Comparison of Composite Curing Parameters: Effects of Light Source and Curing Mode on Conversion, Temperature Rise and Polymerization Shrinkage

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Clinical Relevance

The use of a low intensity light source for photopolymerization based on LED technology provides equivalent final degree conversion with possible flow of the resin composite, similar to when QTH technology is used. At the same time, the lower temperature rise in the sample and the more favorable development of shrinkage kinetics compared to the higher intensities of halogen light may aid in maintaining marginal adaptation while avoiding possible thermal injury.

SUMMARY

This study analyzed the degree of conversion, temperature increase and polymerization shrinkage of two hybrid composite materials polymerized with a halogen lamp using three illumination modes and a photopolymerization device based on blue light emitting diodes. The degree of conversion of Tetric Ceram (TC) (Ivoclar Vivadent) and Filtek Z 250 (F) (3M/ESPE) was measured by Fourier transformation infrared spectroscopy at the surface and 2-mm depth; temperature rise was measured by digital multimeter, and linear polymerization shrinkage was measured during cure by digital laser interferometry. Composite samples were illuminated by quartz-tungsten-halogen curing unit (QTH) (Astralis 7, Ivoclar Vivadent) under the following modes: "high power" (HH) 40 seconds at 750 mW/cm², "low power" (HL) 40 seconds at 400 mW/cm² and "pulse/soft-start" (HP) increasing from 150 to 400 mW/cm² during 15 seconds followed by 25 seconds pulsating between 400 and 750 mW/cm² in 2-second intervals and by light emitting diodes (LED) (Lux-o-Max, Akeda Dental) with emitted intensity 10 seconds at 50 mW/cm²

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and 30 seconds at 150 mW/cm². A significantly higher temperature increase was obtained for both materials using the HH curing mode of halogen light compared to the HP and HL modes and the LED curing unit after 40 seconds. Significantly lower temperature values after 10-second illumination were obtained when LED was used compared to all halogen modes. For all curing modes, there was no significant difference in temperature rise between 20 and 40 seconds of illumination. Results for the degree of conversion measurements show that there is a significant difference in the case of illumination of resin composite samples with LED at the surface and 2 mm depth. For polymerization shrinkage, lower values after 40 seconds were obtained using LED compared to QTH.

INTRODUCTION

Although the mechanical properties of modern composites have been improved, polymerization shrinkage is still an inevitable problem (Uno & others, 2003). The magnitude of shrinkage is determined by the number of covalent bonds formed and the molecular weight of monomers—smaller monomers, lower composite viscosity and increased degree of conversion—but they also increase polymerization shrinkage, whereas adding monomers of greater molecule weight can minimize contraction (Chung & others, 2002). Trifunctional methacrylate with a relatively high molecular weight has been employed in dental composites; it reduces polymerization shrinkage 10% with greater facility compared with the addition of cyclic monomers such as spiroorthoesters, spiroorthocarbonates and cyclic acrylates (Chung & others, 2002; Stansbury, 1992). Contraction behavior can be very specific to a given composite due to its unique combination of filler and resin matrix (Aw & Nicholls, 2001). The shrinkage that occurs in a resin composite is clinically significant, because it can create forces that may disrupt the bond to the cavity walls (Rosin & others, 2002; Cavalcante & others, 2003; Irie, Suzuki & Watts, 2002). This competition between contraction forces within composites and bonds to the cavity wall is one of the main causes of marginal failure and subsequent microleakage that permits the passage of bacteria, fluids and toxins and could encourage dentinal hypersensitivity, secondary caries and pulpal inflammation (Davidson, de Gee & Feilzer, 1984; Opdam & others, 1998). To overcome polymerization shrinkage, several experimental and clinical approaches have been introduced, such as changes in composite composition, incremental placement of composites, application of liners and flowable composites, extraoral polymerization and a decrease in the polymerization reaction rate by an initial reduction

of resin conversion. The photopolymerization method has a profound influence on shrinkage stress. High intensity lights provide higher values for degree of conversion, but they also produce higher contraction stress (Yap & Senevirante, 2001). A slower curing process that permits composite flow may allow for stress relaxation during polymerization (Yap & Senevirante, 2001; Sakaguchi & Berge, 1998).

The spectral emission of conventional curing lights includes wavelengths that induce heating of the resin and tooth during curing (Kurachi & others, 2001). Despite their popularity, conventional halogen light curing units have serious drawbacks, such as a limited effective lifetime (40-100 hours), reflector and filter degradation over time due to high operating temperatures and the significant amount of heat produced during curing (Jandt & others, 2000). Many halogen units have been shown to not reach the minimum power output specified by the manufacturers (Jandt & others, 2000). The most frequently used photoinitiator in resin composites is camphorquinone, which is sensitive to light in the blue region of the visible spectrum, with an absorption peak at 470 nm (Arikawa & others, 1998). Any source of light with reasonable irradiance in this region can be employed as a curing device. To overcome the problems of halogen technology, alternative light sources have been suggested, such as argon laser (Pradhan, Melikechi & Eichmiller, 2002), pulsed laser (Tarle & others, 1998a,b; 1999; Meniga & others, 1997), plasma light (Park, Krejci & Lutz, 2002; Peutzfeldt, Sahafi & Asmussen, 2000; Knezevic & others, 2002) and blue superbright light emitting diodes (Tarle & others, 2002; Knezevic & others, 1999; 2001).

The objective of this research was to analyze the extent of monomer conversion at two different depths—temperature rise and polymerization shrinkage—using the digital laser interferometry (Demoli & others, 2004) of two hybrid composite materials exposed using three modes of conventional QTH curing and one LED light. The research hypothesis (RH) tested was that initial lower curing intensities of the halogen lamp or curing with blue LED strongly influenced temperature development and contraction kinetics in a composite sample and do not significantly influence final cure of the material.

METHODS AND MATERIALS

The composites tested were Tetric Ceram (TC) (Ivoclar Vivadent, Schaan, Liechtenstein; Lot #914760) and Filtek Z 250 (F) (3M/ESPE, St Paul, MN, USA; Lot #20000601) both shade A2.

The first light source tested was the quartz-tungsten-halogen curing unit (QTH) (Astralis 7, Ivoclar Vivadent) with three different modes:

1. High power (HH)–40 seconds at 750 mW/cm^2
2. Low power (HL)–40 seconds at 400 mW/cm^2
3. Pulse/soft-start (HP)–15 seconds intensity increase from 150 to 400 mW/cm^2, and 25 seconds pulsating between 400 mW/cm^2 and 750 mW/cm^2 in 2 seconds intervals.

One of the first commercial blue light emitting diode (LED) curing units (Lux-o-Max, Akeda Dental, Lystrup, Denmark) was used, giving an exposure profile of 10 seconds at 50 mW/cm^2, then 30 seconds at 150 mW/cm^2.

Degree of Conversion Measurement

For degree of conversion (DC) measurement, five samples were prepared for each material. An unpolymerized sample of resin composite was placed between two Mylar foils and compressed between two inox plates under 10 Pa pressure to 0.1-mm thickness. In order to measure DC on the surface of the prepared unpolymerized material placed between the two foils, the light source was pressed against the upper foil and polymerized. For a 2 mm depth of measurement, separate surface layers were made for each material: a 2-mm thick, unpolymerized resin composite sample placed inside the inox ring was covered with Mylar foil on the upper and lower side; it was then compressed to the 2-mm thickness and polymerized for two minutes inside the Spectramat (Model PM 1831, Ivoclar/Vivadent). While simulating measurement at a depth of 2-mm, the 2-mm thick surface layer was placed on the prepared, unpolymerized resin composite sample, and the light source was pressed against that surface layer and polymerized.

DC was measured using a Fourier transformation infrared (FTIR) spectrometer (Model 2000, Perkin Elmer, Beaconsfield, Bucks, UK) (Tarle & others, 1995). The uncured samples were pressed into KBr pellets (Ø=1 cm) using Merck's KBr (Merck AG, Darmstadt, Germany). Absorption spectra were recorded in a transmission mode using 20 scans at a resolution of 4 cm^{-1}. The ratio of absorption maximum was determined using the baseline method according to Rueggeberg (Rueggeberg, Hashinger & Fairhust, 1990), and the degree of conversion was calculated from the equivalent aliphatic/aromatic molar ratio from polymer-

ized (P) and unpoly-merized (U) samples using the formula:

DC (%) = (1-P/U) x 100.

Temperature Measurements

Temperature measurements used K-type thermocouple (Metex M 3850 D, Pirmasens, Winzeln, Germany) and digital multimeter (Metex M 3850 D, Pirmasens). The meter output was connected to a personal computer, and software (Metex Scope View for Windows and Software for DOS V 1.03; Copyright by Metex) displayed real time temperature increases on the screen. Five specimens of each material were made in plastic molds 4 x 4 x 4 mm (open above and from sideway) and illuminated with three modes of QTH and LED light for 40 seconds. A prefabricated probe 1-mm thick was marked at a depth of 1 mm and immersed sideways to that depth in the resin composite material before polymerizing. Resin composite samples were polymerized from above in order to prevent direct warming of the temperature probe by the light source.

Measurement of Polymerization Shrinkage

For measuring linear polymerization shrinkage, a digital interferometry-based device was made, which enabled two types of quantitative measurements, providing data for total displacement and dynamic behavior of displacement. A special carrier made it possible to polymerize specimens from one side, while simultaneously measuring the effects of the polymerization process using the digital interferometry method from
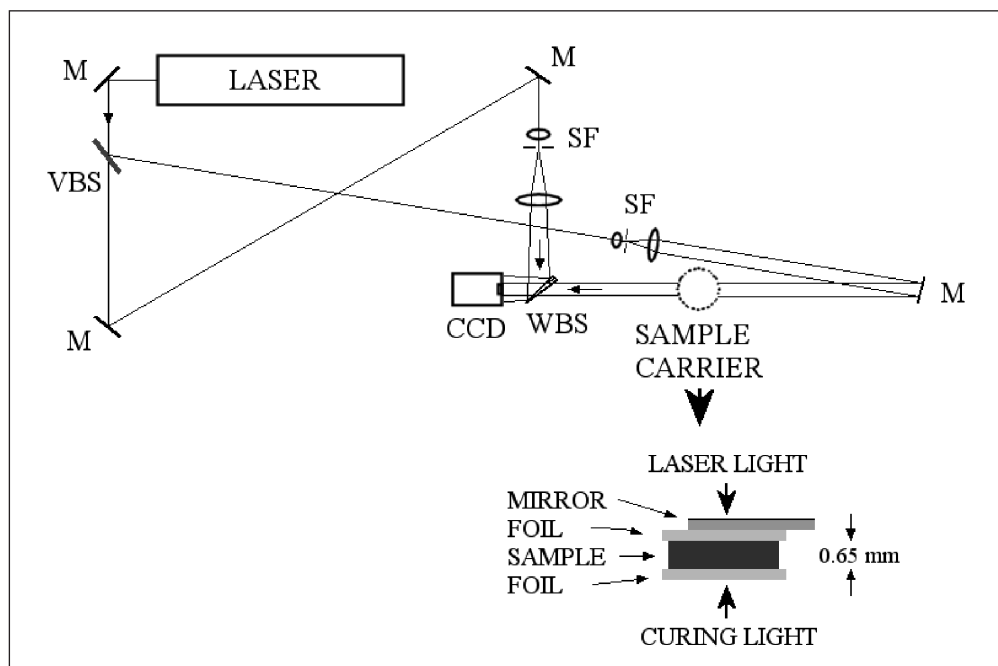

Figure 1: *Experimental setup: M-mirror, VBS-variable beam splitter, WBS-wedge beam splitter, SF-spatial filter, CCD-charge coupled device.*

the other side. In a typical measurement, a series of interferograms was first recorded, then analyzed using a custom computer program. Selected results obtained for various composite dental materials and different curing modes illustrate the effectiveness of the experimental device. Figure 1 illustrates the test setup, which consisted of a laser, beam splitter, beam expander, beam collimator, wedge beam splitter, mirrors, a CCD camera and a sample carrier. The beam emerging from the He-Ne laser (Spectra Physics, model 127, power = 25 mW, wavelength = 632.8 nm) was first split into an object and a reference beam. The reference beam was expanded and steered directly onto the CCD camera. The object beam was first expanded, collimated and steered onto the mirror situated on the upper surface of the sample, reflected under a small angle from the mirror, then directed to the CCD camera. Thus, the CCD camera recorded the fringe pattern formed by the interference of two beams. The CCD camera (Kappa CF 8/4, objective lens removed) had a sensor with 752 x 582 pixels (8.6 x 8.3 µm each) that allowed a rate of 25.8-bit monochrome frames per second. Cylindrical samples were sandwiched between two glass plates with transparent foils between the glass and composite. A micrometer (precise to 2 µm) was mounted, which enabled manual compensation of displacement of the upper specimen surface. The micrometer was used at the end of each measurement to determine the final shrinkage of the specimen (Demoli & others, 2004).

Table 1: *Degree of Conversion of Tetric Ceram Composite Material Using a Halogen or Blue LED Light Source (Mean value [SD])*

| | Degree of Conversion (%), TC | | | |
| | Halogen Curing Unit | | | |
Depth	High Power (HH)	Low Power (HL)	Pulse Mode (HP)	LED Curing Unit
surface	64.3(1.5)[a1]	64.6(0.3)[a1]	65.9(2.9)[a1]	62.3(1.2)[a1]
2 mm	60.9(1.3)[c2]	62.7(1.3)[c2]	62(1.9)[c1]	61.3(1.3)[c1]

n=5 specimens per experimental condition
i. within a row, similar small case letters denote statistically equivalent groups
ii. within a column, similar superscripted numbers denote statistically equivalent groups

Table 2: *Degree of Conversion of Filtec Z250 Composite Material Using a Halogen or Blue LED Light Source (Mean value [SD])*

| | Degree of Conversion (%), F | | | |
| | Halogen Curing Unit | | | |
Depth	High Power (HH)	Low Power (HL)	Pulse Mode (HP)	LED Curing Unit
surface	65.8(1.8)[a1]	64.9(2.0)[a1]	62.8(2.7)[a1]	61.5(0.3)[a1]
2 mm	63.7(2.5)[b1]	62(2.2)[b1]	59.4(0.1)[bc2]	56.5(2.5)[c2]

n=5 specimens per experimental condition
i. within a row, similar small case letters denote statistically equivalent groups
ii. within a column, similar superscripted numbers denote statistically equivalent groups

Table 3: *Temperature Rise Above Baseline of Tetric Ceram Composite Material Using a Halogen or Blue LED Light Source (Mean value [SD])*

| | Temperature Rise Above Baseline (°C), TC | | | |
| | Halogen Curing Unit | | | |
Time (seconds)	High Power (HH)	Low Power (HL)	Pulse Mode (HP)	LED Curing Unit
10	9.4(2.1)[a1]	7.2(1.9)[ab1]	4.6(0.9)[b1]	2.2(1.3)[c1]
20	14(1.7)[d2]	8.4(2.7)[e1]	7(2.0)[e1]	6.4(2.5)[e2]
40	13.4(2.3)[f12]	8(2.8)[fg1]	6.8(2.2)[g1]	6.2(2.0)[g2]

n=5 specimens per experimental condition
i. within a row, similar small case letters denote statistically equivalent groups
ii. within a column, similar superscripted numbers denote statistically equivalent groups

Table 4: *Temperature Rise Above Baseline of Filtec Z250 Composite Material Using a Halogen or Blue LED Light Source (Mean value [SD])*

| | Temperature Rise Above Baseline (°C), F | | | |
| | Halogen Curing Unit | | | |
Time (seconds)	High Power (HH)	Low Power (HL)	Pulse Mode (HP)	LED Curing Unit
10	9.5(2.5)[a1]	8.3(2.2)[a1]	6.3(1.5)[ab1]	1.5(0.6)[b1]
20	11.3(3.9)[c1]	10.3(1.3)[c1]	9(1.4)[c2]	2.5(0.6)[d2]
40	13(3.9)[e1]	9.5(1.7)[e1]	10.3(1.3)[e2]	2.5(0.6)[f2]

n=5 specimens per experimental condition
i. within a row, similar small case letters denote statistically equivalent groups
ii. within a column, similar superscripted numbers denote statistically equivalent groups

Data were analyzed by one-way and two-way analysis of variance for measurement of the degree of conversion, temperature rise and polymerization shrinkage at the $p > 0.08$ level.

RESULTS

Conversion values (Tables 1 and 2) using QTH light varied from 59.4 (0.1) (F, HP mode, 2-mm depth) to 65.9

| Table 5: *Linear Shrinkage of Composite Materials Polymerized Using A Halogen or Blue LED Light Source (Mean value [SD])*											
Polymerization Shrinkage (LIN%)											
	Halogen Curing Unit									**LED Curing Unit**		
Composite Material	**High Power (HH)**			**Low Power (HL)**			**Pulse Mode (HP)**					
	10 seconds	20 seconds	40 seconds	10 seconds	20 seconds	40 seconds	10 seconds	20 seconds	40 seconds	10 seconds	20 seconds	40 seconds
TC	0.31 (0.38)	0.76 (0.01)	1.11 (0.83)	0.08 (0.01)	0.52 (0.01)	0.93 (0.02)	-0.04 (0.02)	0.45 (0.48)	1.09 (0.01)	0.09 (0.01)	0.53 (0.02)	0.93 (0.03)
F	0.39 (0.31)	0.73 (0.01)	.90 (0.01)	0.24 (0.01)	0.55 (0.09)	0.72 (0.45)	0.23 (0.01)	0.55 (0.81)	0.72 (0.08)	0.17 (0.08)	0.56 (0.05)	0.91 (0.01)
n=5 specimens per experimental group												
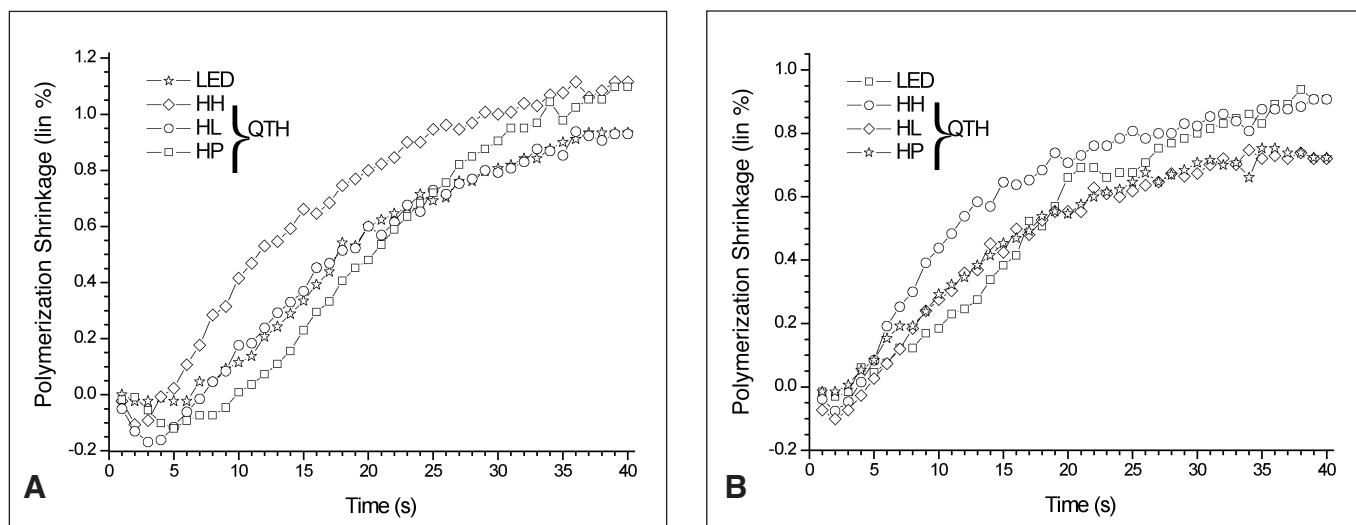
Figure 2. *Development of polymerization shrinkage of TC (Figure 2a) and F (Figure 2b) polymerized using a halogen or blue LED light source.*

(2.9) (TC, HP mode, surface). Using LED, the values varied from 56.5 (2.5) (F, 2-mm depth) to 62.3 (1.2) (TC, surface).

From *p* values, it is obvious that temperature values were significantly lower in the case of LED curing compared to all modes of halogen light. The temperature obtained (Tables 3 and 4) after 10 seconds was the highest for HH mode, then HL mode, HP mode and, finally, LED. Under illumination with QTH, the temperature changed from 4.6 (0.9) (TC, HP mode, 10 seconds) to 14.0 (1.7) (TC, HH mode, 20 seconds); whereas, under illumination with LED, the values increased from 1.5 (0.6) (F, 10 seconds) to 6.4 (2.5) (TC, 20 seconds). It is important to point out that significantly lower temperature values were obtained in the first 10 seconds under illumination with the HP mode of QTH compared to the HH and HL modes.

Polymerization shrinkage values (Table 5 and Figure 2 a,b) using the QTH source varied from –0.06 (0.02) (TC, LED, 10 seconds) to 2.26 (0.30) (TC, HH mode, 40 seconds). Using LED, polymerization shrinkage increased from -0.06 (0.02) (TC, 10 seconds) to 0.32 (0.20) (TC, 40 seconds). The highest final polymerization shrinkage (40 seconds) values were obtained for

HH mode, and the lowest were obtained for the HL mode of QTH and LED. However, these significantly different results were obtained for 10-second illumination with HL, HP and LED, for 20-second illumination with HP mode and for 40-second illumination for HH illumination mode. After 10 seconds, which means the pregelation phase, the lowest values were obtained using the HP mode of QTH and LED. Figures 2a and 2b show polymerization shrinkage development for TC and F cured using all exposure modes of QTH and with the LED for 40 seconds. In the figures, F showed more rapid induction of shrinkage but lower final shrinkage values than TC. In the first eight seconds, the effects of thermal expansion are obvious. Figures 3a and 3b show the fringe patterns obtained by interferometric measurements for LED and QTH (HH mode), respectively. Five images present the fringe patterns recorded at the start of polymerization (left image), at maximum expansion and at polymerization shrinkage in the 10th, 20th and 40th second. Their shape is a result of the interference of two beams, one with plane wavefront and the other with divergent wavefront. As a result, obtained zero fringe is easy to localize and follow its displacements. The right direction shows the extension of the sample (negative values in the tables in the case of 10-
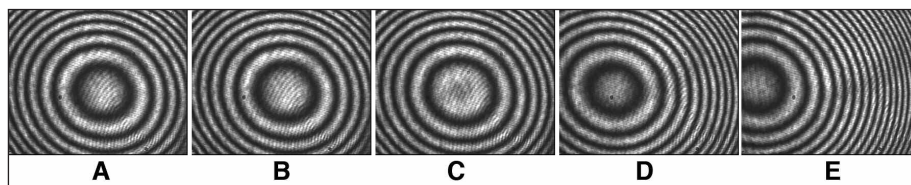
Figure 3A: *Holographic representation of expansion and contraction of the TC cured by HH mode of QTH (a) and LED (b) in 1, 7(8), 10, 20 and 40 seconds.*
A – 1st second of polymerization　　　*B – 7(8)th second of polymerization*
C – 10th second of polymerization　　　*D – 20th second of polymerization*
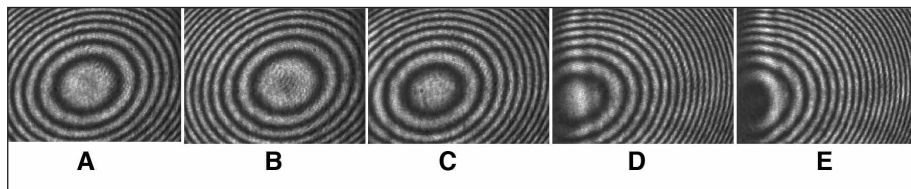*E – 40th second of polymerization*

Figure 3B: *Holographic representation of expansion and contraction of the TC cured by HH mode of QTH (a) and LED (b) in 1, 7(8), 10, 20 and 40 seconds.*
A – 1st second of polymerization　　　*B – 7(8)th second of polymerization*
C – 10th second of polymerization　　　*D – 20th second of polymerization*
E – 40th second of polymerization

second illumination for LED and HP illumination mode), while the left direction indicates shrinkage. Change in the number of fringes indicates the amplitude of the sample thickness (Demoli & others, 2004).

Thermal expansion effects were clearly different for the different curing modes. Induction time also varied with light intensity, but low heat development of the LED-curing device was also clearly visible in this data. The experimental approach showed variation in thickness of the highly viscous composite materials.

DISCUSSION

The RH tested was that different light sources and curing modes do not significantly influence degree of conversion. RH was generally accepted but, in the case of illumination of F on 2-mm depth, it was rejected. In that case, there was a significant difference among polymerization with the LED, HH and HL curing modes. However, there was a significant difference between the degree of conversion on the surface and 2-mm depth for F in the case of illumination with LED and HP illumination mode and for TC in the case of illumination with HH and HL illumination modes. In this case, the RH was rejected.

For temperature measurements, three tests were made and three RH hypotheses were tested. The first test compared temperature values obtained with different curing modes after a 40-second illumination. The RH tested for temperature measurement was that temperature does not significantly increase depending on the curing mode used. The RH was rejected, because materials cured with the HH curing mode represented significantly higher temperature values than any other curing mode. Significantly lower temperature values were obtained in the case of LED curing compared to HH and HL modes for both materials; whereas, compared to HP curing for TC, the temperature value after 40 seconds was lower but not significantly. The second test was a comparison of the temperature rise after 10 and 20 seconds. The RH that was tested was rejected.

The third test represents a comparison of temperature values for the same curing mode in 20 and 40 seconds. The RH hypothesis tested was that there is no significant difference in 20- and 40-second illumination. The RH was accepted for all curing modes at the $p > 0.08$ level.

Observing the results of polymerization shrinkage for both composite materials, there was a significant difference between LED, HH and HL curing modes in the case of 10-second illumination. In the case of 20- and 40-second illumination, there was a significant difference between LED and all programs of QTH. Observing the difference between tested composite materials, there was a significant difference between TC and F composite material in the case of 10-second illumination with LED, HL and HP, in the case of 20-second illumination with HP and LED and in 40-second illumination with the HH program. The RH that curing intensities strongly influence contraction kinetics was accepted. For F composite material, there was a significant difference in the case of 10- and 20-second polymerization with QTH, and, in the case of 20 and 40 seconds, there was not a significant difference. However, in the case of illumination with LED, there was a significant difference in 10, 20 and 40 seconds. The F material was polymerized according to the manufacturer's instructions for 20 seconds. Upon completion of illumination, the authors continued to record polymerization shrinkage for the next 20 seconds. The difference in polymerization shrinkage in the case of LED illumination in 20 and 40 seconds was probably influenced by the ongoing hardening of the material after the end of illumination, due to the internal thermal reactions of the material as a consequence of lower light intensity and incomplete polymerization in the regular interval of 20-second illumination. In the case of TC, there was a significant difference in 10-, 20- and 40-second illumination with HH and HP mode and between 10 and 20 seconds; that is, 40 seconds in the case of illumination with HL and LED.

Few studies have evaluated the relationship between volumetric shrinkage, contraction stress and degree of conversion during polymerization (Lim & others, 2002; Silikas, Eliades & Watts, 2000; Halvorson, Erickson & Davidson, 2002). The conversion values obtained in this study correspond with results cited in studies using conventional QTH curing lights (59–65%) (Yap & Senevirante, 2001; Mills, Jandt & Ashworth, 1999; Imazato & others, 2001; Blankenau & others, 1991). In the current study, the conversion values that were obtained were significantly lower using LED source (56%-62%) at a depth of 2 mm compared to those obtained using the different curing modes of the halogen light. However, higher conversion and better material properties were at the cost of higher shrinkage and contraction stress with all well-known consequences (Bouschlicher, Vargas & Boyer, 1997; Stoll & others, 2000). Lower polymerization shrinkage of TC cured using LED, regardless of the higher degree of conversion, can be explained by slower LED polymerization and by different time frames of measurement. FTIR values were determined after 24 hours, and shrinkage was only measured during and after 40 seconds. The material should be maximally polymerized not only to achieve optimal mechanical properties, but also to avoid a possible pulp response to uncured monomers from composite and dentin bonding agents, which may reach the pulpal space (Chen & others, 2001). The severity of pulpal reactions decreased with an increase in the remaining dentin thickness (Camps & others, 2000).

When a photoactivated process is considered, it is important to know that more than 80% of the total energy of the halogen lamp is outside the useful curing range. In contrast, 100% of light emitted by a blue LED lies within the spectral absorption of camphorquinone (Kurachi & others, 2001; Tarle & others, 2002). The energy of the halogen lamp spreads wider than that of the wavelength needed to cure resin by a factor of three times, when compared to light emitted by blue LED. Curing lamps vary from one to another in the amount of heat generated and the most effective light source gives the highest temperature (Shortall & Harrington, 1998). However, if one takes into account factors such as rise in temperature during curing, then the LED-based device offers a significant advantage. Tarle and others (2002) evaluated temperature rise in composite samples cured by experimental LEDs; Knezevic and others (2001) observed that LEDs obtained slightly lower conversion values and a significantly lower temperature increase when compared to conventional lamps.

For the halogen lamp used, regardless of the curing mode, significantly higher temperatures were obtained when using blue LED light. Slightly higher final polymerization shrinkage values associated with significantly higher temperature in the case of the halogen light used (especially HH mode) may have a negative influence on the development of stress, including adhesive and cohesive failures in materials and tooth structure. Radiation intensity must not be reduced to the degree where complete polymerization is compromised, because this would produce inferior mechanical properties and increase water sorption and residual monomer content (Caughman & others, 1991), which could compromise biocompatibility.

CONCLUSIONS

Photoactivated resin based composite can be successfully polymerized at reduced light intensity, while the final conversion value remains high. The results obtained with blue LED light confirm this theory. Therefore, the purpose of blue LED polymerization or "soft-start" polymerization is to enable, for as long as possible, the pregelation phase to permit slow and ordered chain growth and the reduction of interfacial stresses by internal plasticity during the slow cure. However, according to the results of this study, it can be concluded that significantly lower final temperature increases and polymerization shrinkage are an important advantage of the LED light source compared to a QTH curing unit.

(Received 20 January 2005)

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