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Laboratory Research

Efficiency of Dual-Cured Resin Cement Polymerization Induced by High-Intensity LED Curing Units Through Ceramic Material

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Clinical Relevance

When polymerizing dual-cured resin cements through all-ceramic restorations, high-intensity light-emitting diode curing units are recommended because they can compensate for the reduction in the intensity of the light reaching the cement and achieve adequate polymerization with a shorter irradiation period.

SUMMARY

Objective: This study aimed to evaluate the ability of high-intensity light-emitting diode (LED) and other curing units to cure dual-cured resin cement through ceramic material.

Methods: A halogen curing unit (Jetlite 3000, Morita), a second-generation LED curing unit (Demi, Kerr), and two high-intensity LED curing units (PenCure 2000, Morita; Valo, Ultradent) were tested. Feldspathic ceramic plates (VITABLOCS Mark II, A3; Vita Zahnfabrik) with thicknesses of 1.0, 2.0, and 3.0 mm were prepared. Dual-cured resin cement samples (Clearfil Esthetic Cement, Kuraray Noritake Dental) were irradiated directly or through one of the ceramic plates for different periods (5, 10, 15, or 20 seconds for the high-intensity LED units and 20, 40, 60, or 80 seconds for the others). The Knoop hardness test was used to

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determine the level of photopolymerization that had been induced in the resin cement. Data were analyzed by one-way analysis of variance and Dunnett's post-hoc test to identify test-control (maximum irradiation without a ceramic plate) differences for each curing unit ($p<0.05$).

Results: For all curing units, the curing conditions had a statistically significant effect on the Knoop hardness numbers (KHNs) of the irradiated cement samples ($p<0.001$). In general, the KHN decreased with increasing plate thickness and increased as the irradiation period was extended. Jetlite 3000 achieved control-level KHN values only when the plate thickness was 1.0 mm. At a plate thickness ≥2.0 mm, the LED units (except for PenCure 2000 at 3.0 mm) were able to achieve control-level KHN values when the irradiation time was extended. At a plate thickness of 3.0 mm, irradiation for 20 seconds with the Valo or for 80 seconds with the Demi were the only methods that produced KHN values equivalent to those produced by direct irradiation.

Conclusion: Regardless of the type of curing unit used, indirect irradiation of dual-cured resin cement through a ceramic plate resulted in decreased KHN values compared with direct irradiation. When the irradiation period was extended, only the LED units were able to achieve similar KHN values to those observed under direct irradiation in the presence of plates ≥2.0-mm thick. High-intensity LED units require a shorter irradiation period than halogen and second-generation LED curing units to obtain KHN values similar to those observed during direct irradiation.

INTRODUCTION

Resin cements are commonly used in all-ceramic restorations because of their low solubility, high bond strength, and superior mechanical properties, all of which contribute to the reinforcement of ceramic restorations.[1-4] Adequate curing of resin cement during ceramic restoration is a very important factor in obtaining adequate physical and biological properties.[5,6] When the restoration is thicker than 1.5-2.0 mm and/or its opacity inhibits light transmission, the use of dual-cured resin cements is advocated.[7-9] Dual-cured resin cements have been developed in an attempt to combine the properties of chemical-cured and light-cured materials, thereby providing adequate polymerization in deeper and/or shadowed regions and a shorter setting time.[3] It is also important that dual-cured resin cements are irradiated sufficiently at the time of their application because occlusal adjustment and polishing, during which the restoration is subjected to mechanical stress, are usually performed immediately after the cementation.

During light-curing through a ceramic material, the irradiance of the transmitted light decreases as the thickness of the ceramic material increases.[10] Thus, the actual irradiance reaching the dual-cured resin cement underneath the ceramic material is reduced to a certain extent, depending on the irradiance of the light-curing unit and the thickness, type, and opacity of the ceramic material.[9,11] Reductions in irradiance could adversely affect the physicochemical properties of dual-cured resin cements because they can lead to reductions in cement polymerization, which might not be completely counteracted by the chemical-curing abilities of the resins.[12]

Halogen lights are the most frequently used light sources for inducing polymerization in resin-based dental materials.[13] They emit a continuous spectrum of light, though only a small part of the spectrum is useful for curing. Other wavelengths are filtered out to prevent undesirable side effects.[14] Even after the filtration, however, halogen lights deliver several unwanted wavelengths of light that are highly absorbed by dental materials, resulting in the heating of the tooth and resin during the curing process.[15] Other drawbacks include a decline in irradiance over time, a limited curing depth, and a need for a longer exposure period.[13]

Recently developed light-emitting diode (LED) lights offer a much narrower emission spectrum (a bandwidth of about 20 nm centered on 470 nm),[16] and the spectrum falls closely within the absorption range of camphorquinone, the most frequently used photoinitiator in resin composites.[16] In general, LED lights have the following advantages: extended lifetimes of more than 10,000 hours, little light output degradation over time, and resistance to shock and vibration.[16] Today, most light-curing units use single-peak blue LEDs, which usually have higher irradiances than conventional halogen lights.[13,16] The manufacturers of these high-intensity LED curing units claim that they can reach irradiances of up to 2,000-3,200 mW/cm^2 depending on the chosen mode.[13] More recently, high-intensity third-generation LED curing units have become commercially available. These units are equipped with multiple diodes (violet/blue diodes, polywave)

Table 1: *Materials Used in the Study*

Materials	Manufacturer	Shade	Lot No.	Composition[a]
VITABLOCS Mark II	Vita Zahnfabrik	A2	26630	Fine particle feldspar ceramic: SiO_2 (56-64), Al_2O_3 (20-23), Na_2O (6-9), K_2O (6-8), CaO (0.3-0.6), TiO_2 (0.0-0.1)
Clearfil Esthetic Cement	Kuraray Noritake Dental	A2	016ABA	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, colloidal silica, accelerator, others
				Paste B: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated barium glass filler, colloidal silica, benzoyl peroxide, dl-camphorquinone, initiators, pigments, others

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate.
[a] Weight percentage is in parentheses.

and, thus, are effective not only at curing cements containing camphorquinone but also at curing those containing its alternatives.[13] In addition, many of the newer high-power LED units are said to require shorter irradiation periods;[13,16] however, the ability of these devices to polymerize dual-cured resin cement through ceramic restorations has not been fully investigated.

Thus, the purpose of this study was to evaluate (using microhardness measurements) the ability of high-intensity LEDs, second-generation LEDs, and halogen curing units to induce polymerization in dual-cured resin cement through ceramic plates.

METHODS AND MATERIALS

A block of feldspathic glass ceramic material (VITABLOCS Mark II, shade A2, size I12, lot 26630, Vita Zahnfabrik, Bad Säckingen, Germany) was used to produce the ceramic plates (Table 1). From the block of ceramic material, plates 12.0 mm long and 10.0 mm wide were cut using a low-speed diamond saw (Micro-cutter 201, Maruto, Tokyo, Japan). Both sides of the ceramic plates were then polished under water cooling with a polishing device (Struers A/S, Marumoto Struers, K.K., Denmark) and silicon carbide papers (FEPA P, Marumoto Struers, K.K., Denmark) of descending grit size (#320 to #1200). During the aforementioned polishing, the thickness of the ceramic plates was monitored with a digital micrometer (Mitutoyo PK-1012, Mitutoyo, Kanagawa, Japan), and the plates were polished until the following thicknesses were reached: 1.0, 2.0, and 3.0 mm.

Four different light-curing units were tested: a conventional halogen unit (Jetlite 3000, Morita, Tokyo, Japan), a second-generation LED curing unit (Demi, Kerr, Orange, CA, USA), and two high-intensity LED curing units (PenCure 2000, Morita; Valo, Ultradent, South Jordan, UT, USA) were tested (Table 2). All of the curing units were used in maximum power mode. The emission spectra and the irradiance of each unit were measured with a laboratory-grade spectroradiometer (USR-45DA-14, Ushio, Tokyo, Japan),[17] either without ceramic plates (at a thickness of 0 mm) as a control or through a ceramic plate with a thickness of 1.0, 2.0, or 3.0 mm. The plate was placed between the tip of the curing unit and the aperture of the spectroradiometer, and the resultant light output was detected and recorded using the analytical software supplied with the device. All the results were expressed as the means of five measurements, and distribution of the irradiances (in mW/cm^2) was calculated in 380–525 nm ranges using the analytical software.

A dual-cured resin cement (Clearfil Esthetic Cement, clear, lot 016ABA, Kuraray Noritake Dental, Tokyo, Japan; Table 1) was used in this experiment. To prepare the resin cement specimens, a clear glass slab was used as a supporting surface on top of a black background that decreased the

Table 2: *Curing Units Used in the Study*

Curing Unit	Manufacturer	Type	Serial No.	Light Intensity,[a] mW/cm^2
Jetlite 3000	Morita	QTH	2010889	>400
Demi	Kerr	LED	752020284	1100
PenCure 2000	Morita	LED	2B0085	2000
Valo	Ultradent	LED	V18955	3200

Abbreviatiions: LED, light-emitting diode; QTH, quartz-tungsten-halogen.
[a] According to the manufacturer.

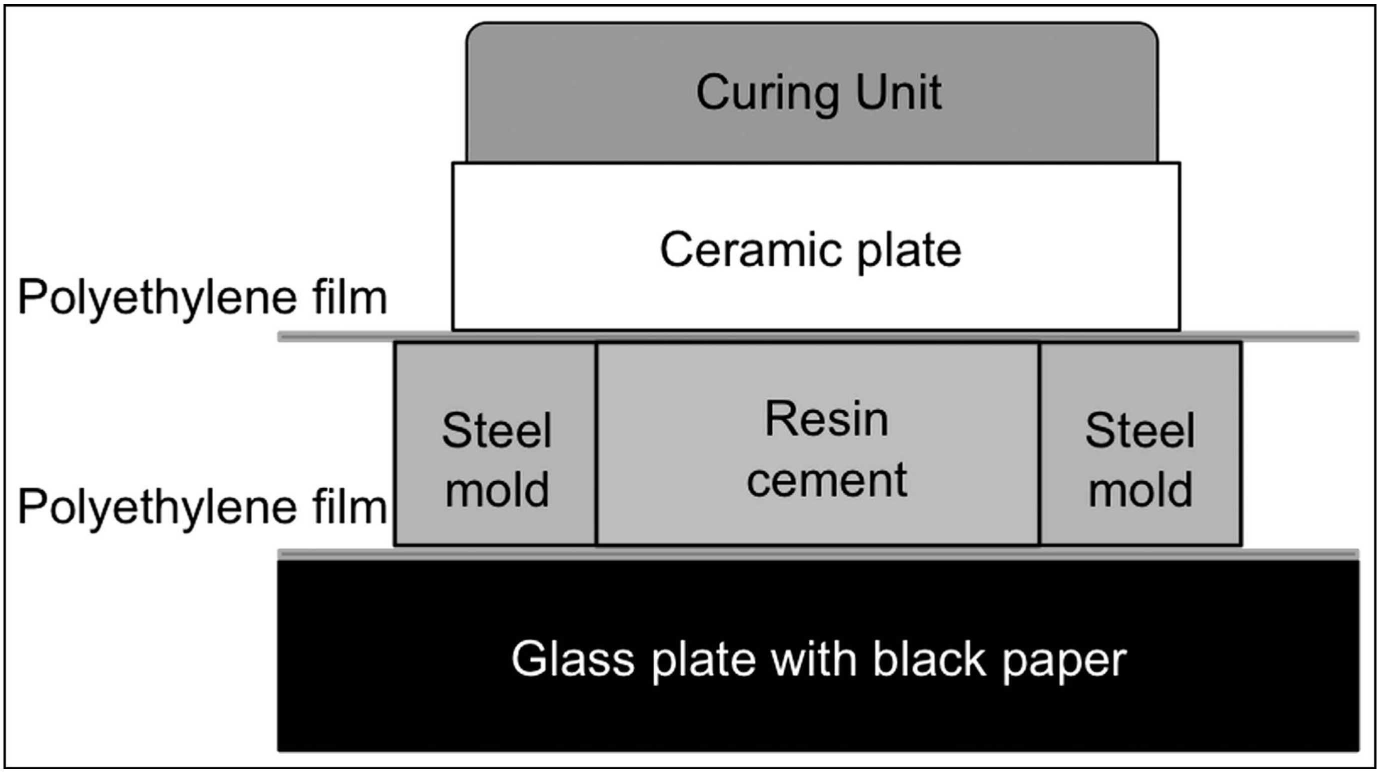

Figure 1. *Preparation of resin cement specimens.*

reflectivity of the underlying surface. A stainless steel mold (internal dimension: 12 mm wide, 2 mm deep, and 2 mm high) was placed on the glass slab. The dual-cured resin cement was mixed using mixing tips according to the manufacturer's instructions and used to fill the mold after a small amount of the mixed paste had been discarded to ensure that equal amounts of the two pastes were pushed out of the mold. A polyethylene film (GC, Tokyo, Japan) was then placed on the top and bottom of the resin cement to isolate it from the ceramic plate and glass slab.

Light-curing was performed either through the polyethylene film (as a control) or through a ceramic plate with a thickness of 1.0, 2.0, or 3.0 mm placed on top of the polyethylene film, whilst the tip of the curing unit was in contact with the ceramic plate (Figure 1). For the Jetlite 3000 and Demi units, the curing time was set at 20, 40, 60, or 80 seconds (n=5, each). The latter three time periods were achieved by performing the appropriate number of 20-second irradiation periods. For the PenCure 2000 and Valo, in which the maximum irradiation period was set to 3 seconds when the device was in maximum power mode to prevent heat generation, the curing time was set to 5 (3 + 2), 10 ([3 $\times$ 2] + [2 $\times$ 2]), 15 (3 $\times$ 5), or 20 (3 $\times$ 6 + 2) seconds (n=5, each). The minimum irradiation period was set according to the recommendation of the manufacturer of the cement used. The maximum irradiation period (80 seconds for the Jetlite 3000 and Demi and 20 seconds for the PenCure 2000 and Valo) was only used when the 3.0-mm-thick ceramic plates were used. As a result, a total of 260 resin cement specimens were evaluated. All of the specimens were stored in lightproof containers in distilled water at 37°C for 24 hours.

A microhardness tester (MVK-E, Akashi Co Ltd, Tokyo, Japan) was used to produce microindentations on the surface of the resin cement specimens. The microindentations were then measured to determine the microhardness of the resin cement specimens, which indicated the extent of the polymerization that had been induced in the resin cement. Knoop hardness measurements were performed under a load of 50g for 15 seconds. For each examined cement sample, three indentations were made in a longitudinal section of the sample taken from a region located 100 μm from the sample's surface (Figure 2). In addition, the distance of the indentations from the specimen's center was set at $\geq$1.0 mm. The mean of the five measurements was recorded as the Knoop hardness number (KHN) (Figure 2).

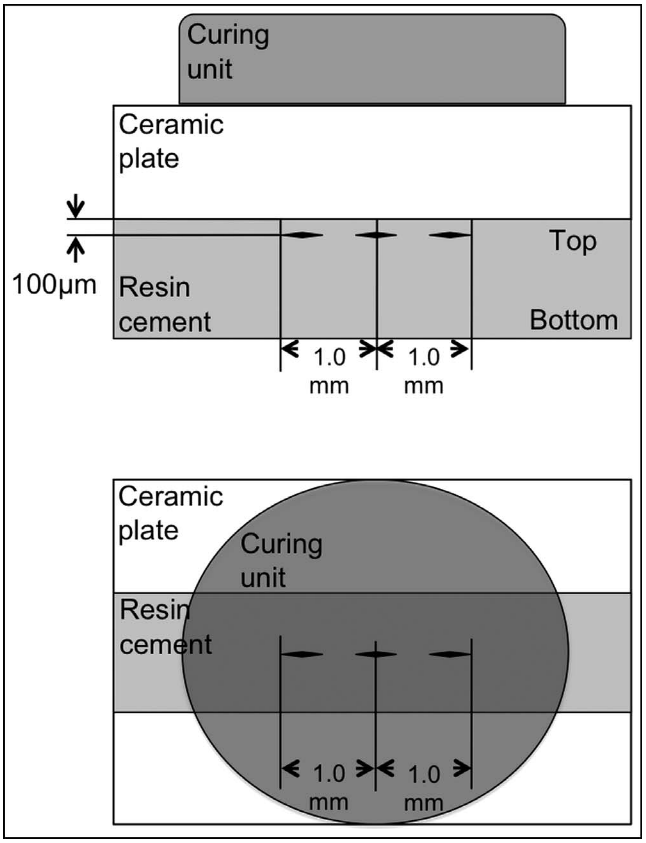

Figure 2. *Position of Knoop hardness measurement (top: side view; bottom: top view).*

The KHN data for the four curing units were analyzed by one-way analysis of variance using the SPSS Base 10.0 statistical software package (SPSS Inc, Tokyo, Japan). The curing condition (13 levels) was treated as an independent variable. Multiple comparisons analysis was performed with Dunnett's test to identify test-control (maximum irradiation without a ceramic plate) differences. All statistical tests were performed using a significance level of α=0.05.

RESULTS

Figure 3 shows spectral distributions obtained for each curing unit in the absence or presence of ceramic plates with different thicknesses. Jetlite 3000 showed broad spectra with a mild peak around 480 nm. Both Demi and Pencure 2000 had distinct single-peak spectra in the wavelength range of 440 to 460 nm. Valo showed dual-peak spectra around 400 nm and between 440 and 470 nm. Almost all emissions were detected in the wavelength range between 380 and 525 nm. Light transmittance values calculated by integrating the irradiance values in the 380–525 nm ranges are shown in Table 3. In the absence of a ceramic plate, the irradiance values exhibited the following order: Jetlite 3000 < Demi < PenCure 2000 < Valo. The overlaid ceramic plates severely reduced the irradiance of the transmitted light; that is, the irradiance values were reduced to approximately 1/3, 1/5, and 1/10 of the relevant positive-control value by the plates with a thickness of 1.0, 2.0, and 3.0 mm, respectively.

The KHN values of the resin cement samples after polymerization had been performed with each curing unit are shown in Table 4. One-way analysis of variance demonstrated that the curing condition had a statistically significant effect on microhardness, regardless of the curing unit used (p<0.001). In general, the KHN values decreased as the plate thickness increased and increased as the curing period was extended. When the Jetlite 3000 was used, control-level KHN values were only obtained at a plate thickness of 1.0 mm. The specimens cured with the Demi exhibited significantly reduced KHN values at a plate thickness ≥2.0 mm and required 80 seconds to produce control-level KHN values at a plate thickness of 3.0 mm. When irradiation was performed with the PenCure 2000, significantly decreased KHN values were observed after 5 seconds' irradiation at all plate thicknesses, and when a plate thickness of 3.0 mm was used, the KHN values were still significantly decreased even after 20 seconds' irradiation. The specimens irradiated with the Valo also showed significantly decreased KHN values after 5 seconds' irradiation at all plate thicknesses. At a plate thickness of 3.0 mm, the Valo required 20 seconds to polymerize the cement to a sufficient extent to produce control-level KHN values.

DISCUSSION

The mechanical properties of resinous materials are dependent on the degree of conversion of the resin matrix, which can be assessed by several methods.[18] Fourier transform infrared spectrometry (FT-IR) and laser Raman spectroscopy, which directly measure the degree of conversion, are regarded as sensitive methods; however, they are time consuming and expensive.[19] Thus, indirect methods, such as assessments of the depth of cure and microhardness testing,[20] are used as practical techniques for assessing the effects of different exposure conditions.[21,22] In this study, we used microhardness testing because it is the most commonly used technique for measuring the degree of conversion of resin cements.[23] It has been reported that surface hardness measurements

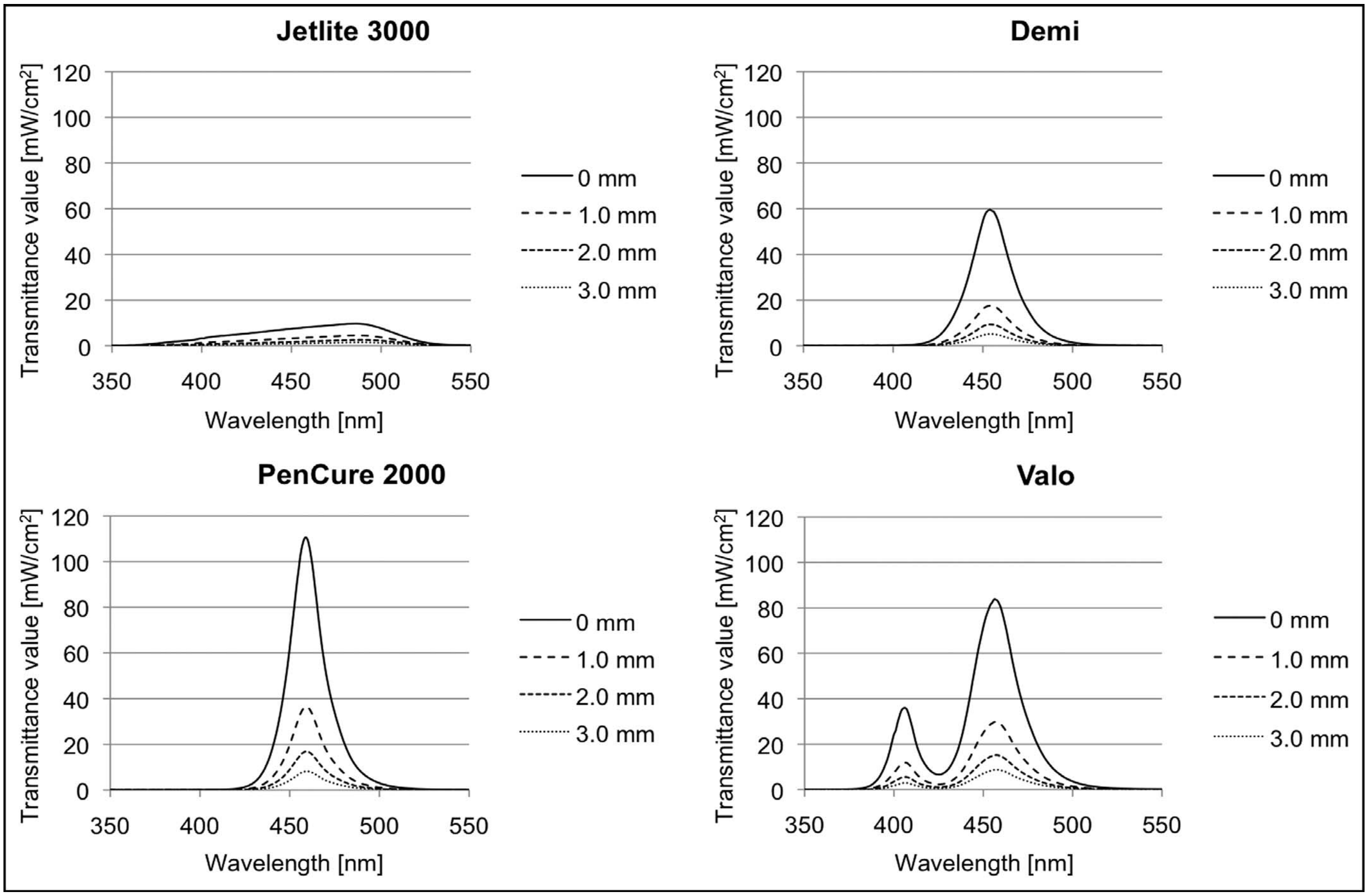

Figure 3. *Spectral distribution of light transmittance values (mW/cm^{2}) of four light-curing units through various thicknesses of ceramic plates.*

produce similar results with FT-IR spectroscopy and that hardness testing is more able to detect small changes in the degree of conversion than FT-IR after the network has been cross-linked.[24]

In this study, we measured the irradiance of the four curing units with a laboratory-grade spectroradiometer.[17] The results demonstrated that light intensity values obtained were equal or higher than those stated by the manufacturer, whereas they decreased as the plate thickness increased (Table 3; Figure 3). All the units showed a peak around 470 nm and thus are compatible to camphorquinone. On the other hand, lights emitted from dental curing units are known to be inhomogeneous,[25,26] which could have some influence on the present results. This point requires further investigation, although one study has reported that LED units emit more homogenous light, and thus yield more uniform distribution of surface hardness (KHN) of a resin composite, than halogen units.[25]

Ceramic thickness is regarded as a critical factor determining the amount of light transmission through all-ceramic restorations,[10] although other factors, such as crystalline structure and light refractive index, also have an effect.[27,28] The results of the present study confirm that ceramic thickness has a strong effect on the irradiance of transmitted light;[10] that is, ceramic disks 2.0 and 3.0 mm thick reduced irradiance by approximately 80% and $\geq$90%, respectively, regardless of the light-curing unit used (Table 2). Similar results were obtained in a previous study, in which leucite-reinforced glass ceramic (IPS Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) and lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) caused reductions in irradiance of 81.9%-83.3% and 85.8%-86.5%, respectively, at a thickness of 1.5 mm and reductions of more than 95% at a thickness of 3.0 mm.[11]

In a clinical setting, a large proportion of all-ceramic crowns are 1.0-3.0 mm thick.[29,30] Furthermore, posterior inlay/onlay restorations have to be at least 1.5 to 2.0 mm thick, and the thickness of the ceramic can increase to 3.0 mm in the proximal box.[31] Thus, a thickness of 3.0 mm is clinically

Table 3: *Integrated Light Transmittance Values (Standard Deviation) (mW/cm²) of Four Curing Units Through Various Thicknesses of Ceramic Plates*

Curing Unit	Ceramic Thickness			
	0 mm	1.0 mm	2.0 mm	3.0 mm
Jetlite 3000	860 (86)	384 (9)	210 (8)	116 (1)
Demi	1812 (108)	538 (55)	284 (23)	157 (13)
PenCure 2000	2790 (79)	927 (52)	431 (12)	210 (10)
Valo	3337 (88)	1181 (15)	595 (16)	337 (13)

relevant, and clinicians should consider the negative effects of light attenuation on the polymerization of dual-cured resin cements.[8] The chemically cured properties of dual-cured resin cements might compensate to some extent for decreased light transmission;[7] however, the actions of chemical catalysts might not be sufficient to allow maximum monomer conversion.[6,32] According to our unpublished data, KHN value of non-light-cured Clearfil Esthetic Cement was 24.6 ± 1.8, and this value is comparable to that obtained for specimens cured with Jetlite 3000 (20 and 40 seconds) through a ceramic plate 3.0 mm thick (Table 4). This may indicate that, in the presence of a plate 3 mm thick, the halogen unit achieves little effect on resin polymerization unless irradiation time is extended.

The duration of irradiation is another important factor affecting the curing of dual-cured resin cement through ceramic materials.[33] This is explained by radiant exposure (J/cm²); that is, the product of irradiance (mW/cm²) and light-curing time (s),[34] which indicates that an increase in light-curing time could be used to compensate for a reduction in irradiance.[34-37] The present results demonstrated that as the thickness of the ceramic plates increased, a longer irradiation period was required to produce positive control-level (no ceramic plate) KHN values. Moreover, previous studies have reported that different combinations of irradiance values and light-curing periods that resulted in similar radiant energy levels produced similar material properties, such as surface hardness,[10,32] degree of conversion,[32-35,37] and flexural strength.[36] Taken together, these findings support the notion that longer irradiation periods are required for the adhesive luting of all-ceramic restorations in order to compensate for attenuated irradiance and to provide sufficient radiant energy for the adequate polymerization of resin cements.

To induce adequate monomer conversion in light-cured composite resin materials, 40 seconds' curing at an irradiance level of 400 mW/cm² is considered to be sufficient for direct irradiation.[38] However, it is apparent that this curing protocol should be modified according to the ceramic material (thickness, composition, shade, etc) and curing unit (light source, irradiance, etc) used. The data presented here indicate that working time can be shortened by the use of high-intensity units. However, in the presence of ceramic material ≥2.0 mm thick, the curing time required for adequate polymerization is markedly longer than the time recommended by the manufacturers, indicating that it is necessary to extend the curing time. A recent study has shown that many practitioners use halogen lights with power outputs <300 mW/cm.[2,39] Thus, the polymer-

Table 4: *Mean (Standard Deviation) Microhardness (Knoop Hardness Number) Values for the Resin Cement Samples Irradiated With Each Curing Unit for Various Curing Periods*

Ceramic Thickness	Curing Time, s	Curing Unit			
		Jetlite 3000	Demi	PenCure 2000	Valo
0 mm	20 or 5	46.3 (2.0)	48.2 (2.1)	43.4 (1.8)*	46.7 (1.3)
	40 or 10	47.8 (2.4)	47.8 (3.4)	48.0 (1.9)	47.0 (3.1)
	60 or 15 (control)	49.2 (1.3)	48.7 (1.9)	48.1 (0.9)	48.2 (1.5)
1.0 mm	20 or 5	38.0 (0.7)*	47.6 (0.9)	41.7 (1.6)*	44.7 (0.6)*
	40 or 10	46.0 (0.8)	48.3 (0.9)	47.1 (0.9)	48.4 (1.2)
	60 or 15	48.0 (1.4)	49.4 (0.7)	47.2 (0.9)	48.9 (0.9)
2.0 mm	20 or 5	37.9 (0.9)*	38.2 (1.5)*	38.3 (1.4)*	39.1 (1.4)*
	40 or 10	38.9 (2.8)*	46.6 (2.2)	43.7 (2.0)*	44.7 (3.7)
	60 or 15	44.0 (3.4)*	46.4 (1.3)	46.1 (1.9)	47.9 (2.4)
3.0 mm	20 or 5	24.3 (0.8)*	35.6 (0.8)*	32.1 (2.3)*	32.6 (1.0)*
	40 or 10	25.3 (0.8)*	38.8 (1.5)*	33.7 (1.3)*	34.6 (1.8)*
	60 or 15	28.0 (0.8)*	42.3 (1.2)*	34.7 (1.2)*	40.4 (1.4)*
	80 or 20	29.3 (1.4)*	46.6 (1.1)	35.9 (2.2)*	48.8 (0.8)

* *Significantly different compared with the control duration (ceramic thickness: 0 mm, curing periods: 60 and 15 seconds, respectively).*

ization efficiency achieved with high irradiance lights might be even more significant than was demonstrated in the present study.

The radiant energy theory also indicates that increasing irradiance by using a high-power light unit shortens the light-curing period by increasing the irradiance of the curing light, which results in more photons available per unit time for absorption;[40] thus, more photoinitiator molecules react with amines, and more free radicals are available for polymerization.[41] Therefore, recently developed high-power curing units (eg, the PenCure 2000 and Valo) are considered to achieve adequate polymerization (as indicated by positive control-level KHN values in this study) within a shorter exposure period than traditional units.

It is worth noting that in the present study the KHN values of the cement samples did not always reach the positive control level, even after the maximum curing duration (four times the minimum light-curing period). In particular, when a plate thickness of 3.0 mm was used, only the Demi and Valo achieved positive control-level KHN values after the maximum curing period (80 seconds and 20 seconds, respectively). These findings indicate that a high-intensity light is required to compensate for the light attenuation that occurs as light passes through a thick ceramic material, even when the irradiation period is extended.[19] In this regard, high-intensity LED curing units might have advantages over conventional halogen units. In the present experimental conditions, however, the PenCure 2000 did not produce positive control-level KHN values in the presence of ceramic plates 3 mm thick. This might have been because, even in high power mode, the shortened irradiation period decreased the amount of radiant energy delivered by the PenCure 2000 to a level that was insufficient for achieving adequate polymerization.

All curing units in this study can produce a painful burning sensation if the light-curing tip is inadvertently placed in contact with surrounding soft tissues when the curing time is extended over the manufacturer's recommendation. An extensive curing time could also cause cytotoxic effects.[42] Thus, care should be taken to avoid direct contact of soft tissues with high-intensity curing ights. Regarding pulpal damage, however, an *in vitro* study reported that light curing a resin cement under feldspathic ceramic caused a temperature increase of 3°C,[43] which is smaller than that necessary to damage the pulp (5.5°C).[44] This suggests a possible insulation effect of the ceramic material. Moreover, the temperature increase in a clinical situation might be smaller because of the effect of blood circulation in the pulp chamber. Nevertheless, clinicians should take the harmful effects into account, and avoid inadvertently extended irradiation of high-intensity LED lights. Air-cooling of the tooth during irradiation may be recommended.

The results of the present study indicate that high-intensity curing units are recommended for polymerizing dual-cured resin cement through all-ceramic restorations because they are able to achieve adequate resin cement polymerization within a shorter period than halogen and second-generation LED curing units. However, even high-intensity LED curing units might require an extended curing period to induce sufficient polymerization in such cements.

CONCLUSION

Regardless of the type of curing unit used, indirect irradiation through a ceramic plate decreased the KHN values of the dual-cured resin cement tested. In the presence of ceramic plates ≥2.0 mm thick, only the LED units achieved KHN values similar to those produced by direct irradiation, even when the irradiation period was extended. High-intensity LED units require a shorter irradiation period to obtain KHN values similar to those produced by direct irradiation compared with halogen and second-generation LED curing units.

Conflict of Interest

The authors 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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