

# Comparison of Linear Polymerization Shrinkage and Microhardness Between QTH-cured & LED-cured Composites

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## Clinical Relevance

The second generation LED is an improvement over the first generation LED. Despite its reported efficiency in composite curing, the second generation LED performs as well as the QTH system for composite curing and does not produce more rapid composite polymerization compared to the QTH unit.

## SUMMARY

**This study evaluated the effectiveness of second generation light emitting diode (2<sup>nd</sup>LED) units in composite curing. In order to compare their effectiveness with that of conventional quartz tungsten halogen light curing units (QTH) and first generation LEDs (1<sup>st</sup>LED), the amount of linear polymerization shrinkage, polymerization speed and microhardness were measured. Linear polymer-**

**ization shrinkage was measured every 0.5-0.55 seconds for 60 seconds when composite specimens (Z250, 3M ESPE Dental Products, St Paul, MN, USA) were light cured with five different light sources: XL 3000 (QTH, 3M ESPE Dental Products), Elipar FreeLight 2 (2<sup>nd</sup>LED, 3M ESPE Dental Products), Ultra-Lume LED2 (2<sup>nd</sup>LED, Ultradent Products, South Jordan, UT, USA), Elipar FreeLight (1<sup>st</sup>LED, 3M ESPE Dental Products) and experimental product X (1<sup>st</sup>LED, Biomedisys, Seoul, Korea). The amount of linear polymerization shrinkage in 60 seconds and the speed of polymerization shrinkage in the first 15 seconds were measured for the different lighting units. The amount of polymerization was compared with one-way ANOVA using Tukey at the 95% confidence level. In order to compare the speed of polymerization, the peak time (PT) showing the highest speed of polymerization and maximum speed of polymerization ( $S_{max}$ ) were determined from the data and compared using one-way ANOVA with Tukey at the 95% confidence level for each material.**

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For microhardness measurements, the microhardness of 2-mm composites, Z250, which had been light cured by XL 3000 (G1), FreeLight 2 (G2), Ultra-Lume LED2 (G3), FreeLight (G4) or experimental product X (G5) were compared on the upper and lower surface. The microhardness of each surface was compared between groups using two-way ANOVA with Tukey test at 95% levels of confidence.

The amount of polymerization shrinkage at 60 seconds was G1, G2, G3 > G4, G5 ( $p < 0.05$ ). PT was G1, G3 < G2 < G4, G5.  $S_{\max}$  was G1, G2 > G3 > G4, G5 ( $p < 0.05$ ).

On the upper composite surface, there was no difference in microhardness between groups ( $p < 0.05$ ).

On the lower surface, the microhardness was G1, G2 > G3 > G4, G5 ( $p < 0.05$ ).

There was no difference in microhardness between the upper and lower surface in G1 and G2; whereas, microhardness of the lower surface was lower in G3, G4 and G5.

It was concluded that 2<sup>nd</sup>LEDs and the conventional QTH unit cured composites more effectively than 1<sup>st</sup>LEDs.

## INTRODUCTION

Adequate polymerization of a resin composite is considered a very important factor in obtaining adequate physical (Asmussen, 1982) and biological properties (Caughman & others, 1991). It is reported that the power density required for effective polymerization of a composite should be more than 280 mW/cm<sup>2</sup> (Rueggeberg, Caughman, & Curtis, 1994). Quartz Tungsten Halogen (QTH) units have been most widely used for curing composites. However, the power density of a QTH system decreases as time passes, and halogen bulbs or filters should be replaced. The problem is that most dentists do not notice the decrease in light-emission if they fail to regularly check the power density of curing lights (Miyazaki & others, 1998; Martin, 1998).

Plasma Arc Curing (PAC) systems have a higher power density than conventional QTH units. They use a high-frequency electrical field to generate plasma energy, and matter is, thereby, transformed into a mixture of ions, electrons and molecules. The large amount of energy released during this process is used for curing photosensitive composites. Although manufacturers recommend that three seconds is sufficient for curing, 12 seconds of light curing is usually necessary for a shallow cavity not exceeding 2 mm (Park, Krejci & Lutz, 2002). In addition, as composites are cured faster in the PAC-cure than the QTH-cure, they may produce more strain in the cavosurface margin (Park & others,

2002). As the light spectrum emitted by the PAC system is narrower (450-470 nm) than the conventional QTH unit, some composites and bonding systems that do not use camphorquinone as a photo-initiator system do not react. The price of the PAC system is also much higher than the QTH unit.

An argon (Ar) laser, which has a more consistent light output over distance, has also been used to cure composites (Blankenau & others, 1991a,b; Kelsey & others, 1989). It emits specific bandwidths of light in ranges of 454 nm to 466 nm, 472 nm to 497 nm and 514 nm. Because it generates little infrared output, not much heat is produced. Even though 10 to 15 seconds is needed to cure composites with an Ar laser, the curing tip is so small that more time is generally needed to cure the restoration. Argon lasers also have narrow spectral outputs, they are expensive, inefficient and occupy too much space, and currently they are not popular (Burgess & others, 2002).

Light emitting diodes (LED) have recently been introduced to cure dental composites. LED curing lights use gallium nitride semiconductors that produce a blue light when subjected to an electrical current. An LED generates a narrower light-output of around 470 nm. As compared with the QTH unit, the LED has considerable merit from the clinician's viewpoint. Regarding its use as a semiconductor for light-emission, the power density of light does not decrease. Therefore, clinicians do not need to be concerned with loss of power density in the curing light. In addition, battery-powered LEDs have excellent battery life, because 1) the power requirements are significantly lower than QTH and PAC lights; 2) LEDs produce little heat during operation, which means that fans are not required to cool the units and 3) LEDs have no moving parts (Burgess & others, 2002). However, it has been reported that the power density of LEDs is so low that they do not sufficiently cure 2-mm composite specimens (Kurachi & others, 2001; Park & others, 2003). The recently introduced second generation LEDs (2<sup>nd</sup>LED) have a higher power density than first generation LEDs (1<sup>st</sup>LED).

A linear relationship between light intensity and polymerization contraction has been demonstrated (Sakaguchi & others, 1992). The contraction rate of light-cured composite is highest during the first 30 to 40 seconds of the polymerization reaction (Sakaguchi & others, 1992). This is clinically important, because the integrity of the tooth-composite interface is rapidly challenged during the early phase of polymerization, when the bond between hard tissue and the composite is still maturing. The speed and amount of polymerization shrinkage of a resin composite is easily and accurately measured by a specially designed linometer (de Gee, Feilzer & Davidson, 1993; Park, Krejci & Lutz, 1999, 2002).

In a resin composite, the physical properties are closely related to the degree of conversion and a hardness measurement is an effective way to evaluate the degree of cure (Rueggeberg & Craig, 1988).

This study evaluated the effectiveness of second generation LEDs (2<sup>nd</sup>LED) for composite curing. To compare its effectiveness with conventional LEDs (1<sup>st</sup>LED) and QTH units, the amount of linear polymerization shrinkage, speed of polymerization and microhardness of composites were compared.

## METHODS AND MATERIALS

### A. Measurement of Linear Polymerization Shrinkage

Z250 was used as the test composite. It was transferred to a Teflon mold to ensure that the same amount of composite was used for each linometer sample. The composite was then transferred to the disk in the custom-made linometer, which had been previously coated with a separating glycerin gel. The resin composite was then covered with a glass slide and loaded under constant pressure. The surface of the glass slide facing the composite had also been coated with separating gel. The composites were light cured with QTH, 1<sup>st</sup>LEDs or 2<sup>nd</sup>LEDs (Table 1). The power density of each curing unit was measured using a Coltolux Light Meter (Coltene, Altstätten, Switzerland) (Table 1). The tip of the curing light was positioned 2-mm above the slide glass and the specimens were light cured for 60 seconds. As the composite under the glass slide was cured, it shrank towards the light source and the aluminum disk under the composite was moved upward. The amount of disk displacement caused by linear shrinkage of the resin composite was measured using a Linear Variable Differential Transformer (LVDT) linometer (R&B Inc, Daejeon, South Korea). The digital data was recorded on a computer for 60 seconds using the Microsoft Excel 2002 program. Park and others (1999, 2002) previously reported the design of the linometer. Fifteen measurements were made for each group and the amount of linear shrinkage occurring over 60 seconds was statistically compared using a one-way ANOVA test, and a Tukey's test was used as a post hoc test at the 95% confidence level.

### B. Determination of Polymerization Speed

From the data in A, the peak time (PT) that showed the highest speed of polymerization and maximum speed of polymerization ( $S_{\max}$ ) in the first 15 seconds of curing were determined.

The speed of polymerization at time =  $t$  was calculated as follows;

$$S_t = (L_{t+\Delta t} - L_{t-\Delta t}) / (T_{t+\Delta t} - T_{t-\Delta t})$$

$S_t$ : Speed of polymerization shrinkage at time  $t$

$L_{t+\Delta t}$ : Amount of linear shrinkage at time  $t = t + \Delta t$

$L_{t-\Delta t}$ : Amount of linear shrinkage at time  $t = t - \Delta t$

$T_{t+\Delta t}$ : The time when  $L_{t+0.5}$  was measured.

$T_{t-\Delta t}$ : The time when  $L_{t-0.5}$  was measured.

( $\Delta t$  was 0.5-0.55s in this study)

PT and  $S_{\max}$  were compared between groups with one way ANOVA test, using a Tukey's test as a post hoc test at the 95% confidence level.

### C. Measurement of Microhardness

A 6-mm diameter hole was made in a 2-mm thick Teflon plate, and a glass slide was positioned along the lower side of the hole. Titanium-coated instruments (Composite Instrument, Coltene, Switzerland) were used to place the Z250 into the mold. The slide glass was placed on top of the composite and pressed flat. The specimens were light cured using QTH, 1<sup>st</sup>LEDs or 2<sup>nd</sup>LEDs (Table 1). Ten specimens were assigned to each group. After the composites were light cured, the specimens were removed from the mold. The upper surface (closer to the light source) and lower surface of the specimens were then marked with a pen. The specimens were stored in the dark in 100% humidity at 37°C for seven days. The microhardness of the upper and lower surfaces were then measured with a Vickers hardness-measuring instrument (Optidur, Göttfert Feinwerktechnik GmbH, Buchen, Germany). The microhardness was compared using a two-way ANOVA test and Tukey test to compute statistical significance at the 95% confidence level. In each group, the hardness ratio was also calculated.

Hardness ratio = (Microhardness of lower surface)/(Microhardness of upper surface)

Table 1: Curing Lights Used in this Study

Groups	Curing Light	Type	Curing Time (seconds)	Manufacturer	Power Density (mW/cm <sup>2</sup> )
1	XL 3000	QTH	60	3M ESPE, St Paul, MN, USA	730
2	Elipar FreeLight2	2 <sup>nd</sup> LED	60	3M ESPE, St Paul, MN, USA	980
3	Ultra Lume LED2	2 <sup>nd</sup> LED	60	Ultradent Products, South Jordan, UT, USA	560
4	Elipar FreeLight	1 <sup>st</sup> LED	60	3M ESPE, St Paul, MN, USA	330
5	X(Experimental Product)	1 <sup>st</sup> LED	60	Biomedisis, Seoul, Korea	310



## RESULTS

### A. Polymerization Shrinkage

Change in the amount of linear polymerization shrinkage versus time is shown in Figure 1.

Table 2 lists the amount of linear polymerization shrinkage measured in 60 seconds.

The order of the amount of polymerization shrinkage in 60 seconds was G1, 2, 3 > G4, 5 ( $p < 0.05$ ).

### B. Polymerization Speed

Changes in the speed of polymerization shrinkage versus time are shown in Figure 2.

The PT and  $S_{max}$  are listed in Table 3. The order of  $S_{max}$  was G1, 2 > G3 > G4, 5 ( $p < 0.05$ ). The order of PT was G1, G3 < G2 < G4, 5 ( $p < 0.05$ ).

### C. Microhardness

In a two-way ANOVA, significant differences in microhardness were observed for curing lights (Group 1 to 5) ( $p < 0.05$ ) and observation surfaces (Upper surface or Lower surface) ( $p < 0.05$ ). A significant interaction existed between the curing lights and observed surface ( $p < 0.05$ ). There was no difference between groups in the microhardness of the upper surface. However, on the lower surface, the order of microhardness was G1, 2 > G3 > G4, 5 ( $p < 0.05$ ) (Table 4). There was no difference in microhardness between the upper and lower surface in G1 and G2; whereas, the microhardness of the upper surface was higher than the lower surface in G3, G4, G5 ( $p < 0.05$ ) (Table 4).

The hardness ratio was more than 0.8 in all groups (Table 4).

## DISCUSSION

Results on the polymerization shrinkage, polymerization speed and microhardness showed that the 2<sup>nd</sup> LED was an improvement over the 1<sup>st</sup> LED and has similar performance to a QTH system.

LEDs were reported to be more effective than a QTH lamp in composite polymerization when power densities of both the LED and QTH lamp were adjusted to 300mW/cm<sup>2</sup>, because LEDs produced a narrower spectrum of light that fell closely within the absorption range of the camphoroquinone (Mills, Jandt & Ashworth, 1999). When this study was first designed, it was assumed that the polymerization speed of G2

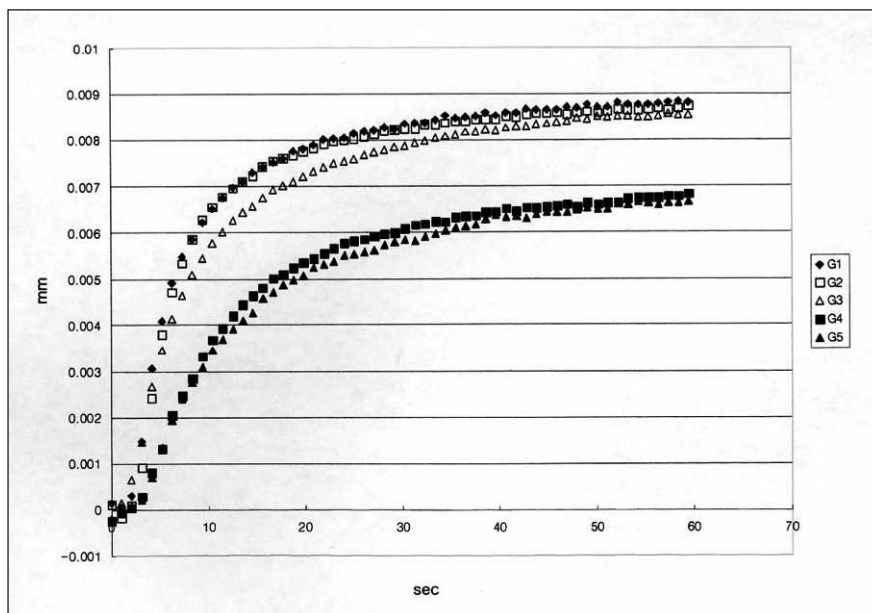


Figure 1: Change in the amount of linear polymerization shrinkage versus time.

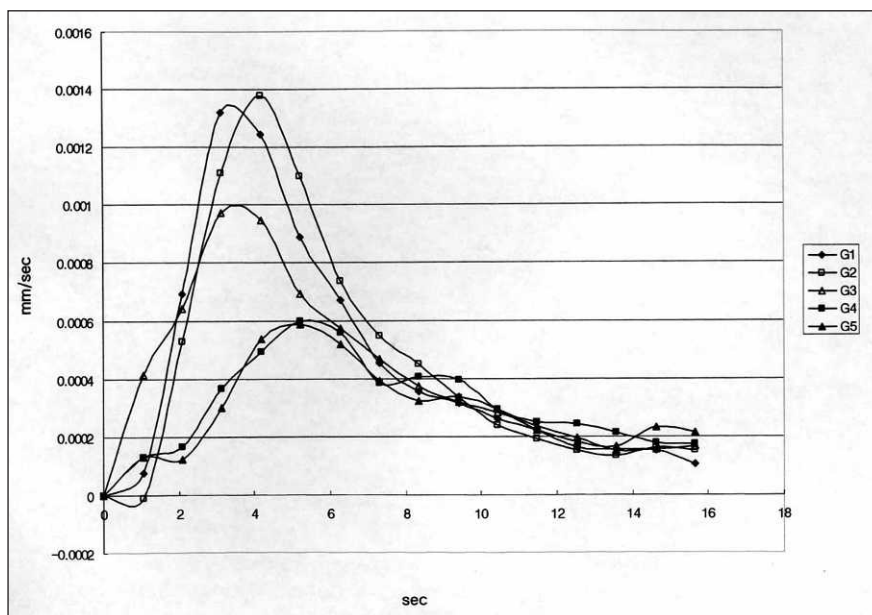


Figure 2: Change in the speed of polymerization versus time.

would be much faster than G1, because G2 was an LED and had a higher power density than G1. However, the shrinkage patterns of G1 and G2 were similar (Table 3, Figures 1 and 2). Some factors may be responsible for this discrepancy. First, the efficacy of the LED lamp may not be tied to the composite polymerization. Even though the efficiency of the LED lamp was 31% greater than the QTH lamp, the scrape-back lengths from the composites polymerized using the LED lamp were only 6% higher than those polymerized using the QTH lamp due to the exponential decay of light through the com-

posites (Halvorson, Erickson & Davidson, 2004). Second, the power density value may be incorrect, because a hand-held radiometer that has an opening with a set diameter was used. It has been reported that a radiometer with a fixed opening does not allow for an accurate measurement of power density (Leonard, Charlton & Hilton, 1999). Burgess and others (2002) reported that the power density of XL3000 (G1) and Elipar FreeLight2 (G2) was 750mW/cm<sup>2</sup> and 808 mW/cm<sup>2</sup>, respectively, when they were measured using a radiometer that did not have a fixed opening and allowed for an accurate measurement. In this study, the power density was 730mW/cm<sup>2</sup> and 980mW/cm<sup>2</sup>, respectively. Another possibility is that this might be a material-specific phenomenon. In Z250, a three-component initiation system (camphorquinone, tertiary amine and Iodonium salt) was introduced to initiate and activate the composite (US patent #5,545,676). Park and others (2002) reported that this initiation system was quite effective in composite polymerization kinetics and induced a more rapid cure with the QTH unit. However, the similarity in the amount of linear polymerization shrinkage and the similar shrinkage patterns between G1 & G2 is currently unclear, because there are many factors that can influence polymerization kinetics. Therefore, further research will be needed.

There was no difference in the amount of linear polymerization shrinkage between G1, G2 and G3 (Table 2, Figure 1). G3 showed a slower shrinkage pattern than G1 and G2 (Table 3, Figure 2), but it achieved the same shrinkage amount in 60 seconds. It has been reported that pre-polymerization at a low power density followed by a final cure at a high power density may allow for increased flow of material and a decrease in polymerization shrinkage stress in a restoration, which may produce a more favorable margin (Mehl, Hickel & Kunzelmann, 1997; Feilzer & others, 1995). G3 showed a slower cure than G1 and G2, but there was no difference in the amount of linear polymerization shrinkage in 60 seconds (Figures 1 and 2). Further research is needed to determine whether G3 provides better marginal adaptation than G1 and G2.

Table 2: Amount of Linear Polymerization Shrinkage ( $\mu\text{m}$ ) at 60 Seconds of Light Curing

Groups	1	2	3	4	5
	8.8(1.1)a	8.7(0.6)a	8.5(0.7)a	6.8(0.8)b	6.6(0.9)b
Different letters indicate different amounts of linear shrinkage at $p < 0.05$ level. Figures in the parenthesis indicate standard deviations.					

Table 3: Peak Time (PT) That Showed the Highest Speed of Polymerization and Maximum Speed of Polymerization ( $S_{\text{max}}$ )

	G1	G2	G3	G4	G5
PT(sec)	3.65(0.05)c	4.20(0.03)b	3.65(0.05)c	5.25(0.05)a	5.25(0.03)a
$S_{\text{max}}$ ( $\mu\text{m}/\text{sec}$ )	1.36(0.30)a	1.38(0.30)a	1.00(0.20)b	0.60(0.20)c	0.59(0.25)c
Different letters indicate different amounts of linear shrinkage at $p < 0.05$ level. Figures in the parenthesis indicate standard deviations.					

Table 4: Microhardness of Upper and Lower Surface

	G1	G2	G3	G4	G5
Upper surface	81.7(1.8)a	81.3(1.5)a	82.0(1.8)a	80.5(1.3)a	80.7(2.7)a
Lower surface	80.5(2.2)a	80.7(2.5)a	77.7(3.9)b	75.0(1.3)c	74.7(2.8)c
Hardness Ratio	0.98(0.02)	0.99(0.01)	0.95(0.02)	0.93(0.01)	0.93(0.03)
*Indicates different microhardness between upper and lower surface at 95% levels of confidence. Different letters indicate different microhardness on the upper or lower surface at the 95% levels of confidence. Hardness Ratio: (Microhardness of lower surface)/(Microhardness of upper surface)					

In this study, there were no differences in the microhardness of upper surfaces between groups. However, the order of microhardness of the lower surfaces was G1, G2 > G3 > G4, 5, and the microhardness of the upper surfaces was higher than the lower surfaces in G3, G4 and G5, whereas, there was no difference in G1 and G2. This is consistent with an article by Kurachi and others (2001). It has been reported that composite curing of a deep cavity layer is considered to be complete if the minimum hardness value is >80% of the maximum value measured on the specimen surface (Lutz, Krejci & Frischknecht, 1992; Breeding, Dixon & Caughman, 1991). Even though microhardness of the lower surface was lower than the upper surface in G3, G4 and G5, the hardness ratio was >0.8 in all groups. Therefore, it is possible that all LED systems used in this study can be used for a 2-mm cavity with 60 seconds of light curing. In a recent study, second generation LED was able to polymerize top and bottom composite surfaces equivalent to or greater than the 40 second-QTH control in only 10 seconds (Rueggeberg, Daronch & de Goes, 2004). Further research will be needed to determine the correct curing depth and curing time.

For Ultra-Lume LED 2 (G3), the power density was 560mW/cm<sup>2</sup>. Due to its unique elliptical shape, exact power-density measuring was difficult. Burgess and others (2002) reported that the power density of Ultra-Lume LED2 was 500 mW/cm<sup>2</sup> when measured using a radiometer that did not have a fixed opening and allowed for accurate measurement. According to the manufacturer, the major emission of Ultra-Lume 2 lies

between 445 and 472, and the peak wavelength of the unit is shifted away from a conventional LED: from 468 nm to 459 nm. Such a shift may provide additional energy within the spectral region of some of the alternative photoinitiators on the market. In this study, the PT was G1, 3<G2<G4, 5 (Table 3). It is not yet clear whether the shift in peak wavelength in G3 induced such a fast PT.

As long as camphorquinone is used as the photoinitiator, LED curing lights have adequate cure. However, photoinitiators, such as 1-phenyl-1,2-propanedione (with a peak absorption of 410 nm), bisacylphosphine oxide or triacylphosphine oxide (with peak absorption of 320 nm to 390 nm) may fall outside this range. Since the wavelength of light that LEDs emit range from 440 nm to 490 nm, some bonding agents and composites that use other agents as photo initiators suffered curing problems (Palmer & others, 2004). Recently, a third generation LED, which has a bimodal emission spectrum and covers a broader spectral range, has been released.

The 1<sup>st</sup>LEDs, G4 & G5, aligned small LEDs of lower power output into arrays. Their power density was much lower than 2<sup>nd</sup>LEDs, G2 & G3. Ultra-Lume LED 2 has two LEDs that are not arranged in array, but they rely on AC power to produce higher output. Of the LEDs used in this study, FreeLight2 provided the highest power density. According to the manufacturer, the 5W Luxeon LED was introduced and the effectiveness of the emitted light is enhanced by the application of a special reflector that increases coupled light.

## CONCLUSIONS

The results of this study show that second generation LED is an improvement over first generation LED and performs as well as a QTH system for composite curing.

## Acknowledgement

This study was supported by a grant (02-PJ3-PG4-PT03A-0018) by the Korea Health 21 R&D Project, Ministry of Health & Welfare, Republic of Korea.

(Received 2 January 2004)

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