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ORIGINAL ARTICLE

The effects of extended curing time and radiant energy on microhardness and temperature rise of conventional and bulk-fill resin composites

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Abstract

Objectives To investigate radiant energy, microhardness, and temperature rise in eight resin composites cured with a blue or violet-blue curing unit, using a curing protocol which exceeded manufacturer recommendations.

Materials and methods Cylindrical composite specimens ($d = 8$ mm, $h = 2$ or 4 mm, $n = 5$ per experimental group) were light-cured for 30 s. Light transmittance through specimens was recorded in real time to calculate radiant energy delivered to the specimen bottom. Vickers microhardness was used to evaluate the polymerization effectiveness at depth. Temperature rise at the bottom of the specimens was measured in real time using a T-type thermocouple.

Results Radiant energy delivered from the blue and violet-blue curing unit amounted to 19.4 and 28.6 J/cm^2, which was 19 and 13% lower than specified by the manufacturer. Radiant energies at bottom surfaces (0.2–7.5 J/cm^2) were significantly affected by material, thickness, and curing unit. All of the composites reached 80% of maximum microhardness at clinically relevant layer thicknesses. The benefit of using the higher-irradiance violet-blue curing unit was identified only in composites containing alternative photoinitiators. Temperature rise during curing ranged from 4.4 to 9.3 °C and was significantly reduced by curing with the lower-intensity blue curing unit and by increasing layer thickness.

Conclusion Curing for 30 s, which can be regarded as extended considering manufacturer specifications, produced radiant energies which are in line with the recommendations from the current scientific literature, leading to adequate curing efficiency and acceptable temperature rise.

Clinical relevance Extended curing time should be used to minimize concerns regarding undercuring of composite restorations.

Keywords Resin composites · Radiant energy · Microhardness · Temperature · Polywave · Monowave

Introduction

The development of high-irradiance light-emitting diode (LED) curing units has led to the shortening of time recommended for light-curing of composite materials for direct restorations. Although the concept of mathematical reciprocity of curing time and irradiance has been invalidated [1], manufacturers of curing units continue to advertise high irradiances coupled with short curing time in an attempt to appeal to the practitioner's demands for time savings [2]. Concerns about whether the curing time recommended by manufacturers is sufficient for an adequate polymerization at the restoration bottom have been raised due to the evidence of inferior cure produced by following manufacturer guidelines regarding layer thickness, curing unit irradiance, and curing time [3–5]. Additionally, it has been noted that curing time advertised by manufacturers is usually minimum time required for a

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sufficient cure in controlled laboratory conditions and not necessarily optimal for a more complex clinical setting [6]. The issue of suboptimal cure at the restoration bottom is clinically relevant because of its negative influence on mechanical properties and biocompatibility [7, 8]. The simplest way a practitioner can overcome these issues is by extending the curing time beyond manufacturer recommendations [9]; this approach has been shown to improve the degree of conversion and microhardness at the bottom of the composite layer [4, 8, 10], while also reducing the amount of elutable species [11, 12]. Also, extended curing time may help to mitigate the effect of spatial heterogeneity of the curing unit beam, thus producing more uniformly cured restorations [13].

On the other hand, curing with high-irradiance units for extended time raises concerns regarding possible overheating of the dental pulp [14]. Much work has been done on this topic, encompassing investigations of a wide variety of composite brands [15, 16], different radiant energies and spectral characteristics of curing units [17–20], simulations of the temperature rise within the pulpal chamber [16, 17, 19, 21, 22], and attempts to define the threshold temperature value which would indicate a potentially irreversible pulpal damage [23, 24]. Despite these efforts, the multiplicity of factors involved in the heat transfer [25] and its effect on the dental pulp makes it difficult to translate laboratory data into clinically meaningful inferences [6]. Consequently, no consensus currently exists on the reference temperature values, reached either within a composite specimen or within a pulpal chamber, which would indicate potential pulpal damage [26]. This renders laboratory investigations unable to determine the actual potential for irreversible pulpal pathoses [27]. However, laboratory studies remain useful for simulating the interplay of various factors and evaluating their influence on temperature rise in the polymerizing composite.

The present study investigated radiant energy, microhardness, and temperature rise in eight composite materials at layer thicknesses of 2 and 4 mm. A blue and a violet-blue LED curing units were activated for the longest curing "program" available (30 s), delivering radiant energies which exceeded most of the manufacturer recommendations. Null hypotheses assumed no effect of composite material, curing unit, and layer thickness on (I) radiant energy, (II) Vickers microhardness (MH), and (III) temperature rise at the bottom of the composite layer. Further null hypotheses assumed (IV) no correlation between radiant energy and MH and (V) no correlation between radiant energy and temperature rise.

Materials and methods

Composite materials and curing units

Four conventional and four bulk-fill resin composites were investigated. Detailed composition and manufacturer recommendations for curing time are shown in Table 1. To evaluate the effect of pigment additives used for the adjustment of shade, composites Tetric EvoCeram (TEC) and Tetric EvoCeram Bulk Fill (TECBF) were investigated in two different shades. These composite types also contained additional photoinitiators besides camphorquinone/amine. Composite specimens were light-cured using either a blue (Bluephase Style M8, Ivoclar Vivadent, Schaan, Liechtenstein) or a violet-blue curing unit (Bluephase Style, Ivoclar Vivadent). The blue curing unit is also known by the name "Monowave" (MW), while the violet-blue is referred to as "Polywave" (PW). Emission spectra and irradiances of the curing units are shown in Fig. 1.

Radiant energy

To determine the radiant energy delivered to the bottom of 2- and 4-mm-thick composite layers, the light intensity which passed through the mold aperture was measured by integrating sphere (IS, Gigahertz Optik GmbH, Puchheim, Germany), and real-time monitoring of the light transmittance during light-curing of composite specimens was performed using a charge-coupled device array fiber spectrometer (HR4000, Ocean Optics, Dunedin, FL, USA). Composite specimens were prepared in black cylindrical Teflon molds ($d = 8$ mm, $h = 2$ or 4 mm), covered with a polyethylene terephthalate (PET) film, and light-cured for 30 s. The light transmitted through the specimen during the light-curing was collected by a lens and led to the spectrometer which recorded irradiance as a function of time at the data collection rate of 20 s^{-1} using custom-made software. Radiant energy (J/cm^2) was calculated by integrating the area under the irradiance vs. time curve.

Microhardness

Composite specimens of the same geometry as described for the radiant energy measurements ($d = 8$ mm, $h = 2$ or 4 mm) were prepared by pressing composite pastes in black Teflon molds, covering mold apertures with a PET film and light-curing for 30 s. The specimens were then stored in dark at 37 ± 1 °C for 24 h in order to complete the post-cure reaction [28]. MH was evaluated at specimen surface (designated as 0 mm) and at the bottom of 2- or 4-mm-thick specimens. Prior to performing indentations, specimen surfaces were wet ground using a silicon carbide paper in a sequence of decreasing roughness (grits 600, 800, and 1200) for 1 min per each roughness. This was followed by a fine polishing using aluminum oxide powder mixed with distilled water in a sequence of particle sizes of 1.0, 0.3, and 0.005 μm (1 min each). The specimens were then thoroughly rinsed with distilled water and blotted with cellulose pads. Five indentations per specimen were performed using a Vickers diamond pyramid in an

Table 1 Manufacturer information about the composite materials investigated

Material (abbreviation)	Shade LOT No. EXP	Composition	Filler load (wt%/vol%)	Manufacturer	Minimum curing time (manufacturer's recommendation)[a]
Tetric EvoCeram (TEC A2)	A2 T26729 2018-07	UDMA, Bis-GMA, Bis-EMA, ytterbium trifluoride	76/54	Ivoclar Vivadent, Schaan, Liechtenstein	20 s for < 1000 mW/cm^2
Tetric EvoCeram (TEC A3)	A3 S12959 2018-07				10 s for > 1000 mW/cm^2
Grandio (GRA)	A3 1428354 2018-01	Ba-Al-Borosilicate glass filler, SiO_2 nanofillers, Bis-GMA, TEGDMA, Bis-EMA	87/71	Voco, Cuxhaven, Germany	20 s for > 500 mW/cm^2
Gradia Direct Posterior (GDP)	A3 1406252 2017-06	UDMA, dimethacrylate, fluoroaluminosilicate glass	77/65	GC Corp., Tokyo, Japan	20 s for < 1200 mW/cm^2
					10 s for > 1200 mW/cm^2
Tetric EvoCeram Bulk Fill (TECBF-IVA)	IVA S21840 2017-05	Dimethacrylate, Ba-Al-Si glass, prepolymer filler (monomer, glass filler, and ytterbium fluoride), spherical mixed oxide	80/61 (including 17% prepolymers)	Ivoclar Vivadent, Schaan, Liechtenstein	10 s for > 1000 mW/cm^2
Tetric EvoCeram Bulk Fill (TECBF-IVB)	IVB R77065 2016-10				
Filtek Bulk Fill (FBF)	A2 N621319 2017-08	Bis-GMA, Bis-EMA, UDMA, zirconia/silica, ytterbium trifluoride	65/43	3M /ESPE, St. Paul, MN, USA	40 s for < 1000 mW/cm^2
					20 s for > 1000 mW/cm^2
X-tra fil (XF)	U 1441587 2017-04	Bis-GMA, UDMA, TEGDMA, Ba-B-Al-Si glass	86/70	Voco, Cuxhaven, Germany	10 s for > 800 mW/cm^2

[a] Manufacturers commonly recommend minimal curing time required for complete polymerization of 2 and 4 mm thick layers for conventional and bulk-fill composites respectively. The recommendation also includes the minimum irradiance of a curing unit for which the listed curing times are advised

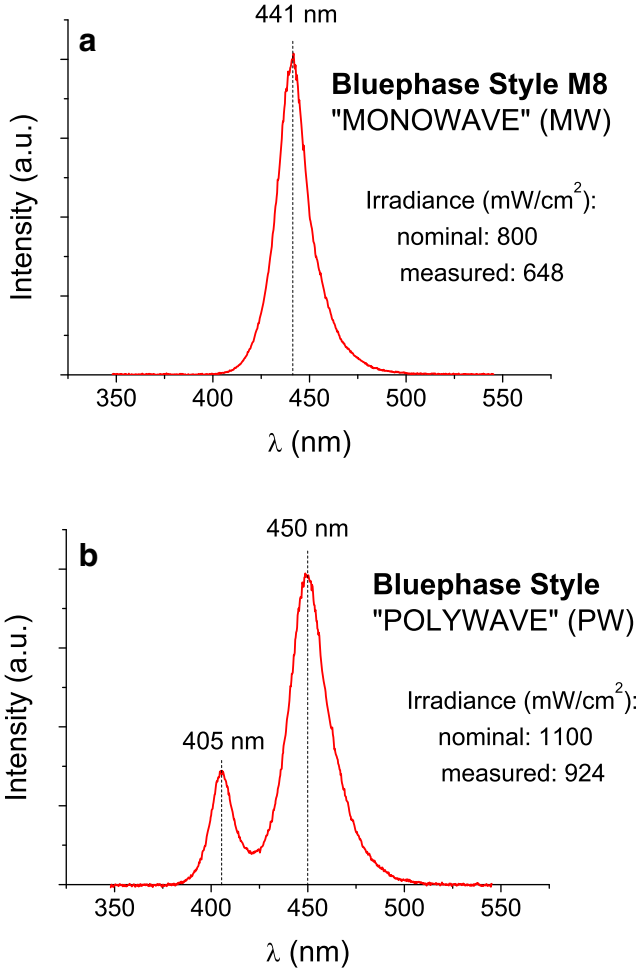

Fig. 1 Emission spectra and irradiances of the blue (**a**) and violet-blue (**b**) curing unit

MH tester (Miniload 2, Leitz, Oberkochen, Germany) with a load of 100 g and a dwell time of 10 s. All of the indentations were made at the central part of specimen surface in order to minimize the variability due to spacial inhomogeneity of the curing light. Vickers MH was calculated using the following equation: $MH = 1.8544 \times F/d^2$, where d is the indentation diagonal and $F = m \times g$ ($g = 9.81$ N/kg, m = load). As a measure of curing efficiency at depth, the following ratio was calculated:

$$\text{Microhardness (\%of maximum value)} = \frac{\text{Microhardness at a particular layer thickness}}{\text{Maximum attainable microhardness}} \times 100.$$

Temperature rise

Composite specimens ($d = 8$ mm, $h = 2$ or 4 mm) were prepared in the same black Teflon molds as used for radiant energy and MH measurements. Specimen volumes were 100.5 and 200.1 mm^3, respectively, corresponding to the

amount of material which would be applied in a single layer in large cavities. Temperature rise was monitored in real time during the light-curing of 30 s by means of a T-type thermocouple which was positioned centrally at the bottom of composite specimens [29]. The custom-made software was used to record real-time temperature changes at the data collection rate of 20 s^{-1}. To assess the heating effect of curing units alone, the temperature rise was measured at the bottom of the empty specimen compartment in the same manner.

The environmental temperature during all the tests was 22 ± 1 °C. For each test, five specimens per experimental group were prepared ($n = 5$).

Statistical analysis

Normality of distribution and homogeneity of variances were verified by Shapiro-Wilk's and Levene's test, respectively. For repeated MH measurements performed on the same specimen, sphericity was validated by Mauchly's test. Mean values of temperature rise and radiant energy were compared by a three-way ANOVA with factors *material*, *curing unit*, and *thickness*. Mean MH values were compared using a mixed model ANOVA with *material*, *curing unit*, and *thickness* as independent factors and repeated measures of $n = 5$ per specimen as dependent observations. The mixed ANOVA was performed instead of averaging the MH data obtained from the same specimen in order to account for the clustering effect of repeated measures [30]. Tukey's post hoc adjustment was used for multiple comparisons in all ANOVA models. Partial eta-squared statistics were used to describe the relative effect size for each factor. Pearson correlation analysis was used to relate radiant energy to the MH and temperature data. Statistical analysis was performed in SPSS 20 (IBM, Armonk, NY, USA) with $\alpha = 0.05$. An exception to the overall $\alpha = 0.05$ was done in the correlation analysis of radiant energy and MH, which used $\alpha = 0.1$ due to the exploratory character of the analysis.

Results

Partial eta-squared values in Table 2 reflect the practical significance of factors *material*, *curing unit*, and *thickness* on radiant energy, MH, and temperature rise. Highly significant influence of all the factors on all dependent variables was observed. Most of the binary and ternary interactions were also significant.

Radiant energy delivered to the top surface of composite specimens amounted to 19.4 J/cm^2 (MW) and 28.6 J/cm^2 (PW). Radiant energies that reached the bottom surfaces were significantly affected by material, thickness, and curing unit and varied from 0.2 to 7.5 J/cm^2 (Table 3).

Table 4 presents MH as a function of *material*, *thickness*, and *curing unit*. Conventional composites showed a significant MH decline at thicker layers, whereas the MH values of bulk-fill composites were statistically homogeneous throughout all layer thicknesses. The effect of *curing unit* was identified in TEC A2: significantly higher MH was attained with PW compared to MW at all layer thicknesses. The same was observed in TEC A3 but only at the surface (0 mm).

Figure 2 shows real-time light transmittance curves for the composites containing alternative initiators (TEC and TECBF). Higher light transmittance was identified for blue light (450 nm) compared to violet light (405 nm). Increasing the layer thickness from 2 to 4 mm diminished the blue light transmittance by 57% (TECBF-IVA) and 71% (TEC A2). For violet light, the corresponding reduction was 93% (TECBF-IVA) and 95% (TEC A2).

Table 5 shows MH values expressed as a percentage of the maximum MH value attained within each material. The dashed line marks the "80% of the maximum" threshold, which was considered an indicator of adequate cure. This threshold was surpassed by all bulk-fill composites, while in some conventional composites inferior cure was identified at 4 mm. Moreover, in two conventional composites (TEC A2 and TEC A3), the "80% of the maximum" requirement was marginally fulfilled at the layer thickness of 2 mm.

MH values measured at 2- and 4-mm-thick layers were correlated with the radiant energies delivered to the corresponding thicknesses. The Pearson correlation analysis was performed for each material separately and the results are shown in Table 6. Moderate correlations, significant at the 0.1 level, were identified for seven out of eight materials.

Maximum values of temperature rise measured at the bottom of composite specimens are summarized in Table 7. Overall, the temperature rise at the bottom of composite specimens was in the range of 4.4–9.3 °C. In the case of MW, the temperature rise measured at the bottom of the empty specimen compartment was significantly lower than the temperature rise at the bottom of composite specimens, while for PW, the temperature rise in the empty specimen compartment was statistically similar to the temperature rise at the bottom of composite specimens. Correlating temperature rise with radiant energy for all combinations of *material* × *thickness* × *curing unit* revealed a high and highly significant linear relationship (Fig. 3).

Real-time temperature curves for each combination of *material* × *thickness* × *curing unit* are shown in Fig. 4. Dashed vertical lines mark the curing time recommended by respective manufacturers, considering the nominal manufacturer-declared irradiances of the curing units. Most of the temperature rise occurred before the marked time, especially in bulk-fill composites, which reached a temperature plateau within the 10 s of curing (Fig. 4e–h). An additional

Table 2 Partial eta-squared values as estimates of the effect size for different factors and their combinations

Factor	Dependent variables					
	Radiant energy		Microhardness		Temperature rise	
	Partial η^2	p	Partial η^2	p	Partial η^2	p
Material	0.944	< 0.001	0.987	< 0.001	0.490	< 0.001
Curing unit	0.963	< 0.001	0.125	< 0.001	0.834	< 0.001
Thickness	0.972	< 0.001	0.622	< 0.001	0.844	< 0.001
Material × curing unit	0.795	< 0.001	0.227	< 0.001	0.254	< 0.001
Material × thickness	0.586	< 0.001	0.413	< 0.001	0.192	< 0.001
Curing unit × thickness	0.877	< 0.001	–	N.S.	0.325	< 0.001
Material × curing unit × thickness	0.308	< 0.001	0.142	0.007	–	N.S.

N.S. not significant

observation from the real-time temperature curves is that for PW, the "30 s" curing mode actually lasted for 31 s.

Discussion

This study investigated the influence of material composition, curing unit type, and layer thickness on radiant energy, MH, and temperature rise. Since all of the factors significantly affected the dependent variables, null hypotheses (I)–(III) were rejected.

Radiant energies required to adequately cure a composite layer are material-dependent and have been reported to vary between 6 and 36 J/cm^2 [1, 31, 32]. Recent recommendations for practitioners mentioned 24–36 J/cm^2 [1], which is comparable to incident radiant energies in the present study (19.4 J/cm^2 for MW and 28.6 J/cm^2 for PW), but exceeds most of the manufacturer recommendations, as these usually range between 10 and 20 J/cm^2 (Table 1). In this sense, curing time, which seems "extended" considering the manufacturer guidelines, was in fact adequate for producing the recommended radiant energies [1]. Radiant energy measured at the bottom of 2- or 4-mm-thick layers was up to two orders of magnitude lower than the incident radiant energy (Table 3). This was caused by light attenuation which was highly influenced by *material*, *thickness*, and *curing unit*, as indicated by partial eta-squared values in Table 2. Significant interactions of the factor *material* with the other two factors indicate that the dependence of radiant energy on layer thickness and curing unit cannot be modeled generally for all materials because each material composition behaved differently. This fact is observable in Table 3: the effect size of *thickness* and *curing unit* on radiant energy differed from material to material. The pronounced dependence of the curing light attenuation on material composition can be understood in the context of the wide variability of optimal radiant energies measured in different studies [1, 31, 32].

Manufacturers are known to overrate curing unit irradiances by specifying values which may be even two to three times higher than the real irradiance [33]. Additionally, spatial heterogeneity of the light beam leads to the discrepancy between the radiant energy received by the specimen and average radiant energy delivered through the whole aperture of the curing unit tip [34], since the inner part of the light guide often

Table 3 Radiant energy in J/cm^2 (± S.D.) delivered to the bottom of 2- and 4-mm-thick specimens

Material	2 mm MW	2 mm PW	4 mm MW	4 mm PW
TEC-A2	2.23 (0.06) a A	4.24 (0.51) b A	0.91 (0.05) c A	1.14 (0.09) c A
TEC-A3	2.25 (0.04) a A	4.02 (0.06) b A	0.81 (0.07) c AB	0.97 (0.12) c A
GRA	0.66 (0.04) a B	3.23 (0.14) b B	0.16 (0.01) c C	0.86 (0.06) d A
GDP	1.58 (0.08) a C	4.51 (0.17) b A	0.27 (0.02) c D	1.09 (0.11) d A
TECBF-IVA	2.76 (0.12) a D	7.35 (0.72) b C	1.29 (0.09) c EF	2.70 (0.05) a B
TECBF-IVB	2.90 (0.06) a E	6.84 (0.67) b CD	1.22 (0.01) c E	3.14 (0.27) a C
FBF	1.83 (0.07) a F	6.11 (0.11) b D	0.76 (0.03) c B	2.28 (0.13) d D
XF	2.90 (0.02) a E	7.55 (0.50) b C	1.33 (0.04) c F	2.94 (0.22) a BC

Same lowercase letters denote statistically similar values within a row. Same uppercase letters denote statistically similar values within a column

MW "Monowave"—Bluephase Style M8, *PW* "Polywave"—Bluephase Style, *TEC* Tetric EvoCeram, *GRA* Grandio, *GDP* Gradia Direct Posterior, *TECBF* Tetric EvoCeram Bulk Fill, *FBF* Filtek Bulk Fill, *XF* X-tra fil

Table 4 Vickers microhardness (± S.D.) at 0 mm (surface), 2 mm, and 4 mm attained by curing with the blue (MW) and violet-blue curing unit (PW)

Material	0 mm MW	0 mm PW	2 mm MW	2 mm PW	4 mm MW	4 mm PW
TEC-A2	73.0 (2.3) a AC	79.4 (2.9) b A	62.1 (4.9) c A	74.2 (3.1) a A	51.9 (3.5) d A	65.6 (4.7) c A
TEC-A3	66.2 (3.7) a AB	77.6 (4.4) b A	64.1 (3.8) a A	62.1 (4.8) a B	55.1 (4.8) c A	54.5 (2.8) c B
GRA	157.2 (3.3) a D	156.0 (4.3) a B	152.2 (3.6) a B	154.8 (3.1) a C	136.9 (4.0) b B	135.2 (4.4) b C
GDP	68.5 (2.7) a AB	64.3 (4.8) ab C	65.2 (3.5) ab A	58.9 (4.6) b B	51.2 (4.8) c A	51.6 (4.5) c B
TECBF-IVA	75.1 (4.9) ab BC	79.2 (4.9) a A	74.5 (4.5) ab C	75.8 (3.5) ab A	74.5 (2.1) ab C	72.3 (2.3) b D
TECBF-IVB	78.9 (4.9) abC	82.6 (4.4) a A	77.8 (3.8) ab C	79.8 (3.9) ab A	74.1 (4.8) b C	76.6 (4.5) b D
FBF	101.9 (4.2) ab E	107.3 (3.9) a D	100.4 (3.9) bc D	105.5 (4.1) ab D	97.4 (4.7) c D	103.4 (3.8) ab E
XF	133.7 (14.9) a F	140.2 (10.1) a E	131.9 (8.8) a E	132.1 (11.0) a E	132.0 (12.2) a B	131.6 (5.7) a C

Same lowercase letters denote statistically similar values within a row. Same uppercase letters denote statistically similar values within a column

MW "Monowave"—Bluephase Style M8, *PW* "Polywave" —Bluephase Style, *TEC* Tetric EvoCeram, *GRA* Grandio, *GDP* Gradia Direct Posterior, *TECBF* Tetric EvoCeram Bulk Fill, *FBF* Filtek Bulk Fill, *XF* X-tra fil

delivers more energy than the peripheral part [26]. Specimen diameter in the present study (8 mm) was close to the functional diameter of the light guide of 9 mm [13]; thus, the radiant energy measured through the mold opening should be similar to the averaged radiant energy of the whole light guide. However, the measured radiant energy values of 19.4 and 28.6 J/cm^2 were 19 and 13% lower than the nominal values calculated from manufacturer specifications (24 and 33 J/cm^2, respectively). Manufacturers' practice of overrating the curing unit performance [6, 9, 33, 35] has practical implications since clinicians usually learn about the curing requirements from manufacturer brochures, which may provide imprecise information.

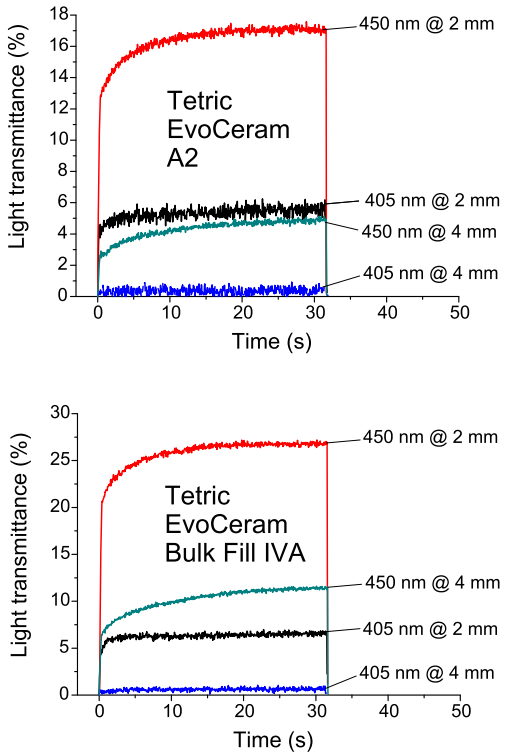

Fig. 2 Real-time light transmittance curves for two composites containing alternative photoinitiators, recorded during curing using the violet-blue curing unit. Wavelengths of 405 and 450 nm, representing two emission peaks of the violet-blue curing unit, were considered separately. The presented curves are representative for all tested shades of TEC and TECBF

The MH values were predominantly influenced by *material* (partial $\eta^2 = 0.987$), followed by *thickness* and *curing unit*, with partial η^2 of 0.622 and 0.125, respectively. Besides having the largest effect size, the factor *material* showed significant interactions with other factors, indicating that different material compositions responded differently to curing units and layer thicknesses. In six out of eight materials, no statistically significant difference in MH was identified between the curing units (Table 4). In contrast, the benefit of using PW over MW was identified in TEC A2 and TEC A3, i.e., in the composites which contained both camphorquinone/amine and Lucirin TPO [6, 36, 37]: PW produced higher MH at all layer thicknesses for TEC A2 but only at the surface for TEC A3. The declining effectiveness of combined photoinitiators at increasing thicknesses has been reported to vary among different materials: the combination of camphorquinone/amine and Lucirin TPO was effective at up to 4–5 mm in model composites [14], while in commercial composites, the benefit of violet light may be completely lost already at 1.6 mm due to high light attenuation of shorter wavelengths [36, 38] (Fig. 2). What is interesting is that the effectiveness of combined photoinitiators in basically the same material composition (TEC) differed due to the shade difference. This can be explained by different ratios of the two photoinitiators [38] or, alternatively, by the effect of pigments which may intensely absorb wavelengths responsible for photoactivation [31]. The former explanation is more likely since the radiant energies at the bottom of composite layers were similar for both shades of TEC (Table 3). It should be noted that the benefit of curing with the violet-

Table 5 Microhardness values expressed as a percentage of the maximum microhardness value attained within each material

Material	0 mm MW	0 mm PW	2 mm MW	2 mm PW	4 mm MW	4 mm PW
TEC-A2	92.0	100.0	78.2*	93.5	65.5*	82.6
TEC-A3	85.2	100.0	82.6	80.0	71.0*	70.2*
GRA	100.0	99.2	96.8	98.5	87.1	86.0
GDP	100.0	93.9	95.2	85.9	74.7*	75.2*
TECBF-IVA	94.9	100.0	94.1	95.7	94.1	91.3
TECBF-IVB	95.5	100.0	94.1	96.6	89.7	92.7
FBF	95.0	100.0	93.6	98.4	90.8	96.4
XF	95.4	100.0	94.1	94.2	94.2	93.9

Values below 80% are marked with an asterisk (*)

MW "Monowave"—Bluephase Style M8, *PW* "Polywave"—Bluephase Style, *TEC* Tetric EvoCeram, *GRA* Grandio, *GDP* Gradia Direct Posterior, *TECBF* Tetric EvoCeram Bulk Fill, *FBF* Filtek Bulk Fill, *XF* X-tra fil

blue spectrum in TEC [32] was identified also at the specimen surface, i.e., that high radiant energies received at the surface did not compensate for the differences between curing units.

Calculating the ratio of MH at a particular layer thickness and the maximum attainable MH is a common means for evaluation of polymerization efficiency at depth (Table 5). The 80% of the maximum MH has been traditionally considered to indicate an acceptable cure [4, 8, 13, 39]; this requirement is expected to be fulfilled by following manufacturer's instructions for use. Since manufacturer recommendations were exceeded in the present study, all of the composites fulfilled the "80% of the maximum" requirement at 2 mm for conventional and 4 mm for bulk-fill composites. However, the "80% of the maximum" criterion was met only marginally in the case of TEC A2 cured with MW and TEC A3 cured with either curing unit. Although this finding does not necessarily imply inferior clinical performance, it highlights the fact that significant inhomogeneity in mechanical properties through depth can exist despite the fact that the manufacturers' recommendations for curing (10 s for > 1000 mW/cm^2) were surpassed by a factor of 3.

Although conventional composites are not designed for use in layers thicker than 2 mm, a homogeneous cure throughout 4 mm was demonstrated to be attainable if sufficient radiant energy is used [14]. In our study, such a "bulk-fill" capability was identified in conventional composites Grandio (GRA) and TEC A2, which reached over 80% of the maximum MH at the bottom of 4-mm layers (Table 5): GRA was equally well cured by both curing units, whereas TEC A2 required the higher-irradiance violet-blue curing unit (PW). However, placing conventional composites at 4-mm-thick layers cannot be recommended on the basis of the MH data alone, as some other considerations, e.g., polymerization shrinkage and associated stress, need to be taken into account. Interestingly, TEC A3 did not reach the 80% of the maximum MH at 4 mm, demonstrating that the minor shade difference (A2/A3) may play an important role in limiting the polymerization efficiency at depth [38].

TECBF comprises camphorquinone/amine and a proprietary germanium-based photoinitiator system with maximum absorption in the violet range [40]. Unlike TEC, TECBF was homogeneously cured for up to 4 mm regardless of the curing unit (Table 5), thus showing no benefit of using PW over MW. The finding of a homogeneous cure in TECBF is in contradiction with several studies which reported a statistically significant MH decline at 4 mm when TECBF was cured using a blue LED curing unit at (1000 mW/cm^2 × 20 s = 20 J/cm^2) [39, 41] and a violet-blue curing unit at (1170 mW/cm^2 × 30 s = 35.1 J/cm^2) [4]. The decline in curing efficiency at depth was also identified in TECBF in a study which assessed MH immediately after light-curing [42], unlike the other mentioned studies [4, 39, 41] in which MH was measured 24 h post-cure. It remains unexplained why the decrease in curing efficiency at depth for TECBF was not observed in the present study, despite similar curing conditions to the studies in which it was identified.

Table 6 Correlation between microhardness values and radiant energy at layer thicknesses of 2 and 4 mm

Material	Pearson's *R*	*p*
TEC A2	0.786	< 0.001
TEC A3	0.634	0.003
GRA	0.637	0.003
GDP	0.395	0.085
TECBF-IVA	0.406	0.076
TECBF-IVB	0.556	0.011
FBF	0.687	0.001
XF	–	N.S.

N.S. not significant at $\alpha = 0.1$

TEC Tetric EvoCeram, *GRA* Grandio, *GDP* Gradia Direct Posterior, *TECBF* Tetric EvoCeram Bulk Fill, *FBF* Filtek Bulk Fill, *XF* X-tra fil

Table 7 Temperature rise in degrees Celsius (± S.D.) measured at the bottom of 2- and 4-mm composite layers, as well as at the bottom of empty specimen compartments (denoted as "empty")

Material	2 mm MW	2 mm PW	4 mm MW	4 mm PW
TEC A2	5.9 (0.3) a A	8.6 (0.3) b ABC	4.5 (0.2) c AB	6.1 (0.4) a ABC
TEC A3	6.1 (0.3) a A	7.7 (0.6) b A	4.6 (0.1) c B	5.5 (0.5) a A
GRA	5.9 (0.1) a A	8.5 (1.0) b ABC	4.4 (0.2) c AB	6.1 (0.5) a ABC
GDP	6.3 (0.3) a AB	9.3 (0.3) b C	4.8 (0.2) c BC	5.9 (0.5) a AB
TECBF-IVA	6.9 (0.3) a B	8.9 (0.6) b BC	6.0 (0.4) c E	6.7 (0.2) ac BC
TECBF-IVB	7.0 (0.7) a B	8.6 (0.5) b ABC	5.7 (0.2) b DE	6.7 (0.5) a BC
FBF	5.9 (0.5) a A	8.5 (0.2) b ABC	5.2 (0.2) c CD	6.8 (0.3) d BC
XF	6.4 (0.2) a AB	7.8 (0.5) b AB	5.3 (0.2) c D	6.1 (0.5) a AC
Empty	4.7 (0.5) a C	8.4 (0.7) b ABC	4.1 (0.2) a A	7.0 (0.6) c C

Same lowercase letters denote statistically similar values within a row. Same uppercase letters denote statistically similar values within a column

MW "Monowave"—Bluephase Style M8, *PW* "Polywave"—Bluephase Style, *TEC* Tetric EvoCeram, *GRA* Grandio, *GDP* Gradia Direct Posterior, *TECBF* Tetric EvoCeram Bulk Fill, *FBF* Filtek Bulk Fill, *XF* X-tra fil

The correlation of MH with the degree of conversion within a composite formulation is the foundation for the use of MH as an indirect measure of polymerization efficiency [6]. MH plotted as a function of radiant energy can be envisioned as asymptotically approaching the plateau at which saturation is reached, i.e., the radiant energy is sufficient to attain the maximum possible MH [43]. Before reaching the saturation point, radiant energy should correlate with MH [5]. This type of correlation was identified in seven out of eight composites (Table 6) and a moderate amount of variability in MH data was attributable to differences in radiant energy (R^2 = 0.16–0.62). The lack of correlation between MH and radiant energy was observed only in X-tra fil (XF), which appears to have reached the saturation point for all combinations of *curing unit* × *thickness*. Thus, the fourth null hypothesis of no correlation between radiant energy and MH was rejected for all materials except XF.

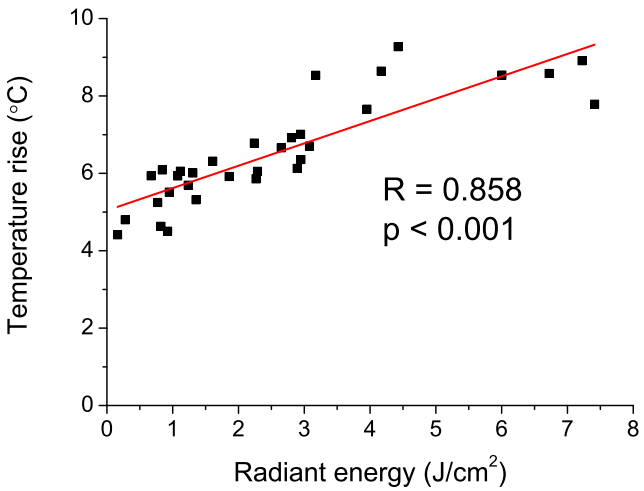

Fig. 3 Correlation between radiant energy and temperature rise measured at the bottom of 2- and 4-mm-thick specimens

As MW and PW differed by radiant energy and spectral emission, the relative contributions of these effects could not be separated. Additionally, two subtle differences between the curing units were identified: (I) the "30-s" curing mode actually lasted 31 s for PW and (II) the blue emission peak in MW was shifted by 9 nm towards lower wavelengths (Fig. 1), thus being less aligned with the camphorquinone absorption maximum at 468 nm [44]. This suggests that the beneficial effect of PW may not be only due to the presence of an additional violet peak but also because its blue peak was more effective in activating the conventional camphorquinone/amine photoinitiator system.

Temperature rise measured at the bottom of composite specimens (4.4–9.3 °C) was comparable to 6–8 °C reported in a study using similar specimen geometry [26]. In another study, 2-mm-thick composite layers separated from the pulpal chamber by 1-mm-thick dentin and cured with radiant energies comparable to those in the present study produced an intrapulpal temperature rise of 7–8 °C [27]. The aforementioned studies [26, 27] used thermocouples; however, other means of temperature measurements may record much higher values [45], e.g., a temperature rise of up to 43 °C was measured using infrared cameras [46]. Temperature rise due to the heating of the curing unit can also vary widely; a recent review article mentioned the range of 1.5–23.2 °C [47]. Such heterogeneity of experimental data suggests that quantitative results obtained in vitro cannot be easily interpreted in terms of the potential pulpal hazard [6, 48]. Considering these limitations, temperature values in this study were used only to assess the relative effects of material composition, curing unit, and layer thickness, with no attempt to relate the absolute temperature values to the potential for pulpal damage.

For MW, the temperature at the bottom of the composite specimens was higher than that of the empty specimen compartment, suggesting that material-dependent reaction exotherm was superimposed on the curing unit heating

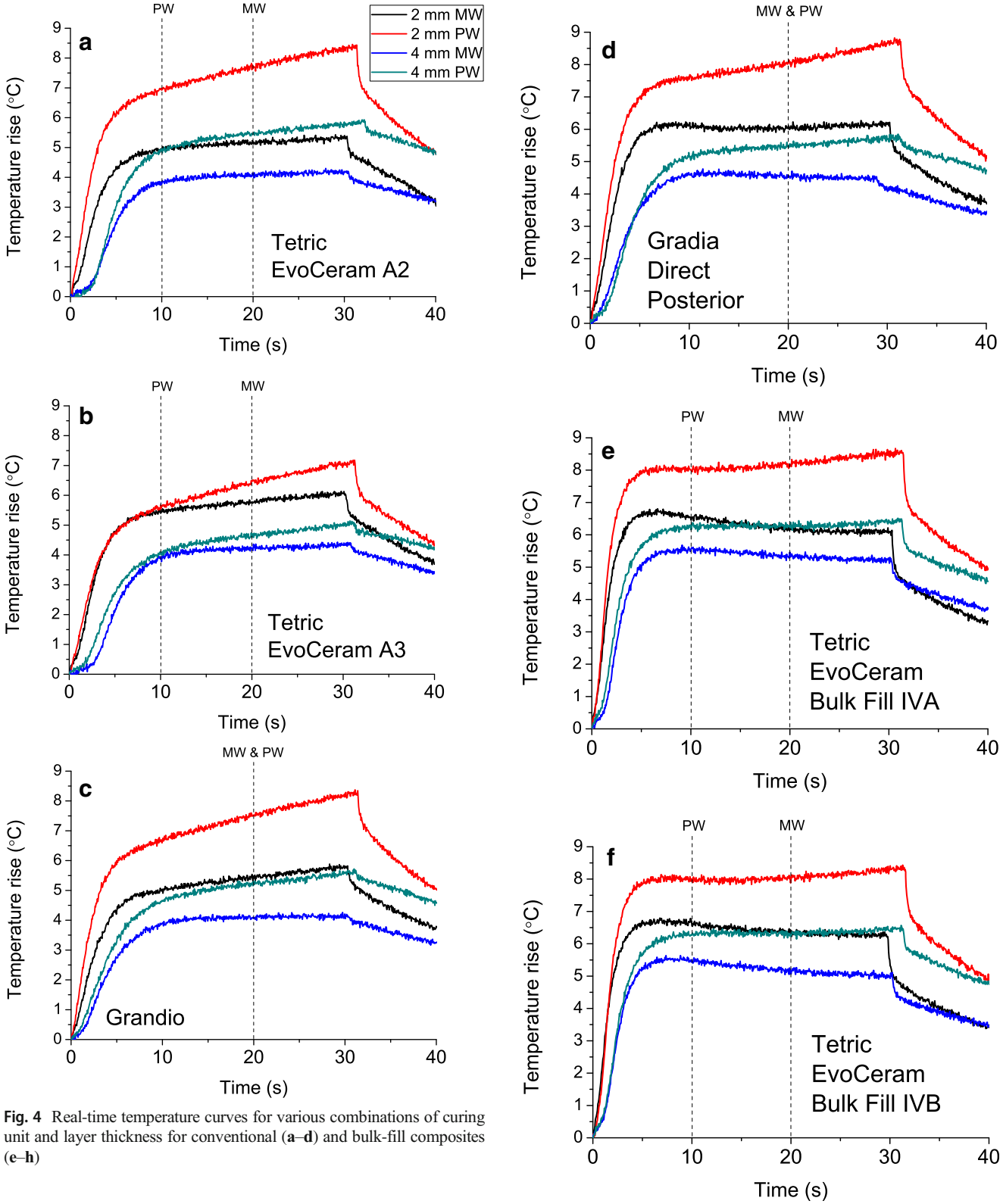

Fig. 4 Real-time temperature curves for various combinations of curing unit and layer thickness for conventional (**a**–**d**) and bulk-fill composites (**e**–**h**)

Fig. 4 continued.

(Table 7). Conversely, in the case of PW, temperature values in the empty specimen compartment were statistically similar to those measured in composite specimens. This is probably because the heating contribution of PW dominated the measured temperature rise thereby masking the effect of reaction exotherm. Only TEC A3 and Gradia Direct Posterior (GDP) cured with PW at 4-mm thickness showed significantly lower

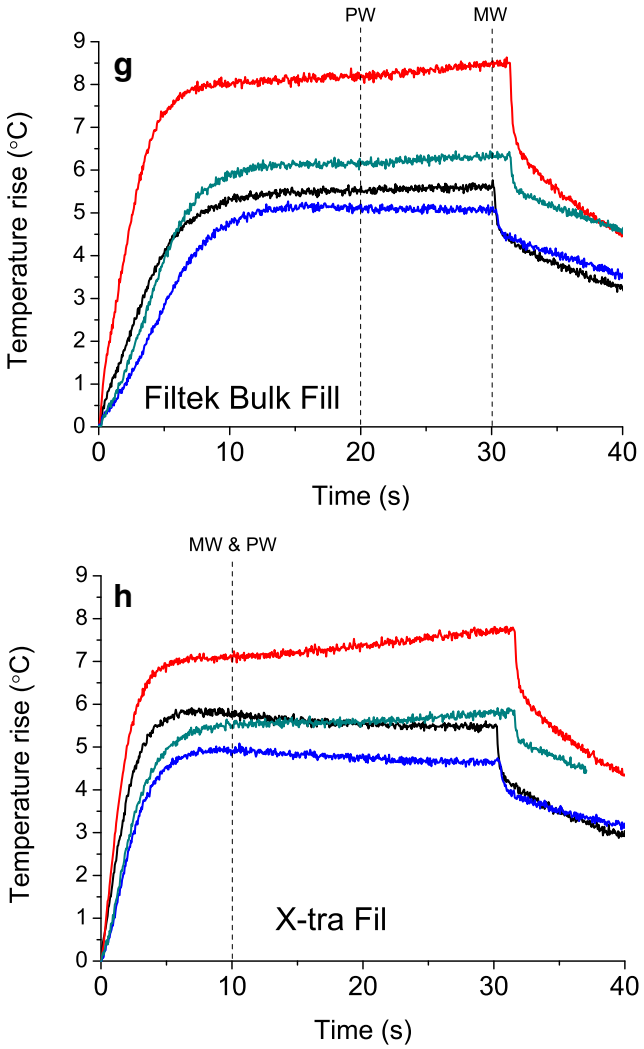

Fig. 4 continued.

temperatures compared to that of the empty specimen compartment, indicating a thermal insulating potential.

The effect of the composite amount on temperature rise is twofold. A larger amount of material represents a thicker insulating layer between the curing unit and specimen bottom at which temperature is measured. However, a larger amount of composite contains more C=C bonds and thus a larger amount of heat is released in the exothermic reaction [49]. The relative strengths of these two effects determine whether the increase in material amount acts to increase or decrease temperature rise during light-curing. Results of the present study suggest that the thermal insulating effect prevailed, as 4-mm layers consistently showed lower temperature rise than 2-mm layers (Table 7).

In ANOVA for temperature rise, curing unit type and layer thickness were more influential (partial η^2 of 0.834 and 0.844, respectively) than material composition (partial $\eta^2 = 0.490$). A high correlation between temperature rise and radiant energy for each *material* × *thickness* × *curing unit* combination (Fig. 3) suggests that temperature rise was mostly determined by variations in radiant energy reaching the specimen bottom; for an obtained $R = 0.858$, the coefficient of determination $R^2 = 0.74$ means that 74% of the variability in temperature rise was attributable to changes in radiant energy. This is in line with previous studies which demonstrated that the highest contribution to the temperature rise originates from the curing unit [22, 50] and identified high linear correlations between radiant energy and temperature rise measured in the composite material [51] or in the pulpal chamber of anesthetized human teeth [52]. The fifth null hypothesis was thus rejected.

The shapes of temperature curves varied among different combinations of *material*, *curing unit*, and *thickness* (Fig. 4). Generally, for 2-mm layers, a steeper initial part and higher maximum values were observed compared to 4-mm layers. The temperature curves for bulk-fill composites reached maximum values within 5–10 s and plateaued thereafter. For conventional composites, temperature curves rose throughout the 30-s period at various slopes; however, most of the increase occurred within the first 10 s. These findings suggest that extending the curing time beyond manufacturer recommendations (dashed vertical lines in Fig. 4) produced a minor additional increase in temperature. This means that the maximum temperature gradient in clinical conditions would be established soon after activating the curing unit. Thus, analyzing peak temperature values without considering curing time provides an incomplete indication about the amount of heat which would be transferred to the dental pulp. For example, if TECBF was evaluated using curing time of 10, 20, and 30 s, almost the same peak temperatures would be recorded for all time points. However, the effect on pulpal tissue would differ due to different time available for heat transfer. Although the actual risk for pulpal health remains indeterminate [6, 45], the effect of time should at least be acknowledged in studies of temperature rise during light-curing of dental composites.

According to the findings of this study, temperature rise at the bottom of a composite layer can be minimized by the following approaches: (I) bulk-filling can help to mitigate the heating effect, as 4-mm layers always produced lower temperature than 2-mm layers, (II) using the lower-intensity curing unit (MW) can be advised because it produced similar cure as the higher-intensity curing unit (PW) at clinically relevant layer thicknesses, while consistently producing lower temperature than PW, and (III) if high-irradiance curing units are used, placing bulk-fill composites in 4-mm layers can be beneficial for minimizing the thermal effect on dental pulp.

Conclusions

Within the limitations of this study which evaluated radiant energy, curing efficiency, and temperature rise in composites which were light-cured for 30 s using a blue (648 mW/cm^2)

and a violet-blue (924 mW/cm^2) LED curing unit, the following can be concluded:

1. Curing with the lower-intensity curing unit produced less heat, whereas both curing units produced adequate cure at 2 mm for conventional composites and 4 mm for bulk-fill composites.
2. The conventional composites containing combined photoinitiators benefitted from the use of violet-blue curing unit, while no benefit was identified in two bulk-fill composites with combined photoinitiators.
3. Thicker composite layers may protect the pulp from thermal insult by producing lower temperature rise at the layer bottom.
4. Temperature rise was mainly determined by radiant energy, suggesting that thermal hazard can be controlled by limiting curing parameters to optimal values.
5. Most of the temperature rise occurred within 5–10 s after the start of light-curing; extending the curing time to 30 s resulted in a minor additional increase.

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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