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ORIGINAL ARTICLE

Temperature rise and degree of photopolymerization conversion of nanocomposites and conventional dental composites

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Abstract The aim of the study was to investigate the temperature rise of a nanocomposite and a conventional hybrid dental composite during photopolymerization when cured with halogen curing lamp (QHT) and light-emitting diode (LED). Temperature rise during photopolymerization of two commercially available composites (Filtek Supreme® and TetricCeram®) were measured using a K-type thermocouple and a digital thermometer. Different curing modes were utilized to cure the composites: a high-intensity QHT unit (Optilux 501) in two different modes (standard and ramp), a low-intensity QHT unit (Coltolux 50), and an LED unit (Ultralume-2). Total temperature rise, polymerization reaction exotherm, and irradiation-induced temperature rise of the composites were determined. Degree of conversion of the specimens was measured using FTIR spectroscopy. The results revealed that the Filtek Supreme® nanocomposite showed lower temperature rise and degree of conversion in comparison with the hybrid composite ($p<0.05$). It was also found that the LED curing unit induced considerable total and irradiation temperature rise without any improvement in the degree of conversion. Ramp curing mode showed lower temperature rise and delayed gel point and was found to be more effective than QHT standard mode and LED units. Although it is claimed that the LED curing units exhibit lower temperature rise during the photopolymerization, the present study showed that the curing units have no advantage over the conventional QHT units regarding the temperature rise and degree of polymerization conversion.

Keywords Dental composites · Temperature rise · Degree of conversion · Light-emitting diodes · QHT units

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Introduction

Dental pulp is a highly vascularized tissue whose viability may be compromised during cavity preparation and restorative procedures [17, 34, 40]. In restorative procedures, temperature may easily exceed the normal values, especially if the procedures are carried out incorrectly [9] and/or in deep cavities in which the dentin thickness is less than 1 mm [2]. Studies concluded that heat generated by restorative procedures can be harmful to the dental pulp. Zach and Cohen, using monkey teeth, showed that a rise of 5.5°C in the pulp caused considerable damage, resulting in complete loss of vitality in 15% of the teeth [41].

Photopolymerization exotherm of the resin-based restorative materials and the heat of irradiation of dental light-curing units (LCUs) are sources of temperature rise in the tooth cavity which may consequently damage the pulp [22, 23]. The temperature rise during photopolymerization is influenced by factors such as intensity of the light [15], chemical composition of restorative resins [10, 20], transmission properties of the resin composites [32], the depth of cavity or restoration [30, 32], and duration of light exposure [13, 27].

At present, halogen light-curing units (QHT) have been the most commonly used light sources for polymerization of the resin-based materials. To enhance the curing efficiency, manufacturers continue to develop light sources with higher intensities. Although the high-intensity halogen

lamps reduce the curing time, they may increase the risk of pulp damage. The literature reports maximum temperature rises of 10–20°C in resin composites during polymerization [16, 20–23, 32]. Moreover, the halogen lamps have a relatively high output in the range of red and infrared wavelengths, which does not match the maximum absorption peak of camphorquinone, the most commonly used photoinitiator in dental composites (at 470 nm), resulting in more temperature rises without significantly improving photopolymerization process [16, 36].

Light-emitting diode curing units (LEDs) are recently introduced photopolymerization devices, which are claimed to be more effective in the curing of resin-based materials. The curing efficiency of LED units is explained by the better match of their emission spectrum with the absorption peak of the camphorquione than the broad spectra of halogen light-curing units [1, 19, 25, 26, 28]. In the LEDs, unlike QHTs, there is no emission in the infrared range [37]. Therefore, it might be expected that LEDs produce less irradiation-induced heat during the photopolymerization of the restorative materials [1, 6, 28]. Furthermore, the LEDs last several times more than conventional QTH light bulbs. These features may have promise to overcome some of the reported drawbacks of QTH curing lights [25, 29, 31]. However, curing performance of the blue LEDs, especially in the case of heat generation during the curing of composite resins, is not well established.

This study was conducted to compare the temperature rise and degree of photopolymerization conversion of two commercially available composite resins using an LED and two QTH units.

Materials and methods

Resin composites and curing units

The characteristics of the resin composites and the light-curing units, which were used in this study, are listed in the Tables 1 and 2. The QHT unit, Optilux 501, was utilized in two different modes: the standard (full intensity from the beginning, 680 mW/cm^2) and ramp mode (exponential rise of light intensity from 100 to 1,030 mW/cm^2 over the first 10 s and full intensity, 1,030 mW/cm^2, over the second 10 s). Coltolux 50, as a curing unit with low-output intensity of 330 mW/cm^2, was also utilized to study the effect of the low light intensity, although it may not be the unit of choice in the clinical practice. The output of curing units was measured using the built-in radiometer of Optilux 501. The radiometer measures the intensity of light from 100 to 1,999 mW/cm^2 in the wavelength range of 400–510 nm.

Temperature rise measurements

Temperature rise during photopolymerization of the composites was measured using a K-type thermocouple and a digital thermometer (Escort 20, Taiwan). The thermocouple was placed under the composite specimen and connected to a computer through the digital thermometer interface. The specimens were irradiated, and temperature rise during the photopolymerization of the specimen was recorded on a computer with respect to the time. The light guide of the curing units and the thermocouple were fixed in a mold set to prevent their displacement during the experiments (Fig. 1). A thin Mylar strip (30 μm thickness, Mani, Japan) was placed over and under the specimens in order to prevent the light guide and thermocouple to be stuck to the resin composites. The size of the specimens was 4 mm in diameter and 2 mm in depth. No dentin disk or other spacer was placed between the thermocouple and the resin composites.

The specimens (five repeats) were cured at room temperature. The ambient temperature (T_a) was then subtracted from the measured values (T_c) to calculate the temperature rise ($\Delta T = T_c - T_a$).

Ten minutes after curing, when the temperature of the specimens reached the ambient temperature, the samples were re-irradiated and temperature rise was measured through the cured composites. The temperature rise during the first exposure is due to the both heat of the exothermic polymerization reaction and the heat generated by irradiation. During the subsequent exposure, however, the most of the polymerization has already been completed, and the

Table 1 Characteristics of the resin composites

Composite	Batch number	Resin monomers	Fillers type	Filler loading (%)
TetricCeram, fine-filled hybrid (Ivoclar-vivadent, Lictenstein)	F61782	Bis-GMA, UDMA TEGDMA	Baricum glass, yetterbium trifluoride, Ba-Al-fluorosilicate glass, highly dispersed silicon oxide, spheroid mixed oxide	79
Filtek Supreme, nanofilled (3M, USA)	3910A2D	Bis-GMA, UDMA TEGDMA, Bis-EMA	Aggregated zirconia/silica cluster nonagglomorated/nonaggregated silica	78.5

Table 2 Specifications of the light-curing units

Light source	Curing mode	Power density[a]
Optilux 501, halogen bulb (Kerr, USA)	Standard	680 mW/cm^2 (40 s)
	Ramp	100 → 1,030 mW/cm^2 (10 s) 1,030 mW/cm^2 (10 s)
Coltolux 50, halogen bulb (Coltene/Whaledent, USA)	Standard	330 mW/cm^2 (40 s)
Ultralume-2, LED, (Ultradent/USA)	Standard	>400 mW/cm^2 (40 s)[b]

[a] The values are the power densities of the units at the time of the tests, which might be different from the values reported by their manufacturer

[b] This is the power density reported by the manufacturer. The power density measured by radiometer of the Optilux 501 was 620 mW/cm^2

observed temperature rise is predominantly due to the heat of irradiation. By superimposition and subtraction of these curves, the temperature rise caused by exothermic heat of the curing reaction was calculated [16, 22, 32]. Typical curves are illustrated in Fig. 2. A blank group was also tested using no composite in the mold to measure the temperature rise due to the irradiation of the light units.

Measurement of degree of conversion

Degree of conversion of the composites was measured using FTIR spectroscopy (EQUINOX 55, Bruker, Germany) at a resolution of 4 cm^{-1} and 32 scans in the range of 4,000–400 cm^{-1}. A small amount of resin composites was placed between two polyethylene films, pressed to form a very thin film, and the absorbance peaks of the uncured samples were obtained. The samples were then light-cured at the same time periods of the temperature rise measurements, and the peaks were collected for the cured samples. Degree of conversion (DC%) was determined from the ratio of absorbance intensities of aliphatic C=C (peak at 1,638 cm^{-1}) against internal reference aromatic C...C (peak at 1,608 cm^{-1}) before and after curing of the specimen. The degree of conversion was then calculated as follows [7]:

$$\mathrm{DC}\% = \left(1 - \frac{(1,637\ \mathrm{cm}^{-1}/1{,}608\ \mathrm{cm}^{-1})\ \text{peak area after curing}}{(1,637\ \mathrm{cm}^{-1}/1{,}608\ \mathrm{cm}^{-1})\ \text{peak area before curing}}\right) \times 100$$

Three replicate specimens were made for each of the uncured and cured conditions.

Statistical analysis

The results were analyzed and compared using one-way ANOVA followed by the Tukey–HSD test at the significance level of 0.05.

Results

Temperature rise due to the light emission of LCUs (blank group)

Temperature rise profiles of the LCUs in the blank group are illustrated in the Fig. 3. Table 3 shows the average of the maximum temperature rise measured with the various LCUs and curing modes. Significant difference was observed among the temperature rise of different LCUs and curing modes ($P<0.05$).

Temperature rise measurements of the composite specimens

Table 4 shows the maximums of total temperature rise (ΔT_t), the temperature rises due to the light irradiation (ΔT_i), and photopolymerization reaction (ΔT_r) of TetricCeram® and Filtek Supreme® resin composites, respectively. Significant difference is observed between ΔT_t, ΔT_i, and ΔT_r of TetricCeram®. There are also significant differences in ΔT_t and ΔT_i of Filtek Supreme® in different curing modes but

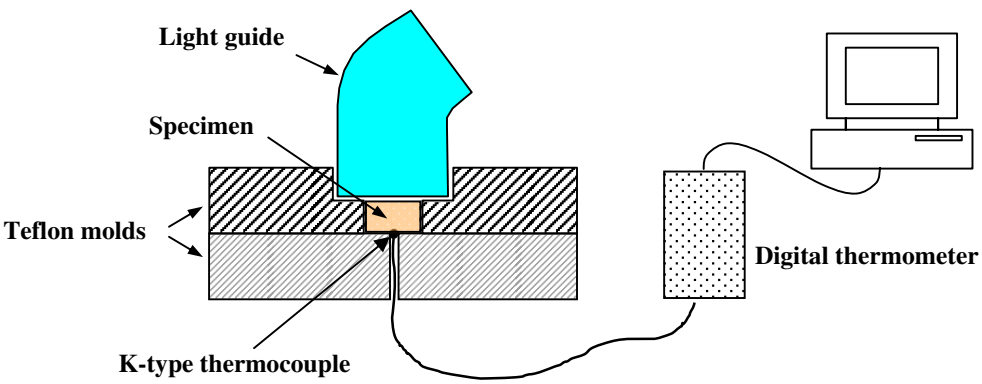

Fig. 1 The schematic of the temperature rise measurement set

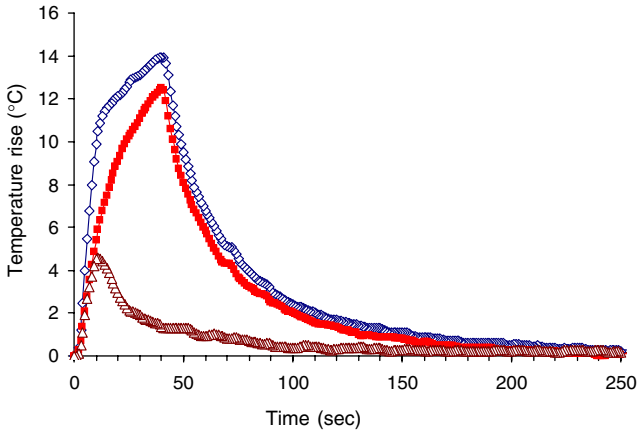

Fig. 2 Typical curve showing total temperature rise (*diamonds*) and temperature rises due to irradiation (*squares*) and polymerization reaction (*triangles*). (The curves of TetricCeram® is shown as an example)

not in ΔT_r. Table 4 shows significant differences between the two resin composites in ΔT_t, ΔT_i, and ΔT_r.

Typical curves of total, irradiation, and reaction temperature rise with respect to the time are illustrated in Fig. 2. Figures 4, 5, and 6 show the corresponding temperature rise of TetricCeram® composite cured with different LCUs. As the temperature rise patterns of the both composites were the same, the curves of the TetricCeram® results are illustrated.

Time to reach the maximum reaction temperature rise

Temperature rise due to the polymerization reaction (ΔT_r) could be an estimation of exothermic polymerization reaction of the resin monomers. The time to reach the maximum temperature rise in ΔT_r, therefore, could approximate the vitrification point in the polymerization reaction.

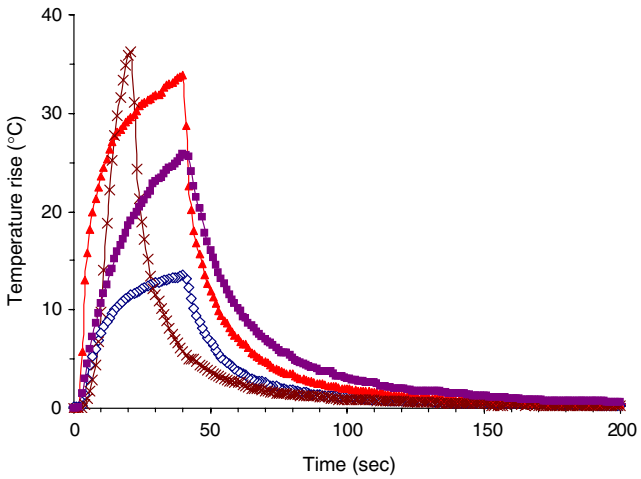

Fig. 3 Temperature rise profiles of LCUs in different irradiation modes (no composites). Optilux 501-Standard (*triangles*), Optilux 501-Ramp (*multiplication signs*), Coltolux 50 (*diamonds*), LED (*squares*)

Table 3 Maximum temperature rise in LCUs and different modes (blank group)

Light sources	Temperature rise, °C (SD)
Optilux 501 (standard)	34.2 (1.3)
Optilux 501 (ramp)	36.3 (0.4)
Coltolux 50	13.0 (0.9)
LED	26.3 (0.3)

Table 5 represents the time to reach the maximum ΔT_r for the composites at different irradiation regimes. Significant differences are observed between LCUs and curing modes in the both resin composites. The time is longer for Filtek Supreme® than TetricCeram® ($p<0.05$).

Degree of polymerization conversion

Table 6 shows the degree of conversion of the resin composites cured with different irradiation modes. The curing condition was the same as the temperature rise measurements.

In TetricCeram®, significant difference is only observed between the specimens cured by Coltolux 50 and other LCUs ($p<0.05$). Degree of conversion of Filtek Supreme® polymerized with various LCUs and modes was not significantly different ($p>0.05$). In comparison, TetricCeram® showed higher degree of polymerization than Filtek Supreme® ($p<0.05$).

Discussion

Thermal emission of LUCs

Thermal emission of Optilux 501 in the ramp mode (36.3°C) and standard mode with an 8-mm tip (34.1°C) were

Table 4 Maximums of total temperature rise (ΔT_t), the temperature rises due to the light irradiation (ΔT_i), and polymerization reaction (ΔT_r) of TetricCeram® and Filteke Supreme®

ΔT	Light irradiation sources and modes	Temperature rise °C (SD)	
		TetricCeram	Filteke Supreme
ΔT_t	Optilux 501(standard)	14.1 (0.7)	12.4 (1)
	Optilux 501(Ramp)	12.9 (0.6)	10.9 (0.7)
	Coltolux 50	6.6 (0.4)	5.8 (0.7)
	LED	14.1 (0.4)	12.6 (0.3)
ΔT_i	Optilux 501(standard)	12.6 (0.5)	11.2 (0.9)
	Optilux 501(Ramp)	9.1 (0.4)	7.3 (0.7)
	Coltolux 50	4.9 (0.4)	4.3 (0.6)
	LED	11.9 (0.4)	11.1 (0.4)
ΔT_r	Optilux 501(standard)	4.8 (0.9)	3 (0.2)
	Optilux 501(Ramp)	4.7 (0.5)	3.4 (0.7)
	Coltolux 50	3.4 (0.5)	2.9 (0.4)
	LED	5.0 (0.7)	2.9 (0.4)

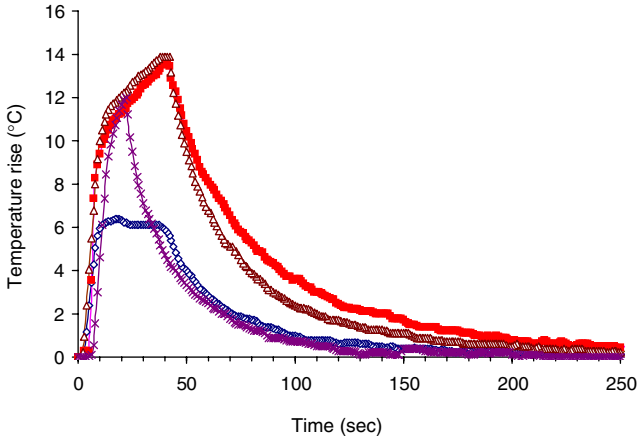

Fig. 4 Total temperature rise in the TetricCeram® cured with different curing units: Optilux 501-Standard (*triangles*), Optilux 501-Ramp (*multiplication signs*), Coltolux 50 (*diamonds*), LED (*squares*)

significantly higher than the other groups ($p<0.05$). This is attributed to the high-power intensity in ramp curing mode and long radiation time in standard mode. In standard mode of Optilux 501 and LED with approximately the same irradiation intensities, the QTH unit showed higher irradiation temperature rise, which could be due to its broader light emission spectrum. The study of Yap and Soh [40] on the temperature rise due to radiation energy at various cavity depths also showed that thermal emission of LED units was significantly lower than halogen lights, which are in agreement with our study. Coltolux 50 showed the lowest temperature rise ($p<0.05$) because of its low-power intensity (Table 3 and Fig. 3).

Temperature rise measurements in the composite specimens

Higher maximum ΔT_t and radiation temperature rise were monitored for the TetricCeram® in comparison with Filtek

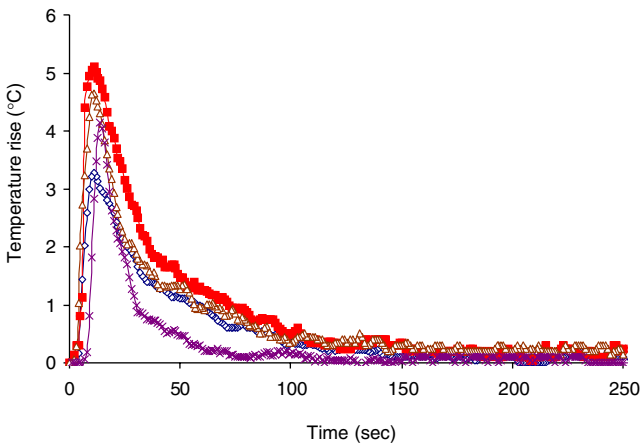

Fig. 5 Temperature rise due to reaction in the TetricCeram® cured with different curing units: Optilux 501-Standard (*triangles*), Optilux 501-Ramp (*multiplication signs*), Coltolux 50 (*diamonds*), LED (*squares*)

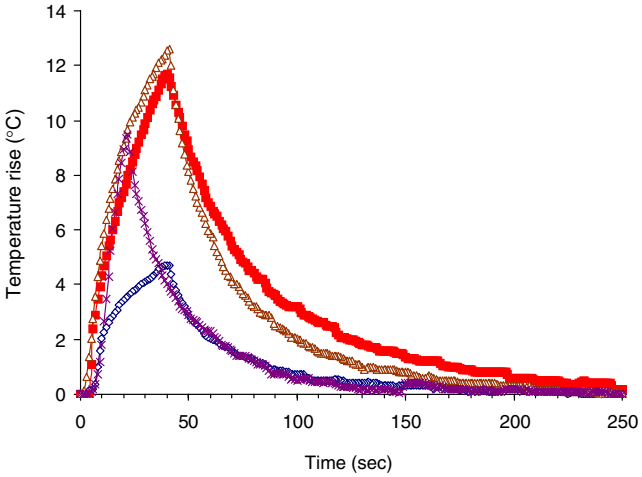

Fig. 6 Temperature rise due to irradiation in the TetricCeram® cured with different curing units: Optilux 501-Standard (*triangles*), Optilux 501-Ramp (*multiplication signs*), Coltolux 50 (*diamonds*), LED (*squares*)

Supreme® ($p<0.05$; Table 4), which is probably due to a combination of higher mobility of molecular radical species and better light transmission in TetricCeram®. Filtek Supreme® is a nanofill composite and contains aggregated zirconia/silica cluster with an average cluster particle size of 0.6 to 1.4 μm and primary particle size of 0.5–20 nm and a nonagglomerated/nonaggregated 20 nm silica fillers, while TetricCeram® is a hybrid composite containing predominantly glass filler in the range of microns. Nanoparticles may restrict the mobility of the resin monomers and (macro)radicals [8, 14], resulting in lower polymerization reaction rates and, therefore, lower temperature rise due to the reaction. The higher irradiation temperature rise in TetricCeram® is probably due to the difference in the opacity of the two composites and/or more scattering of the light passing through the Filtek Supreme® nanocomposite [32].

The total temperature rise, ΔT_t, and radiation-induced temperature rise, ΔT_r, in both composite resins was significantly higher for Optilux 501 in standard curing mode and LED in comparison with other groups (Table 4). Although some LED LCU manufactures claim that the units induce no or very low heat of irradiation, it is not

Table 5 Time to reach the maximum polymerization reaction temperature rise (ΔT_{rm}) of TetricCeram® and Filteke Supreme®

Light irradiation sources and modes	Time to reach ΔT_{rm}, sec (SD)	
	TetricCeram	Filteke Supreme
Optilux 501(standard)	10.2 (0.8)	10 (1)
Optilux 501(Ramp)	14.0 (0.7)	17.6 (1.7)
Coltolux 50	11.4 (0.1)	13 (1.6)
LED	10.6 (0.5)	12 (1)

Table 6 Degree of conversion of the resin composites cured with different irradiation modes

Light irradiation sources and modes	Degree of conversion, % (SD)	
	TetricCeram	Filteke Supreme
Optilux 501(standard)	55.0 (2.6)	42.1 (2.7)
Optilux 501(Ramp)	59.7 (1.5)	44.0 (2.8)
Coltolux 50	44.4 (3.3)	44.4 (1.4)
LED	55.2 (2.6)	43.8 (2.6)

confirmed in this study. The first generation of LED curing units had relatively low-power output, which might be the reason of the lower irradiation-induced heat of the units [12, 21]. New generations of the LED units, however, emit high-power blue light, which may, in turn, cause higher temperature rise due to the light irradiation [5, 38]. It has also been shown that at a standardized total energy density condition, LED units produce greater temperature increase than the halogen units [32].

The maximum temperature rise caused by exothermic polymerization reaction was higher in TetricCeram® than Filtek Supreme® (Table 4), which could be explained considering the lower polymerization rate of the Filtek Supreme® nanocomposite. This is confirmed by the results of the time to reach maximum ΔT and degree of conversion, which are discussed in the following sections.

TetricCeram® specimens, which were cured by Coltolux 50, showed significantly lower ΔT_r ($p<0.05$; Table 4), which is related to the low-power intensity of this LCU. In the case of Filtek Supreme®, there was no significant difference between ΔT_r of the specimens cured with different LCUs and modes. This is presumably due to restriction in molecular and/or radicals mobility of the resin matrix in the nanocomposite, which also results in lower degree of conversion.

Time to reach the maximum reaction temperature rise

As the reaction temperature rise is the result of the exothermic polymerization process, it is expected to follow the polymerization reaction pattern. The time to reach the maximum reaction temperature rise (t_m) could be an estimation of the well-known vitrification point in the polymerization of matrix resin monomers.

In the present study, curing mode and composite resin type had a significant effect on the t_m. The t_m, except for the standard mode of Optilux 501, is higher for Filtek Supreme® (Table 5). The results concur with those of Shortall and Harrington [33] who investigated temperature rise during polymerization of light-activated composite. In their study, higher exothermic temperature rise was observed at later stages in Filtek Supreme® in comparison with TetricCeram®.

The nanosize filler particles in Filtek Supreme® provide a very high filler surface area, which is in contact with the resin monomers in the interface. The mobility of the resin monomers could be restricted in the interface layer leading to a slower polymerization rate adjacent to the fillers surface [8, 14]. This may also result in lower polymerization contraction stress in Filtek Supreme®.

As it can also be seen in Table 5, statistically significant delay in maximum exothermic temperature rise was observed when the ramp curing mode of Optilux 501 was used. In the ramp mode, light is irradiated in lower intensities in the first few seconds that may help in postponing the gel point of the polymerization reaction. The delayed gel point let the polymerization–shrinkage-induced stress be relaxed [11, 18]. Using the standard curing mode of Optilux 501, both composite resins showed higher maximum irradiation temperature rise (Table 4). This is due to longer light irradiation and higher total energy absorbed by the composites. No significant difference was observed in the maximum reaction temperature rise in these two groups. This finding was confirmed by degree-of-conversion measurements (next section). The results suggest that the ramp curing mode is preferred to standard curing mode in Optilux 501. The specimens cured with Coltolux 50 also showed delay to reach the maximum reaction temperature rise, which is due to its lower light intensity. Appling LED, the time was not significantly changed in comparison with the standard mode of Optilux 501.

Figure 4 also illustrates a maximum in the temperature–time curve of the specimen cured with Coltolux 50, which is not seen in the other curing units. The temperature rise in the composites is the result of exothermic reaction and light irradiation. In the light-curing units with high-intensity output, the temperature rise due to the light irradiation is dominant, and it covers the reaction-induced temperature rise. In the specimens cured with the low-intensity irradiation of Coltolux 50, however, the share of the light irradiation is not pronounced, and the effect of reaction-induced temperature rise has appeared in the temperature–time curve.

In the present study, the LED curing unit induced considerable total and radiation temperature rise within the dental composites; however, temperature rise due to the exothermic reaction, which is an indication of polymerization, did not show any significant difference in comparison with the Optilux 501 curing unit (Table 4).

Degree of conversion

The results (Table 6) show a higher degree of polymerization for TetricCeram® compared to Filtek Supreme®, except that the specimens cured with Coltolux 50 in which there is no difference between the degree of conversions.

TetricCeram® specimens cured with Coltolux 50 showed lower DC% ($p<0.05$), while there was no significant difference between the specimens photopolymerized using high-intensity curing unit Optilux 501 or LED ($p>0.05$). Photopolymerization reaction of the resin monomers follows an autocatalytic pattern, which is diffusion-controlled after gel point [3, 4, 7, 8, 39]. It seems that after a limiting light intensity, which is needed for initiation of polymerization reaction, the gel point is reached in the first few seconds, and further increase in the light intensity could not enhance significantly the degree of conversion. This is most probably due to the high viscosity of the polymerizing medium and restrictions in the mobility of the monomers and (macro)radicals. Low-power intensity of the Coltolux 50 light, however, produces lower active radicals in the initiation stage of the polymerization process, leading to lower final degree of conversion because of the lack of enough active sites in the further propagation stage of polymerization process.

Although the Filtek Supreme® specimens received more irradiation energy in the LED and Optilux 501 (standard and ramp curing modes) in comparison with Coltolux 50, no significant difference was observed among the degree of conversion in different groups ($p>0.05$). Very high surface area of the nanoparticles in Filtek Supreme® limits the mobility of a large portion of the resin monomers, which are in contact with the filler surfaces, resulting in lower degree of conversion. It should be mentioned that the DC% may increase after stopping the light irradiation due to postpolymerization process, but it was not the aim of our study.

Results of this study showed that LED curing unit induced considerable total and irradiation temperature rise without any improvement in the degree of conversion. Therefore, it seems that there is no advantage in using the LED compared to the halogen lamps considering degree of conversion and temperature rise. Soh et al. also reported that LEDs and halogen lights showed comparable curing efficiency [35]. Furthermore, in the study of Meyer et al., the LED units showed higher decrease in power output, in comparison with QHT curing devices, with increasing distance to the filling surface [24].

Although the clinical experience with conventional visible light-curing units indicates that the pulp is able to recover from transient heating from light [15], Zach and Cohen [41] reported that 15% of the teeth in rhesus monkeys developed necrosis when healthy pulp was exposed to a temperature increase of only 5.5°C.

In the present study, even the lowest temperature rise, and the case of Filtek Supreme® cured by Coltolux 50 curing unit was higher than 5.5°C.

It should be mentioned that the results of this study cannot be directly applied to in vivo conditions. Heat transfer by conduction through the tooth structure and convection due to the blood circulation in the pulp chamber and fluid motion in the dentinal tubules may compensate part of the heat generated during the photopolymerization of the restorative composites. Nevertheless, clinicians should be aware of the potential thermal damage to the pulp, which may result from the visible light curing of composite resins in deep cavities.

Conclusion

Temperature rise and degree of conversion of two different composite resins cured by various LCUs were investigated. The result showed that:

1. Composite resin type had a statistically significant influence on the temperature rise, and degree of conversion and Filtek Supreme® nanocomposite showed lower values.
2. No significant difference in temperature rise and degree of conversion was observed between the standard mode of Optilux 501 and LED curing units.
3. Ramp curing mode showed lower temperature rise and delayed gel point and was found to be more effective than the standard mode of Optilux 501and LED units.

Conflict of interest The authors declare that they have no conflict of interest.

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