the production of this new generation of light-emitting diodes was the belief that higher density irradiance would improve the curing efficiency of resin composites. Moreover, a broader spectrum of light, which could be obtained by adding near-UV-exposed LED chips, allows LED curing units to polymerize a wider spectrum of resin-based dental materials (12)

Although there have been numerous investigations of resin composites cured with LEDs, the results have varied considerably, probably because of the multiplicity of test configurations, the individual characteristics of each commercial unit, and the assumptions and approximations integrated into the experimental methodologies. Therefore, it is important to obtain additional data on the performance of newly developed LEDs with light intensities higher than 1,000 mW/cm^2. Because QTH devices remain the gold standard for light-curing units, every LED device produced is commonly compared with conventional QTHs.

The aim of the current study was to evaluate a number of high-power LED curing units by comparing their performance characteristics to those of a QTH unit, using parameters related to photopolymerization. The percentage of residual C=C (%RDB), surface microhardness (SM), depth of cure, relative microhardness (DC), percentage of linear shrinkage (%LS), and percentage of wall-to-wall contraction (%WWC) were determined. The research hypotheses were that there were no differences among the high-intensity LED units and that the curing performance of high-intensity LED units would be superior to that of the QTH unit.

Materials and Methods

The hybrid resin-based composite Spectrum TPH (Detrey/Dentsply, Konstanz, Germany, Batch number: 0005000063), Compule system, shade A2 was used throughout the study. This material contains Bis-GMA-adduct, Bis-EMA, TEGDMA, camphorquinone as photo initiator, stabilizers, barium aluminum borosilicate (mean particle size <1.5 μm), and highly dispersed silicon dioxide (particle size, 0.04 μm)

The resin composite was photopolymerized by 5 different light-curing units 4 high-intensity cordless light-emitting diode (LED) devices, and 1 halogen (QTH) device. The units are listed in Table 1, together with their intensity and total energy values. The intensity of each unit was measured 3 times before the experiment, using the LED radiometer (Demetron Corp., Danbury, CT, USA), and the intensity was calculated as the average of these 3 readings. The total energy delivered (intensity × time) was calculated by integrating intensity of output versus time. All units were used in constant light-intensity mode, with a light guide

Table 1 The light-curing units evaluated

Light-curing unit*	Manufacturer	Intensity (mW/cm^2)**	Total energy (J)
Elipar TriLight (QTH)	3M/ESPE, St Paul, MN, USA	800	32
Allegro (LED)	Denmat, Santa Maria, CA, USA	1250	50
Bluephase (LED)	Ivoclar Vivadent, Amhest, NY, USA	1100	44
FreeLight 2 (LED)	3M/ESPE, St Paul, MN, USA	1000	40
The Cure TC-01 (LED)	Spring Health Products, USA	1250	50

* (LED) light-emitting diode, (QTH) quartz tungsten halogen lamp
** exposure time 40s; light guide tip, 8 mm in diameter

tip 8 mm in diameter and an irradiation time of 40 s for all experiments. The curing tip of the units was in contact with each specimen.

The percentage of residual C=C (%RDB) of the irradiated specimens was measured by micro-multiple internal reflectance Fourier transform infrared spectroscopy (micro-MIR FTIR) after 24 h storage in dark and dry conditions at 37°C. Five rectangular specimens ($4 \times 2 \times 0.5$ mm) were prepared per light-curing unit, between 2 translucent glass slides covered with transparent polystyrene strips. The cured specimens were pressed against the crystal with a torque wrench device. Spectra of the unset paste and of the directly irradiated surfaces were acquired using a micro-MIR cell attached to an FTIR spectrometer (micro-MIR accessory and Spectrum GX FTIR spectrometer, Perkin-Elmer, Norwalk, CT, USA) under the following conditions: range from 4,000 to 400 cm^{-1}, 4 cm^{-1} resolution, 450 para edge KRS-5 minicrystal of 7 internal reflections, 30 scans coaddition at 20 ± 2°C. The %RDB of each specimen was estimated as a relative percentage by using the 2-frequency method and the tangent baseline technique. Aliphatic (C=C) bond stretching vibrations at 1638 cm^{-1} were chosen as the analytical frequency, whereas the aromatic (C..C) bond stretching vibrations at 1605 cm^{-1}, which are not affected by the polymerization reaction, were selected as the reference frequency. The net peak absorbance areas of these peaks were used to quantify the extent of C=C remaining in the directly irradiated resin surfaces relative to the unexposed surfaces.

Differences in microhardness by depth were used to determine the depth of cure of the resin composite. Two different depths were evaluated in this study: 3 mm and 5 mm. Ten cylindrical specimens (4 mm in diameter) were prepared per light-curing unit using black Plexiglas molds of 2 different heights (3 mm and 5 mm). The molds were filled with the resin composite, covered with a transparent celluloid strip. Then, a glass microscope plate was used to remove the excess material, after which the specimens were photopolymerized. Immediately after photopolymerization, the specimens were lightly polished with 600-grit wet silicon carbide paper to remove the superficial resin-rich layer and stored for 24 h in dark and dry conditions at 37°C. The microhardness measurements were made using a Vickers indenter (HMV 2000, Shimadzu, Tokyo, Japan) at the upper (surface microhardness, SM) and lower surfaces (depths of 3 mm and 5 mm). Six measurements per surface for each specimen were made, under a 200 g load, for 10 s. The depth of cure was expressed as the ratio of microhardness values at 3 mm and 5 mm to the corresponding surface microhardness (relative microhardness, DC).

The percentage of shrinkage-strain was measured by using the bonded disk method (13). Resin disks 1 mm in height were mounted on a glass plate. A linear variable differential transformer (LVDT) displacement transducer (Type GT 2000, RDP Electronics, Wolverhampton, UK) was positioned in contact with the center of a flexible ultrathin cover slip ($25 \times 25 \times 0.1$ mm thick) that was initially pressed flat to ensure it was in contact with the supporting 15-mm-diameter brass ring. The LVDT was connected to a microcomputer transient recorder and a data logging system (Bioman, Biomaterials Science Unit, University of Manchester School of Dentistry, UK). The resin specimens were irradiated directly from the bottom, and the cover slip was pulled axially and downward as shrinkage was taking place; the displacement of the LVDT (in mV) at the center of the cover slip was monitored for up to 30 min, at a constant temperature of 37°C. The shrinkage-strain deflection, in mm, of the cover slip and the specimen ($\Delta L = L_0 - L$), where L_0 is the original specimen thickness (1 mm) and L is the final thickness, was determined from the data via voltage/displacement calibration. The percentage of shrinkage-strain [$(\Delta L / L_0) \times 100$] was calculated as a function of time. This deflection was represented as a shrinkage-strain curve over time, and percentage of linear shrinkage (%LS) was calculated for a period up to 30 min after initiation of the light exposure. Three specimens were tested per light-curing unit.

For the estimation of wall-to-wall contraction, 25 freshly extracted human intact molars were used. The teeth were sectioned in the horizontal buccal-lingual direction, below the dentin-enamel junction, using a hard tissue microtome (Isomet, Buhler, Lake Bluff, ILL, USA). Another parallel section was made at a distance of 3 mm from the first, thus producing flat, intact, dentin specimens. In the center of the dentin disks, cylindrical cavities were prepared by using air-rotor carbide burs (End-cutting 956, Jet Co, Kerr, Orange, CA, USA) under continuous water cooling. The cavities were 2 mm in depth and 3 mm in diameter. The cavity dimensions were measured by digital caliper to ensure that standardized cavities were used throughout the study.

The cavities were randomly divided into 5 groups with 5 specimens each and stored in tap water at 37 ± 1°C for 10 min, to obtain full hydration at a clinically relevant temperature. Each group corresponded to a different light-curing unit. Then, the specimens were air-dried with oil-free air and filled with a single layer of each resin composite, without any conditioning, priming, or bonding treatment of the dentin cavity walls. The cavities with the unset composite pastes were stored for 1 min at 37 ± 1°C to simulate an average intraoral handling period. Immediately

after, each composite was covered with a transparent cellulose strip, a microscopic cover slip was used to level the surface by removing residual material, and the composite was then photopolymerized. Then, the surface of each specimen was polished with wet silicon carbide paper of 360-, 600-, and 1,000-grit until the margins were exposed, after which the specimens were rinsed with distilled water to remove debris. The patterns were stored in a dark humid environment for 24 h in 37°C. The specimens were imaged by high-resolution micro-CT (SkyScan-1072, SkyScan, Aartselaar, Belgium) operated under the following conditions: 100 kV accelerating voltage, 98 μA beam current, 1 mm Al filter, 8.09 μm pixel size at 1,024 × 1,024 resolution, 180° rotation at 0.23° step, 6 s exposure time per step, and averaging by 2 frames. For each restoration, 250 horizontal tomographic sections were recorded and reconstructed using the 2D software package provided with the device (CTAn, SkyScan). Eleven sequential sections taken at the top and bottom sites (depth, 2 mm) of the restorations, relative to the axial wall, and the interfacial gap fractions were calculated by using the software. The parameter investigated was the percentage length of the debonded margins, relative to the cavity periphery for each restoration (%WWC).

Statistical analysis of the %RDB, SM, and %LS values was performed using one-Way analysis of variance (ANOVA) and the Student-Newman-Keuls test. The results of DC and %WWC were analyzed using 2-way ANOVA, in which the independent parameters were the depth and the light-curing unit, followed by the Student-Newman-Keuls test. A P value of 0.05 or less was considered to indicate statistical significance; all calculations were performed using the Sigma Stat 3.0 software package (Jandel Scientific).

Table 2 The percentage of residual C=C (%RDB) of the light-curing units (mean ± SD)

Light-curing unit	%RDB
Elipar TriLight	46.8 ± 4.4[a]
Allegro	49.3 ± 3.7[a]
Bluephase	46.2 ± 4.0[a]
FreeLight 2	49.2 ± 4.8[a]
The Cure TC-01	54.6 ± 6.0[a]

Values with the same superscript did not significantly differ.

Results

Table 2 shows the %RDB for all the light-curing units. There were no statistically significant differences among the units.

The values for SM and DC at both depths are shown in Table 3. The DC values were influenced by the depth interaction of the light-curing units. As compared with the other units, the Bluephase and Allegro units had significantly higher SM values; there were no differences in SM among the Elipar TriLight, FreeLight 2, and The Cure TC-01 LED units. The Bluephase and The Cure TC-01 LEDs had lower DC values than the other units at both depths (3 mm and 5 mm). There were no significant differences among the Elipar TriLight, FreeLight 2, and Allegro devices with respect to relative microhardness.

Figure 1 shows representative shrinkage-strain rate curves for the period up to 100 s from the initiation of light-exposure; the %LS values are displayed in Table 4. There were no significant differences in %LS among the units.

Figure 2 shows representative x-ray micro-CT images and Table 5 lists the %WWC values at depths of 0 mm and 2 mm. The Elipar TriLight had the lowest values at both measured cavity sites. There were no significant differences among the LED units at either depth. In addition, %WWC values were significantly higher at a depth of 2 mm, as compared with those for the directly exposed surface (0 mm), regardless of the light-curing unit used.

Discussion

Both hypotheses of the present research were rejected. The high-intensity LED units differed significantly with respect to many of the curing characteristics assessed and were not superior to the QTH unit in curing efficiency.

Table 3 Surface microhardness (SM) and relative microhardness (DC) of the light-curing units (mean ± SD)

Light-curing unit	SM	DC	
		H3/H0	H5/H0
Elipar TriLight	248 ± 21[a]	0.85 ± 0.07[a]	0.85 ± 0.09[a]
Allegro	310 ± 23[b]	0.89 ± 0.07[a]	0.87 ± 0.09[a]
Bluephase	330 ± 40[b]	0.72 ± 0.06[b]	0.75 ± 0.10[b]
FreeLight 2	285 ± 32[a]	0.88 ± 0.09[a]	0.82 ± 0.05[a]
The Cure TC-01	280 ± 39[a]	0.76 ± 0.04[b]	0.77 ± 0.08[b]

Values with the same superscript did not significantly differ.

Table 4 The percentage of linear shrinkage (%LS) of the light-curing units (mean ± SD)

Light-curing unit	%LS
Elipar TriLight	2.6 ± 0.10[a]
Allegro	2.7 ± 0.30[a]
Bluephase	2.6 ± 0.30[a]
FreeLight 2	2.7 ± 0.30[a]
The Cure TC-01	2.5 ± 0.20[a]

Values with the same superscript did not significantly differ.

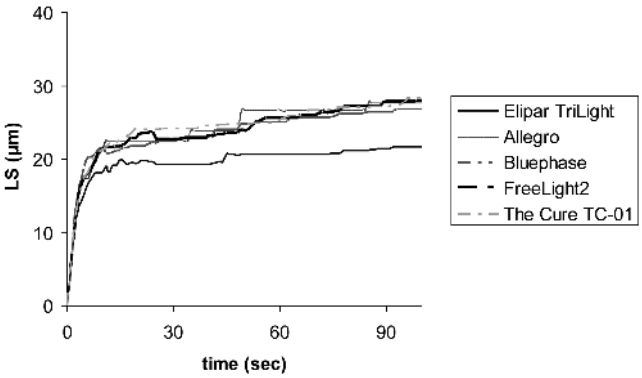

Fig. 1 Representative shrinkage-strain rate curves for the period up to 100 s from the initiation of light-exposure.

Micro-MIR FTIR has been advocated as an analytical technique in quantifying C=C conversion of resin composites (14). In general, the %RDB in commercially available resin composites is between 50% and 60% (15). The present findings show that the evaluated high-intensity LED units can result in a C=C conversion of the resin composite within that range. However, the higher total energy delivered by the LED units, as compared with the QTH, was not correlated with the extent of the reacted double bonds. This finding corroborates the results of several studies, which found no difference between QTH and LED units with respect to C=C conversion, regardless of the intensity of the energy emitted by the LEDs (16-20).

Generally, increased curing intensity leads to a better conversion rate, assuming that the spectrum of the curing unit, irradiation time, and light-guide tip diameter are very similar (4). Regarding spectra emission, LED units differ greatly from conventional QTHs (21). It would be expected that the narrow light spectrum of LEDs, with a peak intensity at 465 to 475 nm, better fits the absorption peak of camphorquinone than the broader spectrum of QHT light-curing units. Theoretically, LED units should be

Table 5 The percentage of wall-to-wall contraction (WWC) at the surface (0 mm) and at a depth of 2 mm (mean ± SD)

Light-curing unit	0 mm	2 mm
Elipar TriLight	0 ± 0[a]	20 ± 10[a]
Allegro	5 ± 2.5[b]	50 ± 20[b]
Bluephase	5 ± 5[b]	50 ± 12.5[b]
FreeLight 2	5 ± 5[b]	40 ± 15[b]
The Cure TC-01	10 ± 5[b]	50 ± 15[b]

Values with the same superscript did not significantly differ.

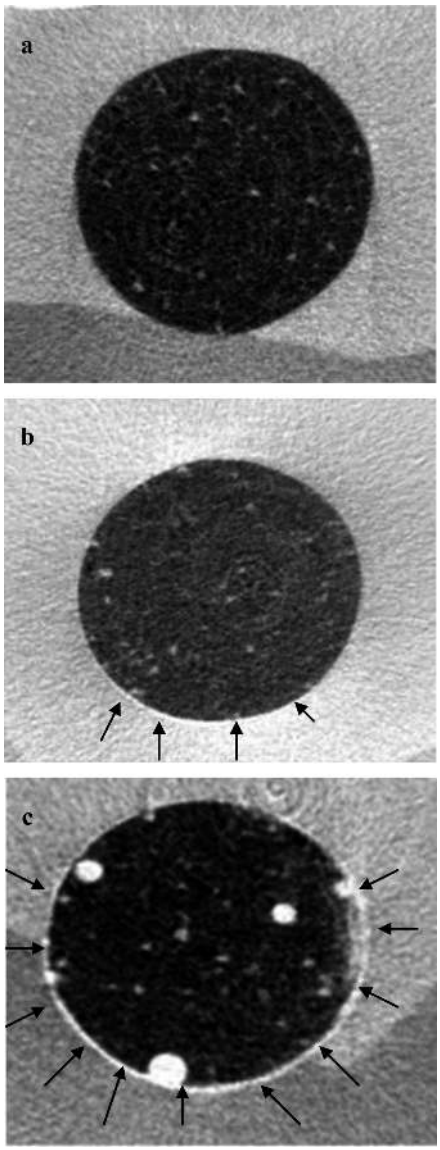

Fig. 2 Typical images of a micro-CT section of a resin composite restoration photopolymerized by the Elipar TriLight unit: (a) top site and (b) bottom site; (c) a restoration photopolymerized by the Allegro unit (bottom site, arrows show interfacial debonding).

more efficient than QTH units in curing activation. (20,22,23). However, neither the higher light intensity nor the narrower light spectrum of the tested LEDs resulted in a higher C=C conversion rate.

Moreover, differences in light intensity among the LED units were not associated with the degree of C=C conversion. Watts (24) reported that the photopolymerization reaction of resin monomers is diffusion-controlled after the gel point. Therefore, after a critical threshold of light intensity – which is necessary for the initiation of the polymerization reaction in a resin composite – the gel point is reached in the first few seconds, and any further increase in light intensity does not significantly enhance the degree of conversion. Thus, it can be assumed that all the units tested, both the LEDs and the QTH, emitted the energy necessary for the resin composite used (2,25). Furthermore, Martin (26) reported that in order to adequately cure resins, a minimum intensity of 400 mW/cm^2 was required, because much of the electrical energy is converted into heat and the emitted light includes many superfluous wavelengths.

Even though the results regarding the degree of C=C conversion were inconsistent with the first, low-intensity (100-200 mW/cm^2), generation of LEDs, it is generally accepted that the inferior C=C conversion, relative to conventional QTH units, was an important drawback (11,27). The results of the present study confirm that high-intensity LED units provide better C=C conversion than do low-output analogs, as well as curing performance identical to that of QTH units (as demonstrated by %RDB).

In this study, surface microhardness varied significantly, regardless of the type (QTH or LED) or power density of the unit. The QTH unit had an SM comparable with those of 2 of the 4 LED units and lower than the other 2. Comparisons of previous generations of LEDs (up to 800 mW/cm^2) with QTH units yielded contradictory results. The surface hardness of LED units has been found to be higher, lower, and similar to that of QTHs, which indicates that each commercial device exhibits unique curing performance (28-31).

The ratio of bottom-to-top surface microhardness, or relative microhardness, of a resin composite is an indirect method of assessing the depth of cure. Pilo and Cardash proposed that the ratio should exceed 0.8 for adequate deep polymerization (32). Although a 2-mm layer is the commonly accepted value for clinical applications, we prepared 3-mm- and 5-mm-thick specimens (33). Because multi-layer placement is a time-consuming and sensitive technique, we attempted to determine the effectiveness of the newly developed high-power LEDs in curing increments thicker than 2 mm.

The Allegro, FreeLight2, and Elipar TriLight light-curing units all had adequate relative microhardness (>0.8 at a depth of 5 mm); The Cure TC-01 and Bluephase LEDs failed to reach this threshold, although their relative microhardness was close to 0.8 at both depths. With respect to clinical applications, it is important to note that the use of even the latter 2 units resulted in mostly acceptable curing at 5 mm. The transmittance of light through composites is wavelength-dependent. One study showed that longer wavelengths penetrate composites more deeply than do shorter ones and therefore result in greater curing depth (34). Thus, differences in spectra emission among the light-curing units evaluated in the present study might partially explain our findings.

The Bluephase and Allegro units had the highest surface microhardness values. In the case of the Bluephase unit, it appears that this rapid network formation in the superficial layer of the resin composite might inhibit light transmittance through the bulk of the material because of changes in the optical properties of this layer. However, the Allegro unit had similarly high microhardness surface values, as well as a better deep-cure profile, which may be due to its higher emission intensity. Calheiros et al. reported that an increase in irradiant energy led to a significant increase in the depth of cure (35). This finding, in combination with the possibly superior spectrum of emission and the photo initiator's absorption of the particular light-curing unit, could explain the better performance.

First-generation LED units were unable to provide clinically satisfactory depth of cure, even at 2 mm (21,36-38). The depth-of-cure performance of the high-intensity LED units confirms the superiority of these newly introduced devices.

The percentage of linear polymerization shrinkage was examined indirectly by using deflecting disks (13). This method measured the linear polymerization during a period of 30 min, which included pre- and post-gelation shrinkage. The %LS of the curing units was similar. The type and concentration of the monomers and fillers in a resin composite, as well as the degree of monomer conversion, account for the polymerization shrinkage value (39). Because only one resin composite brand was used in the study, the principal contributor factor accounting for differences in %LS among the light-curing units was %RDB. As there were no significant differences in %RDB among the units, %LS was similar as well. The fact that higher %LS values were noted in the current study, as compared with a previous study (11) with the same experimental conditions, can be attributed to the much higher total energy delivered by the LED units investigated in the present study.

In the use of bonding systems, marginal adaptation is a parameter related to the degree of polymerization, polymerization shrinkage, the flow capacity of resin during the curing process, the mode of curing, and bonding with the periphery (11,40). Because the same commercial resin composite was used in all groups in the present study, the differences in observed %WWC directly reflect the performance of the light-curing units assessed. In the subsequent experimental model, the resin composite was applied without cavity treatments, to evaluate *in situ* shrinkage. Although the use of non-bonded margins is not clinically relevant, it allows for the assessment of the actual shrinkage profile, without interference from adhesive systems. The generated forces are transferred to the periphery, or, where the resistance to deformation is smaller, to free surfaces (41,42). Consequently, shrinkage compensation is expected in the underlying underexposed areas, a process which leads to restoration movement towards the free surface and an increase in interfacial porosity at the bottom sites (40,43). Thus, less satisfactory adaptation was noted at a depth of 2 mm, as compared with the free-surface sites, in all groups, irrespective of the type of light-curing unit used. Under the conditions of the present study, marginal debonding was the outcome of the competition of dentin wetting and setting shrinkage of the resin composite tested, as mediated by the different light-curing units. Interestingly, although there were no significant differences between the QTH and LED units concerning the depth of cure or polymerization shrinkage, the use of the QTH unit resulted in superior marginal and interfacial integrity at the top and bottom sites. It seems that the higher light intensity of the LEDs may cause fast polymerization with a very short pre-gel phase, resulting in greater contraction strain, which is compensated for by flow (44). D'Alpino et al. reported that an LED unit with 700 mW/cm^2 irradiance provided a higher rate of conversion in a shorter period of time than a QTH unit with an irradiance of 540 mW/cm^2 (45).

Currently, clinicians have at their disposal an unprecedented variety of materials and light-curing units. Therefore, thorough evaluations are required if informed decisions are to be reached. In this study, all light-curing units performed at a high level, indicating that a slight increase in irradiance does not result in an increase in performance.

1. The %RDB and %LS were material-dependent and were not influenced either by the type of light (LED or QTH) or by the light intensity emitted.
2. The LED unit with the highest light intensity (Allegro) resulted in the highest surface microhardness, in combination with sufficient depth of cure up to 5 mm.
3. Three light-curing units (Elipar TriLight, Allegro, and FreeLight 2) had a relative microhardness that exceeded the threshold of 0.8, up to a depth of 5 mm, which indicates sufficient depth of cure.
4. The QTH unit, which had the lowest light intensity, resulted in better adaptation than did the high-intensity LEDs.
5. The LED units had similar wall-to-wall contraction performance.

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