Composite conversion and temperature rise using a conventional, plasma arc, and an experimental blue LED curing unit

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SUMMARY The objective of this study was to evaluate the degree of conversion and temperature rise in three different composite materials when illuminated by an experimental light source [blue superbright light emitting diodes (LEDs)] and compared with plasma light and traditional photopolymerization unit. The degree of conversion and temperature rise were measured using Fourier transform infrared (FTIR) spectroscopy and digital multimeter, respectively. The results revealed significantly higher degree of conversion values in case of conventional curing than with other two light sources whereas temperature rise was significantly lower when blue LEDs and plasma light were used. There were great differences in light intensities between blue LEDs of only 9 mW cm^{-2} compared with plasma light of 1370 mW cm^{-2} and Elipar II of 560 mW cm^{-2}. Better match of LED spectral distribution peak to camphorquinone absorption distribution peak probably explains much lower intensities used for similar photopolymerization effect like in the case of rapid plasma lamp curing.

KEYWORDS: composite photopolymerization, blue LEDs, plasma light

Introduction

Despite considerable improvements of dental composite materials, present-day composites still suffer from inadequate degree of conversion and problems of marginal adaptation. Importance is attached to the development of new dentine bonding agents, chemistry of resins and fillers rather than to any increase in intrapulpal temperature that occurs in composite resin placement from the material itself and from light-curing units (Peutzfeldt, 1997; Stansbury *et al.*, 1995; Meredith & Setchell, 1997).

At present, the halogen lamp is the most often used light source for composite photopolymerization. However, heat generation is a major disadvantage of using the halogen lamp as a light source (Fujibayashi *et al.*, 1998). A higher degree of conversion which is primarily related to curing light intensity and exposure time, conflict with the objective of achieving optimal marginal integrity because of the increased contraction (Feilzer *et al.*, 1995). Curing lights differ in intensity output and they range from < 200 to 1000 mW cm^{-2} or more. Temperature rise during the curing of light activated restoratives relates both to the exothermic polymerization of the material and to the heat output from the dental light curing units and increases with increasing radiation time and decreased material thickness (Lloyd *et al.*, 1986). Masutani *et al.* (1988) concluded that the resin itself had a greater influence upon the temperature rise during the curing than the light source, whereas Strang *et al.* (1988) found the light activation unit as the most significant source of heat during polymerization. Recent studies revealed that the marginal adaptation of light cured composites is enhanced when initial period of irradiation is conducted at low light intensity (Feilzer *et al.*, 1990a). Very closely connected to temperature rise is polymerization shrinkage stress in the material which can be divided into the phases corresponding to the development of elastic modulus (Sakaguchi *et al.*, 1997). In the pre-gel phase, the composite is able to flow, which relieves the

Table 1. Composite materials tested in the experiment

Composite material	Manufacturer	Abbreviation	Shade	Batch no.
Filtek Z 250	3M Dental Products (St Paul, MN, USA)	Z 250	A3	6020A3 9AK
Tetric Ceram	Vivadent (Schaan, Liechtenstein)	TC	A3	B12275
Pertac II	ESPE (Seefeld, Germany)	P II	A3	0123 A3 019

stress within the restoration (De Gee *et al.*, 1997; Feilzer *et al.*, 1990b). After the gelation, composite develops shrinkage stresses because of the advancement of elastic modulus. Internal shrinkage stresses occur because flow is not able to compensate for the contraction. It was the reason for the appearance of marketed 'soft cure' light activation units that have the option of operating with an initial period of low intensity illumination and which should reduce stress development during composite cure (Mehl *et al.*, 1997). Blankenau refers to the patterns as stepping (low level followed by high level), ramping (continuous increase from low to high), and interval or pulsing (successive on and off patterns) (Blankenau *et al.*, 1998). Some new light sources aimed at enhancing the conversion and diminishing temperature rise and consequently polymerization shrinkage (e.g., continuous argon laser and pulsed blue laser have already been tested) (Zakariasen, 1993; Tarle *et al.*, 1995; Meniga *et al.*, 1997; Tarle *et al.*, 1998a,b).

The objective of this study was to evaluate the degree of conversion and temperature rise in three different composite materials when illuminated by three different light sources: an experimental light source [blue superbright light emitting diodes (LEDs)], rapid plasma arc light and conventional halogen lamp. The hypothesis is that minimal energy of solid state, highly durable blue LEDs could ensure satisfying degree of conversion and minimal temperature rise in composite resin sample using the most efficient wavelength at 470 nm, which corresponds to absorption coefficient maximum of camphorquinone.

Materials and methods

Three different hybrid composites of the same universal A3 colour have been used for the degree of conversion and temperature rise measurements. Materials, manufacturers, abbreviations, shades and batch numbers are given in Table 1.

Three different light sources used in the experiment were: three blue superbright LEDs NSPB500S* with
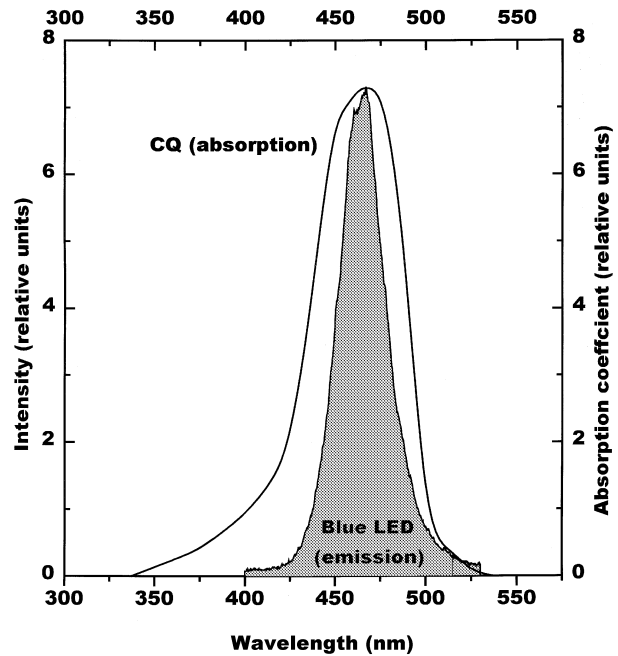
Fig. 1. Spectral distribution of blue LED compared with camphorquinone absorption peak.

only 9 mW cm^{-2} and tip diameter of 10 mm glued together in a triangle, plasma light Apollo 95 E[†] with 1370 mW cm^{-2} and 14 mm diameter tip and conventional halogen lamp Elipar II[‡] with intensity of 560 mW cm^{-2} and 10 mm diameter tip.

Superbright blue LEDs with narrow spectral distribution have the peak that coincides with the camphorquinone absorption curve peak at 470 nm (Fig. 1). The power of a single superbright blue LED is of about 3 mW, and its luminous intensity is of about 3 Cd at the DC forward electric current of 20 mW and the DC forward voltage of only 3·6 V. The power dissipation is of about 120 mW. At room temperature the coefficient of temperature increase per input electric power is about 0·5 °C mW^{-1} at the LEDs active layer. When LEDs are densely mounted, the overall heat generation gets higher. The chromatic coordinates of NSPB500S are $x = 0.130$ and $y = 0.075$ [referred to Commission

International de l'Eclairage (CIE) (International Commission of Illumination) 1931 chromaticity diagram].

The plasma lamp Apollo 95 E produces a visible plasma light between 400 and 500 nm for photopolymerization of dental composite materials. Power supply voltage is 230 V and average value of light power is 1370 mW cm^{-2}. Any variation in voltage has no effect on the lamp intensity. It is possible to select curing mode for 1, 2, 3 s or step polymerization meaning 1·5 s at 150 W and 2·5 s at 300 W.

The degree of conversion and temperature rise were determined after 20 and 40 s illumination for blue LEDs and Elipar II, whereas for the plasma light, all curing modes were tested. In case of plasma light polymerization, temperature rise was controlled after 1, 2, 3, 5, 10, 15 and 20 s. For all the materials and all light sources observed, five consecutive measurements have been made. Statistical analysis was performed using multiple analysis of variance ($P < 0.05$).

Degree of conversion measurements

Composite resin samples were prepared in the same manner for all the photopolymerization techniques used. The intention was to simulate the wafer at surface and at the 2 mm depth using previously cured underlays and overlays (Tarle *et al.*, 1995). Fourier transform infrared (FTIR) spectra of the cured samples, as prepared, were recorded in a transmission mode at room temperature using Perkin Elmer model 2000 spectrometer[§]. The uncured samples were pressed into KBr pellets ($\varnothing = 1$ cm) using Merck's spectroscopically pure KBr with a Carver press[†]. A total of 20 scans were measured at the resolution of 4 cm^{-1}. The spectra were recorded by subtracting the background and conversion into the absorbance mode. RUEGGEBERG's baseline method was used for the peak ratios calculation (Fig. 1c; Rueggeberg *et al.*, 1990). For all the materials aliphatic C=C peak at 1637 cm^{-1} and aromatic C=C at 1608 cm^{-1} were used. RUEGGEBERG found that the spectra of some commercial composites had troughs after 1608 cm^{-1} that were lower in absorbance than the one after 1582 cm^{-1}, making a baseline connection impossible. The conversion rate was obtained from equivalent aliphatic/aromatic molar ratios of cured (C)

[§]Perkin Elmer, Beaconsfield, Bucks, UK.
[†]Specac Ltd., Orpington, Kent, UK.

Table 2. Degree of conversion (%) of composites cured at surface and 2 mm depth for 20 and 40 s with blue light emitting diodes (LEDs) and Elipar II halogen light

Curing units		Composite material		
		Z 250	TC	P II
Blue LEDs				
Surface	20 s	57·7 ± 3·5	57·6 ± 1·9	61·1 ± 1·4
	40 s	54·9 ± 1·3	61·3 ± 2·2	65·4 ± 2·4
2 mm	20 s	56·7 ± 4·8	–	–
	40 s	59·1 ± 7·8	58·8 ± 4·4	–
Elipar II halogen light				
Surface	20 s	60·0 ± 2·5	65·7 ± 2·1	70·3 ± 3·1
	40 s	63·5 ± 1·8	68·3 ± 0·5	73·5 ± 1·4
2 mm	20 s	60·6 ± 2·1	64·7 ± 2·4	60·3 ± 2·6
	40 s	61·6 ± 0·8	67·8 ± 0·8	68·6 ± 3·1

and uncured (U) samples using the following formula: % conversion = $(1 - C/U) \times 100$.

Temperature rise measurements

The temperature rise in composite samples was measured in the $4 \times 4 \times 4$ mm thick samples, positioned in the Teflon moulds of the same dimensions. Thermocouple tip (K type, 1 mm diameter) was inserted into the composite sample to the depth of 2 mm from the front side. Temperature rise was measured using digital multimeter (Metex, M-3850 D[*]) connected to laboratory personal computer using Metex software. Temperature profile could be obtained online, on the PC monitor and written on a file and as a printed hardcopy with all data.

Results

Table 2 shows the mean and standard deviation (s.d.) of the degree of conversion for three composite materials after illumination of 20 and 40 s at the surface and at the simulated depth of 2 mm with blue LEDs and conventional halogen lamp Elipar II. Degree of conversion values varied from 54·9 ± 1·3 to 65·4 ± 2·4 in case of blue LEDs curing and from 60·0 ± 2·5 to 73·5 ± 1·4 when halogen lamp was used.

Table 3 shows mean and s.d. of the degree of conversion of composites cured for 1, 2, 3 s and using step curing mode at surface and at 2 mm depth with plasma light Apollo 95 E which varied from 53·8 ± 2·7 to 69·7 ± 5·5. Results obtained revealed mostly the

[*]Pirmasens, Winzeln, Germany.

Table 3. Degree of conversion (%) of composites cured at surface and 2 mm depth for 1, 2, 3 s and using step curing mode with plasma light

		Composite material		
		Z 250	TC	P II
Surface	1 s	55·5 ± 2·9	58·0 ± 6·3	62·4 ± 2·4
	2 s	57·8 ± 5·6	58·9 ± 3·2	60·8 ± 2·8
	3 s	57·2 ± 4·4	62·1 ± 3·4	60·7 ± 2·6
	Step	60·4 ± 3·2	61·9 ± 1·8	69·7 ± 5·5
2 mm	1 s	–	–	–
	2 s	57·1 ± 8·5	59·6 ± 2·7	57·1 ± 4·3
	3 s	53·8 ± 2·7	58·2 ± 4·2	59·1 ± 1·7
	Step	58·5 ± 2·0	59·1 ± 4·5	57·8 ± 1·7

Table 4. Temperature increase (°C) of composites during 20 and 40 s illumination with blue LEDs and Elipar II halogen light and during 20 s measuring after plasma light curing

		Composite material		
		Z 250	TC	P II
Blue LEDs	20 s	5·6 ± 0·9	3·6 ± 0·9	5·0 ± 1·2
	40 s	5·6 ± 1·1	4·6 ± 0·5	7·8 ± 0·8
Elipar II	20 s	10·5 ± 2·4	10·8 ± 1·8	14·0 ± 5·0
halogen light	40 s	10·5 ± 2·5	9·2 ± 1·8	13·0 ± 2·7
Plasma light	1 s	5·4 ± 1·8	6·2 ± 0·8	3·2 ± 1·1
	2 s	5·2 ± 2·3	3·6 ± 1·1	4·2 ± 1·9
	3 s	2·6 ± 0·8	5·0 ± 1·0	5·2 ± 0·8
	Step	2·8 ± 1·1	6·0 ± 2·5	6·2 ± 3·2

highest polymerization values for Pertac II (P II) composite for experimental and control curing methods. Significantly higher degree of conversion ($P < 0.05$) was obtained for conventional curing unit Elipar II compared with other light sources.

Table 4 shows mean and s.d. of temperature increase in composite samples polymerized by blue LEDs, conventional curing unit Elipar II and plasma light. It is obvious that significant differences in temperature values ($P < 0.05$) exist among conventional curing method and blue LEDs as well as plasma arc. For the quartz unit and the blue LED, temperature values were presented with respect to time. Most temperature changes occur after 20 s. For all the materials temperature rise values were significantly higher ($P < 0.05$) in case of conventional curing technique, whether compared with blue LEDs or plasma light. The values ranged from 9.2 ± 1.8 to 14.0 ± 5.0 °C. For the blue LEDs all the values were significantly lower ($P < 0.05$) after 20 and 40 s of

polymerization and varied from 3.6 ± 0.9 to 7.8 ± 0.8 °C. In case of plasma light illumination, temperature rise was measured for all four curing modes after 1, 2, 3, 5, 10, 15 and 20 s. Plasma arc temperature values were presented with respect to time, while the illumination duration was very short 1, 2, 3 s and step-curing (4 s) and the temperature rises after the finishing of exposure and finishing of termination of cross-linking (15–20 s). It was the reason to leave the thermocouple inserted in the composite material after the end of exposure. For all the materials the highest temperature values were obtained between 10 and 15 s after illumination with plasma light regardless of the curing mode used and were significantly lower ($P < 0.05$) compared with standard curing unit. The values varied from 2.6 ± 0.8 to 6.2 ± 3.2. It can be noticed that significantly lower temperature rise ($P < 0.05$) was obtained for all the materials with plasma light as well as with blue LEDs compared with conventional Elipar II halogen light.

Discussion

On the basis of published literature it is often difficult to know what caused measured differences in material properties because investigated materials are different in several aspects. In the present study, the colour of the materials was universal (A3) for all the composites tested because differences in light transmittance through different shades may affect their clinical properties (Arikawa *et al.*, 1998). Two variables have been changed: light source and composite materials. Significantly higher degree of conversion values ($P < 0.05$) were obtained in case of conventional curing after 20 and 40 s polymerization of both measured depths compared with blue LED results and after all curing modes with plasma light. At the depth of 2 mm, blue LEDs were unable to adequately cure the composite layer for all the materials except for the Filtek Z 250 after 20 and 40 s illumination and Tetric ceram (TC) after 40 s; namely, conventional curing unit had intensity of 560 mW cm^{-2}, blue LEDs had only 9 mW cm^{-2} and plasma light 1370 mW cm^{-2}. Slightly lower values of the degree of conversion at the surface in the case of blue LEDs curing could be explained with 60 times lower intensity compared with conventional curing unit. On the other hand 152 times lower intensity of blue LEDs than high power plasma light did not influence on the degree of conversion values. The results obtained after blue LEDs and plasma arc illumination were almost equal regardless of the intensities and exposure time

applied. It is surprising that high intensity plasma light suggested for rapid and more successful polymerization also had lower degree of conversion for all the materials at both depths than conventional curing unit and almost equal conversion at the surface as low intensity blue LEDs. Temperature values are often neglected although they are very important not only for shrinkage stress appearance in the material and on the composite cavity interface but also for the tooth pulp injury (Davidson & Feilzer, 1997; Shortall & Harrington, 1998). For all the materials, temperature rise values were significantly higher ($P < 0.05$) in case of conventional curing technique whether compared with blue LEDs or plasma light. In the case of plasma light curing, the highest temperature increase was obtained between 10 and 15 s after illumination regardless of the curing mode used and were significantly lower ($P < 0.05$) compared with standard curing unit. As already noticed the peak of temperature did not occur immediately but 10–15 s later and after 20 s the temperature values were again lower and stabilized.

All efforts are being devoted to the development of new monomer systems intended to reduce shrinkage stress such as oxybismethacrylate monomers and oligomers, as compared with dimethacrylate commonly used in dentistry (Peutzfeldt & Asmussen, 1996; Peutzfeldt, 1994). Because of the favourable stereochemistry, long chain flexible dymethilacrylate glycols such as ethylene glycol dimethacrylate (EGDMA) or triethyleneglycol dimethacrylate (TEGDMA) have been found to exhibit relatively high degree of conversion of the methacrylate double bonds (Asmussen & Peutzfeldt, 1998). It could explain the higher conversion values obtained for P II compared with other materials illuminated by the same light sources. Other resin composites contain UEDMA-urethan dimethacrylate (TC, Z 250) which has the advantage of lower viscosity and greater flexibility of the urethane linkage that may improve toughness of resin composite (Kalliyana & Yamuna, 1998). General differences between resin composites based on urethane dimethacrilates and conventional bisphenol A glycidyl methacrylate based resin composites are camouflaged by the fact that the materials differ in many other aspects, e.g. type and amount of filler, initiators and silanization of the filler particles. The Z 250 contain new monomer system UEDMA and BIS-EMA of higher molecular mass and fewer double bonds, which enables more efficacious polymerization and shorter polymerization of only 20 s per layer. Results of this study confirmed almost equal degree of conversion after 20 and 40 s for the mentioned material and even possible polymerization at the 2 mm depth with low power blue LEDs. Temperature rise has also not changed after 20 and 40 s illumination.

Halogen lamp is more efficient in the red and infrared light and is only slightly energetic in the zone of camphorquinone absorption of maximum at 470 nm. It is common knowledge that red light produces more heat than violet light. Unfortunately, this means that the temperature rises without significantly improving photopolymerization. Important advantage of blue LEDs is the possibility to choose the most efficient wavelength of 470 nm, justifying the very narrow wavelength preference of camphorquinone. Any wavelengths below about 430 nm and above 500 nm are not utilized in the electron promotion of the ketone groups in camphorquinone and therefore it can be said that camphorquinone ignores these wavelengths. The unwanted wavelengths do produce additional heat, affecting the kinetics of the reaction and may thereby influence the reaction. Conventional light sources produce a white light, which is then filtered in an effort to remove the unwanted wavelengths. Blankenau et al. (1999) studied the output intensity of different conventional light sources. While filtering systems do not increase the peak wavelength close to optimum, there is a large amount of energy that falls outside the utilization envelope. The major disadvantage of blue LEDs is low light intensity per element and it is necessary to use more than one LED to obtain sufficient intensity for curing units and to ensure convergence and focusing of their light.

The unexpected result was that high power (1370 mW cm^{-2}) plasma light did not ensure significantly higher degree of conversion ($P < 0.05$) than only three blue LEDs and it was not capable to ensure polymerization at 2 mm depth using 1 s curing mode as claimed by the manufacturer. However, definitely very low temperature increase was obtained for all the materials with the high power plasma light because of very short exposition time. It means that more energy obviously does not lead to higher thermal effects. Plasma light rapid curing conflicts the suggested theory that it is better to polymerize slowly to achieve minimal internal tension (Kelsey et al., 1989). The analysis of rapid polymerization showed that composite behaves like a heterogeneous body, allowing a global distribution of internal stresses.

Presently, it is definite that the only commercially available light source halogen lamp has too many

drawbacks including very short lifetime. The future will show whether the attention needs to be turned to the minimal energy of solid state, highly durable blue superbright LEDs or to rapid polymerization with high-energy plasma light.

Acknowledgments

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