

Effect of Irradiance and Exposure Duration on Temperature and Degree of Conversion of Dual-Cure Resin Cement for Ceramic Restorations

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Clinical Relevance

Providing longer exposure durations can be an efficient strategy to compensate for attenuated light through ceramic restorative materials in order to achieve a high degree of conversion. However, continuous irradiation with high irradiances for long durations will yield a higher temperature. Providing pauses in the irradiation to avoid continuous accumulation of energy that increases the temperature is recommended.

SUMMARY

This study investigated the effects of irradiance and exposure duration on dual-cured resin cements irradiated through ceramic restorative materials. A single light-curing unit was calibrated to three different irradiances (500, 1000, and 1500 mW/cm²) and irradiated to three different attenuating materials (transparent acryl, lithium disilicate, zirconia) with 1-mm thicknesses for 20 or 60 seconds. The

changes in irradiance and temperature were measured with a radiometer (or digital thermometer) under the attenuating materials. The degree of conversion (DC) of dual-cure resin cement after irradiation at different irradiances and exposure durations was measured with Fourier transform near infrared spectroscopy. Two-way analysis of variance revealed that irradiance ($p < 0.001$) and exposure duration ($p < 0.001$) significantly affected temperature and DC. All groups showed higher DCs with increased exposure times ($p < 0.05$),

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but there were no statistically significant differences between the groups irradiated with 1000 mW/cm² and 1500 mW/cm² ($p > 0.05$). Higher-intensity irradiances yielded higher temperatures ($p < 0.05$), but exposure time did not affect temperature when materials were irradiated at 500 mW/cm² ($p > 0.05$).

INTRODUCTION

Dual-cure resin composite uses both light and chemical initiation to activate polymerization. Adequate light curing is essential to achieve the sufficient polymerization rates of dual-cure resins, as autopolymerization without photopolymerization yields lower polymerization rates compared with dual polymerization.^{1,2} Moreover, adequate initial polymerization through light curing is critical for the success of restorations, given that restorations are exposed to masticatory force, saliva, and foods before autopolymerization is sufficiently processed, and these clinical circumstances may lead to material resorption and degradation.^{3,4}

Dual-cure resin cements are commonly used to cement ceramic restorative materials, given their color stability and sufficient working times.⁵ In this case, initial polymerization of dual-cure resin cements depends on the curing light that reaches luting materials after passage through the ceramics.⁶ The irradiance from curing lights is attenuated by ceramics, and clinical trials to compensate for the lack of degree of conversion (DC) are necessary. Using higher irradiances or providing longer exposure durations may be the solution.^{7,8}

Currently, light-emitting diode (LED) units are popularly applied in clinical practice. LED units are smaller and wireless⁹ and have a longer lifetime (1000 hours with constant light output).¹⁰ Although first-generation LED units have a limited output with an irradiance less than 400 mW/cm²,¹¹ LED units recently released on the market can deliver much higher light irradiances (greater than 2000 mW/cm²), facilitating reduced exposure times. Previous studies evaluating the effects of curing light irradiance on polymerization modes of resin composites used different light sources; quartz tungsten halogen and LED were used as lower-irradiance light sources, and plasma-arc lamps and argon-ion laser were used as higher-irradiance light sources. Each light source has typical wavelengths and characteristics,¹² and comparing the effects of irradiance with different light sources cannot be accurately indicative of the relation between irradiance and polymerization because polymerization aspects

can be affected by the spectral emission of light sources.^{13,14}

Temperature can be a critical factor affecting polymerization kinetics and the biologic homeostasis of pulp. Increasing temperatures during polymerization promote free radical and monomer mobility¹⁵ and lead to higher polymerization rates and elevated DCs. In addition, external thermal irritation may cause irreversible changes of pulp tissue,¹⁶ and temperature increases in restorative materials have substantial effects because a thinner dentin wall remains between the restorative material and pulp after tooth preparation. Increasing irradiance and exposure duration causes temperatures to rise,¹⁷ and measuring temperature changes, as a result of irradiating at various irradiances and exposure durations, is significant to the clinical outcome.

The purpose of this study was to evaluate the effects of irradiance and exposure duration on the DCs of dual-cure resin cements and temperature changes of restorative materials. For this, three irradiances (500, 1000, 1500 mW/cm²) were simulated with a single LED light-curing unit (LCU) by combining various ceramic restorations and exposure times, and the degrees of conversion and changes to temperature were evaluated. The null hypothesis was that irradiance and exposure duration have no effect on DC or temperature.

METHODS AND MATERIALS

Specimen Preparation

Figure 1 shows the methods for preparing specimens and irradiating with attenuating materials. A glass slide was positioned at the center of an infrared spectroscopy measuring block and fixed with cellulose tape. Mixed resin cements were applied to the center, and a second glass slide was used to cover the resin cement. The thickness of resin cement was determined by the thickness of the cellulose tape (40 μ m). A dual-cure resin cement, G-CEM LinkAce (GC Corporation, Tokyo, Japan; Lot 1604071), was used with Automix mixing tips.

Light Curing With Various Materials and Measuring Irradiance and Temperature

A single LED light LCU (Dr's Light, Good Doctors Co, Incheon, Korea) was used in this study. The output of the LCU was manually calibrated, varying the pulse width of LED using a microcontroller controlling the duty cycle of LED. The pulse width modulation is the efficient method to control the output of LED with high resolution.¹⁸ The calibrated

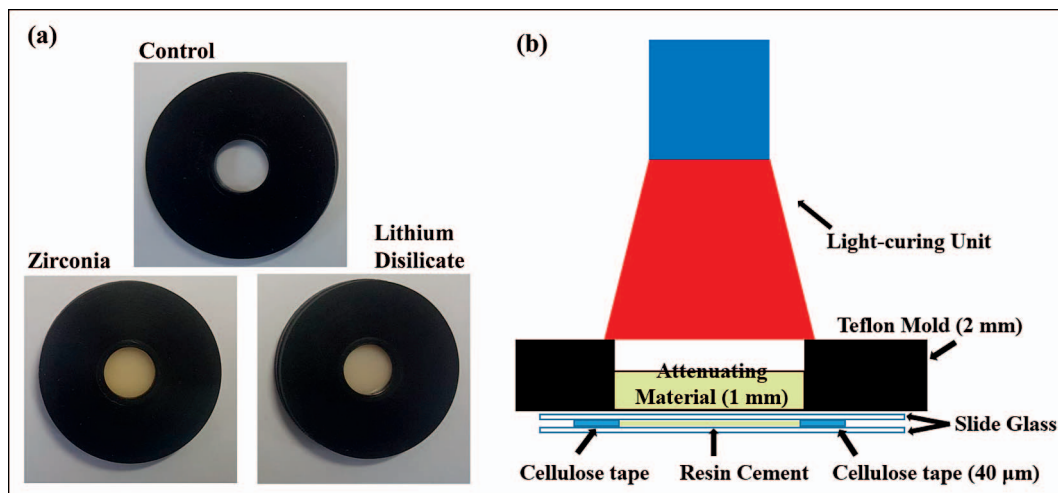


Figure 1. (a): Teflon molds with different attenuating materials. (b): An illustration of the methods for specimen preparation and irradiation (sagittal view). Measuring irradiance (or temperature) was performed with the same molds, not specimens, overlying a radiometer (or digital thermometer).

irradiances were confirmed with a radiometer (LED radiometer; Good Doctors Co). The LCU was set up to emit three different light irradiances: low (≈ 500 mW/cm²), medium (≈ 1000 mW/cm²), and high (≈ 1500 mW/cm²). Temperature changes after irradiation were measured with a contact-type digital thermometer (digital thermometer 367DN, TPI, Inchon, Korea) contacting the tip of the thermometer under the center of attenuating material. The temperature was measured at room temperature (24°C), and irradiance and temperature were measured six times each. Light curing was always performed with Teflon molds, and a 2-mm distance from the curing unit tip to each object was provided.

As the three light irradiances were exposed to specimens for 20 or 60 seconds, six different irradiating methods were used in this study. Twenty-seven specimens were prepared for each irradiation method, divided into three groups (n=9) depending on the materials overlying the specimens during light exposure: control, zirconia, and lithium disilicate (Table 1). The control group specimens were light cured through a transparent acrylic plate. For the zirconia and lithium disilicate groups, light curing was applied through the discs with A2 shades

composed of translucent zirconia (LAVA Plus, 3M Deutschland GmbH, Neuss, Germany) or lithium disilicate (IPS e.max Press HT ingots, Ivoclar-Vivadent AG, Schaan, Liechtenstein). All attenuating materials had a thickness of 1 mm.

Infrared Spectroscopy Measurements

The DC of the resin cement was evaluated using Fourier transform near infrared spectroscopy (NIR-Solutions, BUCHI, Flawil, Switzerland). Absorbance spectrums were examined by scanning the specimens 10 times over a 10,000- to 4000-cm⁻¹ range, with a resolution of 4 cm⁻¹. To calculate DCs, the area of the peak corresponding to vinyl stretching at 6165 cm⁻¹ was used.¹⁹ The DCs were calculated from the ratio of the peak area in the monomeric to polymeric states, using the following formula:

$$DC(\%) = (1 - \text{area}_{\text{polymer}}/\text{area}_{\text{monomer}}) \times 100$$

Statistical Analysis

The mean and standard deviation (SD) temperatures and DCs were acquired for each group. Data were evaluated for homogeneity of variance based on

Table 1: Experiment Study Design^a

Attenuating Materials/ Irradiating Methods	Low Irradiance (500 mW/cm ²)		Medium Irradiance (1000 mW/cm ²)		High Irradiance (1500 mW/cm ²)	
	20 s	60 s	20 s	60 s	20 s	60 s
Transparent acryl (control)	C-L20 (n=9)	C-L60 (n=9)	C-M20 (n=9)	C-M60 (n=9)	C-H20 (n=9)	C-H60 (n=9)
Lithium disilicate	L-L20 (n=9)	L-L60 (n=9)	L-M20 (n=9)	L-M60 (n=9)	L-H20 (n=9)	L-H60 (n=9)
Zirconia	Z-L20 (n=9)	Z-L60 (n=9)	Z-M20 (n=9)	Z-M60 (n=9)	Z-H20 (n=9)	Z-H60 (n=9)

^a n indicates the number of specimens.

Table 2: Irradiance Reaching Sensor for Light Curing Through Different Attenuating Materials (Mean [SD]), and the Reduction of Irradiance by Ceramic Materials

Attenuating Material	Irradiance, mW/cm ²					
	Low Irradiance		Medium Irradiance		High Irradiance	
Control	494.2 (15.1)	Reduction, % ^a	1034.6 (11.9)	Reduction, % ^a	1486.2 (9.7)	Reduction, % ^a
Lithium disilicate	135.5 (2.1)	27.4	329.3 (3.1)	31.8	484.7 (5.8)	32.6
Zirconia	92.5 (1.8)	18.7	225.8 (10.5)	21.8	322.3 (2.7)	21.7

^a Reduction values are calculated compared to control.

Levene’s tests ($\alpha=0.05$). The influences of independent variables, including irradiance and exposure duration, to temperature and DC were analyzed using two-way analyses of variance (ANOVAs; $\alpha=0.001$). Between-group comparisons of temperatures and DCs were conducted using one-way ANOVA and Tukey’s multiple-comparison tests ($\alpha=0.05$). All statistical analyses were conducted using SPSS for Windows (release 12.01; SPSS Inc, Chicago, IL, USA).

RESULTS

Table 2 shows the mean and SD irradiances with different light-curing methods, and Table 4 provides calculated radiant exposures. Temperature and DC results, including means, SDs, and statistical significances, are given in Tables 4 and 5, respectively. Two-way ANOVA revealed significant differences in temperature and DC due to irradiance and exposure duration ($p<0.001$; Table 3).

Light irradiance was reduced by lithium disilicate and zirconia, to 26.4%, 31.8%, and 32.6% and 19.7%, 21.8%, and 21.7% at low, medium, and high irradiances, respectively. Temperatures with control, lithium disilicate, and zirconia were increased to 47.1°C, 39.2°C, and 42.2°C, respectively, at room temperature (24°C) by irradiation for 60 seconds at high irradiance (1500 mW/cm²). Higher irradiance led to significantly higher temperatures, regardless of exposure duration and attenuating materials ($p<0.05$). Longer exposure durations caused higher temperatures at medium (1000 mW/cm²) and high (1500 mW/cm²) irradiances ($p<0.05$), but exposure duration did not affect temperature when materials were irradiated with low irradiance (500 mW/cm²; $p>0.05$).

Groups irradiated for 60 seconds had significantly higher DCs compared with groups irradiated for 20 seconds, regardless of the irradiance and attenuating material ($p<0.05$). In the control groups, statistical differences in DC were as follows: C-L20 < C-M20 = C-H20; C-L60 = C-M60 = C-H60 ($p<0.05$). In the lithium disilicate groups, statistical differences

in DC were as follows: L-L20 < L-M20 = L-H20 = L-L60 < L-M60 = L-H60 ($p<0.05$). In the zirconia groups, statistical differences in DC were as follows: Z-L20 < Z-M20 = Z-H20 = Z-L60 < Z-M60 = Z-H60 ($p<0.05$).

Irradiating with low irradiance for 20 seconds caused statistically different DCs in the attenuating materials; higher DCs occurred in the following order: C-L20 > L-L20 > Z-L20 ($p<0.05$). The control group (C-L60) showed higher DCs compared with other attenuating material groups (L-L60 and Z-L60) when they were irradiated with low-intensity irradiance for 60 seconds ($p<0.05$). Irradiating with medium or high irradiance for 60 seconds caused no differences between the attenuating materials ($p>0.05$).

DISCUSSION

The present study aimed to determine the effect of LED light-curing methods on dual-cure resin cement under different restorative materials. To determine the effects of irradiance and exposure duration to DC and temperature, a single LCU was used and irradiance was manually calibrated with a radiometer. Two-way ANOVA showed that irradiance and exposure duration affected both DCs and temperature changes and the null hypothesis was rejected. The results of this study show that irradiating with higher than 1000 mW/cm² irradiance for 60 seconds can achieve sufficient DCs despite the attenuating effect of ceramics with 1-mm thickness. Interestingly, DCs and temperatures were affected by irradiance and exposure duration in different ways. All groups showed higher DCs with increased exposure times, but there were no statistically significant

Table 3: Results of the Two-Way Analysis of Variance for Each Attenuating Material

Independent Variable	Significance	
	Degree of Conversion	Temperature
Irradiance	$p<0.001$	$p<0.001$
Exposure duration	$p<0.001$	$p<0.001$

Table 4: Radiant Exposure of Each Group and the Means (SD) and Statistical Differences Between Groups for Temperature^a

		Low Irradiance (500 mW/cm ²)			Medium Irradiance (1000 mW/cm ²)			High Irradiance (1500 mW/cm ²)		
	Group	Radiant Exposure, mJ/cm ²	Temperature, °C	Group	Radiant Exposure, mJ/cm ²	Temperature, °C	Group	Radiant Exposure, mJ/cm ²	Temperature, °C	
Control										
20 s	C-L20	9884	28.4 (0.3) ^{A,a}	C-M20	29,724	32,6 (0.8) ^{A,b}	C-H20	29,724	39.3 (2.1) ^{A, c}	
60 s	C-L60	29,652	28.4 (0.6) ^{A,a}	C-M60	89,172	39.3 (2.3) ^{B,b}	C-H60	89,172	47.1 (2.3) ^{B, c}	
Lithium disilicate										
20 s	L-L20	2710	27.8 (0.7) ^{A,a}	L-M20	9694	29.5 (0.3) ^{A,b}	L-H20	9694	31.4 (0.3) ^{A, c}	
60 s	L-L60	8130	28.2 (1.1) ^{A,a}	L-M60	29,082	33.2 (1.4) ^{B,b}	L-H60	29,082	39.2 (1.2) ^{B, c}	
Zirconia										
20 s	Z-L20	1850	27.3 (1.1) ^{A,a}	Z-M20	6446	29.3 (0.2) ^{A,b}	Z-H20	6446	34.5(1.7) ^{A, c}	
60 s	Z-L60	5550	28.2 (1.3) ^{A,a}	Z-M60	19,338	34.2 (3.0) ^{B,b}	Z-H60	19,338	42.2(2.7) ^{B, c}	

^a Temperature was measured at room temperature (24°C). Similar superscript letters (uppercase for columns and lowercase for rows) indicate homogenous subsets among the experimental groups (p>0.05).

differences between the groups irradiated with 1000 mW/cm² and 1500 mW/cm². Thus, 1000 mW/cm² of irradiance was sufficient to achieve DCs through ceramics if sufficient exposure durations were provided. In conclusion, sufficient exposure duration was prerequisite to achieve a high DC. On the other hand, longer exposure durations, with 500 mW/cm² of irradiance, did not cause increases in temperature, although higher irradiance caused higher temperature changes. Thus, exposure durations, at low irradiances, did not affect temperatures, and irradiance was more critical to temperature changes than exposure duration.

The concept of the law of reciprocity, that high-irradiance exposure for short durations and low-irradiance exposure for long durations cause similar polymerization to resin composites,²⁰ is controversial.²¹⁻²³ In this study, similar to previous experiments,^{24,25} exposure duration was more critical than irradiance. The results, that C-L60 showed a

statistically higher DC compared with C-H20, despite both groups providing the same radiant exposure, confirm this. Total radiant exposure is indicated by the number of photons reaching the light beam area,¹² and excessive photons delivered in a short time is not necessary to activate photo-initiators and free radicals.^{26,27} Fast saturation of a photo-initiating system restricts the available monomers causing lower DCs and increases the frequency of crosslinking and short polymeric chains, causing the lower mechanical properties of resin composites.²⁸ Restorative materials attenuate irradiance in certain proportions (lithium disilicate: about 70%; zirconia: about 80%); for example, only 322 mW/cm² of irradiance reached the specimens through zirconia when they were exposed to an irradiance of 1500 mW/cm². Therefore, there is a limit to the amount of photons that may be delivered to resin composites through ceramics, despite increases in irradiance,

Table 5: Mean (SD) Degrees of Conversion and Statistical Differences Between the Groups^a

		Low Irradiance	Medium Irradiance	High Irradiance
Control				
20 s		58.25 (1.35) ^{A,c}	62.87 (3.62) ^{B,b}	64.35 (1.84) ^{B,a}
60 s		70.07 (0.83) ^{C,b}	70.80 (1.29) ^{C,a}	69.88 (0.51) ^{C,a}
Lithium disilicate				
20 s		54.52 (1.93) ^{A,b}	60.71 (5.47) ^{B,ab}	62.88 (7.26) ^{B,a}
60 s		59.62 (3.43) ^{B,a}	70.87 (1.90) ^{C,a}	70.23 (2.42) ^{C,a}
Zirconia				
20 s		51.84 (2.76) ^{A,a}	56.96 (5.48) ^{B,a}	60.08 (4.48) ^{B,a}
60 s		58.68 (1.57) ^{B,a}	68.75 (3.73) ^{C,a}	69.47 (2.16) ^{C,a}

^a Similar superscript letters (uppercase: statistical differences in irradiance and exposure times; lowercase: statistical differences in attenuating materials) indicate homogenous subsets among the experimental groups (p>0.05).

because of the limited output of LCUs and the possibility of curing light burns.²⁹

Although increasing radiant exposure is necessary to compensate for the attenuating effect of restorative materials, higher radiant exposure may cause temperature rises.³⁰ In this study, exposure times did not statistically affect temperatures with low irradiances (500 mW/cm²), but longer exposure durations caused higher temperatures when materials were irradiated with higher than medium irradiance (1000 and 1500 mW/cm²). Especially, irradiation with 1500 mW/cm² for 60 seconds to zirconia caused temperature rises of 18.2°C. The thermal stimulus generated by LCU can cause the thermal response of the dentin-pulp complex.³¹ The physiological change of pulp by external heat depends on the intensity and duration of the thermal stimulus to pulp, the fluid motion of the dentinal tubule, the thickness of enamel and dentin, and intervening substrates between heat and tooth, including composite resins. Intrapulpal temperature rise of more than 5.5°C can cause irreversible pulp damage,³² and thermal stimulus for 60 seconds with increasing temperature of 5°C is critical for the vital pulp.³³ The fluid of the dentinal tubule transmits the sensation to pulp, and the intimate stimuli to the dentinal tubule can cause more physiological changes of the dentin-pulp complex.³⁴ As the thermal diffusivity of dentin (1.87×10^{-3} cm²/s) and enamel (4.79×10^{-3} cm²/s) is relatively low,³⁵ the intrapulpal temperature change by external heat is less to the tooth that has thicker dentin and enamel walls. Similarly, composite resin acts as an insulator rather than heat container,³⁰ and thinner composite resins cause higher temperature rises of pulp.^{16,36} A previous *in vivo* study showed that LED irradiation with 1200 mW/cm² for 60 seconds on a human tooth with a 3-mm-thick enamel-dentin wall caused a temperature rise over the threshold of 5.5°C in the pulp chamber.³⁷ The results of this study represent the temperature rise of resin cements during cementation of a ceramic crown. In clinical situations, a relatively thin dentin wall remains, dentinal tubules may be exposed, and a comparatively thin resin composite intervenes between the restoration and tooth. Therefore, the results of this study show that lengthy light curing with high irradiance for the polymerization of resin cement under ceramic restorations may cause harmful effects to the pulp. To avoid unfavorable effects, alternative curing techniques, such as providing interrupted light exposure (dividing total exposure duration) is

recommended; for example, three periods of interrupted irradiation at 1500 mW/cm² for 20 seconds to zirconia (Z-H20) can decrease temperatures by 7.7°C compared with irradiation at 1500 mW/cm² for 60 seconds to zirconia (Z-H60).

In this study, a single LED light LCU was used, and the output of the LCU was manually adjusted with a radiometer. Using a single light source and LCU is important to draw concise conclusions for evaluating the effects of light intensity. Despite similar irradiances among different LCUs, polymerization kinetics can be affected by the type of LCU.^{12,38} Irradiating kinetics are affected by the design of the LCU; for example, tip diameter affects light dispersion distances from the light tip,³⁸ and collimating irradiance with distance was different due to commercial differences in LCUs. Moreover, the time to peak output irradiance or homogeneity of irradiance also can differ according to the type of LCU.¹² In addition, the radiant to active light beam area is less than the LCU tip area,^{39,40} and measuring the irradiance of light simulating the experimental situation may prevent errors. In this study, all experimental procedures of light exposure were performed with Teflon molds, providing determined distances from curing unit tips to objects. A control group was necessary in this study to provide comparable data and to evaluate the effects of ceramic materials on the resin cement. Measuring temperature on the surface of objects and in the air is technically different; a different thermometer should be used, and measuring temperature in the air is much more sensitive. To use the same method for measuring temperature, transparent acryl was used as a control group instead of direct irradiation, although it is not used in clinical situations.

However, the limitations of this study were the differences in experimental conditions compared with the patients' intraoral conditions. Flat attenuating materials were used, and the irradiation angles and distances in this study may be different from those through anatomic fixed prostheses. Therefore, laboratory research with anatomic crowns or clinical research may be necessary to verify these results. Although irradiating with 500 mW/cm² achieved lower DC than the control group and did not affect the temperature in this study design, providing longer exposure durations can derive different results as they provide more energy. More studies with various exposure times may be beneficial to verify the results.

CONCLUSIONS

1. Providing longer exposure durations is more efficient to achieve higher DC compared to irradiating with higher irradiances.
2. While longer exposure durations with 500 mW/cm² of irradiance do not cause temperature rises, an irradiance with greater than 1000 mW/cm² for longer exposure yields higher temperature changes, which can cause clinically unfavorable results.
3. LED irradiance with 1000 mW/cm² for 60 seconds achieves appropriate DC of dual-cure resin cement underlying lithium disilicate or zirconia crown with 1-mm thickness, but providing a pause between irradiances may be necessary to prevent temperature rise.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the Korea University Anam Hospital.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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